Progress Report 2008 - 2010

1956 IEA foundation

1958 IEA-R1 nuclear reactor startup

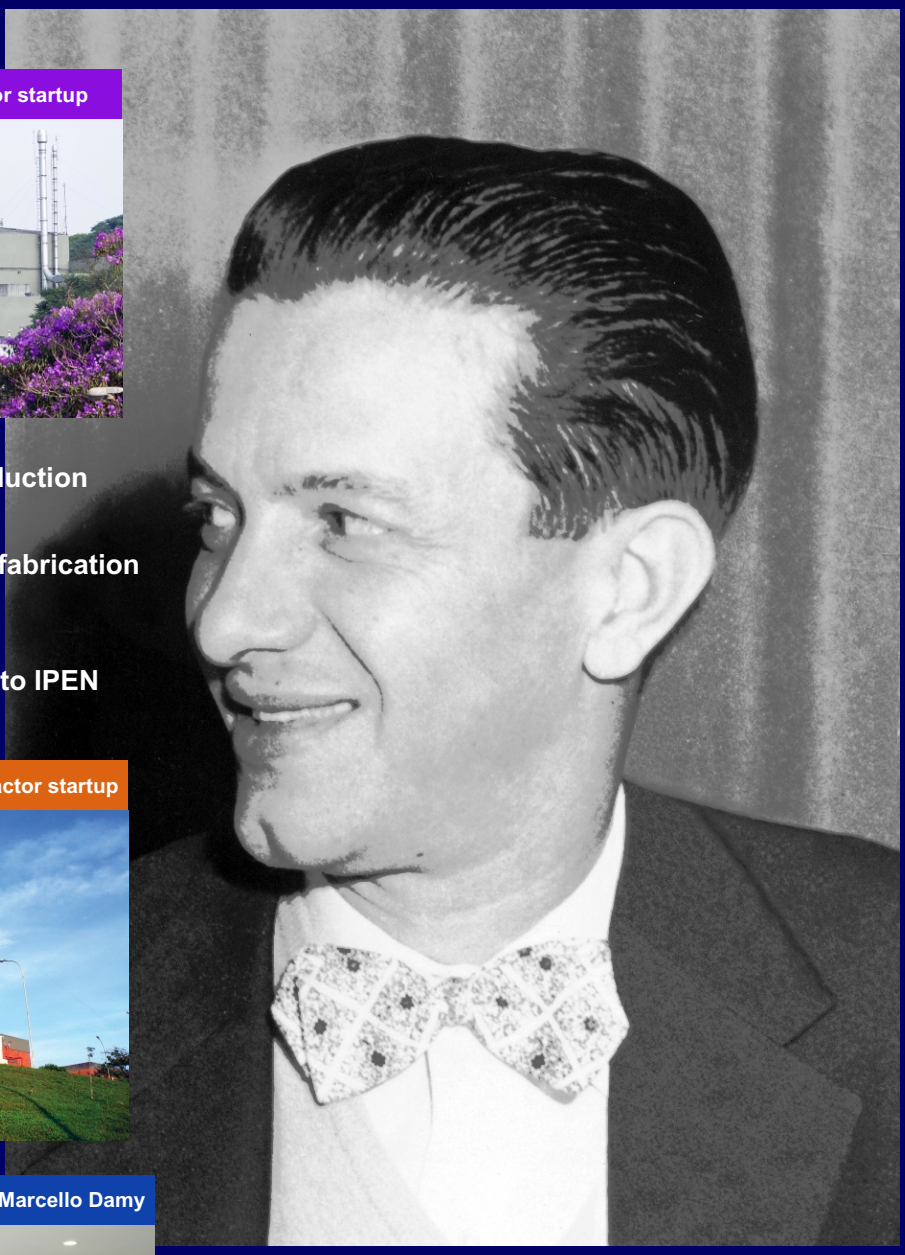
1963 Radioisotopes production

1964 Nuclear fuel elements fabrication

1979 IEA changed its name to IPEN

1988 IPEN/MB-01 nuclear reactor startup

2010 Espaço Cultural Prof. Marcello Damy



Marcello Damy de Souza Santos

IEA's founder and National Commission for Nuclear Energy president

Instituto de Pesquisas Energéticas e Nucleares
São Paulo - Brasil
2011

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Progress Report *2008-2010 is an extraordinary publication with limited edition.*

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Mission

Our mission is to improve the Brazilian people quality of life by producing scientific knowledge, developing technologies and services and promoting human resources for nuclear and correlated areas.

Short Profile

IPEN - Nuclear and Energy Research Institute is a State of São Paulo autarchy, associated to the University of São Paulo - USP for educational purposes and supported and operated technically and administratively by the National Nuclear Energy Commission - CNEN, a federal agency of the Ministry of Science, Technology and Innovation.

The Institute was founded in 1956 with the main purpose of doing research and development in the fields of nuclear energy and its applications. It is located at the campus of USP, in the city of São Paulo, in an area of nearly 500,000 m^2. It has over 1000 employees and 40% of them have qualification at master or doctor level.

IPEN is recognized as a national leader institution in research and development in the areas of radiopharmaceuticals, industrial applications of radiation, basic nuclear research, nuclear reactor operation and nuclear applications, materials science and technology, laser technology and applications.

Along with the R&D, it has a strong educational activity, having a graduate program in Nuclear Technology, in association with the University of São Paulo, USP, ranked as the best university in the country. The Federal Government Evaluation institution CAPES, granted to this course grade 6, considering it a program of Excellence. This program started at 1976 and has awarded 575 doctorates and 1238 master's degrees since them. The actual graduate enrollment is around 550 students.

The internal organization structure comprehends two levels, one for strategic and political issues and for decision making composed by its a Board and a Technical and Administrative Council - CTA and another for the executive and operational actions, coordinated by the Research and Development Centers, grouped according to their research, development and innovation activities.

IPEN has a rigorous program of radiological control and nuclear safety for the activities related to nuclear and radiological aspects. This program comprises personal and environmental monitoring and radiological emergency assistance.

O *Ipen* é uma autarquia vinculada à Secretaria de Desenvolvimento Econômico, Ciência e Tecnologia do Estado de São Paulo, associada à Universidade de São Paulo e gerida técnica, administrativa e financeiramente pela Comissão Nacional de Energia Nuclear, órgão do Ministério da Ciência, Tecnologia e Inovação.

Foreword

The period covered by this report, from 2008 to 2010, is marked by the renascence of the Nuclear Area, meaning for IPEN, as the most important Nuclear Research Institute of Brazil, a retake of its origins. The institute is ruled by a Convenium between the Federal government and the São Paulo State government that started in 1982 and lasted 25 years, ending in 2007. This rebirth of the importance of nuclear activity came to provide a firm direction to the efforts of the Institute and, as a consequence, a new term for this agreement was designed, and it is underway to be signed, to restore the necessary conditions for the success of the 25 years ahead.

The role of IPEN, responsible for distributing more than 95% of radiopharmaceuticals used in the country, was highlighted during the international crisis of ^{99}Mo supply that hit Brazil, letting the country in a very difficult situation. The reason for this situation is that Brazil has a nuclear medicine of first world level (~5% from the total attendance of the world), with 1,5 million patients attended per year. The response of the institution was crucial for mitigating the crisis, providing new ways to acquire ^{99}Mo from different suppliers, and, at that moment, Argentina was our main supplier.

This deal was accomplished in a week, a rare event for the public service. The crisis also pointed out our fragility by the dependence on foreign supply for such a large demand, and was the main reason for defining the need of a Brazilian Multipurpose Reactor. IPEN was the responsible for convincing the society and the government leaders of the urgency to construct our reactor, with the main purpose of producing the ^{99}Mo. This structural project is reorganizing the nuclear area, particularly at IPEN, that has the advantage of taking part of the graduate program of University of São Paulo and achieved the mark of 1813 titles concluded: 1238 masters and 575 doctorates and still keeps its excellence mark (grade 6) granted by the CAPES evaluation program. Its technological programs gained new energy, with the upgrade of the IEA-R1 reactor boosting its power to 3.5 MW and aiming to 5 MW. Also, the ^{99}Mo supply challenge is orienting the whole institute to cooperate in the development of alternative means for supplying ^{99}Mo because the situation is still fragile.

This new situation is already shown in this progress report, where the results of the R&D centers are presented according to the main programs: Biotechnology, Lasers Technology, Renewable Energies, Nuclear Reactors and Fuel Cycle, Applications of Ionizing Radiations, Nuclear Science and Technology, Materials and Nanotechnology, Environmental Science and Technology and Radiopharmacy. It must be stressed that all these results were accomplished due the efforts and dedication of the IPEN staff and were supported by CNEN, MCTI, SDECT/SP, University of São Paulo, IAEA, FAPESP, CAPES and CNPq.

Dr. Nilson Dias Vieira Junior
IPEN Superintendent

Organization Chart

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Contents

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Biotechnology

Research aiming to develop and manufacture products of pharmaceutical interest

Biotechnology

Introduction

The guidelines of the Biotechnology Program are research and development aiming to develop and manufacture products of pharmaceutical interest. This Program has two main research areas, namely Pituitary Hormones and Biopharmaceuticals.

The first one comprises a group with a long experience on Recombinant Human Pituitary Hormone synthesis, purification and characterization. Up to now they have worked mostly with human growth hormone (hGH), human prolactin (hPRL), human thyrotropin (hTSH) and more recently with human follicle stimulating hormone (hFSH) and human luteotropin (hLH). An important research line is devoted to Growth Hormone Gene Therapy, working mostly on animal models: immunocompetent and immunodeficient-dwarf mice. For several years this development has been based on “ex vivo” grafiting of transduced keratinocytes, while more recently very promising results have been obtained with the injections and electroporation of naked plasmid DNA. Besides research, they have also activities in the Biotechnological Production and Downstream Processing of the same recombinant hormones, which are produced in both *E. coli* and mammalian cells and in the development of joint-ventures with the National Industry. The biological effects of radiation on cells are also studied, especially concerning the administration of 131-I together with thyroid-stimulating hormone in thyroid cancer.

The Biopharmaceutical area is dedicated to the research of isolation, structural analysis and biological activities in different biological systems of macromolecules. These macromolecules are peptides or proteins, either native or recombinant with medical or pharmaceutical interest. During this period new proteins related to serine protease activity, breast cancer development and angiogenesis were described. The effects of ionizing radiation on macromolecules have also been investigated to detoxify animal venoms in order to improve antigens for anti-sera production, or even modify microorganisms for vaccination. Recently, we started investigating the peptide fractions of several venoms, identifying many serine-protease and metallo-protease inhibitors. Ideally, these inhibitors will be co-crystalyzed with the target enzyme, aiming to characterize the inhibitor-enzyme interaction. Such data could provide knowledge to develop new drugs against coagulopathies and other endogenous protease related diseases.

The Animal Laboratory Division of IPEN is responsible for the breeding and production of small laboratory animals. In this facility Specific Pathogen Free (SPF) animals are bred and maintained, under controlled sanitary conditions, to be used for testing of the radioisotopes production and research. This facility also produces different mutant mice, severely immunodeficient mice and their offspring, besides other mice lineages as well as normal rats.

Biotechnology

Pituitary Hormones

The research and production activities related to the five human pituitary hormones, namely growth hormone (hGH), prolactin (hPRL), thyrotropin (hTSH), follitropin (hFSH) and luteotropin (hLH), still constitute the basic field of the group. Our goal is the development of applied research, especially emphasizing the interaction between the Academic and the Industrial world, an aspect that has been neglected for so long in Brazil. Under this aspect the joint-venture already set up at the end of 2007 with a successful biotechnology company (FK-Biotecnologia), that demonstrated great interest in hormone and antibody production for diagnostic and therapeutic applications, has continued. At the same time new collaborations have been set up with clinicians from FMUSP, especially in the field of Gene Therapy, and with EMBRAPA, for the purpose of cloning Arapaima gigans (Pirarucu) gonadotropins. As always, the main emphasis of the group was given to scientific production and to collaboration with well known national and international research groups. Thus, in this 3-year period, 19 scientific papers have been published all in international journal whose impact factor was always between 1 and 7.5.

Human growth hormone (hGH)

Human growth hormone (hGH) production and quality control has been already established at the laboratory level and, as stated several times, is only waiting for the "industrial decision", that unfortunately is sometimes independent from our will. However, an important research line in Gene Therapy has been carried out using the hGH gene. In this period, our group gave continuity to the research in the Gene Therapy field, using the human (hGH) and the mouse (mGH) growth hormone genes. The goal of this approach is the development of an animal model, based on immunodeficient dwarf (lit/scid) mice, in which it can be possible to obtain useful and sustained circulatory levels of these hormones together with phenotypic corrections, such as body weight gain and longitudinal growth (Journal of Gene Medicine, 2008). The efforts were concentrated on the use of an *in vivo* system based on naked hGH DNA administration followed by electroporation in the quadriceps muscle of lit/scid mice. An important paper was published (Journal of Gene Medicine, 2010), that describes a sustained secretion of hGH during a 60-day assay together with a highly significant increase in the body weight of these animals. We are preparing another manuscript where the paracrine/endocrine effects of this type of administration are compared to those obtained after regular injection of recombinant hGH. In this regard, the two different strategies provided a similar response in terms of weight variation, when comparing the slopes of both growth curves. We also can emphasize our participation to one of the most prestigious International Meetings, such as the "12th Annual Meeting of the American Society of Gene Therapy" in San Diego, 2009, which originated a publication in Molecular Therapy and to the "Gordon Research Conference on Bioelectrochemistry" in 2010, together with a presentation at the "6th Conference on Recombinant Protein Production" in Vienna. Recently, we also started working with lentiviral vectors, carrying the hGH or mGH gene, which can be used to transduce keratinocytes *ex vivo* and transplant these genetically modified cells into the same animal model. This attempt, which was already studied in our laboratory with retroviral vectors, also intends to improve the sustainability of *in vivo* hormone secretion. These studies are being carried out in collaboration with the Endocrinology Division of the FMUSP/São Paulo. Considering that hGH is positively influencing muscular dystrophy, a collaboration with Dr. Mayana Zatz from the "Centro de Estudos do Genoma Humano" IBUSP was set up. Dystrophic mice of four distinct mutated strains obtained from the Jackson Laboratory (Maine, USA) were maintained in the animal house of the Biotechnology Center. A series of mating were carried out and techniques for the determination of dystrophy and growth hormone deficiency by DNA sequencing were established. Other activities were also carried out under this collaboration, based on the *in vivo* use of human adipose multipotent mesenchymal stromal cells (Stem Cells, 2008; Stem Cell Reviews and Reports, 2010).

Human pituitary glycoprotein hormones

Human pituitary glycoprotein hormones include thyrotropin (hTSH), follitropin (hFSH) and luteotropin (hLH), all heterodimers formed by an alpha and a beta subunit. This hormone is related to thyroid function and metabolism, and is used in the diagnosis and therapy of thyroid cancer, while hFSH and hLH are mostly used for the treatment of human infertility. These recombinant products are among those with the highest aggregate value, their purified forms reaching prices up to US$ 12.000/mg! Considering their carbohydrate moiety, which is strictly related to their *in vivo* bioactivity, these proteins must be synthesized in mammalian cells, the most commonly used for their industrial production being CHO cells. Our laboratory has synthesized and characterized hTSH, having also the know-how for synthesizing hFSH and hLH. During this period (2008-2010) the laboratory completed the study concerning recombinant hTSH synthesis in a reduced CO_2 environment, which led to a higher productivity, characterizing for the first time the N-glycan structures of our recombinant preparations in comparison with the only commercial preparation available: ThyrogenR from Genzyme (Molecular Biotechnology, 2008). Aiming at an efficient and careful quality control of biopharmaceuticals, we studied and characterized the individual subunits (alpha and beta) of the three recombinant and pituitary glycoprotein hormones (hTSH, hFSH and hLH), obtained via a particularly efficient and mild dissociation technique of the intact heterodimeric hormones. The 12 different subunits were characterized comparatively for

purity, hydrophobicity, molecular mass and charge distribution by HPLC, mass spectrometry, SDS-PAGE and isoelectric focusing (Journal of Chromatography A, 2009). For the first time a human-like sialylated recombinant hTSH, with sialic acid bound to galactose by both alpha2,3 and alpha2,6 linkages, was synthesized (Protein Expression and Purification, 2009). The new product which was obtained by genetic modification of the carbohydrate moiety, was found similar to traditional recombinant hTSH for what concerns molecular mass, distribution of charge isomers, hydrophobicity and bioactivity, although several differences were observed in N-glycan structures. The *in vivo* influence of these modifications on the pharmacokinetic properties are now being studied. Finally a new RP-HPLC methodology, related to hLH and hCG identification, characterization and qualitative and quantitative analysis of 11 preparations of different origin (pituitary, urinary and recombinant) was also set up (Journal of Pharmaceutical and Biomedical Analysis, 2010). Our laboratory also participated of an International Collaborative Study organized by the World Health Organization (WHO) for the definition of the new International Standard of Recombinant hFSH for bioassay being, together with our collaborators from the Federal University of Santa Maria (RGS), the only Latin American group and also the unique to determine the mass content of hFSH by physical-chemical methods. It should be emphasized that for the case of recombinant glycoproteins, while the protein moiety is identical to the natural, the carbohydrate moiety is necessarily different. Such difference must be studied and evaluated, especially considering the repeated parentheral use of these biopharmaceuticals. More work is in progress concerning this aspect, especially considering the comparison of the N-glycan structures of the recombinant proteins with those of the native ones. Some of these studies are being carried out in collaboration with the University of Oslo (Norway) and of Vienna (Austria). Also more work is in progress related to the substitution of *in vivo* bioassays with physical-chemical techniques and to the cloning and synthesis of Arapaima Gigans (Pirarucu) gonadotropins, in collaboration with EMBRAPA, aiming at increasing the fertility of this species for alimentary purposes.

Prolactin (hPRL)

Prolactin is the second (after hGH) unmodified protein hormone secreted by the anterior pituitary. Its therapeutic use is still quite limited, while it is important for diagnostic applications. A different importance has been attributed to its analogs/antagonists, whose anti-proliferative activity especially concerns breast and prostate cancer. Two hPRL antagonists (S179D-hPRL and G129R-hPRL), discovered by two leading laboratories in the US and in France, have been synthesized in our laboratory for the first time in their authentic form. Giving thus continuity to the studies carried out in collaboration with the University of California at Riverside, where S179D-hPRL has been first studied and synthesized, *in vivo* antitumor and anti-angiogenic activities have been demonstrated, together with a particular conformational change due to an increased positive surface charge that activates different signaling molecules. In a few words this is what makes the difference between hPRL and its antagonist (Biochemistry, 2009).
Another study has focused directly hPRL production in *E.coli*, where expression levels had always been extremely low. This problem was resolved by carrying out activation at a lower temperature (37° C instead of 42° C), thus using the lambda PL promoter in a constitutive way, without its repressor. This way an approximately 30-fold higher expression was obtained, showing also that the bacterial cytoplasmic environment is apparently very harmful to hPRL stability, especially at temperatures > 37° C (Journal of Biotechnology, 2008). Besides in bacteria, hPRL synthesis had been also increased (~2-fold) in CHO cells, by the addition of an appropriate amount of sodium butyrate (1mM) to the culture medium, a method that we are now considering also for application to other hormones, as for example hTSH (Journal of Biomedicine and Biotechnology, 2010). Another type of addition has been also studied and experimented for CHO cell culture medium: cycloheximide. This compound has the property of slowing the protein synthesis, thereby extending the time available for the glycosylation machinery. As a result glycosylation site occupancy is also increased. The results obtained have been quite rewarding, with an increase of ~4-fold in the concentration of glycosylated prolactin (G-hPRL) and of ~7-fold in the glycosylated versus non-glycosylated hPRL (NG-hPRL) concentration ratio (Journal of Biotechnology, 2010). This way it will be easier to obtain higher amounts of this isoform and better study its physiological role, which is at present still unknown. Finally, a more clinical research was also carried out, in collaboration with the Neuroendocrine Unit of the FMUSP/São Paulo, in patients affected by systemic lupus erythematosus (SLE) hyperprolactinemia. The results indicated that lymphocytic hPRL does not contribute to hyperprolacinemia of SLE, supporting a pituitary origin for hPRL hypersecretion (Clinical and Experimental Rheumatology, 2010).

Cellular response to ionizing radiation

Ionizing radiation is a physical agent known to induce mutation and cancer being also used as a widespread therapeutic modality for cancer treatment. Thus, one of the challenges in radiobiology and oncology is to understand how the cells respond to oxidative stress resulting from exposure to radiation and the pathway they will follow. On the basis of the above considerations, the present study has been developed by the group focusing three interlinked aspects. The objective of the first one (cytogenetic aspect) consists on a

comparative study of the effects of different radiation types and radionuclides of medical interest (^{153}Sm, ^{177}Lu, ^{131}I), in human and rodent cells, by cytogenetic and biochemical techniques. Radiation damage to DNA and repair capacity in cell lines originating from breast cancer were also analyzed (International Journal of Low Radiation, 2009). A collaboration with the Parasitology and Malacology Laboratory of Butantan Institute has been developed for the evaluation of the genotoxic effect of environmental mutagens, using freshwater snail *Biomphalaria glabrata*, irradiated with ^{60}Co (Mutation Research, 2008). The second aspect of our study consists in the establishment of dose-response curves for different types of radiations (γ, β and neutron) for biological dosimetry (dosimetric aspect) directed to the quantitative estimate of absorbed dose, according to the criteria adopted by IAEA (2001). The calibration curves for the γ radiation of ^{60}Co and ^{137}Cs and for the β radiation of ^{90}Sr have already been establishment by chromosome aberration, micronucleus and comet assays. More recently, curves for fission neutrons produced in the Reactor R1 of IPEN-CNEN/SP were obtained in collaboration with the "Centro de Engenharia Nuclear" (INAC 2009). A third aspect (therapeutic approach) consists in evaluating the cytogenetic effects of radiopharmaceuticals used in nuclear medicine, e.g. ^{131}I, administered to patients with differentiated thyroid carcinoma, with or without use of recombinant human thyrotropin (Thyrogen^{R} or rhTSH), that is being carried out in collaboration with the Nuclear Medicine Center of FMUSP. This study was approved by FAPESP and new equipments were granted, among them a modern optic microscope Nikon Eclipse 80i, with image system. A previous Project, based on animal model, also granted by FAPESP, had already been concluded, the related results having been published (Radiation Environmental Biophysics, 2008). Finally, a study about the radiomodificator effect of propolis in normal and human prostate cancer cells is in progress, having already published data on the "Protective effect of propolis in radiation induced chromosomal damage in Chinese hamster ovary cells (CHO-K1) irradiated with ^{60}Co" (INAC 2009). The data obtained via micronucleus test showed a radioprotector effect on the induction of DNA damage and an anti-proliferative effect on tumor cells were demonstrated by cytotoxicity tests. These data indicate a potential promising use of propolis as a natural, nontoxic, effective substance for protection against genotoxic and cytotoxic damages, induced by ionizing radiation

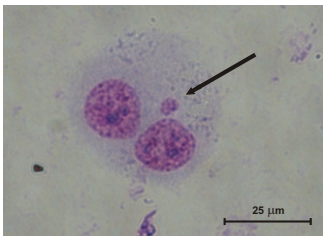

Figure 1. Binucleated cell with one micronucleus

Protein radioiodination

The Group of Hormones of IPEN has a long and well-known expertise on Protein Radioiodination, mostly with the use of ^{125}I. At least ten studies on pituitary hormone radioiodination have been published, before the year 2000, in International Journals of high impact. In the period 2001-2007, five more papers have been published, providing precious collaborations to other Brazilian research groups, in prestigious journals as well. During the period 2008-2010, object of the present Progress Report, four more papers have been published in Pancreas, European Journal of Pain, and Toxicon, in collaboration with the Departments of Pharmacology of Unicamp (Campinas - SP) and of the University of São Paulo (São Paulo - SP). This excellent specialty, in a specific area of the Nuclear Field so long neglected, deserves a proper emphasis, especially considering the work of all the technicians and researchers that dedicated themselves, to the study and manipulation of this extremely useful radioisotope.

Biotechnology

Biopharmaceuticals

Identification, isolation and characterization of new compounds of clinical interest, from plants and animal molecules have been developed. Additionally, studies involving ionizing radiation have been done in order to detoxify or even modify molecules mainly to improve antiserum production and vaccines. Many of pre-clinical assays, the main tool, employed to characterize the candidate to a new medicine have now been standardized to get an accreditation.

Biological screening of snake venoms and toxins

Snake venoms are an extremely rich source of biologically active substances modulating several aspects of the regulation of homeostasis. The investigation of potential new drugs in biological samples has been a major field of research in many laboratories. This activity has focused part of its efforts in screening and isolating toxins with potential therapeutic uses. Amongst those, toxins from the Brazilian rattlesnake which seem to be involved in the down regulation of the sodium/potassium pump have been investigated. Such molecules have a potential for further understanding the fine physiology of many cell types and for guiding the rationale design of cellular metabolism modulators. During investigation of the action mechanism of snake neurotoxins, we developed a model of excitable medium, the Belousov-Zabotinsky reaction, was developed. It allows understanding many aspects of neuronal transmission and membrane physiology. Among several experiments a set of reactions to microgravity, using a sounding rocket provided by a collaboration between Brazilian and German Space Agencies was done, and it was possible to observe that, indeed gravity seems to be able to act at molecular levels, modulating reactions which can be compared to those ruling neuronal transmission, onset of migraine and epileptic crises and regulation of heart beating. Also toxins with modulatory activity on the blood clotting system (Snake Venom Serine Protease - SVSP) are under investigation. Such toxins might be employed for the treatment of coagulation disorders and as auxiliary drugs in major surgeries where unexpected activation of the blood clotting cascade could put the patient at risk. These proteases are also key players in a wide range of biological processes; for example, in regulating the cell cycle, cell growth and differentiation, affect the haemostatic system, antigen processing and angiogenesis. In addition, it is becoming apparent that the aberrant functioning of certain proteases may be involved in several disease states, including Alzheimer's disease, in cancer metastasis and in inflammation (over-reactive inflammatory reactions in CNS often cause irreversible neuronal damage). In this period it was reported the molecular cloning of five new nucleotide sequences of SVSP (GenBank accession number AY954040 EU360951; EU360952; EU360953; EU360954) that were retrieved from a cDNA library constructed with the venom gland of a single specimen of Brazilian rattlesnake *Crotalus durissus terrificus*. These sequences have been analyzed in silico with respect to their cDNA organization, similarity in relation to others SVSPs, their probable biological functions and the overall particularities of these nucleotide sequences. The functional dendrogram was generated to group the serine protease activities in relation to others snake venom thrombin-like enzymes. Moreover, a rapid and efficient method for screening vectors for mammalian cell expression was developed. It is based on the fact that SVSPs are difficult-to-express toxins since they contain several disulfide bounds and are glycosylated. The biochemical properties and the molecular weight of recombinant toxin were compared to native gyroxin purified from the venom and are essentially identical.

Biological evaluation of new products for health

This activity is mainly based on the biological evaluation of substances and biomaterials performing *in vitro* and *in vivo* tests. These tests are carried out in compliance with the rules of ISO-10.993 and some other international directives. Such tests include: cytotoxicity, genotoxicity, hemocompatibility etc and some others tests of systemic toxicity and implants. Synthesis of polymeric biomaterials was also done, resulting in three patents submitted to INPI in 2007. A biomaterial can be defined as a substance (with the exception of drugs) or a combination of substances (either synthetic or natural), employed in the treatment, improvement or substitution of organism tissues, organs or function. Since interaction with the biological system is involved, biocompatibility implies the capability of the material to exhibit in the host the appropriate functional and "biomimetic" qualifications. In recent years, interest in biomedical applications of natural and synthetic polymers has grown steadily, with a substantial contribution to the quality and duration of human life. Presently, novel porous biologically active composites based on hydroxyapatite (HA) and poly(caprolactone) (PCL) have been developed and tested, with potential for use in scaffolds for bone tissue engineering. The experiments are focused on the synthesis and biological response of bone to the PCL/HA composite. Such work resulted in a partnership with the Biosintesis Company which received a financial support from FAPESP (PIPE project). Another approved FAPESP project about lyophilization process of biological tissues to make cardiac valves, includes IPEN, INCOR, UNICAMP and the Pharmaceutical Sciences Faculty of University of São Paulo. The biofunctionality of the bovine pericardium with endothelial cells has been tested.

Recombinant proteins - refolding from inclusion bodies using high hydrostatic pressure

The bacteria *Escherichia coli* is the most efficient and cost-effective host for transgenic protein production for therapeutic as well as for research purposes. However, *E. coli* is often unable to fully process the recombinant foreign proteins during overexpression and thus misfolded proteins aggregate in bacteria cytoplasm. These aggregated proteins are known as inclusion bodies (IBs). Utilization of high hydrostatic pressure is a novel and robust method to disaggregate and refold proteins from inclusion bodies, by solubilization of the aggregates in mild conditions, maintaining the existing secondary and tertiary structure of the insoluble and mostly inactive proteins produced in bacteria. The Green Fluorescent Protein (GFP) is a monomeric protein that was initially extracted from the jellyfish *Aequorea Victoria*. It has the ability to convert ultraviolet light into a bright green fluorescence. The fact that the native structure must be present for emission of the characteristic fluorescence of this protein and the simplicity of monitoring the GFP bioactivity make this protein an excellent model system for refolding studies, enabling determination of an efficient protocol for protein refolding. In fact, contrary to what has been described in previous studies, we demonstrated that the pressure level that induced dissolution of eGFP IBs aggregates (2.4 kbar) was 7 times higher than the ideal conditions for refolding of this monomeric protein. By utilization of the Green Fluorescent Protein (GFP) IBs, as well as IBs of other proteins, as endostatin, QM, bothropstoxin 1 and cruzain, we demonstrated that high pressure can successfully convert insoluble protein from inclusion bodies to a preparation with native tertiary structure and fully biological activity. Endostatin can specifically inhibit endothelial cell proliferation and thus potently inhibit angiogenesis and tumor growth. QM or ribosomal protein L10 is originally identified as a tumor suppressor protein.

Structural analyses of soluble human QM protein

The ribosomal protein QM belongs to the L10 family of ribosomal proteins, which is highly conserved from yeast to humans. The presence of the QM protein is necessary for joining the 60S and 40S subunits in a late step of the initiation of mRNA translation. Although the exact extra-ribosomal functions of QM are not yet fully understood, it has been identified as a putative tumor suppressor. We investigated the effect of growth temperature on the expression of the soluble form of the 24.5-kDa QM protein. QM was expressed in a soluble form in culture at temperatures of 25°C and 30°C for 16 h. Structural analysis of the soluble protein fraction by circular dichroism showed that this protein has less alpha helix than beta sheet, and a fluorescence assay with zinc incorporation showed that this fraction displays tertiary structure, which has been described previously in the literature. The circular dichroism and fluorescence analyses were made in National Laboratory of Synchrotron Light, Brazil.

Biological effects of ionizing radiation

Ionizing radiation, in aqueous solution, produces several highly reactive species. The most important are hydroxyl radical and hydrated electron. Theses products interact with peptides and proteins causing several modifications such as fragmentation, aggregation or oxidation, which are responsible for detoxification or even few modifications on proteins. These properties of ionizing radiation make it a good tool to improve antiserum production and vaccination process. Additionally, some substances called scavenger can be used to modulate these effects. It was found that the irradiated protein could be selectively incorporated to the cells, due to specific receptor for oxidized protein, the scavenger receptors. This increased uptake could also result in better antigen presentation and high immune response, either humoral, as demonstrated with purified crotoxin or cellular, as recombinant *M. leprae*. Rp 18 heat shock protein. Ionizing radiation can also modify biological and structural properties of toxins as crotamine, used here as a model. Biological alterations occurred in irradiated crotamine were observed with intravital microscopy; native crotamine causes a time-dependent vasoconstriction that suffers inversion with irradiated toxin (2 kGy) leading to the vasodilatation. Structural analysis suggested alteration in tertiary structure, keeping the primary structure unbroken.

Biotechnology

Animal Laboratory Division

The Animal Laboratory Division is a facility having 1.040 m^2 of built area, distributed in production and stock areas of animal models for IPEN as well as for other institutions. Some of these models bred in this division are unique in Brazil, thus providing extremely useful tools for many investigators. The goal of this division is to act as an animal breeding and experimentation facility, sterilizing products and providing services to guarantee the genetic and sanitary quality of animals employed in investigations focusing mostly on the development of new drugs and radiopharmaceuticals.

Figure 2. Specific Pathogen Free animals kept under genetic, sanitary and environmental controlled conditions

Besides breeding animals for use in our institution, this facility also sells animals for other laboratories and offers housing of special care requiring mice and rats upon request. For further information, contact nnascime@ipen.br.

Biotechnology

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Honor Mention and Awards

The Brazilian Society of Science in Laboratory Animals (SBCAL) honored the work "Influence of ionizing radiation on well being of animals producing anti ophidic serum", realized by Nanci do Nascimento, Miriam C. Guarnieri, Pedro C.L. Oliveira and Roberto Rogero, during the XI Brazilian Congress of Science in Laboratory Animals and the II Forum of Ethic Committee on Animal Use, 2009.

Lasers Technology

Single crystal fibers growth

Lasers Technology

Introduction

The Lasers Technology Program of IPEN is committed to the development of new lasers based on the research of optical materials and new technologies, as well to laser applications in several areas: Nuclear, Medicine, Dentistry, Industry, Environment and Advanced Research. Additional goals of the Program are human resource development and innovation, in association with Brazilian Universities and commercial partners. The Program is basically divided into two main areas.
"Material and Laser Development", includes crystal growth of optical materials (laser crystals); characterization, modeling and optical spectroscopy of solids, plasmas and biological materials; development of compact diode pumped-solid state lasers and the development of a high power (TW) laser system, one of the main projects of the Center for Lasers and Applications. High power ultrashort pulses lasers based on Chirped Pulse Amplification technologies and Ti:Sapphire gain media, allowed the generation of TW peak power on top of conventional optical tables and the realization of relativistic intensities (10^{18} W/cm^2) at modest costs.
The main area, "Laser Applications", is concerned with technological laser uses such as laser processing, laser remote sensing, development of new diagnostic and therapeutic methods such as optical coherence tomography (OCT), laser Doppler flowmetry, photosensitization, prevention of dental caries, plus other advanced applications of high intensity lasers.
Recent activities are highlighted bellow:

- Development of single crystal fibers for compact laser systems;
- Growth of a solid solution $LiGd_{0.232}Lu_{0.75}Nd_{0.018}F_4$:Nd crystal suitable to obtain a laser medium for mode-locking purposes;
- Characterization, modeling and optical spectroscopy of rare-earth doped crystals and glasses for the development of solid laser medium;
- First single crystal Nd:YLF fiber laser;
- Evaluation of the performance of fs laser-induced Breakdown Spectroscopy (fs-LIBS) for the determination of elements in animal tissues.
- New method for the evaluation of microvascular functionality using low-frequency fluctuations in the laser Doppler flow signal;
- Construction of an automatized workstation with ultrashort laser pulses (femtoseconds) for the study of thermal and non-thermal processes in dielectrics, semiconductors and metals;
- Study of a therapeutic method combining Nd:YAG laser and topical fluoride treatment for effective reduction of caries incidence in patient.
- Development of studies showing that photodynamic antimicrobial therapy is able to reduce 99% of multi-resistant bacteria in burn wounds.
- Analysis of Optical Coherent Tomography applied to dermatology (research work winner of the Natura Campus 2010 Premium for Technological Innovation),
- New LIDAR system for Industrial Emission and Detection installed in Cubatão/SP (collaboration in The National Institutes of Science and Technology Program /INCT).
- Studies for isotope enrichment by ultrashort laser pulses.

Lasers Technology

Lasers Development

Crystal growth

The Crystal Growth Laboratory works on bulk, micro and nanocrystals research for materials properties studies and development of new lasers including lamp and diode pumped systems from fluoride single crystals, as $LiREF_4$ and KRE_3F_{10} (where RE = rare earth ions), and double tungstates single crystal fibers, as $AB(WO_4)_2$ (where A = Na, Li and B = La, Gd, Nd, Yb, Eu).
The research on crystal growth performed in this period was supported by CNPq, Fapesp and CAPES including collaboration projects with Federal University of Sergipe - UFS (CNPq *"casadinho"* project) and Federal University of Pernambuco - UFPE (CNPq-INCT program in National Institute for Science and Technology in Photonics).

Bulk single crystals growth

Thulium ions (Tm^{3+}) are suitable for frequency upconversion of infrared to visible light, because these ions have long-lived high excited $4f^n$ states that give rise to strong blue luminescence. In this period the effect of Nd and Yb used as sensitizers to pump Tm ions has been studied. Two matrices have been utilized as host for these ions: $LiYF_4$ (YLF) and KY_3F_{10} (KY3F), which have the scheelite and fluorite structures, respectively.
YLF:Yb:Nd:Tm crystals were grown by the Czochralski method and doped with 0.5 mol% Tm and/or 20 mol% Yb, and/or 1.3 mol% Nd.
KY3F samples of good quality were obtained using a simple synthesis method consisted by the slow cooling of liquid charges under a HF+Ar atmosphere. This method reduced the time to produce samples with sufficient transparency for spectroscopic studies. KY3F:Yb:Nd:Tm crystals were doped with 0.5 mol% Tm, and/or 1.3 mol% Nd and different concentrations of Yb (5, 10, 20 and 30 mol%). Spectroscopic studies determined the mechanisms of energy transfer that lead to the thulium upconversion emissions in the blue and ultraviolet regions.
YLF:Er (15 mol%), YLF:Nd (1.3 mol%) and YLF:Yb (10 mol%):Tm(1 mol%) crystals grown by the Czochralski method were also utilized to obtain lasers with new features.
A solid solution $LiGd_{0.232}Lu_{0.75}Nd_{0.018}F_4$:Nd crystal was successfully grown and the spectroscopic studies showed that Nd presents larger bandwidth at 792 and 797 nm, which can be suitable to obtain a laser medium for mode-locking purposes.
Inspired in the results obtained in the growth of solid solutions of $LiGd_{1-x}Lu_xF_4$ by zone melting and by crystal growth from the melt, it is in course the study of the LiF - GdF_3 - LuF_3 phase diagram by computational methods, using the commercial simulation program FactSage.

Single crystal fibers growth

Despite new developments for compact laser devices, the Nd^{3+} ion continues to be the most widely used active laser ion. In 2004 we started a program on single fiber growth by the micro-pulling down (μ-PD) method aiming the development of laser materials. The objective was to obtain single crystal fibers with constant diameter and good optical quality for laser tests. This project that included the installation of two μ-PD furnaces started with fluorides fibers growth, resulted in 2009/2010 in the demonstration, for the first time, of laser action from Nd:$LiYF_4$ (Nd:YLF) single crystal fibers. At the present other materials are under investigation as fluoride fibers of Nd:BaY_2F_8 and rare earth double tungstates fibers also for laser tests. We have prepared Nd:$NaLa(WO_4)_2$, Nd:$NaGd(WO_4)_2$ and Eu,Yb and Nd doped $LiLa(WO_4)_2$. The structural and optical characterizations of these materials are under investigation to improve their quality for future laser tests.

Figure 1. Crystal fiber growing in the micro-pulling down system

Development of compact diode-pumped solid-state laser sources

This activity comprises the development of new laser sources based on diode pumped solid state lasers (DPSSL) for applications in research, industry, medical and pollution control. Our investigations are focused mainly on controlling the temporal, spectral and spatial features of the laser beam. In some cases it also includes the production design of such systems including the reliability tests and application experiments. We have built several DPSSL systems emitting from the blue up to the far infrared. Some highlights of the last period are shown below:
- A high power 2.3 μm Yb:Tm:YLF laser diode-pumped simultaneously at 685 and 960 nm. The achieved output power of 620 mW is the highest reported so far.

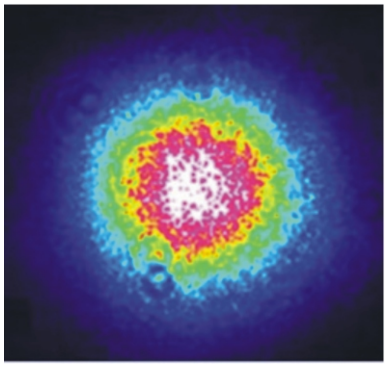


Figure 2. Gaussain beam quality obtained from the Yb:Tm:YLF laser

- Compact, diode-side-pumped Nd^{3+}:YLF laser at 1053 nm with 45% efficiency and diffraction-limited quality by mode controlling. The highest efficiency obtained to date and 9.5 W of output power in a transversely diode-pumped Nd^{3+}:YLF slab laser operating at 1053 nm were obtained.

- 620 mW Single-Frequency Nd:YVO_4/BiB_3O_6 Red Laser. Using a type-I critically phase-matched bismuth borate crystal, a record 620 mW single-frequency red laser at 671 nm is achieved from intra-cavity second harmonic generation (SHG) of a π-polarized single-end pumped Nd:YVO_4 ring laser oscillating on the lambda similar to 1342 nm transition.

- Record 1.3 W single-frequency red laser at 661 nm. Achieved from intra-cavity second harmonic generation of a Nd:YLF ring laser oscillating on the π-polarized transition (lambda similar to 1321 nm).

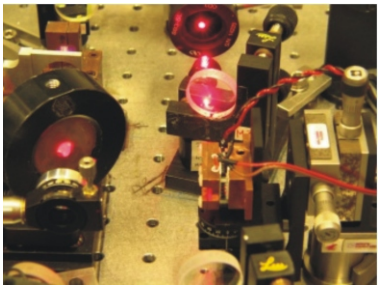


Figure 3. 1.3 Watt single-frequency Nd:YLF/ppKTP red laser

- High pulse power Er:YLF laser operating at 2.8 μm wavelength (laser cavity shown in Fig. 4).

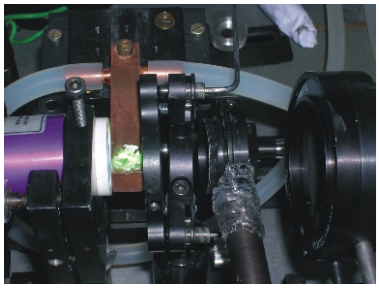


Figure 4. We reported 60% higher average output power for short pulse duration

- First single crystal Nd:YLF fiber laser. Fiber orientation (Fig. 5) and the optical arrangement used in diode laser pumping (Fig. 6) are shown.

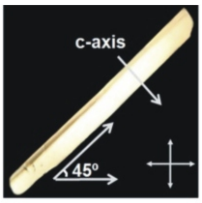


Figure 5. 700 μm diameter Nd:YLF fiber

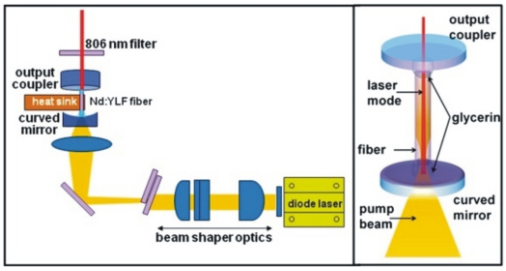


Figure 6. 300 mW of output power and 37% slope efficiency were achieved

Production and optical characterization of active laser media based on nanopowders and metamaterials

This activity comprises the development of new laser sources based on diode pumped dispersive media. Lasing action in Nd: YVO_4 nanopowder has been analyzed by investigating the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition. A method to quantitatively determine the upconversion rate and the contribution of the spontaneous emission in the samples backscattering emission that includes the random laser emission as a function of pump power has been created.

Characterization, modeling and optical spectroscopy of rare-earth doped solid laser media

A luminescence spectroscopic system with spectral and temporal discrimination that uses a Box-car technique and tunable laser excitations of 4 ns (10 Hz) in the range of 420 to 2000 nm (10mJ), was used for lifetime measurements of rare-earth ions in glasses and fluorides crystals. These measurements allowed determining the rate constant of the non-radiative energy transfer that happens due to dipole-dipole interactions between donor and acceptor ions in solids. Energy transfer mechanism involving two interacting erbium (and holmium) ions in the first (and second) excited state, energy-transfer up-conversion has been observed and the rate constant determined. The aim of this study is the development of solid laser medium emitting in the mid-infrared (~2750 nm) and to improve light signal amplifiers based on thulium-doped materials that operate in S-band of telecommunication (1470-1500 nm). A detailed investigation of the energy transfer process from the first excited state of holmium caused by Pr^{3+} ions in fluorozirconate (ZBLAN) glass was carried out. Solving the rate

equations one can verify the potential gain of the laser medium and know how it can be affected by dopant concentration (activator and sensitizer ions) and pumping intensity. ZBLAN glasses doped with 4 mol% of Ho^{3+} and co-doped with 0.1, 0.2 and 0.3 mol% of Pr^{3+} were used to evaluate the 2850 nm emission gain under 650 nm continuous pumping regime. Only the ZBLAN glass doped with 4 mol% of Ho^{3+} and co-doped with 0.3 mol% of Pr^{3+} showed a positive small signal gain, which indicates that Ho^{3+}, Pr^{3+}-codoped ZBLAN glass is a promasing candidate for high power laser operation at 2850 nm using diode laser pumping at 650 nm.
ZBLAN glasses doped with 0.5, 1, 2, 3 and 4 mol% of Dy^{3+} were investigated using the time-resolved luminescence spectroscopy. Selective laser excitation at 1125 and 1358 nm established that the energy levels above the second excited state of Dy^{3+} are entirely quenched by non-radiative multiphonon emission in ZBLAN glass. Only two emissions are present with peaks at 1700 nm and 2885 nm with low quantum efficiencies. Excited state absorption (ESA) was detected by monitoring the rise time of the 1700 nm luminescence after tuning the probe wavelength across the spectral range from 1100 to 1400 nm. As a result of nonradiative decay of the higher excited states, ESA contributes to the heating of 3 microns fiber laser based on Dy^{3+}-doped fluoride glass. It was establihed that pump ESA not the short decay times of the metastable energy levels has the biggest impact on the performance of Dy^{3+}-doped ZBLAN fiber laser operation at the previously demonstrated pump wavelengths of 1100 and 1300 nm. These results suggest that fiber laser operation is temperature dependent, which may account for large difference in performance of Dy^{3+}- doped ZBLAN fiber lasers pumped at either 1100 nm or 1300 nm because 40% more heat is generated at the former pump wavelength.

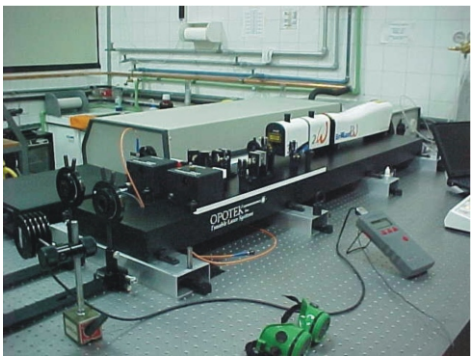


Figure 7. The decay characteristics of the excited states of rare earth ions were measured using an pulsed (4 ns) laser excitation from a tunable double optical parametric oscillator (OPO) pumped by the third (or second) harmonic of a Q-switched Nd:YAG laser working in the visible from 420 to 685 nm and in the infrared from (1) 670 to 990 nm and (2) 1150 to 2050 nm

Characterization, modeling and optical spectroscopy of plasmas

Laser plasma spectroscopy technique was applied to ultrashort pulses generated plasmas to evaluate the performance of femtosecond laser-induced Breakdown Spectroscopy (fs-LIBS) for the determination of elements in animal tissues. Sample pellets were prepared from certified reference materials, such as liver, kidney, muscle, hepatopancreas, and oyster, after cryogenic grinding assisted homogenization. Individual samples were placed in two-axis computer-controlled translation stage that moved in the plane orthogonal to a beam originating from a Ti:Saphire chirped-pulse amplification (CPA) laser system operating at 800 nm and producing a train of 840 μJ, 40 fs pulses at 90 Hz. The plasma emission was coupled into the optical fiber of a high-resolution intensified charge-coupled device (ICCD)-echelle spectrometer. Time resolved characteristics of the laser-produced plasmas showed that the best results were obtained with delay times between 80 and 120 ns. Data obtained indicate that both are matrix-independent sampling process and that fs-LIBS can be used for the determination of Ca, Cu, Fe, K, Mg, Na, and P elements. The same technique and apparatus were used to verify the applicability of the technique to monitor the migration of amalgam elements (mercury, tin, silver, copper and zinc) to the neighbor dental tissue in human samples with a spatial resolution of 100 μm. It was possible to determine the penetration depth of mercury, silver, cooper and tin. We also observed a deeper migration of these elements on permanent teeth when compared with deciduous ones. Although the presence of zinc could not be detected under the experimental conditions, the emission lines corresponding to many other elements such as calcium, sodium, phospor, among others, were observed, so configuring the femtosecond LIBS technique as a valuable tool to map the distribution of endogenous and exogenous tooth elements.

Microvascular function evaluation by low-frequency fluctuations in the laser Doppler flow signal

The laser Doppler flowmetry has been used to study microvascular dysfunctions, common in diabetics and chronic smokers. Low-frequency fluctuations in the laser Doppler flow signal (LDFS) from the skin are related to microvascular mechanisms of flow control. Wavelet spectral analysis has been used to correlate fluctuations in the LDFS with the endothelial, neurogenic and myogenic mechanisms of control in the frequency intervals 0.0095-0.02 Hz, 0.02-0.06 Hz and 0.06-0.16 Hz, respectively. Generally the signal power, in each frequency interval, derived from the respective wavelet coefficients, is used as a measure of the activity of the related mechanism of microvascular control. However, the time-domain characteristics of the fluctuations in the LDFS in each frequency interval are poorly known. As a consequence, there is a lack of objective criteria to properly measure, in each frequency interval, the related hemodynamic parameters. A time-domain method was developed to analyze and quantify fluctuations in the LDFS in each frequency band. Baseline and thermally stimulated LDFS of forearms from healthy volunteers were collected and analyzed. The data obtained indicate that inappropriate time windows, frequently used for measurements, increase the variability of the measured signal power, diminishing the capability of the method when assessing microvascular dynamics and dysfunctions. Objective criteria were proposed to diminish the measured hemodynamic parameters variability, improving the method sensitivity. Potential applications are assessing endothelial, neurogenic and myogenic dysfunctions.

Laser processing of special materials: thermal and non-thermal process

Modern technological advances have demanded the development of new materials like high mechanical strength steels, superalloys, ceramics and composites, besides very small pieces with complex geometrical forms. Consequently, traditional milling and welding processes can no longer fulfill the requirements demanded by modern applications. Hence, laser processing comes as very useful and versatile alternative method, and has been used here for cutting welding, heat treating and ablating of some materials for important technological applications. In welding, a pulsed Nd:YAG laser has been used to join very thin foils of alloys highly resistant to corrosion with the purpose of using then as protective shields for sensors against harsh media . Pressure, flow and temperature sensors used in many industrial and nuclear plants must withstand extreme conditions of pressure, temperature and corrosive environments. This is done by covering these devices with foils of special alloys with 100 μm of thickness. To accomplish this task, welding of AISI 316L stainless steel, Hastelloy C-276, tantalum, Ti6Al4V and Monel 400 superalloy has been developed in pure material and dissimilar combinations. The experiments lead to hermetic and sound laser welds in several geometries between these thin foils and also between thin and thick base metal. Dissimilar welding of AISI 304 stainless steel and Inconel 600 Ni alloy was developed and the study of the morphology of solidification was used to explain its microstructure development. Also the effects of gas protection during Ti6Al4V welding was studied and optimized. Laser heat treatment is another area of study and it was used in hardening of gray cast iron and aluminum-silicon alloy used in internal parts of combustion engines for the automobile industry. Besides thermal processes accomplished by traditional lasers, another program has been developed where thermal effects can be absent in the region of interaction between laser focus and processed piece. This occurs when laser pulses of very short temporal length, in the order of femtoseconds (10^{-14} to 10^{-13} s) are employed. To use such pulses a workstation was built and automatized and process parameters for thermal and non-thermal regions were obtained for many dielectrics, semiconductors and metals. These data are essential for machining in micron or sub-micron scale and have been used to obtain such structures in optical glasses and ceramics. Microfluidic devices were directly milled in BK7 optical glass with excellent precision and geometrical form. Texturing in ceramics surface was also done with femtosecond pulses and used to improve cellular growth and adhesion in medical implants.

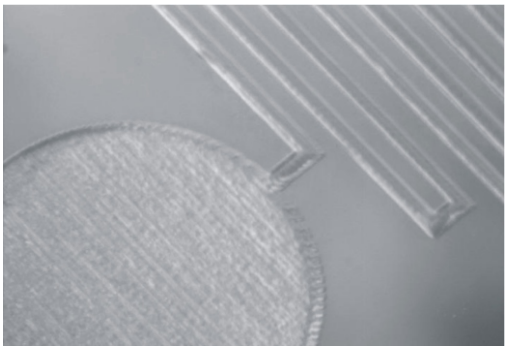


Figure 8. Microfluidic device machined with femtosecond laser pulses. Each channel has a 100 μm square profile

Physical characterization of biological tissues for the development of new diagnostic and therapeutic methods

The physical characterization of biological tissues, mainly the study of their optical properties and thermal responses, allows the development of new processes using high intensity lasers, which can be useful as new diagnostic or therapeutic methods in Dentistry and Medicine. Protocol were developed and tested for several diseases. The caries

prevention with laser was also studied *in vitro* and a clinical trial was carried out to prove the safety and effectiveness of the method. A cream for topical use for the clinical photodynamic therapy of skin cancer was developed, tested and patented in Veterinary Medicine. We are also studying the potential treatment of burned skin with high intensity femtosecond laser as well as the potential use of erbium laser to cut bone during surgery.

Prevention of dental caries with lasers "in vitro"

The decline in dental caries over the last few decades has been attributed to the extensive use of fluoride. Although fluoride is the most powerful treatment to prevent tooth decay, the development of new methods to completely control this disease is still necessary, mainly in developing countries. In this way, lasers combined or not with fluoride, have been tested on teeth to improve dental enamel properties in order to enhance its resistance to demineralization. We investigated the compositional and crystallographic changes *in vitro* on enamel when irradiated by Er,Cr:YSGG or Nd:YAG associated with black coating, its resistance to demineralization when irradiation is associated with fluoride, and CaF2-like material formation and retention. Sample surfaces were analyzed by ATR-FTIR. Irradiation with Er,Cr:YSGG laser promoted a significant decrease on carbonate content of enamel. After Nd:YAG irradiation, it was observed a significant decrease of carbonate and amides I and II. X-ray diffraction measured at Synchrotron facility showed that both laser irradiations promoted formation of α-tricalcium phosphate and tetracalcium phosphate, and a significant increase on the crystal growth of the enamel apatite. These changes can explain the improved resistance of enamel to demineralization observed in another study, in which enamel slices received professional fluoride gel applied before or after irradiation. Both lasers significantly reduced enamel demineralization, and the previous APF-gel application followed by laser showed the higher reduction of enamel demineralization. CaF_2 formed before pH-cycling was significantly higher in groups were APF was associated with laser irradiation. After demineralization *in vitro*, these groups also presented higher CaF_2 retention in respect to isolated treatments (only APF or only laser). The combined treatment of laser irradiation with fluoride propitiates an expressive fluoride uptake, reducing the progression of carieslike lesions, and this treatment is more effective than laser or fluoride alone.

Prevention of dental caries with lasers clinical trial

After all *in vitro* results described above, a double-blind crossover clinical trial was developed, in which 121 teeth of 33 volunteers were selected. In all volunteers, the right side teeth were selected for Nd:YAG laser + APF application (lased group) and the left side teeth were kept as control group (only APF application). Nd:YAG laser irradiated teeth painted with a black organic ink; after that, topical APF was applied. Recalls were made after 1 year in order to evaluate the formation of white-spot lesions or caries cavities. After 1 year, this clinical experiment showed an overall reduction of 60.2% in caries incidence in (lased group + APF) when compared with control (no treatment) and 39,2% of reduction when compared with only fluoride group. As a conclusion, combined Nd:YAG laser and topical fluoride treatment was effective for reducing the incidence of caries *in vivo*.

Lasers in Periodontology - clinical trial

The Nd: YAG laser efficacy associated with conventional treatment for bacterial reduction has been evaluate after Nd:YAG laser irradiation was associated with scaling and root planning in class II furcation defects in patients with chronic periodontitis. Thirty-four furcation lesions were selected from 17 subjects. The control group received conventional treatment, and the experimental group received the same treatment followed by Nd:YAG laser irradiation. Both treatments resulted in improvements of most clinical parameters. A significant reduction of colony forming unit (CFU) of total bacteria number was observed in both groups. The highest reduction was noted in the experimental group immediately after the treatment. The number of dark pigmented bacteria and the percentage of patients with *Porphyromonas gingivalis*, *Prevotella intermedia*, and *Actinobacillus actinomycetemcomitans* reduced immediately after the treatment and returned to values close to the initial ones 6 weeks after the baseline for both groups. The Nd:YAG laser associated with conventional treatment promoted significant bacterial reduction in class II furcation immediately after irradiation, although this reduction was not observed 6 weeks after the baseline.

Development of therapeutic processes of photosensitization and photobiomodulation

The aim of this activity is to develop new therapeutic processes using innovative technology through low-power lasers and light-emitting diodes to provide a nonthermal, noninvasive, environmental safe treatment that can be useful in the Health Sciences. The overall mission of this activity is to develop phototherapeutic processes for human health through of the physical, chemical and biological knowledge of the low intensity light tissue interaction. Our major interests are to investigate the effects of low intensity laser therapy (LILT) and photodynamic antimicrobial therapy (PAT) on biosystems.

Photobiomodulation

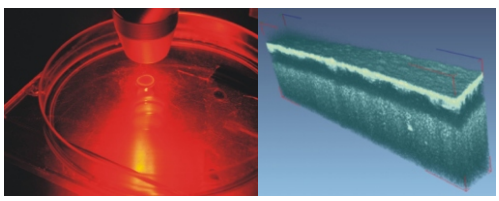
LILT is a treatment modality that is becoming more useful in Health Sciences, although mechanisms underlying light effects are still not completely understood. Our group develops researches on light dosimetry and tissue optics to provide to the health professionals scientific background for using this technology to benefit patients. Studies were carried out *in vitro* and *in vivo* to investigate the influence of LILT on cell cultures, wound healing, and edema. Our results show that LILT promotes a faster differentiation of human dental pulp stem cells into osteoblast cells compared to non-irradiated control cells opening new possibilities mostly on tissue engineering techniques; preventive laser application is effective in the prevention and treatment of oral mucositis and its daily use contributed to the relief of painful symptoms collaborating to improve the life quality of oncologic patients; near-infrared LILT showed to be efficient on edema prevention and treatment when lymph nodes were irradiated being a potential alternative to anti-inflammatory drugs; light attenuation coefficient changes during wound healing and inflammatory process suggesting that this parameter is suitable to optimize low intensity laser therapy.

Photosensitization

PAT is based on the application of a photosensitizer and a light source to kill microorganisms. PAT can be useful in life sciences, like odontology, where along with conventional treatment provides a higher efficiency to care for local infections. Alone, neither photosensitizer nor light has the capacity to produce deleterious effect on the biological system. However, when combined, they can eliminate bacteria and fungi, including those resistant to conventional antibiotics and antifungals. Our results show that PAT was able to reduce 99% of multi-resistant bacteria in burn wounds, delayed bacteremia, kept the bacterial levels lower compared to control group and increased the animal survival for 24 h; light parameters play an important role on yeast inactivation and the same energy density under different irradiation parameters presents dissimilar quantity of cell death; methylene blue transport thought yeast membrane can change PAT effects, as well as the photosensitizer (PS) preferential bind site inside the cell; microbial strain characteristics can directly interfere on PAT results and cells appear to be killed by an apoptotic-like effect; PAT can kill fungal cells *in vivo* and reduce its recovery from infected site; it is possible to induce dental caries in a rat model since demineralization areas were identified by optical coherence tomography, and PAT significantly reduced the number of total bacteria compared to control without treatment and bacterial cells level remained lower than control group until 10 days post-treatment; treatment of herpes labialis with PAT is effective, has no side effects, and when associated with LILT accelerates the healing process.

Optical coherence tomography applications

Optical coherence tomography (OCT) is a diagnostic imaging technology based on low length coherence interferometry in which the coherence features of photons are explored, leading to an imaging technology that is capable of producing non-contact, non-destructive, high-resolution cross-sectional images of internal microstructures of living tissues. We implemented several OCT systems. There are available OCT's in several wavelengths (830, 903 and 1300 nm) with polarization and Doppler flow capabilities. With a Polarization Sensitive OCT (PS-OCT) birefringence images can be performed, in this way the differences between the refraction indices can be analyzed as an image, making diagnoses simple to be performed. Besides PS-OCT images, a more complex, but also more complete, way to study the polarization properties of light can be done using the Mueller Matrix theory. OCT with micron resolution allows tiny and delicate structures precise study such as hair stream. Hair streams have about 70 microns in diameter, in such a manner that the cosmetic field can also take advantage of this technology by evaluating physical and optical effects caused in the hair streams by the chemical treatment. We have used OCT to determine the medium refraction index of different hair groups, blond and Caucasian before and after a chemical treatment. In collaboration with a cosmetic industry we developed non-invasive methods, for in vivo visualization and access the skin topography changes during a cosmetic treatment of periorbital region with an anti-aging product. We found that this method was able to quantify small variations in skin surface roughness (1%). After 28 days, significant reductions in roughness (6.2%) were observed in treated areas whereas non-treated areas showed no changes in skin roughness. Frequency depth distribution of OCT values (5 to 600 μm) showed a reduction in the frequency of 170 - 300 μm events and an increase in the frequency of 5 - 170 μm events. These results are consistent with the wrinkle reduction profile expected from anti-aging treatments. OCT proved to be a highly sensitive method to detect skin topography variation. We also have used the OCT technique to follow in real time optical changes in yeasts organized in biofilm simultaneous to PDT.



(a) (b)

Figure 9. (a) Biofilm in SE disks during PAT, (b) 3D OCT image after PAT with 1 mM MB and red laser. Image generated by VGStudioMax 1.2®

Laser remote sensing of the atmosphere

LIDAR is the acronym for Light Detection and Ranging and like RADAR operates on the same principles of sending electromagnetic radiation into the atmosphere and detecting the returned light signal. This technique is powerful dues its high temporal (seconds to minutes) and spatial (below 10 m) resolutions. With a few laser shots the system is able to characterize the atmospheric dynamics, the presence of pollutants and study dispersion patterns important features in pollution monitoring. Also this system is capable of tracing mid- and longrange transport of biomass burning activities in rural areas of the country. Also the LIDAR is an important climatological tool for studying cloud-base altitudes, water vapor atmospheric content and aerosol optical properties, which have been investigated since 2001, and the Laboratory of Environmental Laser Application was created. The LIDAR system at IPEN operated on physical principles of elastic scattering, namely Rayleigh and Mie scattering, and inelastic scattering, Raman scattering. The former is used to monitor aerosols in the atmosphere, and besides their optical properties, aerosols act as tracers for atmospheric dynamics and cloud formation. The Raman channels in the system are used to perform water vapor, nitrogen and oxygen concentration profiling. Besides in a collaborative effort with Howard University and Goddard Space Flight Center NASA we have developed an independent calibration methodology to obtain a vertical distribution of water vapor mixing ratio in the atmosphere based on physical principles. The Laboratory of Environmental Laser Applications has started in 2008 a joint project with Instituto Politécnico at University of São Paulo SP to develop a LIDAR like system to evaluate oil refining processes based on the particle distribution exhaust from refineries and the system is part of a The National Institutes of Science and Technology Program (INCT). The latest project involves the understanding of the so-called aerosol indirect effects based on the particle higroscopicity an important process in climate change.

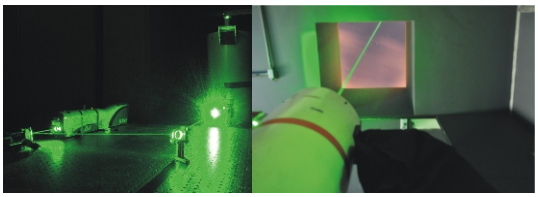


Figure 10. LIDAR system in operation at IPEN, which shows the second harmonic laser beam from Q-switched Nd:YAG laser (left) and the telescope (right)

Operation and optimization of the TW peakpower laser and applications

High power ultrashort pulses lasers based on CPA (Chirped Pulse Amplification) technologies that allow the study, in conventional laboratory scales, of phenomena that only 10 years ago were restricted to national laboratories with annual budgets of billions of dollars. In the Center for Lasers and Applications at IPEN, a hybrid Ti:Sapphire/Cr:LiSAF TW peak power laser system is under continuous development. A flashlamp pumping cavity for a Cr:LiSAF gain medium in the shape of a rod was built. The pumping cavity was developed aiming to minimize the thermal load on the Cr:LiSAF crystal by the use of absorption filters between the filters and the gain medium, allowing the amplification of ultrashort pulses to the terawatt peak power region at high repetition rates. The pumping cavity was initially used in a laser configuration, and generated 60 μs pulses with energy up to 2.8 J, with an average power of 30 W at 15 Hz repetition rate, the highest reported to date. The utilization of the pumping cavity in a hybrid Ti:Sapphire/Cr:LiSAF CPA system produced pulses with 30 mJ of energy and 60 fs of duration at 5 Hz repetition rate, reaching 0.5 TW of peak power, the highest in the southern hemisphere. Our laboratory also has another amplified laser system capable of generating up to 800 μJ, 25 fs pulses or 300 μJ, 5 fs pulses. Even at lower peak powers, the ultrashort character of the pulses generates nonlinear phenomena, particularly those initiated by multiphotonic processes that generate free electrons. Ultrashort pulses with up to 30 GW peak power were utilized to crest defects (color centers, vacancies) in crystals, glasses and polymers, to measure nonlinear effects in solid and liquid samples, and to ablate and machine technological materials with precision on the micrometer scales without heat affected zones. This nonthermal ablation is being studied for the removal of necrosed material from burned animals and its effects on the tissue regeneration. Also, the generation of High Harmonics by ultrashort lasers pulses is under study to produce in the VUV and soft X-ray region with durations into the attosecond regime.

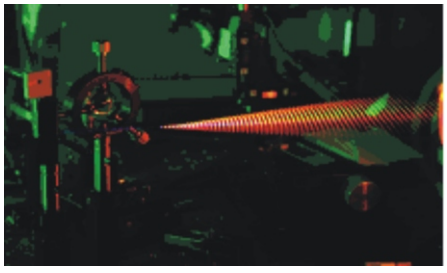


Figure 11. Harmonics generated by 1 Hz trains of pulses focused in air

Color center production by high intensity ultra short laser pulses

Ultrashort pulses with peak powers up to 30 GW were used to create color centers in fluoride crystals grown in our facilities, in glasses and polymers. It was possible to determine that the centers generation begins with multiphotonic absorption, favored by the high intensity due to the short pulse duration that neutralizes negative ions, which are kicked out of their sites. Once the centers are formed by electrons trapped in the vacancies, they act as a probe to study the material structure. The confined characteristic of the laser beam creates a high density of basic color centers that results in its aggregation in secondary, more complex, centers. These secondary centers are generated with a higher concentration than attainable with traditional ways of creating centers, such as exposure to ionizing radiation. Also, the laser excites the centers during its formation process, allowing emission spectroscopy to be performed during its creation. Centers were created in LiF (Lithium Fluoride), pure, Tm and O doped YLF (Yttrium Lithium Fluoride) crystals, and also in BaLiF crystals grown by different methods. Color centers were similarly created in ZBLAN glasses and PMMA polymer samples, and its properties investigated.

Pump and probe studies of ultrafast magnetization dynamics in the femtosecond timescale

Since the advent of reliable solid state lasers in the 90's producing ultrashort light pulses routinely, the field of time-resolved optical spectroscopy has experienced a boom. The availability of new sources, wavelengths and increased time resolution has allowed better comprehension of different phenomena occurring in chemistry, condensed matter physics and biology. In this context one of the most widespread used techniques to study ultrafast phenomena is the pump and probe optical technique. In this kind of experiment one stronger ultrashort light pulse interacts with the medium under investigation, disturbing the system. The return of the system to equilibrium is then monitored by probing the optical properties of the system with a weaker delayed ultrashort probe pulse. The arrival of the light probe is controlled in a stroboscopically way, following the return of the system to equilibrium. After the pump pulse excites the system, the gained energy will be redistributed in the medium in different channels, and the internal forces governing this redistribution of the energy can be investigated. A pump and probe setup was bult in our ultrafast laser laboratory, to study the ultrafast dynamics of the magnetization in technological materials by the simultaneous measurement of the electron and spin dynamics with time resolution limited by the duration of the pump pulse. Amplitude and the direction of the magnetization vector M were measured to retrieve its trajectory from tens of femtoseconds up to a few

nanoseconds in Co-rare earth doped films. Our focus is the investigation of the role of the rare earth doping in the ultrafast demagnetization and damping of the precessation motion in transiction metals.

Determination of the ultrashort pulses ablation threshold of solid samples

Ultrashort pulse laser ablation of solids is due to an electron avalanche induced breakdown process that occurs when seed electrons are accelerated in the laser field, exponentially generating free electrons by collisions. The breakdown takes place when the plasma originated by the avalanche electrons reaches a critical density and transfers energy to lattice ions, which expand away from the surface after the pulse has passed. In metals, the seed electrons are always present (conduction band free electrons), and in dielectrics and semiconductors they are excited from the valence band to the conduction band by the pulse leading edge. Although the seed electrons have dissimilar origins in different material types, a metallization occurs in dielectrics and semiconductor after they are produced, and the avalanche evolves deterministically in time in the same way in all solid materials that now behave like metals. These mechanisms confer a nonselective characteristic to the ultrashort pulse ablation, and the intensity ablation threshold of a material is the only parameter relevant to the etching process. In our labs we created a new method to determine the ablation threshold by ultrashort laser pulses, based on scanning the sample diagonally across the beamwaist of a focused beam. The scan etches a profile in the material surface, and the measurement of this profile maximum transversal dimension and the knowledge of the laser pulses power immediately determines the material ablation threshold. We have applied this technique to a variety of materials, ranging from crystals to metals, composite polymers and even biological samples (animal tooth and bone), obtaining results agreeing with the ones reported in the literature.

Generation of high harmonics into the VUV and soft X-ray spectral regions

When an ultrashort pulse impinges on a gas at low pressure, electrons can be freed from its parent atoms by the leading edge of high intensity ultrashort pulses, and then be accelerated by the pulse carrier wave into a quivering motion. When these electrons collide with the atom, its energy is released in the form of harmonics of the exciting field, and if the kinetic energy is sufficiently high, the harmonics can reach the UV and soft X-ray region, generating photons up to a few keV. In the High intensity ultrashort pulses laser laboratory, we are pursuing the generation of these harmonics into the region of the water window, around 2-4 nm, which are proposed to be used in high-resolution radiographies of living tissues. For this goal, pulses with durations of 25 fs and 5 fs will be focused on noble gases at pressures ranging from around 50 mbar up to 500 mbar, inside a vacuum chamber instrumented for the generation, detection and characterization of the harmonics produced.

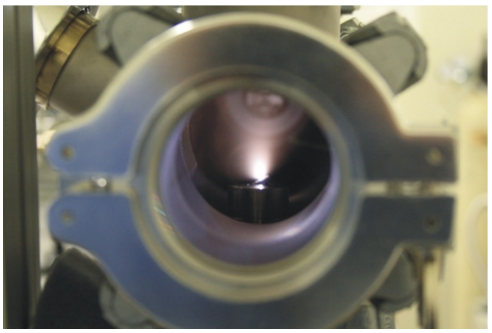


Figure 12. Ablation of a metallic sample under vacuum, to study the isotope enrichment due to the separation that occurs in the ablation plume

Lasers Technology

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Honor Mention and Awards

Claudia Rodrigues Emilio and Denise Maria Zezell - "Comparison between two different photodymanic therapy of tumor protocols applied to the treatment of skin cancer in feline", doctorate project was selected to participate at the Campus of Excellence 2008, Spain - veterinarian medical area. In this event, more than 10 Nobel Prizes discussed and choosed the best 100 graduate projects from Latin America and Africa.

Anderson Zanardi de Freitas, Nilson Dias Vieira Junior, André Rolim Baby, Telma Mary Kaneko, Maria Valéria Robel Velasco, Monica Beatriz Mathor and Valcinir Bedin - "Optical coherence tomography applied to cosmetology: preliminary structural characterization of hair fibers", Best Work Award (Third Place) - XXII Congresso Brasileiro de Cosmetologia, ABC - Associação Brasileira de Cosmetologia, 2008.

Anderson Zanardi de Freitas, Marcus Paulo Raele, Hindra Colodetti and Luciana Sanglard - "Measurement by optical coherence tomography of wear promoted in the enamel of deciduous teeth by abrasion technique", Prof. Guedes-Pinto Award (Third Place) - IV Congresso de Odontopediatria - Associação Paulista de Cirurgiões-Dentistas, 2008.

Honor Mention and Awards

Fábio Juliano da Silva Lopes and Eduardo Landulfo - "Measurements of tropospheric aerosol in São Paulo - Brazil using a Lidar system from Centro de Lasers e Aplicações do IPEN and CALIPSO satellite", LAMP Poster Award SPIE; Best Poster Award (Third Place) - Winter College on Optics in Environmental Science - International Centre for Theoretical Physics - Trieste, Italy, 2009.

Marcus Paulo Raele, Marcello Magri Amaral, Nilson Dias Vieira Junior and Anderson Zanardi de Freitas - "PS-OCT Birrefringent Meassurements", Best Poster Award (Optics) - XXXII Encontro Nacional de Física da Matéria Condensada, Águas de Lindóia - São Paulo, 2009.

IPEN was engaged in the Projeto Cenpe-Cana financed by Petrobrás to measure the level of atmospherical pollution in the interior of São Paulo state using a LIDAR movel station. The physicist Eduardo Landulfo gave an interview to EPTV, subsidiary of TV Globo (Rio Claro), to explain the project objectives that uses a LIDAR mobile station for analising the pollution caused by cane burning, 2009.

Luciana Faria Sanglard, H. Colodetti, Célia R. M. D. Rodrigues, Nilson Dias Vieira Junior, Marcus Paulo Raele and Anderson Zanardi de Freitas - "Optical coherence tomography and scanning electron microscopy analysis of micro abrasion effects in deciduous teeth enamel", Best Oral Presentation (Second Place) - I Encontro da Divisão Sul Americana da Federação Mundial de Laser em Odontologia e do V Congresso da Associação Brasileira de Laser em Odontologia - São Paulo, SP, 2009.

The physicist Ricardo Elgul Samad won the 4th edition of the After Image Photo Contest sponsored by the Optics and Photonics News (OPN), an international magazine. The magazine, which publishes the latest developments in the field of optics, is a monthly publication of the Optical Society of America and is directed at researchers, engineers, entrepreneurs and students. In 2009 the magazine received 81 entries to compete with images produced in laboratories, a record number of participants. In the winning shot, the effect produced by the motion of a sheet of paper across the pulses from a high power laser installed at IPEN is observed.

Anderson Zanardi de Freitas - "Avaliação do uso da tomografia de coerência óptica em dermatologia", Inovação Tecnológica Natura Campus 2010 Award (First Place) - ceremony realized in the Natura company centre. Freitas won also a total free course in the Massachusetts Institute of Technology (USA) and a MAC Book Pro13 microcomputer.

Fuel Cell and Hydrogen laboratory

Renewable Energies

Introduction

Hydrogen is seen by many as a key energetic vector for the 21st century. Its utilization in fuel cells enables a clean and efficient production of electricity. The different ways of obtaining hydrogen and the different types of fuel cells application have called the attention and investment of developed countries. European Union, United States, Canada and Japan have important programs that establish tied goals for the utilization of fuel cells technology in transport and distributed energy. Aware of the importance of this technology for the energetic future of Brazil, IPEN started 10 years ago the development of fuel cells for stationary and distributed energy applications. Preliminary studies were carried out at the Materials Research Center due to IPEN expertise on nuclear materials development. Based on both the good initial results and the proposition of the Brazilian Fuel Cell Program ($ProH_2$) by the Ministry of Science and Technology (MCT), IPEN decided to organize an institutional program on the subject, conducted at the Fuel Cell and Hydrogen Center.

The objectives of the IPEN program are based on the MCT national program, contributing significantly to the national development in this area. The R&D Program was structured in a cross-cutting way involving human and infrastructure resources from many IPEN Technical Departments. The program comprises three main areas of interests: PEMFC (Proton Exchange Membrane Fuel Cell); SOFC (Solid Oxide Fuel Cell); and H_2-PRODUCTION, mainly from ethanol reforming. More than 50 professionals were engaged at this development, although some in part time, including PhDs, MSc, and both undergraduate and graduate students.

Important scientific and technological results have been obtained and the main achievements can be observed by the patents, published international papers, the post-graduated courses given, and the graduate student's thesis advisory. Since 2004, the PEMFC Laboratory was transferred to a new site, improving its research capabilities, which includes catalyst and MEA preparations and fuel cell stack test up to 5 kW electric power. In the period of 2005-2007 new laboratories of SOFC, Hydrogen and Fuel Cell Systems have been implemented. In the period of 2008-2010 our attention turned also to scaling up, reliabilities studies and small demonstration projects. A new building for housing additional personal was built during this period.

The financial resources were based on scientific funds from federal and state government agencies (FINEP-MCT- $ProH_2$, FAPESP, CNPq and CAPES). Today, IPEN is considered as an important partner within the R&D networks established by the MCT-$ProH_2$ Program. Partnership with emerging enterprises from CIETEC (Incubator Center) and others led to advances and autonomous technological domain in some areas.

Proton Exchange Membrane Fuel Cell (PEMFC)

The activities of the Proton Exchange Membrane Fuel Cell (PEMFC) Group are focused on both the basic and technological developments of hydrogen fueled PEMFCs and the direct oxidation of alcohols, such as methanol (DMFC), ethanol (DEFC). The main goal concerns stationary and portable applications for distributed electric power generation.

Amongst the main research subjects are: the development of new methods of electrocatalysts production and new electrocatalysts systems; development, production, and characterization of new composites electrolytes for high operating temperatures (130°C); production, characterization and optimization of membrane electrode assembly (MEA); modeling and simulation of PEMFCs; unit cells tests in laboratory and pilot scales; development of low power fuel cell stacks; development of innovative radiation grafting techniques, combining direct and indirect processes; development of styrene grafted PP, PVDF, ETFE, FEP, PFA and PTFE films; development of sulfonated films of styrene grafted PVDF, ETFE, FEP, PFA and PTFE; polypropylene membrane base performed useful life of 300 hours, operating at 40°C; ETFE membrane based on fluorinated polymer performed useful life of 53 hours, operating at 80°C; and education.

Highlights 2008-2010:

- Development of new method for the preparation of skeletal-type PtSn/C electrocatalyst by chemical dealloying.
- Development of new electrocatalysts formulations like PtSn/CeO_2-C (Fig. 1) and $Pt/Sb_2O_5.SnO_2$-C for ethanol direct electron-oxidation.
- Scaling up of PEM-electrocatalysts production up to 50 g, including the formulations Pt/C and PtRu/C, using the IPEN alcohol-reduction process (Patent BR200304121-A), in cooperation with Evonik Brasil Ltda.
- Development of Nafion-Titanate Nanotube, Nafion-TiO_2 and Nafion-SiO_2 Composite Membranes for PEMFC and DEFC, operating at 130°C (Fig. 2).
- Scaling up of Membrane-Electrode-Assembly (MEA) by sieve printing method, up to 250 cm^2 of electrode area. Delivery of 100 MEAs to Electrocell company for demonstration in a 5 kW PEMFC Stack (Fig. 3).
- Modeling and Simulation of PEMFC and DMFC components using CFD techniques.
- Development of a PEMFC stack of 1 kW electric power using technology developed at IPEN (Fig. 4).
- Reliability studies of PEMFC components.

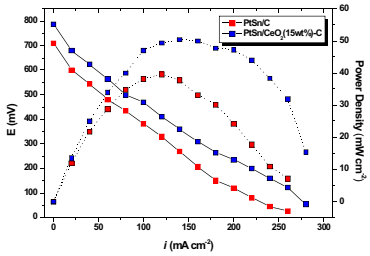


Figure 1. Electrical performances of a 5 cm^2 DEFC (Direct Ethanol Fuel Cell) at 100°C using PtSn/C and PtSn/CeO_2-C electrocatalysts anodes (1 mg Pt cm^{-2}catalyst loading) and Pt/C E-TEK cathode (1 mg Pt cm^{-2}catalyst loading, 20wt% catalyst on carbon), Nafion 117 membrane, ethanol flow rate of 2.0 mol L^{-1} and 2 bar oxygen pressure

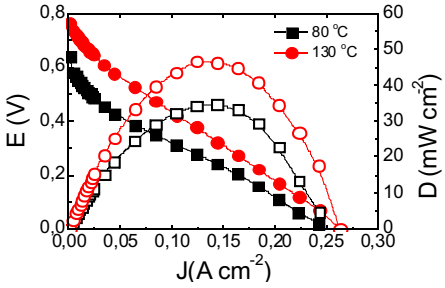


Figure 2. Polarization curve of a DEFC, using composite Nafion-TiO_2 membranes and PtSn/C electrocatalysts produced at IPEN, operating at 80°C and 130°C

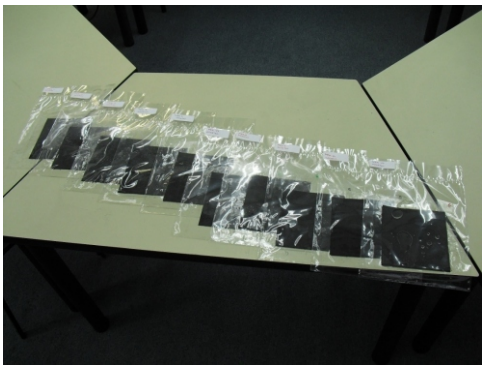


Figure 3. Membrane-Electrode-Assembly (MEA) with 250 cm^2 electrode area fabricated by sieve printing

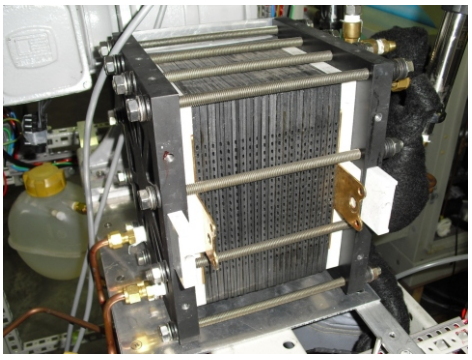


Figure 4. PEM fuel cell stack of 1 kW electric power, using technology developed at IPEN

Solid Oxide Fuel Cell (SOFC)

Solid Oxide Fuel Cells (SOFCs) are the most efficient electrochemical device to convert the chemical energy of fuels into electricity. Such fuel cells are regarded as a promising power source for several applications due to important characteristics such as: i) wide range of power outputs (from centralized power plants of MWatt to auxiliary portable units of a few Watt); ii) fuel flexibility, SOFCs potentially run on different fuels such as hydrogen, natural gas, and ethanol; and iii) environmentally friendly energy generation with rather low noise and harmful emissions.

Among the various possible SOFC designs, the planar type is claimed to have the advantages of high power density per unit volume and low production costs. Two main configurations of planar SOFCs are under development worldwide: electrolyte supported and anode supported. Each of them has particular characteristics, but the anode supported cell is regarded as the most promising technology due to its low operating temperature. Essentially, SOFCs consist of two porous electrodes separated by a dense electrolyte. Such a ceramic fuel cell requires complex fabrication technologies and each component must fulfill several different criteria. Physical and chemical compatibility and stability at high temperature and oxidizing/reducing environments, and good electrochemical properties are key issues for the materials used for this technology. Important tasks in SOFC research are the development of fuel flex anodes and the reduction of the operating temperature from 800-1000ºC down to 500-800ºC range, in order to reduce degradation of cell components, improve flexibility in cell design, and lower the material and manufacturing costs by the use of cheaper and readily available materials.

The research activities at IPEN are primarily concerned with the development of the SOFC materials, aiming at the use of simple and low-cost methods for high-performance planar SOFC components. The activities of the SOFC research group at IPEN have been focused on the synthesis, processing, and characterization of the SOFC components, and single cell testing. Several different synthesis techniques have been used for the preparation of SOFC components. The main constituents of a SOFC have been investigated at IPEN, including ceramic-metal composites for anodes, doped lanthanum manganites for cathodes, zirconia based solid electrolytes and doped chromite interconnects. In addition, alternative materials for all components have been studied, such as ceria based electrolytes, ceramic anodes, and cobalt-ferrite cathodes. The synthesized materials are tailored according to the requirements of different processing techniques such as tape casting, spin coating, and spray deposition, in order to fabricate ceramic layers for SOFC.

Ceramic electrolyte powders, such as yttria-stabilized zirconia (YSZ) and samaria or gadolinia-doped ceria, have been synthesized by chemical and hydrothermal techniques for SOFC component manufacturing. Such synthesis techniques allow the control of microstructural properties, such as grain shape and grain size distribution, that are important to achieve the necessary requirements for the processing of ceramic powders into SOFCs components. Tape casting is widely used for electrolyte and anode forming (Fig. 5) into layers for SOFC, and ceramic powders synthesized at IPEN were tailored for such technique. Figure shows a YSZ flexible green tape (Fig. 5a), and a cross section of the sintered (1500 °C) YSZ tape (Fig. 5b), evidencing a homogeneous and dense layer with thickness of ~80μm.

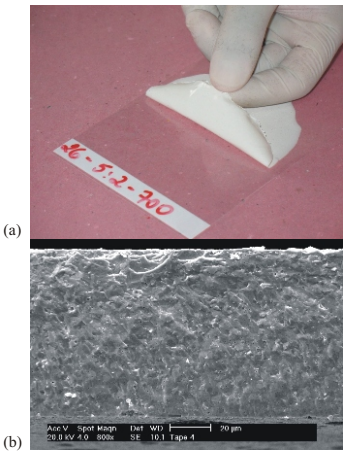


Figure 5. Yttria-stabilized zirconia tape for application as SOFC electrolyte: (a) after tape casting processing and (b) scanning electron micrograph of the cross section of the sintered YSZ tape

A spin coating method for the production of thin (10 μm) and dense yttria-stabilized zirconia (YSZ) was developed for the fabrication of anode supported SOFCs, as shown in Fig. 6a. Specially designed YSZ suspensions were developed in order to obtain 10 μm layers with a minimal number of deposition steps. The fabricated electrolyte layers are homogeneous and dense, with thickness within the desired range, as shown in the Figure 6b.

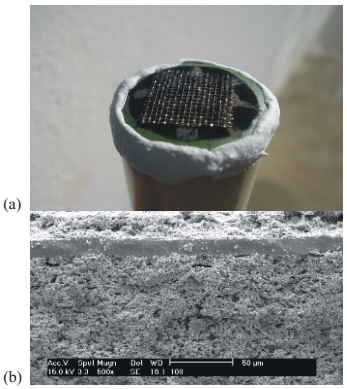


Figure 6. (a) Electrolyte supported SOFC (20 mm diameter, with Pt grid attached to the surface) sealed on the top of an alumina tube for single cell testing. (b) Scanning electron image of the fractured cross section of a single SOFC with spin coated dense YSZ electrolyte layer deposited by spin coating

In both electrolyte and anode supported SOFCs, controlling the morphology of the anodes is key issue for high performance devices. In order to fabricate an optimized microstructure with controlled grain size for both phases and porous, several techniques have been used for the production of the anode. The Figure 7 shows scanning electron micrograph of YSZ / NiO anode produced by combustion synthesis and sintered at 1300°C with pore former (rice starch) addition. The development of fuel flex anodes is a key point to advance the SOFC technology. In that context, direct ethanol SOFCs have been investigated. The main activities in this area are the development of active anodes for ethanol conversion, without coke formation, and the optimization of operating conditions for stable and high performance ethanol fueled SOFC. Preliminary tests of ethanol fueled SOFC evidenced the higher activity of ceria-based cermets than YSZ-based anodes at temperatures < 800°C.

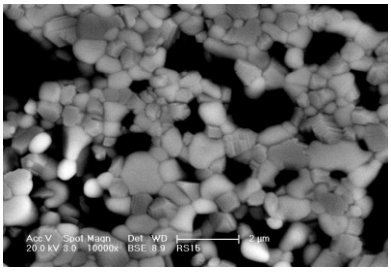


Figure 7. Scanning electron micrograph of porous ceramic yttria-stabilized zirconia / NiO composites for SOFC anode

New cathode materials have been investigated for intermediate temperature SOFCs operating in the 500-800ºC range. The perovskite oxides $Ba_{0,6}Sr_{0,4}Co_{0,2}Fe_{0,8}O_{3-\delta}$ and $La_{0,6}Sr_{0,4}Co_{0,2}Fe_{0,8}O_{3-\delta}$ have been prepared by the citrate-EDTA and the polymeric precursor technique, respectively. The micrographs in Fig. 8 show the morphology of single phase powders heat treated at 800°C.

(a)

(b)

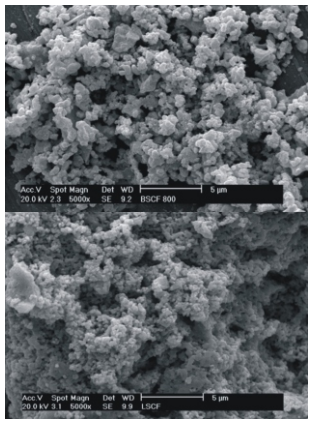


Figure 8. Cathode materials for intermediate temperature SOFCs prepared by chemical synthesis methods. (a) $Ba_{0,6}Sr_{0,4}Co_{0,2}Fe_{0,8}O_{3-\delta}$, and (b) $La_{0,6}Sr_{0,4}Co_{0,2}Fe_{0,8}O_{3-\delta}$

Hydrogen production

The widespread agricultural activities in Brazil led Hydrogen Production group to develop processes using biomass from agriculture and landfills as hydrogen source. Such efforts include bioethanol steam reforming, ammonia cracking of waste from chicken and eggs farming, and gasification of biomass originated from agricultural residues such as sugarcane bagasse, coffee straw, and cashew nut shells, always seeking out ways to mitigate environmental burden of energy production.

Catalysts development

Obtaining suitable catalysts for processes like ethanol steam reforming is an essential knowledge for hydrogen production for PEM fuel cells. Using active metals such as nickel, cobalt and copper supported onto either zirconia or alumina microspheres (Fig.9), the Hydrogen group prepares highly active catalysts for ethanol conversion showing good hydrogen yield.

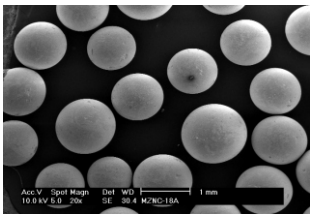


Figure 9. Cu/Ni catalysts supported on zirconia microspheres

Development of selective membranes for the purification of hydrogen

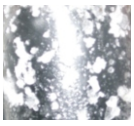
During ethanol reforming, secondary products such as CH_4, CO, CO_2 and oxygenated compounds like acetaldehyde, ethyl acetate, acetic acid are produced along with hydrogen. The purification of hydrogen can be achieved by using selective membranes made from ceramic or porous metallic substrates coated with palladium or silver-palladium alloys.

Development of gasification processes

Coffee straw, sugarcane bagasse and cashew nut shells are currently studied as viable sources for hydrogen production processes that include CO_2 capture techniques. (Fig.10)

(a)

(b)



(c)

Figure 10. (a) sugarcane bagasse; (b) gasificationsystem; (c) crystals obtained by CO_2 capture during gasification process

Project and construction of thermoreactors
Hydrogen Laboratory activities include prototyping and upscaling of laboratory thermoreactors using primary fuels and membranes for the production of high purity hydrogen (Fig. 11).

Figure 11. Ethanol reforming reactor

Fuel Cell systems

Activities of the Systems Group were substantially increased after personnel expansion by the enrollment of three additional permanent collaborators. A second unit for hydrogen and oxygen generator has been acquired to accommodate for ample production of such gases. Total production of ~30 m^3/h (20 m^3/h H_2 and 10 m^3/h O_2) should be enough to feed PEM fuel cell systems in excess of 20 kW (Fig.12).

Figure 12. Hydrogen and oxygen generators on their installation site

A 1kW PEM fuel cell system was assembled using the technology developed at IPEN (Fig.13).

Figure 13. 1kW PEMFC system made with IPEN technology

PEMFC life cycle testing facilities were expanded by the installation of two brand new test benches (Fig.14).

Figure 14. New test benches improved their previous versions by the inclusion of mass flow meters and a PLC controller

Renewable Energies

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Honor Mention and Awards

Best poster in the I Simposium Iberico of Hydrogen, Fuel Cells and Advanced Batteries (HYCELTEC 2008), held in Bilbao, Spain, from the 1st to the 4th of July, 2008. The paper “Graft polymerization of sulfonated styrene from carbon black surface and its use as electrocatalysts support for PEMFC and DMFC applications” was developed as a collaboration between the Instituto de Pesquisas Tecnológicas (IPT) and IPEN, and is co-authored by Marcelo Carmo, João Guilherme Rocha Poco, and Marcelo Linardi.

Nuclear Reactors and Fuel Cycle

Nuclear Reactor IEA-R1

Nuclear Reactors and Fuel Cycle

Introduction

The Center for Nuclear Engineering has shown, along several years, expertise in the field of nuclear and energy systems and correlated areas. Due to the experience obtained over decades in research and technological development at Brazilian Nuclear Program, personnel has been trained and started to actively participate in the design of the main system that will compose the Brazilian Multipurpose Reactor (RMB) which will make Brazil self-sufficient in the production of radiopharmaceuticals. The institution has participated in the monitoring and technical support concerning the safety, licensing and modernization of the research reactors IPEN/MB-01 and IEA-R1. Along the years from 2008 to 2010 numerous specialized services of engineering for the nuclear power plants Angra 1 and Angra 2 were carried out, in addition to the development of many related technologies applied to nuclear engineering, thus enabling the institute to fulfill its mission, that is to contribute in improving the quality of life of Brazilian people.

The Nuclear Fuel Center is responsible for the production of the nuclear fuel necessary for the continuous operation of the IEA-R1 research reactor. Development of new fuel technologies is also a permanent concern. A program for autonomous serial fuel element production started in the 80's, motivated by the political constraints for buying these fuels abroad in order to keep the reactor in operation. The fuel element fabricated at IPEN is a dispersion LEW (Low Enriched Uranium) Material Testing Reactor fuel type, and uses 20 wt% enriched uranium. Both the U_3O_8-Al and U_3Si_2-Al fuel are well qualified for reactor operation up to an average burn-up of 45 %. The U_3Si_2 fuel has been used in the IEA-R1 reactor since 1999, with good performance. The Institution is capable of fabricating U_3Si_2 enriched powder, allowing the domestic fabrication cycle of the dispersion fuel for research reactors, completing the fuel cell cycle from mining to the pellet/plate production. Therefore, Brazil is totally independent in materials and technology to fabricate nuclear fuels for its own research and test reactors. This achievement has placed our country among a few commercial manufacturer countries of LEU uranium for fuel elements of nuclear research reactors. In the last years, IPEN-CNEN/SP, besides its own fabrication needs, is fully accomplished to develop U-Mo and other new nuclear alloys for the next generation of fuel elements.

In the triennium considered, the IEA-R1 research reactor has been operated most of the time at a power of 3.5 MW and operation schedule of 62 hours per week. Other activities were also carried out to extend the lifetime of the reactor, to improve the conditions to comply with the user needs and to allow operation at higher powers.

Nuclear Research Reactors Fuels

Uranium-Molybdenum technology

For the last 30 years high uranium density dispersion fuels have been developed in order to accomplish the low enrichment goals of the Reduced Enrichment for Research and Test Reactors (RERTR) Program. Gamma U-Mo alloys, particularly with 7 to 10 wt% Mo, as a fuel phase dispersed in aluminum matrix, have shown good results concerning its performance under irradiation tests. That's why this fissile phase is considered to be used in the nuclear fuel of the Brazilian Multipurpose Reactor (RMB), currently being designed. For that reason efforts are under way at IPEN-CNEN/SP for developing the fabrication technology of this new fuel.

Gamma U-Mo alloys have been for long considered as fuel phase in research and test reactors using dispersion fuel in aluminum matrix. Promising results concerning performance under irradiation tests of U-Mo alloys, especially with molybdenum content ranging from 6 to 10 wt.% Mo, have encouraged IPEN-CNEN/SP to consider this fuel phase for the second stage of the RMB reactor operation, since uranium silicide compound, U_3Si_2, already produced, will be used at first.

The main challenge is the powder production from these ductile alloys. As a fuel based on dispersion concept U-Mo alloys must be used in powder form. At least three main fabrication routes for U-Mo powders could be listed: atomization (mainly centrifugal atomization by rotating disk method or even rotating electrode process, mechanical comminution, i.e., machining or grinding, and chemical comminution, i.e. hydride-dehydride process, also known by its acronym HDH. HDH of gamma U-Mo alloys can be accomplished by heating the alloy at temperatures where it decomposes in two phases, i.e. alpha and gamma' (U_2Mo). Since alpha uranium is easily hydrated, generating a very fine powder, its content must be carefully controlled (changing soaking time) in order to obtain particles within the desired size range. A variation of the last route, named HMD, combines hydriding-dehydriding with milling process. According to the authors, gamma U-Mo alloys can form a U-Mo hydride (A-15 structure) that embrittles the alloy, but not intensively, so powder could only be produced by interposing a milling operation, before dehydriding.

Each one of the previous routes are held by commercial or potential suppliers based on features like particle size range yield of the powder, costs, and the more important one, irradiation behavior. Since there are some controversial arguments, an investigation at local process conditions is necessary to find the best technological solution.

The focus of the preliminary work was to compare the characteristics of U-Mo alloy powders (10 wt% Mo) fabricated by two routes: mechanical grinding and HMDH. In this particular case, HMDH stands for hydrogenation-milling-dehydrogenation, since a hydride phase was not formed. It was reported before that gamma U-Mo alloys (particularly U-10wt% Mo) suffered a loss of ductility when submitted to a hydrogen atmosphere, by incorporating hydrogen interstitially. This fact was used to provide enough brittleness to the alloy to allow comminution.

Ingots of U-Mo alloy with 10 wt% Mo were induction melted into a magnesia-stabilized zirconia crucible. Metallic uranium and metallic molybdenum were used as raw materials. Metallic uranium was home produced through magnesiothermic reduction and the metallic molybdenum was supplied with 99.95% purity, as small cylinders with 3 mm in diameter and 3 mm in height. Both materials were charged inside the zirconia crucible and heated by induction under high-purity argon atmosphere up to melting. Melting temperature was maintained for 3 minutes providing homogenization, than the furnace was turned off allowing the alloy to be solidified inside the crucible. The solid material was a cylindrical piece with near 40 mm in diameter and 50 mm in height, weighting around 1200 g with a density of 16,87 g/cm^3. The ingot was treated at 1000^{0}C for 72 hours under pure argon and quickly cooled for retention of the gamma phase. It was cut in pieces for studying the two routes for powder preparation, namely mechanical grinding (MG) and hydrogenation-milling-dehydrogenation (HMDH).

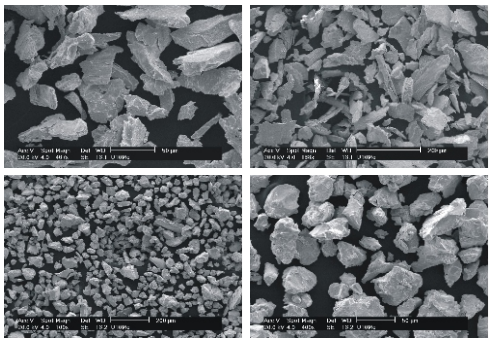
For the mechanical grinding route, the powder was produced by using high-speed grinding (15000 rpm) with diamond abrasive wheel. The abrasive wheel was 4 mm in diameter; having impregnated diamond particles with mean diameter of about 100 μm. Grinding was accomplished inside a glove-box under protective argon atmosphere.

For the HMDH route, small pieces were taken from the U-Mo ingot (with approximately 10x50x5 mm in thickness) and were individually heated at 400^{0}C for 3 hours under high purity hydrogen (99.9999%) at 3 bar. A Sievert type apparatus was used and no measurable hydrogen intake could be noticed, with the pressure gauge used (precision of 0.5 bar). At this temperature and time, alpha phase is not supposed to be formed, since about 40 hours would be necessary to start the gamma decomposition according to published TTT diagram. Next the pieces were manually crushed in a stainless steel mortar. The resulting granules were 3 mm in length. For comparison, crushing of pieces not hydrogenated treated was carried out but not succeeded due to ductile behavior of the parts. This was taken as an indication that some hydrogen intake must be occurred in former pieces. U-Mo granules were milled in a planetary ball mill at 400 rpm for 10 hours, with ball-to-powder weight ratio of 20:1. The vial and the balls were made from hardened steel. Loading and opening of the vial occurred inside a glove-box with protective argon

atmosphere. After milling the powder was heat treated under vacuum at 400^{0}C for dehydrogenization.

Figure 1 shows the morphology of the powders prepared by both routes, mechanical grinding (MG) and hydrogenation-milling-dehydrogenation (HMDH). It was observed that the powder prepared by MG route (left column in Fig. 1) presents particles with acicular and flake shapes, while the particles from HMDH route (right column in Fig. 1) are more regular and equiaxial. The powder prepared by MG route presented particles sensibly larger than the ones prepared by the HMDH route. The mean particle size (50 wt%) was about 100 µm for MG powder and 50 µm for HMDH powder. Furthermore HMDH powder particles fit very well the size requirements of dispersion fuels, with practically 100 wt% below 150 µm and about 30 wt% below 45 µm, while MG powder particles were shown to be larger, with more than 20 wt% above 150 µm. Other important difference was the aspect ratio of the particles (maximum to minimum Ferret's diameters). The aspect ratio reached 10 for the MG powder, much higher than the maximum ratio measured for HMDH powder, close to 4.

The partial results indicate the technical feasibility for producing powders from both investigated routes. The control of variables of the MG route, such as the size of the diamond granules of the abrasive wheel, the pressure of the tool under the alloy surface and the rotation of the grinding wheel machine, should promote the necessary adjustment in the particle size distribution. Powder produced by the HMDH present a particle size distribution compatible to be used as a dispersion fuel. Further work is necessary to increase the yields in order to evaluate both process routes as real technological alternatives for nuclear fuel powder production to research reactors. This project has an important cooperation of the staff of Materials Science and Technology Center of IPEN-CNEN/SP. This project was partially financed supported by FAPESP



Mechanical Grinding (MG) Hydrogenation-Milling-Dehydrogenation (HMDH)

Figure 1. Scanning electron micrographs of powder particles from both investigated routes

Advances in dispersion fuel fabrication technology

The use of radioisotopes in medicine is certainly one of the most important social uses of nuclear energy and IPEN-CNEN/SP has a special place in the history of nuclear medicine in Brazil. The CNEN institutes are the only officially allowed producers of radioisotopes and radiopharmaceuticals for use in nuclear medicine in Brazil. The production of IPEN-CNEN/SP represents nearly 98% of the total produced. Due to the serious international crisis in the supply of radioisotopes, Brazil has decided to build a new nuclear research reactor, the Brazilian Multipurpose Research Reactor - RMB, in order to ensure the delivery of radioisotopes to the Brazilian market.

Since 1988, IPEN-CNEN/SP has fabricated the fuel for the IEA-R1 research reactor. The current fuel produced at IPEN today allows the incorporation of 3gU/cm^3 by using the uranium silicide (U_3Si_2) technology. This concentration is sufficient to operate the research reactor IEA-R1 running at the power up to 5 MW. However, this level of uranium concentration is not enough for the efficient supply of reactors with higher powers and, therefore, higher neutron fluxes, as the Brazilian Multipurpose Reactor - RMB. Another difficulty inherent to low uranium concentration fuel is the generation of higher quantities of burned fuel. This is due to the low operation life of the fuel with low concentrations of uranium, requiring its frequent replacement.

Based on previous experience by IPEN-CNEN/SP in developing and manufacturing the silicide dispersion fuel, it was decided that the new reactor RMB will use the same type of fuel that is used in the IEA-R1 research reactor, with an increase in uranium concentration from 3.0 to 4.8 g/cm^3 using silicide technology. Developing activities were started to promote an adjustment of the current manufacturing procedures, allowing the incorporation of higher concentrations of uranium. The objective was the increase in the uranium concentration into the fuel from up to 4.8 gU/cm^3 using the U_3Si_2, and 3.2gU/cm^3 using the U_3O_8-Al. These concentrations are the maximum possible to reach if adopting the dispersion technology.

From the important parameters to qualify a fuel plate, it was found that meat length and width of all the produced fuel plates met the specification. However, difficulties arose with regard to the quality of homogeneity in the uranium distribution inside the fuel meat, bonding quality between the meat and the cladding and cladding and meat thicknesses. The initial fabrication tests for high uranium concentration U_3Si_2-Al plates (4.8 gU/cm^3) showed lower values for the cladding thickness as 0.33 mm, which resulted in fuel plates rejection. Another problem found refers to the uranium

segregation of mixture towards the underside of pressed briquette. Especially in the case of the U_3O_8-Al dispersion, it was observed that during loading of the pressing die towards the cavity with powder, the nuclear material, which is more dense, tended to segregate at the bottom of the cavity, causing a localized increase in the uranium concentration and exceeding the maximum value of 45% by volume for the nuclear phase. In this case, the aluminum matrix ceased to act as a continuous dispersion, severely hindering the welding between the meat and cladding. Finally, for both fuel studied U_3Si_2 or U_3O_8, when the volume fraction of the nuclear compound was elevated to its maximum, there were difficulties with respect to homogeneity of the uranium distribution inside the fuel meat.

The maximum particle size of the U_3Si_2 powder was lowered from 150 to 125 µm. This change solved the problem regarding the penetration of U_3Si_2 particles into the cladding. The segregation problems observed previously were solved by changing the particle size of aluminum powder used in the manufacture of fuel meats. It was used a finer powder with smaller maximum size (45 µm) and with a size distribution more open. The aluminum powder used previously had a maximum particle size of 150 µm.

Miniplates with high uranium concentration were prepared to be irradiated at the IEA-R1 reactor of IPEN. It was decided to include in the irradiation program 3 miniplates fabricated with U_3Si_2-Al dispersion (4.8 gU/cm^3) and 3 miniplates fabricated with U_3O_8-Al dispersion (3.2 gU/cm^3). Four miniplates, fabricated according to the usual manufacturing technology adopted at IPEN for the routine fabrication of fuel for the reactor IEA-R1, were also included. This technology was extensively proven as feasibly once there were irradiated several fuel plates in IEA-R1 reactor up to 50 wt% ^{235}U burn-up. These miniplates, two with U_3Si_2-Al 3.0gU/cm^3 and two with U_3O_8-Al 2.3 gU/cm^3, will serve as a benchmark for assessing the irradiation performance of high uranium concentration miniplates. The miniplates were fabricated and qualified under the same procedures adopted to produce normal size fuel plates. All miniplates have been approved for operation in reactor. Figure 2 shows photographs of miniplates to be irradiated. The start of the irradiation is planned to be on June 2011.

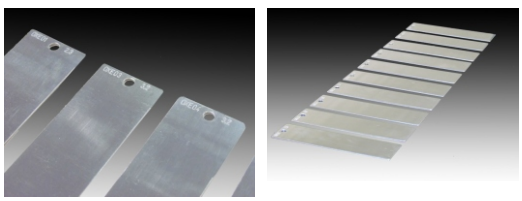


Figure 2. Photos illustrating the miniplates to be irradiated

Fabrication of targets for ^{99}Mo production by fission

As part of the Brazilian Multipurpose Reactor Project, a project aiming at developing the fabrication of targets based on UAl_x-Al dispersion technology started. The targets when irradiated produce mainly the pair 99Mo-99mTc. Brazil has a great demand for this product, which is imported. Currently, the Brazilian nuclear medicine suffers a impact due to the limited availability of these radionuclides in the foreign market. IPEN-CNEN/SP has not yet developed the technique for manufacturing this kind of targets. There are, nowadays, some techniques used worldwide. As a first step, the technique being developed at IPEN is based mainly on metallurgical production of UAl_x powders and UAl_x-Al dispersion miniplates, following the production procedures basically adopted for manufacturing of fuel elements.

UAl_2/UAl_3 powder was fabricated by melting in induction furnace. A slightly hypostoichiometric composition in relation to the nominal composition of UAl_2 was used. The XRD pattern of the powder produced confirmed the predominant presence of the UAl_2 phase, with small amount of UAl_3. However, it was detected the presence of impurities which have not yet been identified. Those peaks are indicated in the diffractogram shown in Figure 3. More studies are needed to identify this contaminant in order to eliminate it.

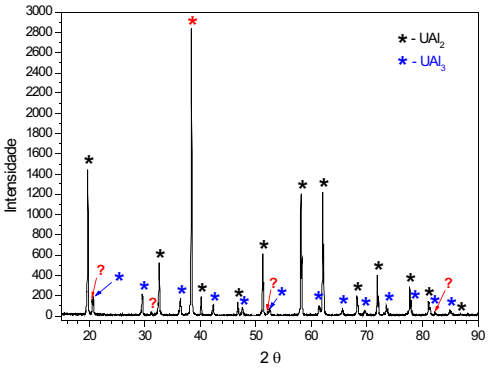


Figure 3. XRD pattern of UAl_x powder produced

The grinding of the material was done manually according the method usually adopted in manufacturing fuels by IPEN-CNEN/SP. The grinding operation was very difficult due to the ductile nature of the material, which was imposed by the presence of the ductile UAl_3. Despite the difficulty, it was possible to manually get enough powder to manufacture prototypes of the first targets, which were used for testing the dissolution and extraction of Mo99.

Eight prototype UAl_x-Al targets with uranium density of 2.8gU/cm^3 were fabricated. The x-value in this intermetallic is close to 3. This amount is the maximum density of uranium that can be incorporated by using the dispersion technology,

which corresponds to 45% by volume of the nuclear phase.

At this stage, it was concluded the feasibility of manufacturing UAl_x-Al targets with uranium concentration of 2.8gU/cm^3. Figure 4 shows the targets fabricated. These are just prototypes manufactured for testing dissolution. The development of the final specification for the target is necessary to continue the work. Additional fabrication tests will be necessary, with a view to meeting the specification that is still under development.

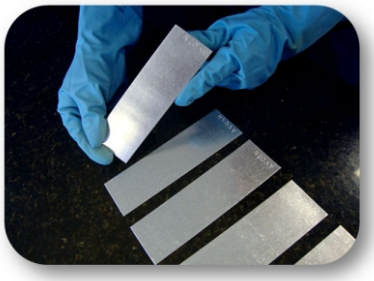


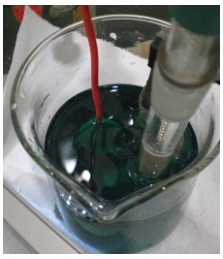
Figure 4. Photograph illustrating the targets UAl_x Al-manufactured

The after-irradiation processing of ^{99}Mo-^{99m}Tc targets could be either done by alkaline or acid route. The alkaline route is used for UAl_x-target, but it penalizes deeply the rejection procedure because it keeps great amounts of highly corrosive liquid radioactive rejection. On the other hand, the acid dissolution route is much more adequate in terms of rejection. This acid route is utilized when metallic uranium, not alloyed with aluminum, is used as target. Normally, the uranium comes involved in nickel covering, which could be removed and separated from ^{99}Mo easier.

There is an IAEA/CPR project planning to use LEU uranium foil (135µm) involved in nickel thin foil (15µm) inserted in an aluminum tube for irradiation. The target fabrication group of CCN/IPEN decided to study other possibilities to produce uranium targets involving electrochemistry. One of the routes could be the compacting of uranium powder and recovering it by electroplating of nickel. Some simulations using iron-powder compacts have been made and are shown in Figure 5. So, LEU metallic uranium powder could be produced by hydrating and then compacted to small coins and then be plated with nickel, using chemical pickling and electrochemistry techniques.

Other possibility is to use small chips of metallic uranium recovered with nickel. The cutting could be made in a cut-off into thin disks with less than half millimeter of uranium. The range of disk diameter could be between 10-30mm. This could be made by straightforward routine in IPEN-CNEN/SP. An example of the metallic uranium chip is shown in Figure 6. Then, the U-chip receives an electroplated layer of nickel and placed inside an aluminum case to be irradiated. The material is set inside the case in such a way it could be removed easily during post-processing manufacture of ^{99}Mo-^{99m}Tc targets following the acid route.

The main purpose of the electroplating is to avoid all possible contact of uranium with aluminum, in order to prevent promptly reactions resulting in U-Al intermetallic and, so, involving alkaline post-irradiation dissolution. Since all nuclear reactions produce radioactive gases, mainly Xe, it is also interesting to have this radioactive product confined inside small volume, which is met by using the nickel electroplated layer over the U-target, helping the fabrication handling inside hot cells and diminishing the hazards occurrences.



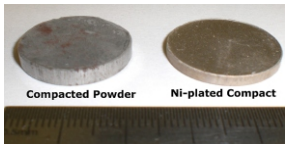


Figure 5. Nickel Electrodeposition over compacted briquette (simulation with 400µm iron powder)

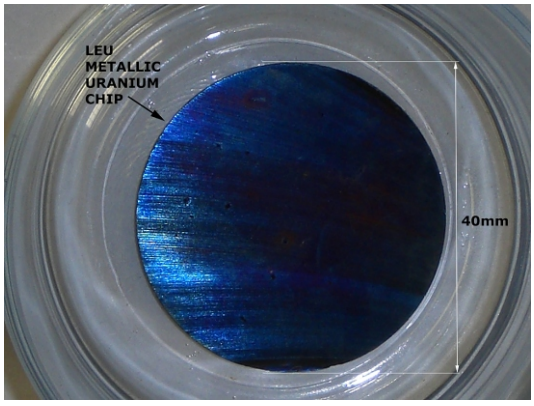


Figure 6. LEU metallic uranium chip (1 mm thick) ready to receive Ni-platting

Treatment of effluent generated by the precipitation of uranium tetrafluoride

The uranium metal used as raw material for uranium silicide production (U_3Si_2) is obtained from the reduction of uranium tetrafluoride (UF_4). There are basically two pathways for the UF_4 production: the first is called the dry route where the UO_2 is treated at 400°C in a resistive furnace under hydrogen and fluoride atmosphere (H_2 and hydrofluoric acid HF); the second is known as the wet route, where the UF_4 is precipitated in hydrofluoric acid using a reducing agent: stannous chloride II ($SnCl_2$). The uranium source for the UF_4 precipitation is uranyl fluoride (UO_2F_2), which can be obtained by the hydrolysis of uranium hexafluoride - UF_6 or from the dissolution of uranium trioxide - UO_3 with HF. In both cases, the

After the precipitation the UF_4 is filtered and washed. The washing solution is collected and used in the UF_6 hydrolysis or to dissolve of UO_3 in the next batch. The filtrate is rich in fluoride ion (F^-), which is treated with calcium oxide (CaO) for the precipitation of calcium fluoride (CaF_2). When the UF_4 is got by hydrolysis of UF_6, the average concentration of F^- ion in the filtrate is 100 g/L. But when the UF_4 is precipitated from the dissolution of UO_3, the average F^- concentration is at the level of 180 g/L. The diffractogram is shown in Figure 7.

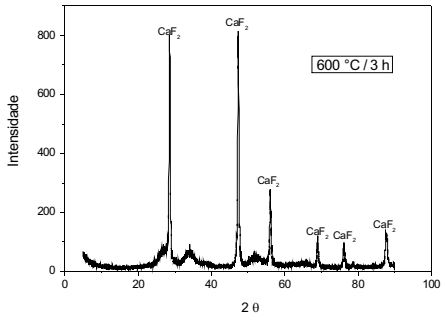


Figure 7. The diffraction diagram of obtained CaF_2

Three main parameters have been studied for the precipitation of CaF_2: initial fluoride concentration, precipitation temperature and final pH of precipitation. The initial fluoride concentration should be between 40 and 50 g/L, because the precipitate decants at this the point. With this initial concentration the filtration is better made. The best temperature was founded to be 90°C and the optimal pH was between 2.5 and 3.0. The combination of temperature and pH produced a filtrate with the fluoride concentration between 2.0 to 3.0 g/ml. The following up step is to calcinate CaF_2 at 600°C for 3 hours.

Computational simulation on reduction process of UF_4 to metallic uranium

The production of metallic uranium is essential for production of fuel elements for using in nuclear reactors manufacturing of radioisotopes and radiopharmaceuticals. In IPEN, metallic uranium is produced by magnesiothermic reduction of UF_4. This reaction is performed in a closed graphite crucible inserted in a sealed metal reactor with no contact with the outside environment. The set is gradually heated in an oven pit, until it reaches the ignition temperature of the reaction (between 600-650°C).

The modeling of the heating profile of the system can be made using simulation programs by finite element method. Through the thermal profiles in the load, we can have a notion of heating period required for the reaction to occur, allowing the identification of greater or smaller yield in metallic uranium production just by recognizing the level of residence time to promote the ignition of the system. Normally, longer time for reaction is accompanied with higher temperature isotherms curves and more homogeneous thermal conditions, but with worse metallic yield. The modeling results were compared to routine production and revealed pertinent in helping the operation routine towards achieving better metallic yield for uranium.

Design and construction of a new fuel fabrication plant

Between 1985 and 1988, IPEN-CNEN/SP worked aiming at assembling a small fuel element fabrication plant with production capacity of only 6 fuel element a year, for demonstration purpose, but produced the necessary quantity to supply its IEA-R1 research reactor that operated at this time in power level of 2 MW with a of regime of 40 hours a week. In August 1988, IPEN-CNEN/SP supplied the IEA-R1 reactor with the first national fuel element. Ever since, IPEN-CNEN/SP began a continuous fuel production, which continues up today.

During 1997, IPEN-CNEN/SP raised the fuel production capacity of the small plant from 6 to 10 fuel element a year, representing the maximum production capacity in laboratorial scale for the available facilities at that time, which was enough to keep IEA-R1 reactor operating at 3-4MW power level in 64 hours regime a week. Due to the emergent increase in radiopharmaceuticals products demand and the consequent increase in the IEA-R1 power, the reactor needed an increasing number of fuel elements to operate properly at 5MW and 120 hours a week. The number should be raised from 6 (U_3O_8-Al) to 18 elements (U_3Si_2-Al) a year. In addition, a new reactor to produce radioisotopes will be built (Multipurpose Reactor Brazilian) and aims to make the country independent in the production of radioactive isotopes for medicine, material testing reactors for energy production and possession of a neutron beam line for scientific use. The new reactor (20MW) would consume about 50 yearly U_3Si_2-Al fuel elements. The RMB is a project that will contribute decisively to the country's strategic objectives for increasing the production of radioisotopes for medical applications among others. The ^{99}Mo and ^{131}I are the main radioisotopes for application in health and will be a priority in the reactor irradiation. The ^{99}Mo and ^{131}I will be produced from targets irradiated in the reactor core consisting of miniature fuel plates. The production of ^{99}Mo and ^{131}I require weekly, with the reactor operating without interruption. It is estimated a consumption of 20 uranium targets per week, which equals 1000Ci/week.

Based on this forecast demand for fuel elements and uranium targets, a project in IPEN-CNEN/SP began in 2001 in order to adapt the production facilities seeking on improving the production capacity. The first phase of that project is now concluded (Figure 8). The fabrication step for

dispersion preparation is made by: cermet core pressing, fuel plate rolling, fuel element assembling and all fuel qualification steps are properly built in the new fabrication plant. The second phase of that project is still going on. Once ready, the whole project will be constituted of an integrated production line using in industrial level facilities, which will operate according to international nuclear quality and safety standards, established by IAEA. The new facility is planned to have nominal capacity for producing 50 fuel elements yearly. That will attend entirely the fuel element demand in 4-5 years. The production capacity of the new facility could be incremented to reach 80 fuel elements in a year, to full supply the new multipurpose reactor which has been planned to be constructed. The conclusion of the physical project and equipment installation is foreseen to happen by 2012-2013.

Figure 8. Partial view- concluded parts of the new CCN plant for fuel element fabrication

Fuel element fabrication

The Nuclear and Energy Research Institute IPEN-CNEN/SP has worked in the area of Nuclear Fuel Cycle practically since its founding in 1956. In the 60s, a golden age in the area of manufacturing technology of nuclear fuel began at the Division of Nuclear Metallurgy. It started, at this period, the development of dispersion based fuel, with applications in pool type research reactors. This fuel type was assembled from fuel plates containing uranium compounds dispersed in aluminum. Between 1964 and 1965, the fuel elements for the Argonauta Reactor of Institute of Nuclear Engineering (IEN) were manufactured in this division of Nuclear and Energy Research Institute IPEN-CNEN/SP. The fuel used had a dispersion of U_3O_8-Al. The employed U_3O_8 powder was enriched to 20wt% of ^{235}U, imported from United States, through the program Atoms for Peace (UN/IEAE). Despite the relatively simple technological requirement to produce the fuel for the Argonauta reactor, it was a great start-up since this technological seed germinated strongly after 1980.

The development program for producing the fuel elements began in the mid 80s. It was motivated by "cold war" restraint purchases policy, which hindered the acquisition of fuel out of the country. The internal necessary fuel demand to promote the continuous operation of IPEN´s reactor IEA-R1 was certainly the most stimulating cause for developing skills to produce the fuel elements. In September 1988, the first fuel element produced at IPEN-CNEN/SP, were placed in our reactor. Since then, IPEN-CNEN/SP has made its own fuel elements based on plate type, initially using U_3O_8-Al dispersions in density of 2.3 gU/cm^3.

Due to the increasing demand of the radioisotopes production in Brazil, the reactor IEA-R1 increased its power from the original 2MW to 5MW. Also, the reactor routine became ready to increase up to 120 operating hours per week, requesting other fuel material types to bring more U-density to the fuel meat. To carry out this increasing in fuel meat density, a new fuel element was developed based on U_3Si_2-Al dispersion with a density of 3.0 g/cm^3, which called for a gradual change inside the IEA-R1 from U_3O_8-Al towards Al-U_3Si_2.

The intermetallic U_3Si_2 fuel was imported until 2002. Presently, it is fully produced in IPEN having the conversion chemical cycle included using enriched LEU UF_6 supplied by Navy Technology Center (CTMSP). Currently the Nuclear Fuel Center is also manufacturer of powdered U_3Si_2 passing through producing UF_4, metallic uranium and then the silicide by induction smelting. The entire fuel cycle is actually nationalized.

The fuel element, which is manufactured by IPEN, is dispersion MTR-type enriched to 20 wt% ^{235}U. The two types of dispersion are well qualified for operation up to 30% burns, on average. However, practice has shown a performance superior to that amount, leading to 50% burn-ups in many fuel elements, without any nuclear hazard.

Brazil became totally independent in terms of materials and technology to fabricate its fuel elements for their research reactors in order to produce radioisotopes. This was an important technological know-how acquired by our research institute. It places our country inside the international market, as part of a restrict group of commercial suppliers of this kind of fuel. Besides, the Al-dispersed fuel technology was established as a developing basis to further going on to reach advanced fuel material and projects for higher performance nuclear research reactors, such as our future RMB reactor.

Up to now, IPEN has manufactured 63 fuel elements of U_3O_8 (26 having meat density of 1.9 to gU/cm^3 and 37 having 2.3 gU/cm^3 and 29 fuel elements with U_3Si_2 having a concentration of 3.0 gU/cm^3. So, a total of 92 fuel elements were produced to date.

From 2010 onwards, it was installed in the reactor a new "instrumented element" system, developed by IPEN aiming at providing information and data parameters to promote better studies on thermohydraulics during operation of IEA-R1 reactor. The new device had its first operation at a power of 3.5MW in February 2010 and has produced ever since important experimental data.

This device had special difficulties to fabricate fuel plates with thermocouples attached to its surface. The IEA-R1 reactor, instrumented in this way, became a world reference for many thermohydraulic and accident analysis codes. Figure 9 shows the instrumented element (EC 208) fabricated.

Figure 9. Instrumented Element (EC 208)

Chemical characterization of nuclear fuel

The nuclear fuel cycle is a series of steps involved in the production and use of fuel for nuclear reactors. The laboratories of Chemistry and Environmental Diagnosis Center, support the demand of Nuclear Fuel Cycle Program providing chemical characterization of uranium compounds and other related materials.

Among these, one can highlight the determination of uranium content in U_3Si_2, UF_4, U_3Si_2-Al compounds and its impurities (metals and rare earths elements). The last ones are determined using extraction chromatography and ICP-OES measurement.

The method development to quantify the uranium content was based on Davies & Gray methodology as used at NBL (New Brunswick Laboratory) adding an improvement on lowering the mass used in the previous methodology. To the analyst, less radiation exposition is a safer condition to work. Also, lower quantities of chemical and radioactive wastes are produced. Another improvement achieved was the reducing of time consumed on determination of silicon and uranium in the respective alloy.

The XRF laboratory runs routinely analyses related to nuclear materials (U_3Si_2, Al powder and its alloys, AgInCd alloys, U and Th compounds). The XRF technique allows developing methods with a minimum or no chemical treatment.

The laboratory participated of an Interlaboratory Comparison for the verification of international target value of uranium content in several uranium compounds using Davies and Gray methodology supported by ABACC (Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials) and NBL (New Brunswick Laboratories).

The Group of Chemical and Isotopic Characterization supports IPEN's nuclear fuel production program to IEA-R1 research reactor providing isotopic analysis for uranium compounds. Isotopic analysis are performed by using a high resolution inductively coupled plasma mass spectrometer. By means of a technical cooperation with the Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials (ABACC) and with the United States Department of Energy an intensive program, based on laboratorial inter-comparison exercises, for improvement of results from analysis of nuclear samples has been in progress. New protocols for safeguards purpose were established. Among then environmental sampling based on swipe samples for the identification of uranium and plutonium in nuclear facilities.

Whenever possible, the laboratories of Chemistry and Environmental Diagnosis Center are engaged to update or develop new methods to become the activities greener as currently expected.

Reactor physics benchmarks at the IPEN/MB-01 reactor

Since 2004, the reactor physics group of the Nuclear Engineering Center of IPEN is participating on two international programs for the elaboration of benchmarks experiments on critical facilities. The programs are the working groups ICSBEP (International Criticality Safety Benchmark Evaluation Project) and IRPhE (International Reactor Physics Evaluation Program) both sponsored by INL (Idaho National Laboratory, EUA) and NEA (Nuclear Energy Agency). ICSBEP is devoted to criticality safety benchmarks and IRPhE is more related to reactor physics experiments in general. The purpose of the ICSBEP is to: a) Identify a comprehensive set of critical benchmark data and, to the extent possible, verify the data by reviewing original and subsequently revised documentation, and by talking with the experimenters or individuals who are familiar with the experimenters or the experimental facility, b) Evaluate the data and quantify overall uncertainties through various types of sensitivity analysis, c) Compile the data into a standardized format, d) Perform calculations of each experiment with standard criticality safety codes, e) Formally document the work into a single source of verified benchmark critical data. The work of the ICSBEP group is documented as an International Handbook of Evaluated Criticality Safety Benchmark Experiments. Currently, the handbook spans over 42,000 pages and contains 464 evaluations representing 4092 critical, near-critical, or subcritical configurations, 21 criticality alarm placement/shielding configurations with multiple dose points for each, and 46 configurations that have been categorized as fundamental physics measurements that are relevant to criticality safety applications. The International Reactor Physics Benchmark Experiments (IRPhE) Project aims to provide the nuclear community with qualified benchmark data sets by collecting reactor physics experimental data from nuclear facilities, worldwide. More specifically the objectives of the expert group are as follows: a) maintaining an inventory of the experiments that have been carried out and documented; b) archiving the primary documents and data released in computer-readable form; c) promoting the use of the format and methods developed and seek to have them adopted as a standard.

The experiments are being performed at the IPEN/MB-01 research reactor facility. During the last three years, several experiments have been designed, executed and analyzed at the IPEN/MB-01 Reactor. They were documented in a proper format and submitted to the working groups. More than 100 critical configurations have been approved to be included in the ICSBEP DVD handbook. From these experiments, we can mention the critical configurations with borated stainless steel used in the storage pool of ANGRA-I and II to save storage space. Another very interesting experiment was a central void simulation with a aluminum block. More recently the reactor physics group completes a series of experiments with a heavy reflector made of SS-304 to give support to the EPR development in Europe. In the reactor physics area (IRPhE) we complete a series of benchmark experimental problems on the isothermal reactivity coefficient of light water reactors. This experiment was very important to give support to the nuclear data evaluation of ^{235}U in the thermal energy region of the neutron. This was a long standing problem in the reactor physics area and recently it was made very good progress in the C/E comparisons of several integral parameters of interest in the physics of reactors. Another experiment complete at the end of 2008 was the measurement of several effective delayed neutron parameters without the need of any correction factor or data from other experiments. More recently the reactor physics area of IPEN added more three contributions to the IRPhE project; the reaction rate experiments, the fission density and the power distributions of the IPEN/MB-01 core. The experiments were approved to be included in the handbook of reactor physics benchmarks issued in March 2009 and March 2010. This project is being supported by FAPESP and CNPq.

Development of a reactivity meter at the IPEN/MB-01 reactor

The development of a reactivity meter at IPEN was performed to fulfill the needs of the IPEN/MB-01 reactor researchers. Due to the good results presented by the reactivity meter, a preliminary test was done using the data of ANGRA 1 unit in the course of the start up P9 in 2001. These data (voltage signals of neutron detectors, temperature signals and bank position signals) were acquired by the Framatome staff and analyzed at IPEN by the staff of the IPEN/MB-01 reactor. The results were in good agreement with those obtained by Framatome.

Since then, the reactivity meter was tested in all start up tests of ANGRA 1 in parallel to the Framatome acquisition and analyses. In these tests, however, all the signals of interest were acquired independently and from different sensors and/or acquisition points. In all start up tests, the results were totally consistent to the Framatome ones.

As an example, Table 1 shows the comparison of results for the reactivity of the control banks and the isothermal reactivity coefficient for the ANGRA 1 P14 start up tests performed in 2007.

Parameter	Calculated Value	Framatome Measurement	IPEN Measurement
Isothermal Temperature Coefficient	-13,87 pcm/°C	-14,405 pcm/°C (heating) -14,391 pcm/°C (cooling) -14,398 pcm/°C (average) deviation = -0,528 pcm/°C	-14,208 pcm/°C (heating) -14,409 pcm/°C (cooling) -14,301 pcm/°C (average) deviation = -0,431 pcm/°C
Bank D	821 pcm	832,90 pcm (-1,43 %)	843,02 pcm (-2,61 %)
Bank C	937 pcm	974,25 pcm (-3,82 %)	957,40 pcm (-2,13 %)
Bank B	1040 pcm	1097,46 pcm (-5,24 %)	1045,78 pcm (-0,55 %)
Bank A	434 pcm	479,43 pcm (-9,50 %)	461,88 pcm (-6,04 %)
The deviation of the bank reactivities is given by: σ = [(VC - VM) /VM]*100 where VC is the calculated value and VM is the measured value			

After the good results obtained in all these start up tests, the researchers of IPEN performed the ANGRA 1-P15A , 1-P16, 1-P17 start up tests in 2008, 2009, and 2010 respectively, without the Framatome staff nor supervision of any kind. The results obtained were again totally consistent with the calculated ones and the tests were approved. It is important to mention that the reactivity meter has also been used in the IEA - R1 reactor at IPEN for almost 6 years, providing a great economy of working time, and that the reactor physics staff of ANGRA 2 unit has interest to install the reactivity meter in this power plant.

Software for medical image based PHANTOM MODELING

Latest treatment planning systems depends strongly on CT images, so the tendency is that the dosimetry procedures in nuclear medicine therapy be also based on images, such as magnetic resonance imaging (MRI) or computed tomography (CT), to extract anatomical and histological information, as well as, functional imaging or activities map as PET or SPECT. This information associated with the simulation of radiation transport software is used to estimate internal dose in patients undergoing treatment in nuclear medicine.

This work aims to re-engineer the software SCMS, which is an interface software between the Monte Carlo code MCNP, and the medical images, that carry information from the patient in treatment. In other words, the necessary information contained in the images are interpreted and presented in a specific format to the Monte Carlo MCNP code to perform the simulation of radiation transport. Therefore, the user does not need to understand complex process of inputting data on MCNP, as the SCMS is responsible for automatically constructing anatomical data from the patient, as well as the radioactive source data.

The SCMS was originally developed in Fortran-77. In this work it was rewritten in an object-oriented language (JAVA). New features and data options have also been incorporated into the software. Thus, the new software has a number of improvements, such as intuitive GUI and a menu for the selection of the energy spectra correspondent to a specific radioisotope stored in a XML data bank. The new version called AMIGO also supports new materials and the user can specify an image region of interest for the calculation of absorbed dose. The main window of the AMIGO software presented as a combo box to set the image and simulation parameters by the user. Several parameters and options are already predefined with default values, but the user can modify them by introducing new parameters or configurations according to each problem and the last modifications will be saved to the next program execution. Several tests have been done successfully using a diversity of examples of anatomic and functional virtual images. The AMIGO software is part of the development of an integrated computational dosimetric system which is being developed at IPEN for applications in nuclear medicine and radiotherapy.

Energy degrading and scattering plates for electron beam radiotherapy for skin diseases in small rooms

There are many radiosensitive epidermotropics diseases such as mycosis fungoids and the syndrome of Sézary, coetaneous neoplasics originated from type T lymphocytes. Several studies indicate the eradication of the disease when treated with linear accelerators emitting electron beams with energies between 4 to 10 MeV. In this project we developed a customized single-field electron beam for total skin therapy (TSET) and local irradiations in small treatment rooms with maximum source-surface distance (SSD) of 2.95 meters, using energy degrading and scattering plates.

The energy spectrum and geometric distribution of the 6 MeV electron beam of the VARIAN 2100C accelerator were reconstructed through Monte Carlo simulations, using the MCNP5 Monte Carlo code and based on experimental data. The simulated source was then utilized to test several materials and geometry for scatter and degrader plates, producing scattered field suitable and adequate for local irradiations and whole body TSET procedure. The simulation results were validated by experimental measurements with thermoluminescent dosimeters (TLD), radiochromic films and ionization chambers. Figure 1 shows the experimental configuration for dose distribution measurements at the Hospital das Clínicas de São Paulo, HC-FMUSP/SP.

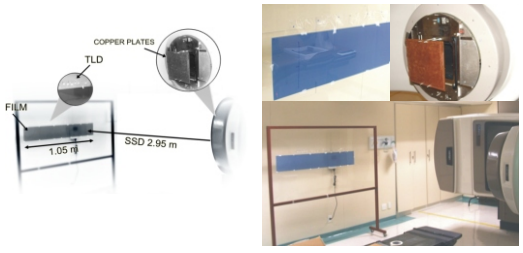


Figure 1. Experimental configuration for TSET dose distribution measurements using TLD and radiochromic films

NSECT applied to the assessment of calcium deposition due to the presence of microcalcifications associated with breast cancer

Breast cancer is the second most common cancer worldwide and the leading cause of death among women in Brazil. According to estimates for the year 2010 it is expected approximately 49,000 new diagnosed cases. One of the main signs of breast cancer at an early diagnosis is the development of

microcalcifications. Because of calcium radiological properties, microcalcifications are associated with non-palpable lesions that can be visualized on mammography, which makes it the primary mode of breast cancer diagnosis. The importance of the detection of microcalcification formations in their early stages is well-known fact and according to the literature, the survival rate of patients who developed breast cancer is inversely proportional to the lesion size.

In recent years, a new technique for in vivo spectrographic imaging of stable isotopes was presented as Neutron Stimulated Emission Computed Tomography (NSECT). In this technique, using multiple projections, a fast neutron beam interacts with the stable isotopes of the irradiated tissue, through inelastic scatterings, making them jump into an excited state. When they return to their ground state, they emit photons which energies are intrinsic to the emitting nuclei. The emitted gamma energy spectra can be used for two purposes: (a) reconstruction of the target tissue image and; (b) determination of the tissue elemental composition.

BNCT research facility experiments

BNCT (Boron Neutron Capture Therapy) is a binary cancer treatment therapy which stands on loading tumor cells with boron following their irradiation with thermal neutrons. As the 10B absorbs the neutron, two high LET particles are emitted (alpha and 7Li) releasing all their kinetic energies (around 2 MeV) in a cell size volume. Its success relies therefore on the capability and specificity of the boron carrier compounds to deliver boron to the target cells and also on tailoring adequate neutron beams to impinge on these cells. Although its idealization and first treatments on brain tumors dates for more than 60 years, it has been felling an increased interest due to good results observed in the treatment of other cancer types.

A BNCT research facility was projected and constructed in IEA-R1m reactor of IPEN-CNEN-SP. It has been used in the development of studies in Radiation Physics and Radiobiology such as: - radiation field (neutrons and gammas) characterization; - neutron beam tailoring through the determination of a moderators and filters configuration which adequate the radiation field to BNCT needs; - dose estimation and development of “in vitro” and “in vivo” biological studies. Work cooperation has also been established to evaluate BNCT efficiency regarding its dependence on cell type cultures and on the use of different boron compound agents.

Multi-modular integral pressurized water reactor control and operational reconfiguration for a flow control loop

The work focused on the International Reactor Innovative and Secure (IRIS) design since this will likely be one of the designs of choice for future deployment in the U.S. and developing countries. With a net 335 MWe output IRIS novel design falls in the “medium” size category and it is a potential candidate for the so called modular reactors, which may be appropriate for base load electricity generation, especially in regions with smaller electricity grids, but especially well suited for more specialized non-electrical energy applications, such as district heating and process steam for desalination. The first objective is to evaluate and quantify the performance of a nuclear power plant comprised of two IRIS reactor modules operating simultaneously with a common steam header, which in turn is connected to a single turbine, resulting in a steam-mixing control problem with respect to “load-following” scenarios, such as varying load during the day or reduced consumption during the weekend. To solve this problem a single-module IRIS SIMULINK model previously developed by another researcher is modified to include a second module and was used to quantify the responses from both modules. This twin-module plant is shown in Figure 2, and the control schematic of a twin-unit IRIS system, with helical coil steam generators connected by the feedwater line, is shown in Figure 3.

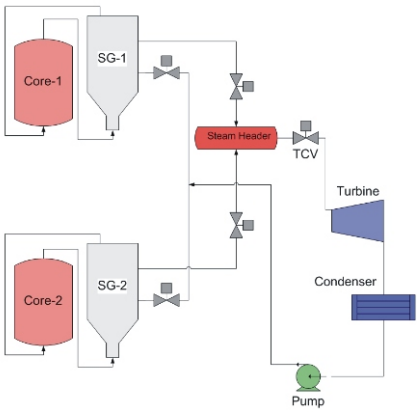


Figure 2. Schematic diagram of an IRIS-type multi-modular power block

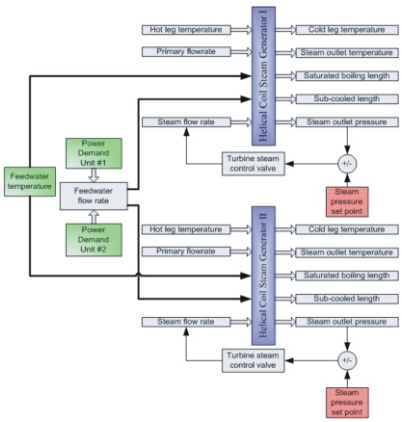


Figure 3. Schematic of a Twin-HCSG control system connected at the feedwater flow rate line

IRIS simulation results

From simulations it is possible to conclude that, for small variations in process variables, both units are somewhat independent of each other even with a common feedwater line connecting both steam generators, and are able to perform well even under such variations, with the real connection between the two units being located in the steam header. Simulations show the feasibility of having two IRIS modules in a single plant. An example of changes in steam temperature at each of the reactor modules and the temperature of the mixed steam in the common header are shown in Figure 4. At first, both steam temperatures are the same, and remained constant in the first unit while it varied in time in the second unit because of the changes in the power demand. As the power decreased, the area available for heat transfer in the steam generator increased, therefore increasing steam temperature, conversely decreasing following power increase. The control strategy of regulating the average reactor temperature and the steam pressure is robust for this load following operation.

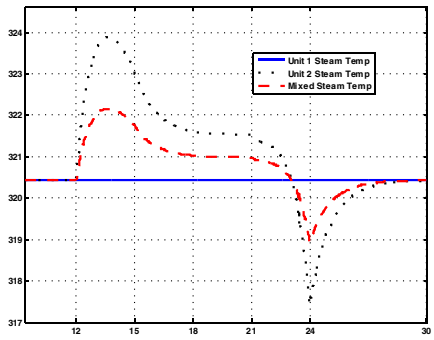


Figure 4. Units #1 & #2 and mixed steam header temperatures changes

In order to develop research related to instrumentation and control, and equipment and sensor monitoring, the second objective is to build a two-tank multivariate loop in the Nuclear Engineering Department at the University of Tennessee. This loop provides the framework necessary to investigate and test control strategies and fault detection in sensors, equipment and actuators. The third objective is to experimentally develop and demonstrate a fault-tolerant control strategy using this loop. Using six correlated variables in a single-tank configuration, five inferential models and one Auto-Associative Kernel Regression (AAKR) model were developed to detect faults in process sensors using Sequential Probability Ratio Test (SPRT). Once detected the faulty measurements were successfully substituted with prediction values, which would provide the necessary flexibility and time to find the source of discrepancy and resolve it, such as in an operating power plant. Finally, using the same empirical models, an actuator failure was simulated and once detected the control was automatically transferred and reconfigured from one tank to another, providing survivability to the system.

Flow control loop results

Actual measurements were successfully substituted with predictions and provided the system the necessary flexibility to keep on operating even under degraded conditions, thus offering survivability to the system and the time necessary to perform corrective procedures, should that be the necessary. These experiments were particularly important because they offered the opportunity to prove that a system like the multivariate loop can survive degraded circumstances, provided the empirical models used are accurate and representative of the system dynamics.

An experiment was performed by adding a drift to the level sensor measurements at a rate of +50 mmH_2O over 3 minutes. In this experiment, once the difference between measured and predicted values reached around 18 mmH_2O, the level SPRT triggered changing from normal to faulty condition causing measured values to be substituted with predicted values, therefore isolating the faulty sensor from the loop.

This work was funded by the US Department of Energy and developed in its entirety at the University of Tennessee in Knoxville, USA. It was presented in October of 2010 as a partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Nuclear Engineering. Graduate research and teaching assistantships were provided by the University of Tennessee Nuclear Engineering Department, under the supervision of UTNE professor Dr. Belle R. Upadhyaya.

Innovative and hybrid reactors

The renascence of Nuclear Energy is bringing new initiatives in the world such as GIF (Generation IV Initiative), and INPRO (International Project on Innovative Nuclear Reactors and Fuel Cycle). These new initiatives are looking for new reactors concepts and associated fuel cycles which take as principle the sustainability of Nuclear Energy for the next centuries. Among several concepts being considered, Fast Spectrum System is the focus of this research group, in particular subcritical reactors driven by external source of neutrons (e. g., spallation neutron source, fusion neutron source). These systems are being considered as dedicated nuclear transuranic burner reactors. In the last years, the calculation methodologies for simulation and analyses of these systems had been developed. We participate in the IAEA technical working group on fast reactors (TWG-FR) and in the Coordinated Research Project (CRP) on Analytical and Experimental benchmark analysis on Accelerator Driven Systems.

The main activities and resulted products during the year 2007-2010 in innovative hybrids reactors are:

- Participating of International Atomic Energy Agency (IAEA) Coordinated Research Project (CRP) on “Analytical and Experimental Benchmark Analyses of Accelerator Driven Systems”.The objective of this CRP is to improve the present understanding of the coupling of an external neutron source [e.g. a spallation source in the case of the accelerator driven system (ADS)] with a multiplicative sub-critical core;

- Participation in cooperative work established to study the feasibility to use Low Enriched Uranium in existing or planned facilities, under the umbrella of the main CRP and with support of DOE of the USA;

- Feasibility studies of the implementation of a compact neutron generator in the IPEN/MB-01 zero power reactor. A numerical calculation of IPEN/MB-01 driven by a compact neutron generator was performed to verify the feasibility of using Low Enriched Uranium in existing or planned facilities;

- Participation in the simulation of Reactor Physics Parameters of the sub-critical assembly YALINA-Booster at the Joint Institute for Power and Nuclear Research Sosny (JIPNR) of the National Academy of Sciences of Belarus. It was performed these calculation benchmark exercise and the results are being compared with the experimental results;

- Participation in Benchmark on Computer Simulation of radioactive nuclides production rate and Heat Generation Rate in a lead target exposed to 660 Me protons, was defined by the participants from Poland. The benchmark model is based on the earlier experiment done within the project SAD, in the Joint Institute of Nuclear Research in Dubna (Russia). In the experiment absolute activities of several long-lived radionuclide's, generated in lead target during its irradiation with 660 Me proton beam, were determined. Thus, the benchmark is oriented to compare simulation predictions, based on different available codes and physical models, with the experimental data;

- Implementation Monte Carlo burnup calculation of subcritical systems;

- Comparison between fusion hybrids subcritical reactors and accelerator driven subcritical reactors, in Figure 5 it is showed Gas Cooled Fast Subcritical Reactor (GCFR) driven by a Tokamak, and in Figure 6 it is showed a neutron flux distribution of these reactor.

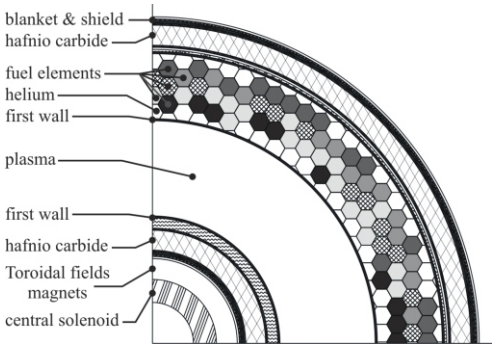


Figure 5. Gas Cooled Fast Subcritical Reactor driven by a Tokamak

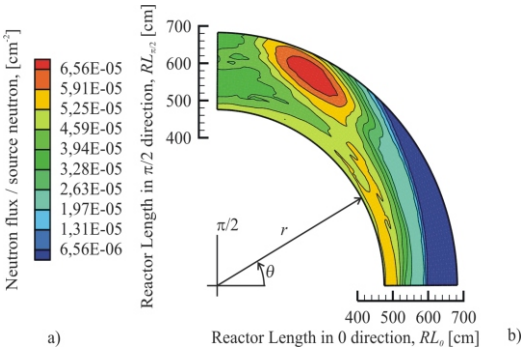


Figure 6. Neutron flux distribution of a Gas Cooled Fast Subcritical Reactor

Collaborative project INPRO/ENV “Environmental Impact Benchmarking Applicable for Nuclear Energy System under Normal Operation”

Since 2010 CEN/Ipen has participated in a IAEA study group of benchmarking of methodologies for the ranking of stressors of interest for given release and pathways scenario. This study presents a comparison of ranking methodologies and will be applied to different source terms. The specific research scopes:

- a benchmark on assessment methodology to rank radio-nuclides in terms of health related impact on human;
- a comparison of the most important radio-nuclides in terms of environmental impact for a given source term;
- the role of retained criteria to obtain this list and the relevance of sensitivity studies;
- reference scenarios for INPRO Assessment Methodology;
- feed back for review of the practical application of the INPRO methodology for environmental protection.

The participating countries are Belarus, Brazil, Czech Republic, France, India, Indonesia, Kazakhstan, Republic of Korea, Russia, Slovak Republic, Ukraine.

MTR instrumented fuel element for thermal-hydraulic analysis of research reactors

With the financial support of the IAEA and IPEN-CNEN/SP, the Nuclear Engineering Center designed an MTR Instrumented Fuel Element for the IEA-R1 research reactor. This Fuel Element was used in order to measure the temperatures on the lateral and central fuel plates, and also to compare them with the calculated temperatures. Thermal hydraulic computer codes such as PARET, COBRA, RELAP, etc were used for the calculations. The Instrumented Fuel Element, called IEA-208, was assembled by the Nuclear Fuel Center, Figures 7 and 8. Fourteen thermocouples were used to acquire the wall and fluid temperature at predefined positions in different cooling channels. IEA-R1 reactor has been operating since February 2011 with this element in different core positions.

Figure 7. Instrumented fuel element assembling

Figure 8. Instrumented fuel element

Natural circulation in nuclear reactors (theoretical and experimental)

One of the most serious problems for a nuclear reactor in accidental conditions is the residual heat removal from the fission products decay. The design of a nuclear reactor provides the establishment of natural circulation in the primary system as an alternative to cooling the reactor core. An experimental circuit was developed in order to understanding the complex phenomena involved in two phase flow natural circulation and also to obtain data for validating computer codes. These studies started at USP in the Departamento de Engenharia Química/Escola Politécnica. Experiments concerning single and two-phase flow natural circulation regimes were performed. Several papers were published. In an agreement between institutions, this circuit was moved from USP to IPEN/CNEN-SP for its better utilization. Some improvements were performed in order to updating instrumentation and data acquisition system. The experimental circuit consists of a rectangular glass loop with an electrical heat source and a coil cooler sink. Glass is used to allow the flow visualization, filming and photographing to identify the flow patterns and to observe the phenomena.

A new user interface using LabVIEW was developed for the data acquisition system. Experiments were performed for single and two-phase flow conditions. RELAP5 code was used to simulate this circuit. Figure 9 shows the comparison between measured and calculated temperatures at two positions of the circuit, T12 and T17, for single phase flow condition. Figure 10 shows the results of measured and calculated temperatures at the same positions of the circuit for two-phase flow condition.

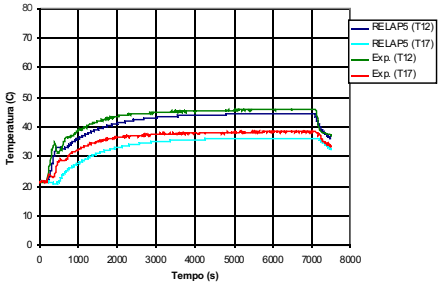


Figure 9. Theoretical and experimental temperatures at heater outlet (T12) and cooler outlet (T17) for single phase flow condition

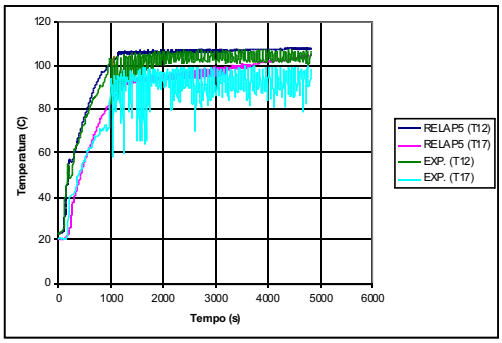


Figure 10. Theoretical and experimental temperatures at heater outlet (T12) and cooler outlet (T17) for two phase flow condition

Theoretical results from the code were compared to the experimental ones in order to validate the RELAP5 models. New instruments will be installed along the circuit for flow rate, void fraction and vibration measurements. Additional tests will be performed and the data will be compared with the code results. A high speed video/photo camera has been used for filming and photographing the two phase flow regimes and other phenomena during the transients. The acquired images are being treated in specific software for void fraction determination and boiling/condensation studies.

Safety analysis of Angra-2 nuclear power plant

The Safety Analysis group is involved with the activities related to the licensing process of nuclear power plant Angra 2. These activities have been developed through cooperation among institutes of CNEN and Pisa University in Italy. In this cooperation the safety analysis group is responsible for activities related to the elaboration of an input data set to RELAP5/MOD3 code, to be used in future independent calculations. IPEN's researchers in this cooperation were qualified to the accident

analysis code in nuclear plants, which has been used in the commissioning of nuclear power plants Angra 1 and 2. The initial task consisted in the preparation of the necessary input data. Some of the Angra 2 components as well as some associated systems of the plant were considered. In a second phase, when the whole plant was modeled, researchers began the simulation of some transients and accidents, according to the description given in the Angra 2 Final Safety Analysis Report. From the results obtained, it was possible to evaluate work and proceed with alterations in order to more realistically represent the analyzed cases.

The simulated accidents will be repeated with the new detailed core nodalization as well as other accidents analyses aiming the validation of the multi-purpose nodalization, which represents a preliminary stage in the process of transient qualification of this input data. Uncertainty calculations were also in development and the results were satisfactory. The uncertainties were calculated by CIAU method. Figure 11 shows the primary loop pressure (theoretical/experimental) and the uncertainties band (CIAU). Figure 12 presents the primary loop mass (theoretical/experimental) and the uncertainties bands (CIAU).

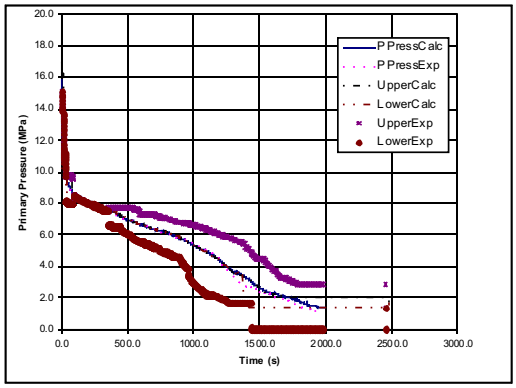


Figure 11. Primary loop pressure and the uncertainties bands

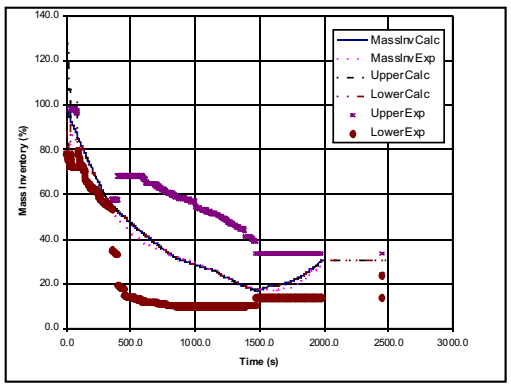


Figure 12. Primary loop mass and the uncertainties bands

Figure 11 shows the results obtained with RELAP5 code to nuclear power plant Angra 2 are into of the uncertainties bands, when compared with the experimental data (LOBI Test A1-93). This work received financial support from Coordinated Research Project (CRP) J7.20.05 of the International Agency Energy Atomic (IAEA).

Computational fluid dynamics studies

The research in this area was focused mainly in the IEA-R1 Heat Exchanger, Figure 13, and the Natural Circulation Loop.

A preliminary model was developed with ANSYS-CFX® code which was used in order to study the flow at the inlet nozzle of the heat exchanger of the primary circuit of the nuclear research reactor IEA-R1. The geometry of the inlet nozzle is basically compounded by a cylinder and two radial rings which are welded on the shell. When doing so there is an offset between the holes through the shell and the inlet nozzle. Since it is not standardized by TEMA, the inlet nozzle was chosen for a preliminary study of the flow. This research established an initial mark to the scientific cooperation between the Mackenzie Presbyterian University and IPEN.

Although with a simplified geometry, the CFD model supplied consistent results for the pressure field, velocity field, Figure 14, streamlines, Figure 15, and vectors. Mesh elements reached approximately 2.2 million, Figure 16, indicating our computational system limits.

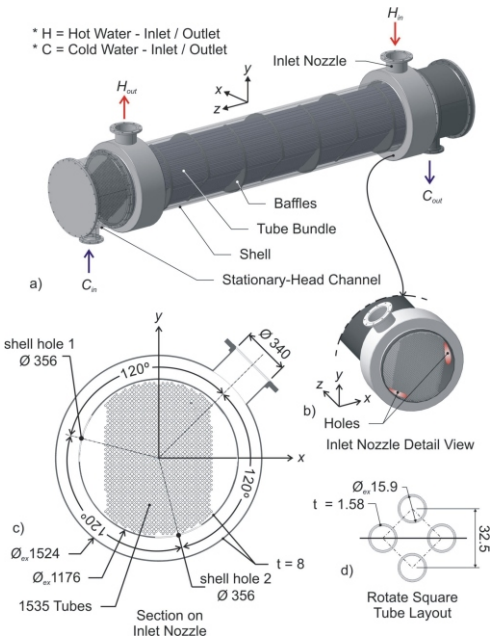


Figure 13. IEA-R1 heat exchanger

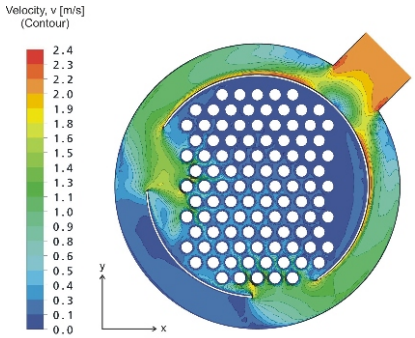


Figure 14. Velocity field

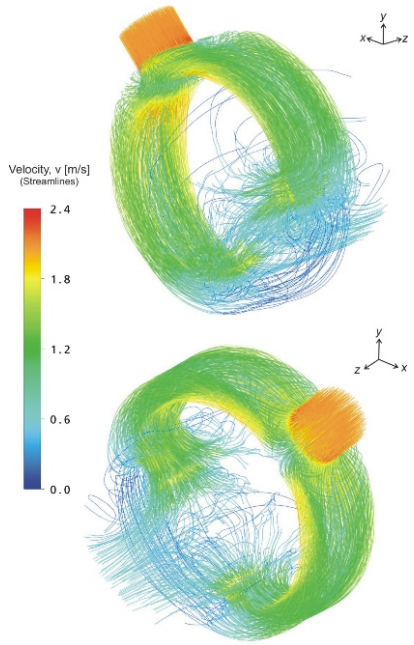


Figure 15. Streamlines

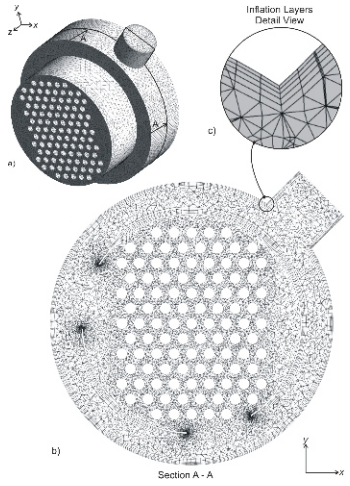


Figure 16. Mesh

Radial equilibrium design of axial flow gas turbine

In the axial flow gas turbine - reaction type - the flow gas has an angular velocity. This causes radial pressure gradient, consequently possible radial flow. The radial flow causes mixing which can result of entropy increase and less efficiency.

The early treatment of the problem is the Free Vortex method which results of constant entropy change in the radial direction and higher blade twist so it was recommended to be used in short blades. Actual measurements show higher entropy change in the tip and the root of the blades.

Later the problem was treated by Flow Through method, which is a numerical method, simplifies the flow in axial and radial directions only. This method was used by Pratt & Whitney (P&W) air craft manufacturer. The simplifying assumption, in order to solve the problem, was that the effect of losses was treated as reversible heat addition, which is not correct.

The approach suggested in our work was to divide the spacing between two successive blades, in the axial direction, in a 51 control volumes. For each control volume is written the governing physical equations, which are conservation of mass, conservation of momentum in axial direction, conservation of momentum in tangential direction, conservation of momentum in radial direction considering zero radial flow, conservation of energy, pressure loss coefficient model with exponent distribution along the axial direction and thermodynamic correlations. Observing that the pressure loss coefficient is a measurable quantity and can be estimated experimentally and there is many experimental works done about that subject.

The result was 612 nonlinear algebraic equations solved by Newton Raphson method, where the Jacobean matrix was calculated analytically forming full matrix of $(612)^2$ elements. The convergent limit can reach any value which the computer can calculate. It is used 10^{-5} as an acceptable limit for each variable.

The output calculates the exit flow angle along the radial direction which satisfies the condition of zero radial flow, also calculated the power and efficiency along radial direction. The results show that the Free Vortex and Flow Through simplifying assumptions was not true for this model.

Calculation of thermal hydraulic and vibration assessment of heat exchanger of research reactor IEA-R1 according as built information of manufacturer

The heat exchanger of IEAR-1 nuclear reactor is designed for 5 MW heat transfer load. The following additional factors were considered in the

1. The hydraulic pressure drop in the secondary side must be in a certain value which can permit flow rate of 2200 GPM in the secondary side pump, valves, filters, etc. without any change in any additional component. The value of 2200 GPM is the flow rate needed for cooling tower for 5 MW load.

2. The hydraulic pressure drop in the primary side must be in a certain value that permits 3500 GPM at least in the primary side without any change in any component of primary circuit.

3. High resistance to vibration damage.

The above three items needed mathematical modeling of hydraulic performance of secondary and primary sides. The third item needed mathematical modeling of cooling towers thermal performance.

The manufacturer IESA suggested many designs. They were checked by calculations done according to HEAT EXCHANGER DESIGN HANDBOOK correlations and TEMA vibration assessment. Finally heat exchanger with 15% cut segmental baffles without tubes in the windows satisfied both calculations done by the manufacturer IESA and IPEN-CNEN/SP. The heat exchanger was fabricated and installed in the IEA-R1 research reactor of IPEN-CNEN/SP.

Development of the reliability database for IEA-R1 Brazilian research reactor

The development of a reliability database for the research reactors located at IPEN-CNEN/SP started in 2001 when Brazil took part in an IAEA Coordinated Research Project (CRP). The IAEA CRP was entitled “CRP to Upgrade and Expand the IAEA Reliability Database for Research Reactor PSAs” and had participants from eleven Member States: Argentina, Australia, Austria, Brazil, Canada, Czech Republic, India, Indonesia, Republic of Korea, Romania and Vietnam.

In the case of Brazil, a specific reliability database for IEA-R1 reactor continued being updated and improved. The reliability database of IEA-R1 reactor consists of a set of connected Microsoft Excel spreadsheets (input data and output/final data) with necessary information to generate estimates of component failure rates/probabilities of failure on demand and accident initiating events frequencies; and to compile human error evidences related to reactor operation and maintenance. The generation of these data aims to give support to several technical areas of IPEN-CNEN/SP for the development of reliability and safety analyses of the local research reactors or other similar facilities. The information gathered in this database mainly covers:

Component Technical/Engineering Data
Technical characteristics of IEA-R1 reactor components are stored in the database (type, size, rating, fluid, manufacturer, model, location, etc.).

Component operational data
Records of continuous operating times between consecutive interruptions (either planned shutdowns or not) and the number of demands of the components per reactor operation are stored in the database. In addition, cumulative operating times and number of demands are also computed.

Component maintenance data
Every maintenance activity (preventive, corrective or predictive), concerning each reactor component, is recorded in the database.

Component failure data
All component failures are reported and verified in order to identify their causes, effects on system / subsystem, actions taken and recovery time.

Data analysis
Part of the data stored in the database can be processed in order to generate estimates of component reliability parameters. The approach implemented in this database is based on the assumption that failure times are exponentially distributed. It generates an estimate of the constant failure rate (that is the inverse of the “mean time to failure”) associated to each time-related component failure mode. The analysis includes the calculation of a 90% confidence interval estimate (uncertainty limits) for each component failure rate or probability of failure on demand.

Accident initiating events and human errors data
Occurrences identified as accident initiating events precursors and/or human errors are stored in the database in order to be investigated and properly grouped. Considering the observation period from January 1999 to December 2007, 557 failures of 108 different component types were compiled. The total operating time of IEA-R1 reactor during that period was 19989,5 hours. Mean values of component failure rates / probabilities of failure on demand and respective confidence intervals are calculated using the algorithms developed during the IAEA CRP and are compiled in a specific spreadsheet. Data stored in the IEA-R1 database can also be used to estimate the frequencies of accident initiating events and to assess occurrences related to human errors during the operational and maintenance procedures. During the nine-year-observation period from 1999 to 2007, over 350 events were identified as initiating events precursors. In addition, 38 human errors were identified and grouped according to event types: failure to follow procedures or maintenance error (26); error of commission (9); and design error (3). Among these 38 events related to human errors, at least 25 could also be classified as precursors of accident initiating events. The scope of this database does not include quantitative derivation of human error data.

Neutronic analysis for production of fission Molybdenum-99 at IEA-R1 and RMB research reactors

The IEA-R1 reactor of IPEN-CNEN/SP is a pool type research reactor cooled and moderated by demineralized water and having Beryllium and Graphite as reflectors. In 1997 the reactor received the operating licensing for 5 MW. A new research reactor is being planning in Brazil to replace the IEA-R1 reactor. This new reactor, the Brazilian Multipurpose Reactor (RMB), planned for 30 MW, is now in the conception design phase. Low enriched uranium (<20% ^{235}U) targets (UAl_x dispersed in Al and metallic U foils) are being considered for the fission Molybdenum-99 (^{99}Mo) production. Neutronic calculations were performed to compare the production of ^{99}Mo for the two type of targets under similar conditions of irradiation (irradiation position, neutron flux and power density) both in the IEA-R1 reactor and RMB.

The UAl_x-Al targets of LEU type proposed and analyzed are aluminum coated miniplates. Each miniplate measures 52 x 170 mm, 1.52 mm thick, corresponding to a total volume of 13.437 mm^3. The UAl_x-Al core fuel is 40 x 118 mm, 0.76 mm thick, leading to a total volume of 3.587 mm^3. Considering this volume and a ^{235}U mass in the target equals to 2.01 g, the ^{235}U density ($\rho_{U\text{-}235}$) in the target core is 0.58 g $^{235}U/cm^3$. For a 19.9% ^{235}U enrichment, the uranium density in the target is ρ_U = 2.91 gU/cm^3.

A special Miniplate Irradiation Device (MID) was designed for the UAl_x-Al miniplates irradiation in the IEA-R1 reactor. Figure 17 shows the MID which has the external dimensions of the IEA-R1 fuel element. The miniplates will be allocated in a box with indented bars placed inside the external part of the MID.

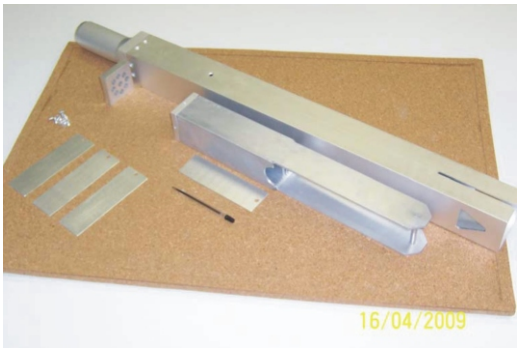


Figure 17. Miniplate Irradiation Device - MID

The targets of metallic U were mounted in cylindrical geometry, in a tubular arrangement. The metallic U foil was covered with a Ni sheet before being placed concentrically inside the aluminum tubes. The dimensions of the target are:

- One foil of uranium (LEU) of 44 cm x 76 mm x 135 µm;
- Coating nickel foil of 15 µm thickness;
- Two aluminum cylinder having 44 cm length, outside diameters of 27.99 and 30.00 mm, and inside diameters of 26.21 and 28.22 mm, respectively;
- ^{235}U mass of 20.1 g, distributed in 10 miniplates, with 19.9% enrichment of ^{235}U.

For the performed calculations, the U-Ni target was located in the same irradiation device, whose external dimensions are 76.2 mm x 76.2 mm x 88.74 cm (with nozzle). A mass equals to 20.1 g of ^{235}U in the metallic U foils was considered for the neutronic calculations.

From the neutronic calculations it was conclude that for the same amount of uranium in the analyzed targets (20.1 g) and the same irradiation conditions, a higher total ^{99}Mo activity was obtained for the U-Ni targets. In the IEA-R1 case, the total ^{99}Mo activity calculated at the end of the irradiation period for U-Ni targets was 1,275.8 Ci, while for the Al-UAl_x targets it was 581.89 Ci. For the RMB, the total ^{99}Mo activity obtained at the end of the irradiation time was 4,409.83 Ci for the Ni-U targets and 2,070.69 Ci for the UAl_x-Al ones. Initially, ^{99m}TC generators will be distributed five (5) days after the end of the irradiation. Consequently, the total ^{99}Mo activity is expected to reach values of 361.35 Ci and 164.7 Ci for the U-Ni and the UAl_x-Al targets irradiated in the IEA-R1, respectively. For the U-Ni and UAl_x-Al targets irradiated in the RMB, the total ^{99}Mo activity at the distribution time is expected to be 1,249.06 Ci and 583.51 Ci, respectively. From these values, it is noted that the Brazilian current demand of 450 Ci of ^{99}Mo per week and the future projected demand of 1,000 Ci may only be addressed by the RMB reactor under conception. This research is part of the IAEA's Coordinated Project (CRP) T1 2018 - Developing Techniques for Small Scale Indigenous Molybdenum 99 Production using Low Enriched Uranium (LEU) Fission or Neutron Activation.

Application of non-destructive methods for qualification of high density fuels in the IEA-R1 reactor

The IEA-R1 reactor of IPEN/CNEN-SP in Brazil is a pool type research reactor cooled and moderated by demineralised water and having Beryllium and Graphite as reflectors. Since 1990, IPEN/CNEN-SP has been fabricating and qualifying its own U_3O_8-Al and U_3Si_2-Al dispersion fuels. The U_3O_8-Al dispersion fuel is qualified to a uranium density of 2.3 gU/cm^3 and the U_3Si_2-Al dispersion fuel up to 3.0 gU/cm^3. The IEA-R1 reactor core is constituted of the fuels above, with low enrichment in U-235 (19.9% of U-235).

Nowadays, IPEN/CNEN-SP is interested in qualifying the above dispersion fuels at higher densities. Fuel miniplates of U_3O_8-Al and U_3Si_2-Al fuels, with densities of, 3.0 gU/cm^3 and 4.8 gU/cm^3, respectively, which are the maximal uranium densities qualified worldwide for these dispersion fuels, were fabricated at IPEN/CNEN-SP. The miniplates were put in an irradiation device, with similar external dimensions of IEA-R1 fuel assemblies (FA), which was placed in a peripheral position of the IEA-R1 reactor core.

IPEN/CNEN-SP has no hot cells to provide destructive analysis of the irradiated fuel. As a consequence, non destructive methods are been used to evaluate irradiation performance of the fuel miniplates:

- monitoring the fuel miniplate performance during the IEA-R1 operation for the following parameters: reactor power, time of operation, neutron flux at the position of each fuel assembly, burnup, inlet and outlet water, and radiochemistry analysis of reactor water;

- periodic underwater visual inspection of fuel miniplates and eventual sipping test for the fuel miniplate suspected of leakage.

The miniplates have been periodically visually inspected by an underwater radiation-resistant camera inside the IEA-R1 reactor pool, to verify its integrity and its general plate surface conditions. A new special system was designed for the fuel miniplate swelling evaluation. The fuel swelling evaluation is being performed by means of the fuel miniplate thickness measurement during the shutdown periods between successive irradiation cycles at the IEA-R1 reactor. During the measuring period, the fuel miniplates are transferred from the reactor core to the measurement system positioned at the pool border. This measurement system was sponsored by FAPESP.

Neutronic, thermal-hydraulics and accident analysis calculations for an irradiation device to be used in the qualification process of dispersion fuels in the IEA-R1 research reactor

Neutronic, thermal-hydraulics and accident analysis calculations were developed to estimate the safety of an irradiation device placed in the IEA-R1 reactor core. The irradiation device will be used to receive miniplates of U_3O_8-Al e U_3Si_2-Al dispersion fuels, LEU type (19.9% of ^{235}U), with uranium densities of, respectively, 3.0 gU/cm^3 and 4.8gU/cm^3. The fuel miniplates will be irradiated to nominal ^{235}U burnup levels of 50% and 80%, in order to qualify the above high-density dispersion fuels to be used in the Brazilian Multipurpose Reactor, now in the conception phase. For the neutronic calculation, the computer code CITATION was utilized. The computer code FLOW was used to calculate the coolant flow rate in the irradiation device, allowing the determination of the fuel miniplate temperatures with the computer model MTRCR-IEA-R1. A postulated Loss of Coolant Accident (LOCA) was analyzed with the computer codes LOSS and TEMPLOCA, allowing the calculation of the fuel miniplate temperatures after the reactor pool draining.

The MID has the external dimensions of the IEA-R1 fuel element. The miniplates will be allocated in a box with indented bars placed inside the external part of the MID. Up to ten miniplates can be placed in the box inside of the MID. The qualification of the U_3O_8-Al and U_3Si_2-Al dispersion fuels with higher ^{235}U density will be made in use, which means that it is based on the irradiation of the dispersion fuel miniplates in the IEA-R1 reactor followed by the use of non-destructive analysis techniques, mainly fuel miniplate visual inspections performed regularly with a radiation-resistant underwater camera. A new special system was designed for the fuel miniplate swelling determination. The swelling determination will be by means of the fuel miniplate thickness measurement during the irradiation time in the IEA-R1 reactor. The so called “Fuel Miniplate Thickness Measurement System” will be located at the fuel storage area of the IEA-R1 reactor pool. It will be operated from the reactor pool border, allowing the measurement of the fuel miniplate thickness along its surface by electronic probes (LVDT). The results will be collected by instrumentation connected to the probes.

The result of neutron calculations showed that the best position for the irradiation in the reactor core is the 36, for having the highest power density, and the inclusion of MID in the reactor core does not affect the operation of the same, since the change in reactivity is irrelevant. Through the thermal-hydraulics calculations it was determined a minimum flow for cooling the fuel miniplates diverting a small fraction of the flow of the reactor core without affecting the cooling of the core. From the analysis of accidents concluded that there is any damage to miniplates in the case of a postulated Primary Coolant Boundary Rupture. This project was partially sponsored by FAPESP.

Development of computer codes for loss of coolant accident analysis of IEA-R1 reactor

Two computer programs, LOSS and TEMPLOCA, were developed to analyze postulated Loss of Coolant Accidents (LOCA) in the IEA-R1 reactor. The LOSS program determines the time to drain the reactor pool down to the level of the bottom of the core. The TEMPLOCA program calculates the maximum temperature reached in the fuel, due to the decay heat of fission products and when there is complete loss of coolant in the core. These programs were used to assess the safety of a

Miniplate Irradiation Device (MID), placed in the IEA-R1 reactor core, during the occurrence of a postulated loss of coolant accident. The MID are being used to receive miniplates of U_3O_8-Al and U_3Si_2-Al dispersion fuels, LEU type (19,9% of ^{235}U) with uranium densities of, respectively, 3.0 gU/cm^3 and 4.8 gU/cm^3. The fuel miniplates will be irradiated to nominal ^{235}U burnup levels of 50% and 80%, in order to qualify the above high-density dispersion fuels to be used in the Brazilian Multipurpose Reactor (RMB), now in the conception phase.

Studies were performed to evaluate possible postulated loss of coolant events, which could lead to the reactor pool emptying. Five of them were considered to be mostly critical: a) Tube rupture of the Irradiation Pneumatic System (IPS); b) Pool drainage failure rupture of the access tubes for the Water Retreatment System (WRS); c) Primary system boundary rupture; d) Undue opening of the WRS drains; e) Failure in the collimator tubes of Beam Hole-3 (BH-3).

Out of the five accidents analyzed with the LOSS program, the primary system boundary rupture was found to be the most critical. The calculations showed that about 7.5 minutes are necessary to drain the reactor pool during a postulated primary system boundary rupture. After the pool draining, the maximal fuel miniplate temperatures calculated with the TEMPLOCA was 125 ºC, below the blistering temperature, which is the fuel temperature design limit.

Fuel miniplate thickness measurement system for dispersion fuel swelling evaluation

A special system was designed and constructed at IPEN/CNEN-SP for fuel swelling evaluation. The system will be used in the qualification process of U_3O_8-Al and U_3Si_2-Al dispersion fuels with 3.0 gU/cm^3 and 4.8 gU/cm^3, respectively. The determination of the fuel swelling will be performed by means of fuel miniplate thickness measurements along the irradiation time, during the shutdown period between the operational cycles of the IEA-R1 reactor. The system will be located at the reactor pool fuel storage area and it will be operated from the reactor pool border, allowing the measurement of the fuel miniplate thickness along its surface by electronic probes (LVDT). The results will be obtained by the instrumentation connected to the probes.

The Figure 18 shows the fuel miniplate thickness measurement system. It consists of a mobile metallic column, held by an X-Y coordinate table system for miniplate thickness measurement. The table is supported by another metallic structure fixed at the border of the reactor pool. The thickness measurement is performed by electronic probes (LVTD). The results are obtained by the instrumentation connected to the probes.

(a) (b)

Figure 18. Fuel miniplate thickness measurement apparatus at IEA-R1: (a) lateral view; (b) profile view (thickness)

For commissioning of the measurement system, two sets of measurements were made. The first set of measurements has consisted of thickness measurements of an aluminum dummy miniplate, outside the reactor pool. These measurements have evaluated the system performance in dry condition. The second set of measurements was performed for another dummy miniplate in the IEA-R1 reactor pool. The fuel miniplate thickness measurement system showed good response. Dimensional results were in good accordance. The results show that the equipment is efficient and accurate, with measurement precision of 1 μm.

Improvement of computer codes used for fuel behaviour simulation - FUMEX III

The Brazilian nuclear organizations are conducting joint efforts toward the coordination of existing personnel and infrastructure in order to create competence to study, design and develop nuclear fuel elements for high performance and extended burnup. As part of this program, IPEN-CNEN/SP joined the FRAPCON/FRAPTRAN users group in order to obtain the permission to utilize the computer codes FRAPCON-3 and FRAPTRAN. Since them, IPEN has made a joint effort between personnel and students to understand fuel rod modeling of these computer codes in both normal and off-normal conditions. With project “IAEA Improvement of Computer Codes Used for Fuel Behaviour Simulation FUMEX III”, IPEN will have the opportunity to test fuel modeling of FRAPCON-3 and FRAPTRAN codes against data and cases provided by IAEA and OECD/NEA. It is IPEN intention to promote with this project interaction, collaboration and discussions amongst Brazilian fuel modelers which will result in a better understanding of physical processes and phenomena, and thus allows improvements to be made in both Brazilian organizations codes and their models. The following Brazilian organization are participating of the FUMMEX III program: IPEN-CNEN/SP, CDTN-CNEN/BH, Eletronuclear (Eletrobrás Termonuclear S.A.), INB (Indústrias Nucleares Brasileiras) and CTMSP (Centro Tecnológico da Marinha em São Paulo). The project is being sponsored by IAEA.

Design, analyses and tests of a nuclear research reactor spent fuel storage and transportation packages

The applied qualification requirements for the packages used in the transportation of nuclear spent fuel elements are very severe due the nature of the radioactive content. They include the so-called normal conditions of transport and the hypothetical accident conditions. The 9 m drop tests are the most critical hypothetical accident conditions. The package qualification under these conditions shall be conducted using full scale models (prototypes), small scale models, numerical simulations and a combination of physical tests and numerical simulations. The choice of the qualification approach depends on economical and safety aspects. To comply with the nuclear safety functions, as the containment of the internal products and biological shielding, the package itself has several components connected to each other in different ways (impact absorbers, welded parts, flanged connections, surface contacts, etc.).

This research involves other groups in Brazil and abroad (mainly, Argentina) and it is sponsored by IAEA. It uses a combination approach with tests and numerical simulations for the structural assessment of a half scale model of a package for transportation of nuclear research reactor spent fuel elements under 9 m drop tests. The numerical simulations of the 9 m free drops over a rigid surface of half scale model of the transportation package under different orientations were conducted using a finite element explicit code considering several nonlinear aspects as the nonlinear materials models and properties, with emphasis on the impact absorbers behavior, the different package materials stiffness, and the different types of the contacts between the package components and between the package and the rigid surface, including the friction in the contacts. Also, several 9 m drop tests were conducted in a half scale model in different drop orientations.

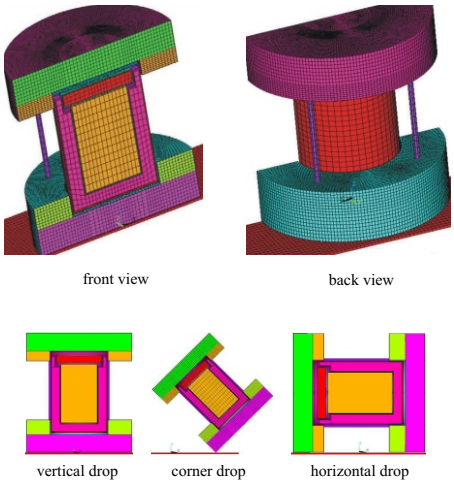


Figure 19. 180° finite element model and drop orientations

Monitoring steam generator tubes using wavelet transforms in eddy current test signals

Eddy current inspection is the standard method for routine steam generator tubes inspection. To improve the rate of correct diagnosis, a time - frequency domain transform, namely the wavelet transform is used to process the signals which are transient by nature, avoiding subjective inspectors decisions. The use of Wavelet Transform allows for automatically denoising the signal from sensor wobble, geometrical and material properties fluctuations and electronic noises. The method has been tested successfully in actual tubes from steam generators as well as laboratory made tubes with known defects implanted. Improved precision in the localization of the fault is also achieved by the right choice of the frequency band.

Integrated analysis of the USEXA postulated accidents

Under request of Centro Tecnológico da Marinha in São Paulo (CTMSP) was developed an integrated analysis of postulated accidental scenarios presented in the Preliminary Safety Analysis Report (PSAR) of the Uranium Hexafluoride Production Facility (USEXA), installed at Iperó, São Paulo, Brazil. The main purpose was to identify the propagation conditions of these accidents, both, on and offsite of process units, as well as to nearby plants (domino effect). Internal and external hazards chemical releases, fire and explosions at the facility were evaluated.

The PHAST Professional Software - version 6.51 (DNV Risk Management Software licensed to CEN) was used to perform consequence assessment. The domino effect analysis was carried out with methodology proposed in MSc. thesis “A Study on Domino Effect in Nuclear Fuel Cycle Facilities”, developed at IPEN/CNEN-SP.

Analysis of the USEXA hydrogen fluoride postulated accidents

Under request of Centro Tecnológico da Marinha in São Paulo (CTMSP) was developed an analysis of accidental scenarios involving Hydrogen Fluoride in the Uranium Hexafluoride Production Facility (USEXA), installed at Iperó, São Paulo, Brazil. The main purpose was to identify the toxic consequences of these accidents, both, on and offsite of process units, as well as to nearby areas. Internal and external hazards releases at the facility were evaluated.

The SAFETI Professional Software - version 6.51 (DNV Risk Management Software licensed to IPEN/CNEN-SP) was used to perform consequence assessment.

Decision support system for major accident prevention in the chemical process industry

The chemical industry today is processing a lot of hazardous substances within densely populated areas. The risks emerging from the processing, storage, handling, and transport of these hazardous materials are becoming more and more complex. Consequently chemical plants worldwide are faced with the growing importance of safety issues. Comprehensive and detailed hazard mapping and an understanding of possible consequences are necessary.

In last years it was developed decision support tools for investigating (internal and external) major hazards in the chemical industry to assist risk management decision makers in implementing organizational decisions on plant safety. Consequently an inherent approach to domino effect prevention in chemical process industries can be applied in early plant design or during modification in existing plant units.

Operation, maintenance and experimental utilization of the IPEN-MB/01 reactor

The first criticalization of the IPEN/MB-01 reactor (Figure 20) was obtained on November 9th, 1988. Since this date more than 2600 operations were made to measure very important in core parameters of the Reactor Physics such as kinetics parameters, spatial and energetic neutron flux distribution, power density, nuclear reaction rate, spectral indices, buckling, criticality rods positions to several and different reactor core (Figure 21) configurations. These experimental parameters are very important to correlate reactor calculation and its nuclear data libraries with experimental results. Thus is possible to improve the accuracy and precision of calculation methodology used to design the nuclear reactors core.

Figure 20. IPEN/MB-01 Reactor building

The IPEN/MB-01 has a maintenance program (each 2 weeks the reactor is shutdown for this purpose) that consists from a simple visual inspection of the reactor systems to a preventive and corrective maintenance made by the operators.

For complex problems is contracted external maintenance. Normally are reserved until 15 weeks by year to maintenance of the IPEN/MB-01 Reactor.

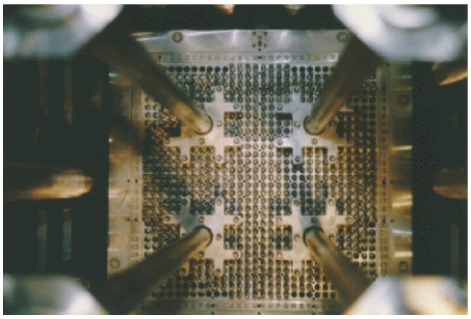


Figure 21. : IPEN/MB-01 Reactor core

Basically the reactor has three kinds of activities: Operational, Experimental and Educational. The operational is to testing the reactor systems and calibration of control rods, nuclear channels, power level, IAEA inspections, and training operators. The experimental activities are to measure reactor physics parameters to several purposes such as academic, benchmarks (OECD-NEA), educational (São Paulo University - Graduated and Post-Graduated) and to internal (IPEN-CNEN/SP) and external (Reactor Operator Training: CTMSP-Brazilian Navy and ELETRONUCLEAR).

The Table shows the number of operations, experiments and criticality time in 2008, 2009 and 2010 years. The total operational time to starting and shutdown of the reactor is about 1 hour and this time in not considered in the table below but may be estimated as being the same that the number of operations by year in hours.

Year	Number of Operations	Time Criticalization (H:Min)	Experimental and Operational Activities
2008	173	236:34	Neutronic characterization at Neutron Flux trap, Spectral Indices, Subcritical experiments, Instrumentation test (electrometers and filters) to noise analysis, Measurements of Spectral Cross Density (CPSD), Spectral ratios using fission chambers, Criticality using gadolinium in fuel rods, Solid detector testing Graduate Experimental Reactor Physics Course, reactivity measurements, training of reactor operators, Radiological emergency simulation-exercise.
2009	137	181:05	Operational testing, Reaction rate along of radial fuel pellet, Instrumentation tests, Neutronic characterization at Neutron Flux trap, Subcritical Reactivity Experiments, Measurements of Spectral Cross Density (CPSD), Buckling, Measurements, Effective neutron temperature, Neutrons box reflector experiments, Out-of-Core experiments, Critical configuration using stainless steel slabs (Benchmarks), Measurements of Neutron Spectrum Energy inside the fuel rods, Post-Graduate Experimental Reactor Physics Course, training of reactor operators (Eletronuclear).
2010	134	133:29	Neutronic characterization at Neutron Flux trap, Reaction rate along of radial fuel pellet, Cross power spectral density (CPSD), Measurements of Neutron Spectrum Energy inside the fuel rods, Reaction Rates measurements, Fission reaction rates measurements, Reactivity temperature coefficient measurements, Experiments using boron acid diluted at water moderator, training of reactor operators, Graduate experimental Reactor Physics Course.

Modernization of the IPEN/MB-01 reactor

The main goal of this modernization project (FINEP Support) is the replacement of the current control board IPEN/MB-01 reactor by a system with Programmable Logic Controllers (PLC). This new system will implement all the existing functions in the control board due to their flexibility and may implement others, so as to facilitate reactor operation and control.

The reactor protection system, consisting primarily through the channels and the nuclear safety interlocks, will not be modified. Only his connections with the current control board will be transferred to the system with PLCs. Being a development model for future reactors to be designed in Brazil, the reactor IPEN/MB-01 must accompany the state of the art control using digital systems. Thus, these digital control systems will be tested and evaluated in the safe operation of a zero nuclear reactor, lends itself to this kind of experience because of their safety, as evidenced in their 23 years of operation

Items controlled by the system with Programmable Logic Controllers (PLC)

The system with Programmable Logic Controllers (PLCs) will be able to act in 237 input variables and output of the scorer's table, with forecast growth of up to 315 variables.

Architecture of the system with PLC

The system with PLCs for data acquisition, display and control of the reactor IPEN/MB-01 will comprise three units interface with the field: Acquisition Unit Signal Command and General, for the acquisition of the variables the scorer's control board and to generate control signals for operating the reactor IPEN/MB-01; Unit Command Bars (UCB) to the command manual/automatic neutron absorbing rods (two control and two safety rods) and Acquisition Unit Position rods, which is to make acquisition of data from 08 position indicators of the reactor rods (04 Absolute Position Indicators and 04 Indicators of Relative Position).

The man-machine interface (HMI) will comprise 03 units, each composed mainly of micro-computer, video terminal, keyboard and mouse. The main unit for operation and control of the reactor called "SCADA/HMI" and two others only for monitoring the reactor called "clients". The connection of these units with units to interface with the field should be done through Ethernet and the communication with the signs of the reactor will be held by the input and output interfaces (I / O) of the PLC.

Researches in energy, environment and development

In 2004 was formed a multidisciplinary group to study several relationships between Energy, Environment and Development with their social reflexes. Researchers from IPEN and others institutions (IEE, UNICAMP and UFRJ) are part of the group qualified by CNPq. The sub-lines studied are: basic concepts, consumption of energy and development, energy scenarios, energy and life quality, environment and development, sustainable development indicators, environmental solutions, alternative energy and energy conservation and consumerism. There are also partnerships to other institutions that already have tradition in this area.

During the last years, several studies have been executed and analyzed by the group, for example: the study of the consumerism as a relevance component in global environmental degradation, the relation between human activities and climate and environment changes since the primordium of the man up to the Industrial Age, study of the Emergy (Embodied-energy) methodology and its scientific fundamentals that takes into account the embodied energy during a process transformation, issues of Climate Change and Global Sustainability based in the Game Theory, analysis and actions aimed to human values as a form of contribution to the planet sustainability, multivariate analysis applied to the study of energy-environment-development relationship, etc.

The sustainable development is no doubt, the most divulged concept in the present time, taking into account the economic effectiveness, simultaneously with the requirements of ecological, social, cultural, technological and politics order. The concept of sustainable development started to be widely used after the United Nations Conference on Environment and Development that occurred in Rio de Janeiro in June of 1992. After that, some countries started presenting the sustainable development as a component of its political strategy conjugating environment, economy and social aspects. One of the challenges of the construction of the sustainable development is to create measurement instruments, such as the sustainable development indicators. Sustainable development Indicators are essential instruments to guide the action and to subsidize the accompaniment and the evaluation of the reached progress toward the sustainable development. One of the critical aspects is the methodology to be adopted in the determination of the indicator, its reading and interpretation. Independent of the choice of the methodology, it must be clear and transparent, about the principles that are in the base of the process.

Services on nuclear engineering and specialized services on nuclear and conventional power plants

Services were performed by the Nuclear Engineering Center in the nuclear engineering field, mainly among activities directly related to power and researches reactors, considering the following specialties: nuclear fuel, waste management, reactor physics, thermal and hydraulics, electricity, structural mechanics, piping, instrumentation, monitoring and diagnosis of reactor systems and components and safety analysis. The expertise of our professionals in the above fields is directed to the identification and analysis of technical problems and proposition of solutions to improve safe operation, better maintenance and efficient upgrade of systems.

Complementing the services described in the nuclear engineering field, other specialized technological services were also performed in risk analysis of fuel cycle installations and benchmarking of conventional fossil fueled power plants. The services were directed to solve technical problems related to safe design, reliable and efficient maintenance operation. The background acquired is also applied to others industrial chemical process and conventional industrial power plants.

All the services and specialized services described above were conducted by a team of 20 (twenty) professionals. A total of approximately 12.000-man-hour per year was spent in all these activities related to experiments, laboratory and engineering services. The outcome of these technical activities was demonstrated through technical reports, engineering documents, calibration sheets, inspections reports and training notes.

The customers were some other departments of the Brazilian Nuclear Energy Commission (CNEN) and other companies such as the Brazilian oil company (named PETROBRAS), Electrical and Energy Institute from São Paulo University, the Brazilian Nuclear Utility (named Eletronuclear) and the Brazilian Navy Technological Center (named CTMSP). All the engineering services are certified with the ISO 9001:2008.

In the triennial 2008-2010 the IEA-R1 Research Reactor has been operated most of the time at a power of 3.5 MW and operation schedule of 62 hours per week, achieving the following results:

- Reactor power: 3,5 MW
- Time of reactor operation: 8090 h
- Energy dissipation: 24697 MWh
- Number of samples irradiated in the grid plate: 3107
- Number of samples irradiated in the pneumatic system: 1950
- Production of ^{131}I: 1958 Ci
- Production of ^{153}Sm: 57 Ci

Besides of the routine operational schedule, other activities were carried out to extend the operational lifetime of the reactor, improve the conditions to comply with user needs and allow the operation in higher powers.

Pneumatic system to transfer irradiated sample from the IEA-R1 reactor to radiopharmaceutical facility and Activation Analysis Laboratory

This system are been installed with the aim to facilitate the transfer of irradiated material to these facilities. The line are installed and a new facility is been built in the reactor to place the terminal station and to link this to the reactor pool.

Pneumatic system to short irradiation in the IEA-R1 reactor

This system needed to be rebuilt due to timing deterioration. The project and construction of the new one was done by IPEN staff. Another station located in a different place and with a different neutron flux is been constructed by the same staff in order to comply with the user needs.

Monitoring systems

Monitoring systems are being improved in order to increase the confidence level in the safety of IEA-R1 reactor operating conditions. These include:

- Data acquisition system (SAD) concluded;
- Neutron flux monitoring system final phase of implementation;
- Online system to monitoring aerosol emission in implementation;
- Core temperature monitoring by using a instrumented fuel element concluded;
- Reactivation of a meteorological station at IPEN site in implementation;
- Pump vibration monitoring system a wireless module is being included.

Many of these equipments were obtained with IAEA financial support.

Ageing program

In order to extend the reactor operation lifetime some equipments and systems are being replaced or rebuilt. These include:

- Cooling tower: Tower B was rebuilt and Tower A will be tested in order to assure it performance if the reactor power is increased to 5 MW;
- Beam hole draining system: totally revised, including the placement of a flange in the BH#4 and BH#12, in order to minimize leak risks and worker exposing when the reactor power is increased to 5 MW;
- Electronic system: control room racks are been replaced and a new table control is been projected.

Physical protection system

New equipments like cameras, 42” televisions, tape records and electronic locks were installed to improve the access control and surveillance system.

Training program

Besides of the internal operator training program, part of the staff participated of scientific visits and training in foreign facilities to learn about different reactor systems. These visits and training were supported by IAEA resources.

Maintenance program

An extensive program of testing, preventive maintenance and calibration was carried out to assure performance and reliable measures by equipments and systems related to reactor operation and control.

Monthly activity report

A report containing the activities and monitored and controlled operational parameters of the reactor are issued monthly. This report encloses the number of reactor operation, dissipated energy, reactor core data, chemical and physical characteristics of the pool water, radioisotopes concentration in the pool water, number of reactor shutdown, number of irradiated samples in the reactor core and pneumatic system and, finally, the radioprotection data.

Management system

Since 2002 the Quality Management System that support the scope “Operation and Maintenance of the IEA-R1 Reactor and Irradiation Services” was considered certified by Fundação Carlos Alberto Vanzolini in compliance with NBR ISO 9001, being submitted to annual internal and external reevaluation.

Nuclear Reactors and Fuel Cycle

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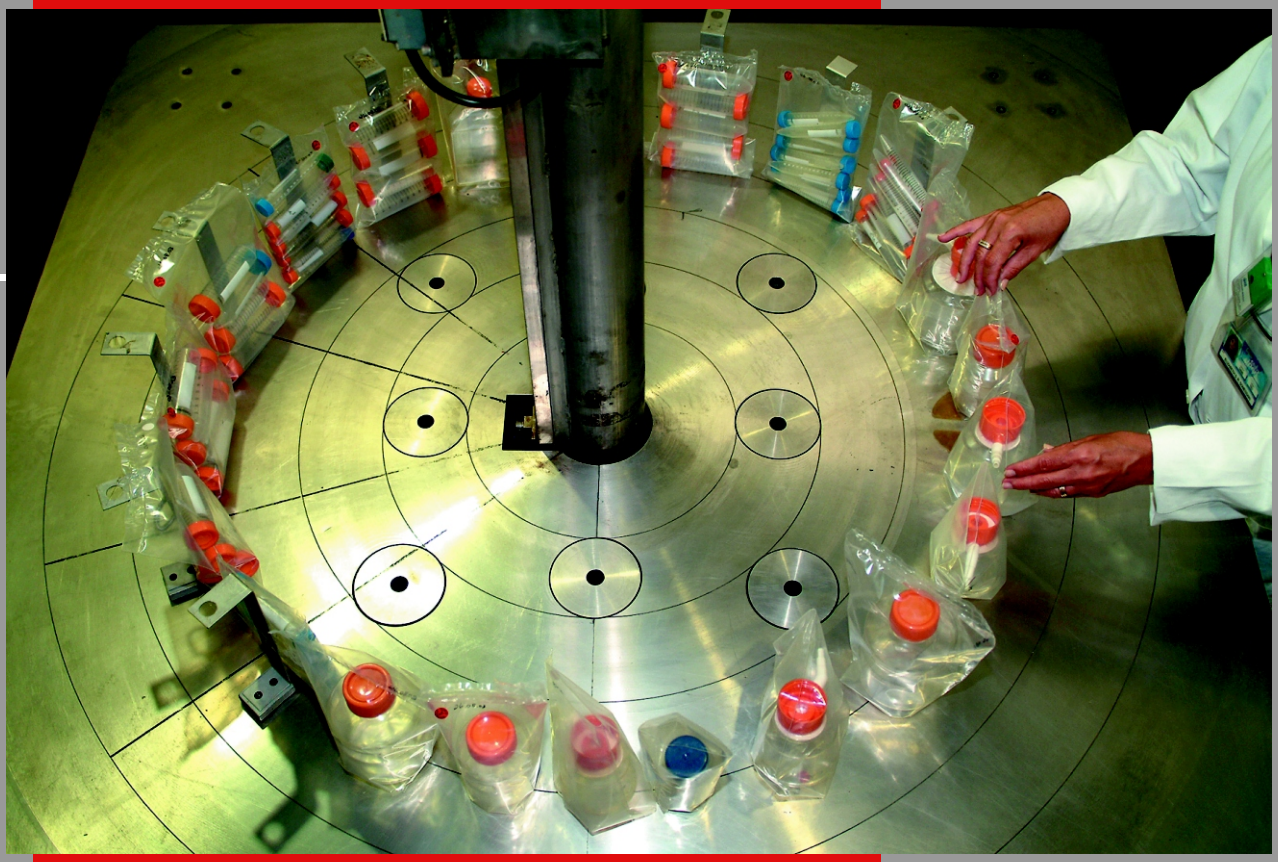
Applications of Ionizing Radiations



Packaging with different materials are sterilized in the cobalt-60 panoramic irradiator

Applications of Ionizing Radiations

Introduction

Ionizing radiation can modify physical, chemical, and biological properties of materials. Today's market dynamics using industrial electron beam, X-ray and gamma ray technologies are changing approaches to radiation sterilization, including an increasing trend towards processing small batches of high value-added, dose sensitive, complex multi-component healthcare products that require rapid turnaround. Significant progress is being made in the development of X-ray systems for large scale, high volume sterilization of medical products.

Many gamma ray (^{60}Co) irradiators have been built and it is estimated that about 300 are currently in operation in Member States of the IAEA. In recent times, the use of electron accelerators as a radiation source (and sometimes equipped with X-ray converter) is increasing. However, gamma irradiators are difficult to replace, especially for non-uniform and high-density products.

Electron beam radiation treatment of polymers continues to be the largest field in the application of radiation. Market surveys indicate there are more than 1,400 high-current electron beam accelerators (EBA) in commercial operation today, providing an estimated added value to numerous products of $85 billion USD or even more. Primarily, EBA are used to treat wire and electric cable, heat-shrinkable tubing and film, and tires, as well as for surfacing curing. Research laboratories and scientists are working together with companies in the development of advanced materials.

For spreading and consolidating techniques that lead to the use of the radiation technology and radioisotopes applications in Industry, Human Health, Agriculture and Environmental Preservation, the Radiation Technology Center was founded in 1972.

The main R&D activities of the Applications of Ionizing Radiations program are in consonance with the IPEN Director Plan (2008-2010) with four subprograms:
- Food and Agricultural Products Irradiation;
- Radiation and Radioisotopes Applications in Industry and Environment;
- Radioactive Sources and Radiation Applications in Human Health; and
- Radioactive Facilities and Equipment for Nuclear Techniques Applications.

During its trajectory of success and achievement it can be highlighted the projects for implantation of the ^{60}Co Multipurpose Irradiator and the EBA, supporting the local scientific and industrial communities on development of process and products; for technological domain production and distribution of ^{125}I seeds for prostate cancer treatment; and also, the assembling of an third-generation industrial computed tomography scanner for multiphase flow system analyses. The improvement of the activities of radioisotope technology application in the petrochemical and chemical industries for processing control and sanitation, and activities in the RadTech South America and ICTR, and organization of the IX ENAN together with the International Nuclear Atlantic Conference (INAC 2009) can also be highlighted.

All realizations and achievements were only possible with the governmental financial support, standing out projects by FAPESP, CNPq, CAPES and FINEP, and international projects, such as, ARCAL, Technical Cooperation (TC) and Research Contract (RC) supported by the IAEA, as well as, the national and international partnership and cooperation with industries, universities and institutions.

Facilities and Devices for Application of Nuclear Techniques

At the IPEN/CNEN/SP there are two Industrial Electron Beam Accelerators of 97.5 kW (1.5 MeV - 65 mA) and 37.5 kW (1.5 MeV - 25 mA), supplied by IBA Industrial Inc. and two Cobalt-60 Irradiators - Gammacell (2,142 Ci Dec - 2010), Panoramic (394 Ci Dec-2010) designs, and Gamma Multipurpose Irradiator covering 76 m^2 of floor area, the irradiator design is product overlap sources and the maximum capacity of cobalt-60 wet sources is 37 PBq (1 MCi). The performed qualification program of this multipurpose irradiator was based on AAMI/ISO 11137 standard, which recommends the inclusion of the following elements: installation and process qualification. The initial load of the multipurpose irradiator was 3.4 PBq (92.1 kCi) with 13 cobalt-60 sources model C-188, supplied by MDS Nordion Ion Technologies - Canada. For irradiator dose optimization, the source distribution was done using the software Cadgamma developed by IPEN-CNEN/SP. The poly-methylmetacrylate (PMMA) dosimeters system, certified by the International Dose Assurance Service (IDAS) of the International Atomic Energy Agency (IAEA) was used for irradiator dose mapping. The economic analysis, performance concerning to dose uniformity and cobalt-60 utilization efficiency were calculated and compared with other commercial gamma irradiators available in the market.

The Electron Beam Accelerator and Cobalt-60 Irradiators are mainly applied for research, development and services of preservation and disinfestations of food and agricultural products; treatment of industrial and domestic effluents, sludge and hospital waste; paints, varnishes, adhesives and coating cure; preservation of art works and books; radiosterilization of bones and human tissues; Brazilian gemstones enhancement; polymer grafting and modification; radiation processing of composite materials and natural polymers. The gamma rays (electromagnetic energy) and electron beam (EB) are very efficient agents for radiosterilization of medical, pharmaceutical, and biological products due to high sensitivity of pathogenic bacteria to radiation. In 2010, 26,340 medical, pharmaceutical, and biological products were radiosterilized and 372,500 semiconductors (diodes) were processed by ionizing radiation in these radioactive facilities. Annually, 3,530 km of wire and electric cables for chemical, automobile, aircraft and electro-electronic companies have been irradiated in the Industrial Electron Beam Accelerator. The radiation processing promotes crosslinking among the polymeric chains, increasing electrical, thermal, mechanical, and chemical properties. The modernization of the installation promotes the elevation of wire and electric cables processing velocity to 300 m/min and polyethylene foams to 15 m/min, becoming the product prices more competitive in the Brazilian market.

Industrial dosimetry in radiation processing

In radiation processing, a well characterized reliable dosimetry system that is traceable for recognized national and international dosimetry standards is the key element of such activities. The Industrial Dosimetry Laboratory has the responsibility to measure the radiation dose absorbed in the processes induced by ionizing radiation at Co-60 gamma ray irradiation (Gammacell, Panoramic and Multipurpose Irradiator) and electron beam (two Industrial Electron Beam Accelerators of 97.5 kW and 37.5 kW) facilities in ordinary services and to develop new products and services by radiation processing. The dosimetry procedures for radiation processing are carried out in agreement with the ISO (International Organization for Standardization) - ASTM (American Society Testing and Materials) standard guides and practices. To establish a reliable dosimetry system, the laboratory has participated of the intercomparisons of gamma dose measures, organized by International Dose Assurance Service (IDAS) offered by the International Agency Energy Atomic (IAEA) and of the national intercomparisons to check on the entire radiation dose measurement system: dosimeters, measurements equipment, and irradiation and data procedures. The dosimetry systems used for the quality control of the radiation process are: Fricke solution as reference standard dosimetry system, Alanina as transfer standard dosimetry system and as routine dosimetry.

Radiation detectors and industrial computed tomography

In the last years, industrial computed tomography (iCT) in Brazil has had its application in non-destructive testing. Nowadays, a wide range of chemical and petrochemical industries have great interest in using iCT for improving design, operation, optimization and troubleshooting of industrial processes. The industrial distillation systems involve fast dynamic processes and, in addition, contain solids, liquids and gas mixtures. The industrial distillation columns are usually built with steel and have large diameters and thicknesses that become unfeasible their analysis with conventional X-ray beam. For these reason gamma radioactive sources in the energies range of 317 keV from ^{192}Ir, 662 keV (^{137}Cs) to ~1250 keV (^{60}Co) are preferable instead of X-ray sources. Gamma ray iCT for multiphase processes is now a promising technique and has been studied for advanced research centres. To follow t his trend and to remain updated, the CTR/IPEN laboratory studies the development of the CT methodology for industrial multiphase systems. Three types of gamma ray iCTs have been developed for different applications:
A first generation iCT, a one detector-one source was developed. The data acquisition board, the mechanical control interface and the software were

developed to be used in this iCT system. The developed tomography system was validated using an IAEA phantom placed between a 2"x2" NaI(Tl) detector and a collimated ^{60}Co source. A good resolution was observed for all images reconstructed from the developed CT systems. A third-generation type CT, where the collimated detectors are arranged in an arc at the center of the radiation source, was developed in our laboratory. The whole assembly of the detectors and the radiation source are mounted on a gantry suitable to be rotated around the axis of the test section through a stepper motor interfaced with a host computer. The electronic system with twelve multichannel boards with their HV supplies, a circuit to control three step motors and data acquisition system were specially developed for the third-generation system.

Figure 1. Electronic system for data acquisition and step motor control

A fourth generation iCT (4D ICT) was proposed and its development is ongoing. It will be mounted on a wooden platform (a lightweight platform easily adaptable for working conditions in industrial plants), using multi small detectors surrounding the distillation column (the object). Among the advantages of this system, it should be emphasized that: (a) this system is fixed around the column, not requiring movements, (b) it is weightless and (c) it is portable, which makes it suitable for its use in industrial plants. While the fourth generation iCT will be more suitable for use in industrial plants, the third generation iCTs are often used in laboratories for demonstration of this technology and/or to establish parameters in the development of a new design for chemical reactors/ columns; the third-generation iCT is, also, useful to promote improvements aiming to increase the production efficiency. As to the first generation iCT, it is being used to establish a methodology to analyse the concentration gradient of uranium in the nuclear fuel plate produced at Ipen-Cnen/SP. This R&D has been supported by the IAEA (RC 12459 and TC BRA8031 Projects) and MCT/CNPq/CT-Petro (505161/04-4 and 620201/2008-8 Projects).

Semiconductor radiation detectors to be operated at room temperature

The lead iodide (HgI_2) and thallium bromide (TlBr) are very promising materials with large technological applicability as a room temperature semiconductor radiation detector for X and gamma rays spectrometry. Several studies have been carried out about the preparation of both crystals and progress has been achieved by the advance of purification techniques, growth and characterization of HgI_2 and TlBr crystals. Material processing, especially purification, was found to have influence on the detector response, suggesting that improvement in the performance of detectors could be possible with more purification. Higher purity crystals exhibit significant enhancement in the detector performance. TlBr crystals were grown by Bridgman technique, while different methods have been studied for establishing the growth of HgI2 crystals, such as, (a) physical vapor transport (PVT) and vapor growth of HgI_2 precipitated from KI and acetone solution and (b) using dimethylsulfoxide solution as a complexion agent. The results suggest PVT to be the technique more suitable for HgI_2 crystals. However, further studies should be carried out to certify this statement. The Bridgman technique was effective to grow TlBr crystals. However, problems still exist in both crystals, used as room temperature semiconductor detectors, due to the low charge carrier collection efficiency, what is being investigated. The energy resolution of these detectors has been, currently, limited by incomplete charge collection. Further improvements of the detector performance will be achieved investigating the structural and surface properties, optimizing the measurement conditions and reducing the electronic noise.

Development of an automation system for ^{125}I brachytherapy seed encapsulated by (ND:YAG) laser welding

The aim of this work is to develop an automation system for Iodine-125 radioactive seed production by (Nd:YAG) laser welding, which has been used successfully in low dose rate (LDR) brachytherapy treatment. This small seed consists of a welded titanium capsule, with 0.8 mm in diameter and 4.5 mm in length, containing Iodine-125 adsorbed onto a silver rod. The Iodine-125 seeds are implanted into the human prostate to irradiate the tumor for cancer treatment. Nowadays, the IPEN-CNEN/SP imports and distributes 36,000 Iodine-125 seeds per year, for the clinics and hospitals in the country. However, the Brazilian market potential is now over 8,000 Iodine-125 seeds per month. The local production of these Iodine-125 radioactive sources became a priority for the Institute, in order to reduce the price and the problems of prostate cancer management. It will permit to spread their use to a larger number of patients in Brazil. On the other hand, the industrial automation plays an important role for Iodine-125 seeds in order to increase the productivity, with high quality and assurance, avoiding human factors, implementing and operating with good manufacturing practices (GMP). The technology consists of appliance electronic and electro-mechanical parts and components to control machines and processes. The automation system technology for Iodine-125

seed production developed in this work was mainly assembled employing a Programmable Logic Controller (PLC), a stepper motor, an (Nd:YAG) laser welding machine and a supervisory. The statistical repeatability of correctly encapsulated sealed sources with this automation system is greater than 95%.

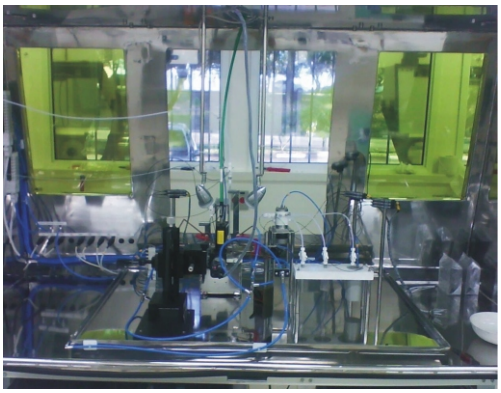


Figure 2. Automation system with electro-mechanical and pneumatic components for Iodine-125 seed production

Radiation-Hard Si Diodes response in radiation processing and clinical beams dosimetry

Epitaxial and FZ silicon diodes processed at the University of Hamburg and Helsinki Institute of Physics has been successfully used as dosimeters in radiation processing, with total doses up to 5 MGy and clinical electron and gamma beams at a total dose of 16 Gy. In this range, the dosimetric response of the diodes is linear.

Measurements of electron transport parameters in avalanche regime

The goal of this work is to measure the first Townsend coefficient and the electron drift Velocity in gases at high uniform electric fields. The heart of the chamber is an RPC like cell with a bulk aluminum anode and a glass cathode. The signal is readout with a fast digitizing oscilloscope to record the fast signal induced by the electron movement in the thin gas gap. A first set of data with nitrogen and isobutane was collected as part of project FAPESP 02/04697-1 and CNPq 78859/2009-0.

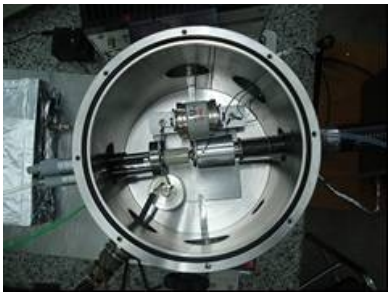


Figure 3. RPC like cell with a bulk aluminum anode and a glass cathode

Use of a low cost pin photodiode for dosimetric purposes in radiation processing

The silicon PIN photodiode (SFH 00206) has been used as a routine dosimeter for irradiation processes performed with a Gamma Cell facility in the. Until now, the diode's response as a function of the gamma-ray doses is linear in the range of 5 Gy to 100 Gy.

Ionizing Radiation Application in Food and Agricultural Products

Irradiated food detection laboratory has been developing different studies in the food irradiation area. These studies embrace many subjects, not only food irradiation detection but also the effects of ionizing radiation on a variety of food stuffs. Works focused on food irradiation detection are based on the application of the microbiological method DEFT/APC and other assays as DNA Comet Assay and germination test. DEFT/APC method and DNA Comet Assay were applied to different minimally processed vegetables. DNA Comet Assay was also used to detect irradiation treatment of soybeans and poultry liver as well as cold chain rupture in food industry control. Moreover, the research also included the detection of genetically modified irradiated and unirradiated food. Several works have been performed to evaluate the effects of ionizing radiation on different kinds of food, such as: meat, through lipid peroxidation analysis in salmon and beef burgers; grains, physical, microbiological and sensorial tests in soybean, peanuts, pistachio; herbs, microbiological and sensorial analysis of medicinal herbs; vegetables, effects of gamma radiation on ready-to-eat vegetables. Besides food analysis, other studies evaluated the decontamination of biological ferment by gamma radiation and the radiation degradation of biological residues (aflatoxins) produced in food laboratories.

During this period, the project (IAEA TC BRA/5/058) related to irradiated mangoes were concluded where a total of 1300 fruits were treated, analyzed and compared. The main activities envolved an international consignment of 600 irradiated mangoes from Brazil to Canada; a regional workshop at Petrolina region where the main mangoes producers of the country are located. The results strongly indicated ionizing radiation as a good alternative for disinfestations purposes.

Studies related to irradiated honey were performed with nutritional, physical-chemical and sensorial evaluations, as well as rheological behavior. Results favorable were obtained with sensory untrained panel and HMF and others relevant contents remained within official limits.

Figure 4. Different honeys irradiated at 5kGy

Considering other applications of irradiation technology, studies on physical-chemical of irradiated tomatoes were realized. Licopene, the most important component of tomatoes, were nutritionally assessed in irradiated tomatoes and also in their sauces. Results showed that irradiation contributed positively.

Figure 5. Study of physical-chemical properties on irradiated tomatoes and its sauces

Industrial and Environmental Application of Ionizing Radiations

Cure of inks, paints, and varnishes by UV/EB technology and evaluation of its degradability

The search for environmentally friendly materials is becoming one of the major focuses of research in the twenty-first century, considering the high level of pollution generated by the inadequate disposal of materials, especially polymers or plastics, in the environment. In addition, environmental legislation already in course in many countries limits the emission of volatile organic compounds (VOC) in the atmosphere. Thus, the technology of curing polymer coatings by radiation is based on the interaction of chemical system with the ultraviolet (UV) or electron beam (EB) incident radiation, forming reactive species capable of inducing polymerization reactions and cross-linking. In this technology, the organic solvents used to reduce the viscosity of the formulations are replaced by reactive monomers that remain in the cured product, providing no VOC emission. The dry/cured film is obtained at room temperature. However, the cured products are insoluble and infusible, increasing the degree of complexity for reprocessing, recycling, and required degradation.

Reduction of environmental impact generated by radiation-cured print inks on post-consuming biodegradable plastic packaging

Research is being done in order to improve the degradability of cured films disposed in the environment. This project aims to evaluate the influence of pro-degrading agents on printing ink formulations, applied on different polymeric substrates or plastic packaging and cured by UV or EB radiation. The degradability of these cured films is being evaluated by the changes in their thermal, mechanical, rheological, and morphological properties during natural weathering and accelerated ageing, as well as biodegradation in simulated soil.

Preservation of cultural heritage by ionizing radiation

Cultural heritage on paper and works of art in general made of organic materials in nature are submitted to a constant process of degradation by ageing through physical, chemical, microbiological, or insect attacks. The use of ionizing radiation aims the disinfestations of works of art and the preservation of artifacts in their original form or in the state in which they are found. The effects of gamma radiation on Brazilian paper and wood based cultural heritage have been investigated concerning some specific characteristics.

The wood is considered a natural composite of extreme complexity, basically composed of cellulose, lignin, hemicelluloses (polyosis) and extractives. Its composition promotes biological attacks from different species. In this context, several techniques have been studies and applied for disinfecting and decontaminating works of art and cultural heritage made of wood, which have been damaged by biodeteriorating organisms (fungi, bacteria, and insects). Gamma radiation is been studied as an alternative to chemical methodologies for wood-made artifacts restoration. By this way, the objective of this project is to evaluate the effect of gamma radiation on some physical-chemical properties of Cedro-rosa and Imbuia wood species. The irradiation has shown itself to be an efficient process to eliminate infestations by both insects and microorganisms, to be fast and not to require quarantine because of the no-generation of toxic residues. Additionally, this process does not protect the irradiated material from re-infestations or re-contamination. In this study, relatively high gamma radiation doses were applied up to 100 kGy so that radiation effects, which are cumulative, could be retrieved. The results showed that gamma radiation, in the studied dose range, does not promote meaningful alterations on the evaluated properties, which allows that artifacts be irradiated multiple times, even if a re-infestation occurs.

Fungi can cause spots or stains on paper and degrade its cellulose fibers affecting paper's integrity. These stains may be due to chemicals produced by fungi in metabolic processes, using cellulose as a nutrient source, and also to the pigmented mycelium and/or spores. Books and documents attacked by fungi and insects have already been treated by radiation for disinfestations purposes. However, there is still need to investigate the influence of radiation on the cellulose paper structure. The aim of this research was to study the effects of radiation on paper properties, especially those related to strength and appearance. Paper sheets for this study were prepared in the laboratory, using bleached eucalyptus pulp as raw material. No additives were used to concentrate the attention only on the effects of irradiation on the pure cellulose matrix. The samples were irradiated at IPEN's ^{60}Co Gammacell irradiator with six radiation doses from 3 to 15kGy, at the dose rate 0.817Gy/s. The properties of paper sheets were tested after irradiation and compared with unirradiated samples according to ISO methods. No significant changes were detected in paper samples irradiated up to 15kGy.

The Institute of Brazilian Studies (IEB) from the University of São Paulo received the charge from São Paulo's Federal Justice to take care of part of the collection from Santos Bank that was severely attacked by insects and micro-organisms (moulds) when stored in a warehouse that was flooded by intense rainfall. A chemical treatment to eliminate the bio-deteriorating agents was tried but it was not effective. As the heap has a large amount of Xylograph wood dye, printings and manuscripts from cordel literature an urgent way to treat was

searched and after the study and previous experience on wood irradiation process it was decided to submit promptly the heap to gamma irradiation. After the treatment the pieces were restored by IEB staff and now are being part of IEB collection and available for the scientific research community.

Modification and preparation of polymeric materials and composites by ionizing radiation

Application on composites based on biodegradable polymers and coconut fiber

The development of polymeric materials that are susceptible to microbiological degradation and have properties similar to the conventional polymers would reduce waste deposit. And, the addition of natural fibers can lead to physical properties improvement and also can reduce cost. Additionally, it will reduce the amount of agribusiness waste disposal in the environment. In Brazil, coconut production is around 1.5 billion fruits by year in a cultivated area of 2.7 million hectares, but the coconut husk fiber has not been used much for industrial applications. Moreover, biodegradable polymers have attracted the attention of the most part of population, due to the environmental issues arising from the increasing use of polymeric materials of low degradability discharged as waste residue. Besides, when considering an application in the medical field, it is necessary that the products are sterilized and, ionizing radiation is widely used to sterilize medical and surgical devices. In this work, it was studied blends and composites based on two commercial polymers: poly (caprolactone), PCL, and poly (lactic acid), PLLA, and coconut fiber. Those polymers are biodegradable as well as biocompatible, so it is important to know the effect of ionizing radiation in these materials. Samples were irradiated with gamma rays from ^{60}Co source and electron beam with radiation doses ranging from 10 kGy up to 1 MGy. The non-irradiated and irradiated samples were studied using several analytical techniques and characterization assays that allowed understanding their properties in order to enable their application as precursors for medical and surgical devices. Thermal processing used to obtain composites and previous acetylation by chemical treatment contributed to the bioburden reduces. Furthermore, reducing initial bioburden it was possible to diminish radiation doses needed to perform sterilization. Enzymatic and soil degradation were not negatively affected by radiation processing. Even though fiber incorporation to the polymer blend slightly reduced degradation process, composites continued degrading through time. Artifacts produced by means of the materials studied here can be radiation processed with doses up to 100 kGy without prejudice of their biodegradability.

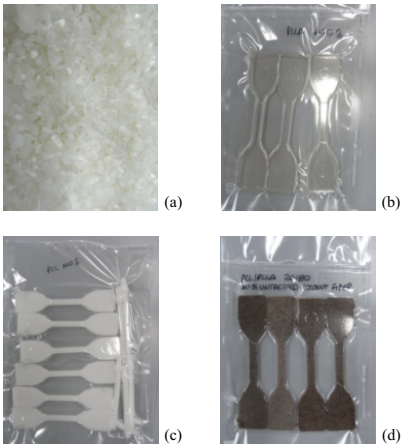


Figure 6. (a) Pellets of PCL:PLLA 20:80 w:w; (b) PLLA; (c) PCL; (d) Composite PCL:PLLA blend and 5% coconut fiber

Utilization of rice husk ash as reinforcement filler for polyamide 6 EB irradiated

New reinforcement fillers like mineral or fiber are developed to improve the dimensional stability, electrical, thermal and chemical resistance, and strength of many kinds of polymers. The aim of this work was to present, dimensional stability, thermal and strength results of the study of amorphous rice husk ash (RHA) like reinforcement filler in a polyamide 6 matrix irradiated by electron beam at different doses and compare it with talc, the most utilized mineral filler by the composes producers. The results showed that the use of the rice husk ash as filler for polyamide 6 composite is technically and economically viable. The irradiation of the studied composite (PA 6 with 30% of rice husk ash) did not provide any improvement for the mechanical and thermal properties previously appraised.

Characterization of crosslinked polyethylene foam by EB irradiation

The foam of polyethylene obtained by crosslinking process by irradiation performs excellent appearance in the surface, which is formed basically by closed cells. The aim of this work was to study the effect of different radiation doses on the low density polyethylene that after irradiation it is thermally expanded for foam obtaining. To certify about the effect of the radiation it was studied the mechanical and thermal properties of the foams. The foams obtained from the crosslinked LDPE by irradiation process (30kGy) presented a smooth and the homogeneous surface and they are formed basically by closed cells.

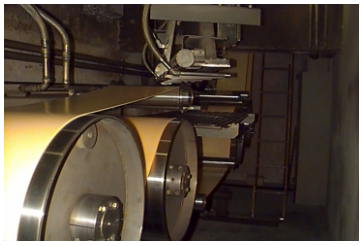


Figure 7. Low density polyethylene foam processed by ionizing radiation

Industrial and Environmental Application of Ionizing Radiations

Polyethylene (terephthalate) - PET recycled post-consumption and of its properties when it is undergone ionizing radiation

The plastic materials have an important role in the life style changing, in the lives of the people and it is more and more utilized in the production and consumption by the world population. By the plastic utilization, it became possible the growth phenomenon of disposable products. As a consequence of this conjecture, the recycling of these materials becomes an obliging option of the modern society. The economy provided by the reutilization of the recycling materials is advantageous not only in consideration to the reutilization of the natural resources involved in these products, but specially for its benefit provided in which concern to the preservation of the environmental matters. The aim of this work was to recycle the PET post-consumption and to evaluate the effects caused by the action of different radiation doses, on the properties of this recycled polymer. The recycling process with super-washing allowed the production of PET post-consumption recycled with lower losses of intrinsic viscosity. It was concluded that the effects caused by the action of the ionizing radiation by mean of the electron beam from electron accelerator and gamma rays emitted by ^{60}Co source in the virgin and recycled PET polymer promoted the predominance of random scission reactions of the main chain, with a consequent decrease in the polymer molecular weight.

The viability of PVC/Al blister reuse and PVC property studies after ionizing radiation processing

The objective of this research was to separate, by means of a process of dissolution, the PVC and the aluminum that compose blister packs, generally used for pharmaceutical pills. It also studied the effect of the ionizing radiation on the PVC, and, finally, the mechanical recycling of the separated PVC, by a process of extrusion. The material it used in this work is the surplus of the pharmaceutical industry, i.e., packs with defects or burrs. It ground the material to facilitate the handling and the homogenization of the system. The system with potassium hydroxide base, concentration of 2M and agitation presented the best relation between time of dissolution and characteristics of the resulting material, without degradation of the PVC. After the dissolution, the samples of the material were submitted to ionizing radiation with doses of 50 kGy, 100 kGy,150 kGy and 200 kGy. In the following, these samples were submitted to traction resistance tests to analyze which modifications the irradiation caused. The last step of the research was the recycling of the PVC separated from the Aluminum. It was made the recycling in industrial equipment, a PVC tube extruder. The material was combined with lubricants, heat stabilizers and pigment in an intensive mixer and processed into the form of rigid PVC electrical conduits. In the irradiated samples, the color of the material changed as well as its extension that was as larger as the radiation dose that they received, indicating the dissociation of the PVC molecules. The extrusion of the PVC was successfully realized: about 200 kg of properly combined were processed. These results presented viability of the whole research.

Electron beam irradiation effect on some properties of aromatic aliphatic copolyester films

Biodegradable plastics and green plastics are the new tendency in the world. The effect of the electron beam irradiation in aromatic aliphatic copolyester and the blend with corn starch films (Ecoflex® and Ecobras®) were studied by tensile strength at break, elongation at break, Scanning Electronic Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FT-IR), Differential Scanning Calorimetry (DSC), crosslinking degree and biodegradability. The Ecobras® material presented crosslinking, when submitted to doses of 10 kGy and 40 kGy. The Ecoflex® material did not present crosslinking when submitted to these doses. The biodegradability of the materials was evaluated by two methods of test: soil simulated and enzymatic. In both methods of assays, the irradiated samples presented faster biodegradation than the references non irradiated.

Rice husk ash utilization as filler in polypropylene matrix and ionization radiation effect on this composite

In the first step of this work, it was evaluated the possibility of using rice husk ash as a filler in polypropylene (PP) making a comparison with talc which is the most used mineral filler in polymers. This comparison was made by using polypropylene with 20% rice husk ash as well as polypropylene with 20% talc measuring their properties. Despite the properties of the PP with 20% rice husk ash decreased compared with the composite of polypropylene with 20% talc. It can be said that the rice husk ash can be used as filler for other utilization less noble of PP. This way it is being given a destination for this residue that it is disposable in the environment contributing to its preservation, moreover reducing the product cost. This work had also as aim to study the ionizing radiation effect in the properties of these composites. It was used the coupling agent, maleic anhydride, to verify a best sample homogenization. According to the results it can be said that PP is a semi crystalline polymer, and so it has its morphology modified when exposed to the irradiation process. This fact is due to the scission mechanisms of the polymeric chains which it is in compliance to the literature.

Comparison of the properties polyamide 6.6 surfaces untreated, treated by plasma and ionizing radiation

The aim of this study was to compare the properties of the polyamide 6.6 surfaces untreated, treated by plasma and ionizing radiation in order to verify if it adheres to the polyacrylic rubber used in seals in

automotive manufacturing. Plasma treatment of polyamide 6.6 was performed using nitrogen gas at a pressure of 1.40 kg/cm^2. The samples irradiation was carried out by electron beam at different doses. After the samples treatment by plasma and by irradiation, they were added to polyacrylic rubber using a hydraulic press. The polyamide 6.6 surfaces were characterized using different techniques. The untreated samples and the irradiated did not adhere to the polyacrylic rubber. The treated samples by plasma adhered to polyacrylic rubber efficiently and presented differences in surface roughness. Also, these samples presented an increase in contact angle when compared with untreated samples.

Development and applications of polymer, matrix composites, and nanocomposites by ionizing radiation

The studies are focused on the development of polymer, matrix composites, and nanocomposites based on natural and synthetic polymers and nanoclays, fibers from the Brazilian biodiversity and wastes arising from Brazil/Uruguay agro-industry, treated by ionizing radiation. The utilization of fiber residues avoids waste through the reuse of materials that would otherwise have been discarded and can bring social and economic benefits for several regions in South America by generating jobs and preventing rural people from having to migrate to the city in search of better conditions of life. CONAP is formed by researchers and students from Radiation Technology Center of IPEN-CNEN/SP, Department of Metallurgy and Materials Engineering of São Paulo University, A. Schulman Brasil-Mash Industry: Technology in Compounds and Masters, the Norte Fluminense State University “Darcy Ribeiro”, and the Technology Laboratory of Uruguay, other universities in South America and recently the starting projects in cooperation with the Center for Advanced Materials (T-CAM) at Tuskegee University, Tuskegee, Alabama, USA. The academic research comprises:

Development of composites and nanocomposites based on thermoplastic polymers, natural biodegradable polymers, Brazilian nanoclays, vegetal fibers from biodiversity and wastes arising from agro-industry

Gelatin/Brazil nut Shell composites processed by casting process and treated by electron-beam irradiation, and Gelatin/Brazil nut Shell/Brazilian clay nanocomposites processed by casting process and treated by electron-beam irradiation.

Figure 8. Gelatin/Brazil nut Shell/Nanoclay

The results showed that the incorporation of those fibers and Brazilian nanoclays to those thermoplastic matrixes represented a gain by up to 200% in tensile strength at break, flexural strength, and flexural module. After electron-beam irradiation treatment, there was an additional gain of around 10 to 30% in those properties, as well as a better interfacial adhesion between the fiber and the polymer matrix.

Ionizing radiation application on renewable energy production

The purpose is the application of ionizing radiation as pretreatment to lignocelluloses conversion in fermentable sugars for ethanol biofuel production. Sugarcane bagasse used has about 42% of cellulose, 31% of hemicelluloses, 20% of lignin, 6% of soluble, and 1% of ash. The moisture maintenance (50%) after irradiation is a positive point for combination with enzymatic or chemical hydrolysis. The main challenge is to obtain the desirable effects applying doses as low as necessary to get some break in the polysaccharides, and at the same time to avoid the glucose loosing due to uncontrolled degradation of polysaccharides.

The obtained results have demonstrated that absorbed doses from 5 to 150 kGy showed to modify the structure and the composition of sugarcane bagasse. The lignin is not degraded completely, but the cellulose and hemicelluloses are cleaved forming cello-oligosaccharides from glucanases and xylanases. About 99% of cellulose and hemicelluloses are converted to oligosaccharides with 70kGy.

The obtained results demonstrate that electron beam treatment enhances the enzymatic hydrolysis of cellulose in SCB. Nowadays the studies are focused on combination of pretreatment technologies, as irradiation with steam explosion or hydrothermal. Combinations of pretreatment technologies are important for economic and feasibility studies, and the purpose is the reduction of processes severity, e.g., decreasing the absorbed dose, the hydrolysis time, as well as, the acidity and enzymes charge. In addition this work will be followed by the use of different complexes of enzymes.

Figure 9. EB-processing of bagasse

Industrial and Environmental Application of Ionizing Radiations

Advanced oxidation processes by ionizing radiation on the treatment of industrial effluent and environmental recovery

The Advanced Oxidation Process by ionizing radiation on removing toxic and refractory pollutants, e.g.,. organic compounds, reactive dyes and pharmaceutical products, in industrial effluent, drinking water, solid wastes, and on destroying pathogenic microorganisms in wastewater and sludge have been studied. These studies are focused on becoming this technology feasible economically for real industrial effluent, which is recalcitrant when treated by conventional methods. Advanced alternative technologies are being developed for effective treatment of herbicide-polluted waters, through the degradation studies of important target molecules such as ametryne methomyl, dimethoate, carbofuran, and methydathion, triazine, thiophos, atrazyne, endosulfan, chlorpyrifos, thriazophos, and trifluralin.

Two reactive dyes as remazol black and remazol orange were also studied. These dyes were selected once cotton is the principal type of fiber to be colored in Brazil and the reactive dyes are suitable for it. More than 10.000 different compounds were commercialized as dyes and pigments. Nearly 12% of processed dyes are lost to aquatic environment, forming mainly three different chemical, such as, sulphatoethylsulphone, vinylsulphone, and hidroxiethyl-sulphone.

The presence of drug residues into waters as environmental contaminants is a new issue and it was selected fluoxetine hydrochloride, and 17α etinilstradiol as pharmaceutical product for radiation degradation and toxicity evaluations.

Besides irradiation application for environmental benefits, two projects were carried out only for water quality and toxicity assessment. They were developed at Jundiai River, Sao Paulo, Br, with the collaboration of Jundiai Water and Wastewater Department. The second study concerned to the effluents and their discharge into Tietê River and with SABESP collaboration. Sediments from Tietê River were submitted to Neutron Activation Analysis in a joint activity with INAA Laboratory of IPEN.

Remediation of soil contaminated with pesticides by treatment with gamma radiation

An understanding of the processes that affect the transport and fate of pesticides is crucial to access their potential for soil and groundwater contamination, and to develop efficient and cost-effective site management and soil remediation strategies. The main objective of this study is the evaluation of the pesticides transferring from contaminated mixture of commercial polymeric packing of high-density polyethylene, HDPE, used in agriculture to soil and their removal by gamma irradiation. Two soil samples of argyles compositions and media composition were exposed to a mixture of commercial polymeric packing contaminated with the pesticides methomyl, dimethoate, carbofuran, methydathion, triazine, thiophos, atrazyne, ametryne, endosulfan, chlorpyrifos, thriazophos and trifluralin. The pesticides leaching from packaging to soil was homogeneous considering a experimental research. The radiation treatment presented high efficiency on removal pesticides from both soil. However it depends on the physical-chemical characteristics of the contaminated soil. The higher efficiency was obtained in soils with higher organic material and humidity. In addition the pesticides removal yield was higher for medium texture soil, than argyles texture soil.

Biological assays for effluent control

In order to measure the efficacy of radiation treatment, luminescent *Vibrio fischeri* bacteria have been used, called as biological assays. Each toxicity assay applies one given specie of living organisms and its sensitivity for contaminants is of concern when applying this type of assessment for effluents treatment. During our researches the priority is given to Cladocera, Luminescent Bacteria and Amphipods when sediments are included for toxicity evaluation.

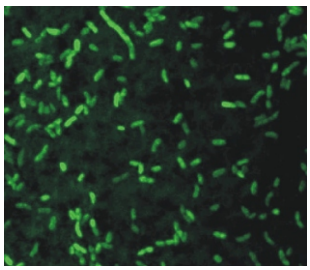


Figure 10. *V. fischeri* bacterial cells as luminescent response to toxins in effluents (toxicity assays)

Gemstones enhancement using gamma radiation

Enhancement services through gamma radiation of colorless quartz and other types of gemstones samples for the companies Murta Gems Trade Gemstones (BH), Stoll Precious Stones of Brazil (RS) and Geoscience Institute of USP. Research of process to induce or enhance the color in several gemstones, and their dosimetry using gamma radiation.

Figure 11. Colored varieties of quartz

Industrial and Environmental Application of Ionizing Radiations

Sealed source production for gammagraphy and industrial process control

Radioactive sealed sources production for radiography and industrial process control. The gamma writing is an important non-destructive technique to analyze metallic components from small to large ones that need high performance and security in operation. Then on-existence of internal failures is checked by gamma rays radiography, because of its great penetration characteristics that allows obtaining the photographic record of failures. This non-destructive analysis is used for quality control of welded components in chemical, nuclear, and mechanical industries, such as pipelines, turbines, reservoirs, and pressure vessels. According to the International Atomic Energy Agency (IAEA) information, the petrochemical and chemical process industries are the mains users and beneficiaries of the radioisotope technology. Radioisotope techniques are very competitive and are largely applied for troubleshooting and process analysis of technically complex, continuously operating industrial plants. Due to this fact, the application of sealed sources becomes more diversified, including for gamma scanning of columns, vessels and pipes, level and interface detection. Since 1983, IPEN-CNEN/SP has supplied industrial gamma sealed sources to more than 25 customers in Brazil and other countries in Latin America and Caribbean. Annually, the laboratory produces 280 sealed sources, with activities ranging from 740 GBq (20 Ci) to 4,444 GBq (120 Ci) of Iridium-192 and from 0.37 GBq (10 mCi) to 18.49 Gbq (500 mCi) of Cobalt-60. The CTR has made 290 inspections in irradiators, command cables and guide pipes annually and also Selenium-75 sources loading services. These supplies allow taking more than 100,000 radiographies per year. The principal CTR's customers are Arctest, ASNDT, Brasitest,CBC, JLM, Confab, Nuclep, TopCheck, Gamatron, Qualitec, Engisa, Capaz, Endlabor, NDT, Sperj, Polyteste, Usiminas, Real WDR, Nuclep, Radiolab,Voigth Hydro, Metaltec, Startec, Accend and Sertech.

Use of radioisotopes as tracers in the environmental and industrial process control

Radioactive tracers, as bromine-82, and dye tracer, as rhodamine WT, are applied to grounded pipe flow rate measurements. Flow rate order of magnitude: 0.1 m^3/s up to 3.5 m^3/s. Radioactive tracer, iodine-131, applications for mean residence time determinations in tanks and digesters of domestic and industrial wastewater treatment plants. Volume order of magnitude: 7,000 m^3 up to 12,000 m^3.

Health Application of Ionizing Radiation and Radioactive Sources

Radiosterilization for tissue banks

In Latin America, the industrial level ionizing radiation sterilization has been used for more than three decades for foods and medical, pharmaceutical and cosmetics products are treated. Later, this activity was extended to the sterilization of human tissues for graft and reinforced in some countries by the technical cooperation and International Agency of Atomic Energy - IAEA financial support.

Brazil was incorporated to this project in 1998 through the Clinical Hospital of Sao Paulo, where the Tissue Bank was installed and the Energetic and Nuclear Research Institute, where the tissues are being irradiated. Since 2009, Brazil has the coordination of IAEA-ARCAL CVIII "Consolidation of Tissue Banks In Latin America And Radiation Sterilization of Tissue Allograft" project with 12 Latin-Americans countries participation. In the last few years, preserved tissue allograft, such as bone, cartilage, skin, amnion and other not viable tissues, have been used in reconstructive surgery by many clinical specialties, like orthopedic and plastic surgery.

The transmission risk of infectious diseases by allograft, however, is a constant concern. To this end, many steps should be taken, including tissue sterilization. This technique is used to minimize the immunogenicity, to kill bacteria and to reduce the contagious diseases transferring risk. As an example, the skin glycerol preservation has a bacteriostatic effect after certain time, on the other hand, skin sterilization by ionizing radiation may reduce the quarantine period for transplantation in patients, and their safety is considered excellent.

The ionizing radiation is a very efficient sterilization technique; nevertheless, its deployment is still contested since there is few data on its effects upon the tissue allograft. At IPEN-CNEN/SP, procedures using two sources of ionizing radiation for sterilization of human skin allograft, and to evaluate the skin after gamma and electron beam irradiation, were established. Besides implanting the irradiation services routine to the tissue banks of the country, the researchers developed irradiation devices for human tissues; implanted dosimetry procedures for irradiation processes control; implanted the quality warranty program for tissue irradiation; optimizing type and dose to be supplied according to the preservation process which the tissue was submitted.

The research group has been collaborating with the implementation of quality systems of the Tissue Banks, as well as with experimental and clinical applications of irradiated tissues. Tissue samples were submitted to 15, 25 and 50 kGy doses and the impact of the irradiation on the mechanical properties was evaluated through the analysis of stress-strain and the morphology was accomplished by and ultra-structure studies, immune histology and others histological tests. Also in the current work, the studies using non destructives tests, like optical coherence tomography, OCT, with the Laser Program collaboration, has been carried out.

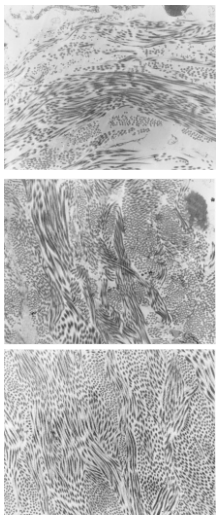


Figure 12. Transmission Electronic Microscopy. Human skin processed in glycerol not irradiated (a); Human skin processed in glycerol gamma irradiated (b) and human skin processed in glycerol submitted to electron beam (c)

Development and production of radioactive sources for brachytherapy application

The number of prostate cancer cases in Brazil is increasing and only a small part of the patients are submitted to brachytherapy treatment using Iodine-125 radioactive seeds. Nowadays, these seeds are imported at a high cost, restricting this application. The local production of these radioactive sources became a priority in order to reduce the problems of prostate cancer management for end users. Such action will permit to spread the use to a larger number of patients. Due to such reasons, the Nuclear Energy Research Institute established a program, in order to produce Iodine-125 radioactive seeds. In brachytherapy, small seeds with Iodine-125 are implanted into the prostate to irradiate the tumor. The Iodine-125 seeds consist of a welded titanium capsule (0.8 mm diameter and 4.5 mm length) containing Iodine-125 adsorbed onto a silver rod. During the project execution, the following methods were developed: the seed core (silver) cutting, the titanium tube cutting, the iodine immobilization through its deposition in silver substrate and the sealing of the seeds through welding process, so that the classification of the seeds, as sealed sources, and the leakage tests can be done according to the international norms. In the moment, the routine production line is settling up. The production line consists in three gloves-boxes. In the first one the Iodine-125 will be adsorbed in the silver core. In the second one, the titanium tube will be sealed. And finally, in the third one, all the assurance tests will be carried out. All the

automation process of the welding glove and the quality control glove are finished.

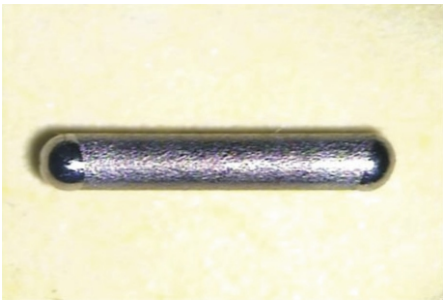


Figure 13. Radioactive seed of Iodine -125

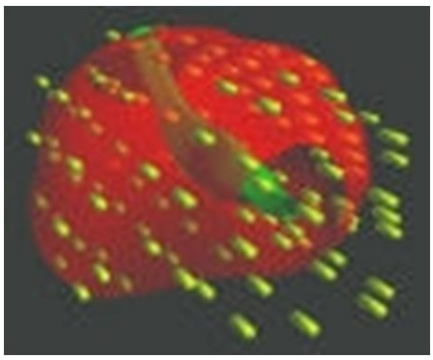


Figure 14. Implanted seeds for prostate cancer treatment

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Honor Mentions and Awards

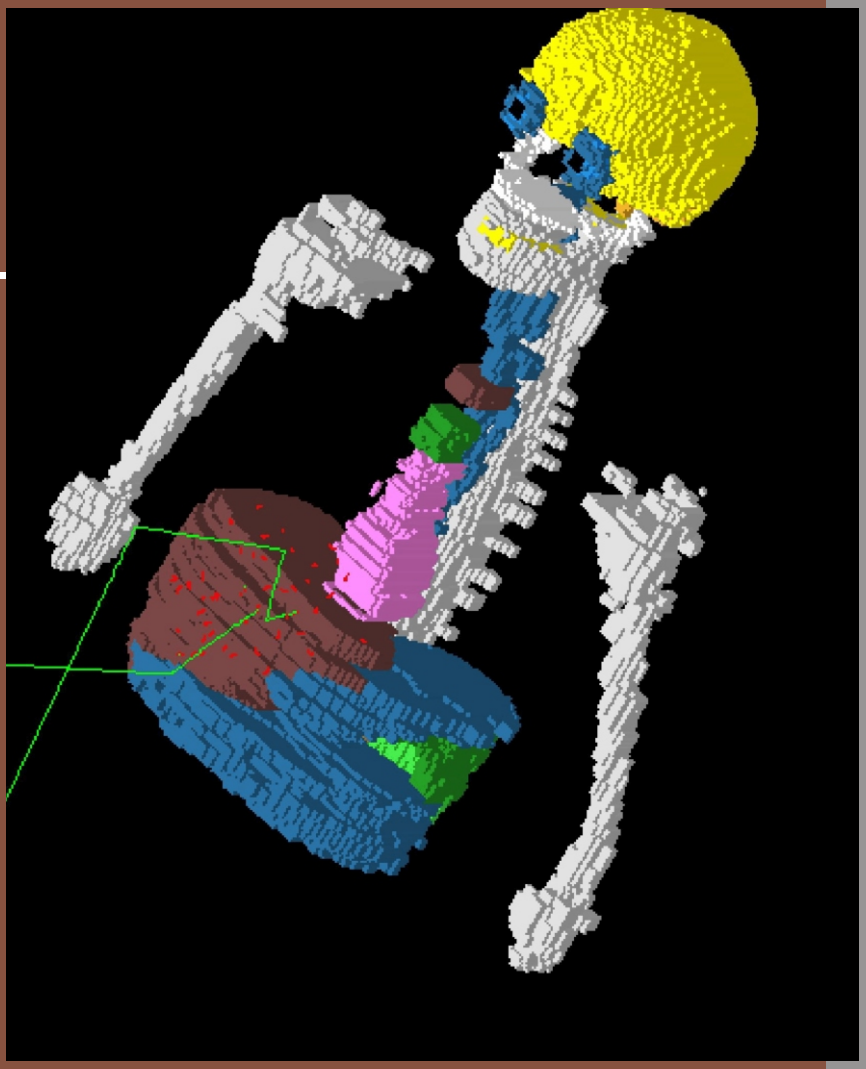
The paper "Study of physical, chemical, and structural effects caused by ionizing radiation and preservation on human costal cartilage" was the best poster winner on the 5th World Congress on Tissue Banking and 12th International Conference of Asia Pacific Association of Surgical Tissue Banks (APASTB), in Kuala Lumpur, Malasia, 2008. The authors are A. C. Martinho Junior, L. D. B. Machado, M. R. Herson e Monica Beatriz Mathor, the research coordinator.

The researcher Luci Diva Brocardo Machado, has received the Honor Member title of the Thermoanalytical Branch of Hungarian Chemical Society, 2008.

The paper "Development and production of radioactive sources used for cancer treatment in Brazil" (NUKLEONICA 2008, 53, Supplement 2), received the Premium Publication of the Year (2009) award of the Latin American Session of American Nuclear Society. The authors are Maria Elisa C. M. Rostelato, Paulo R. Rela, Carlos A. Zeituni, Anselmo Feher, José E. Manzoli, João A. Moura, Eduardo S. Moura and Constância P. G. Silva.

The paper "Effect of gamma irradiation on the vitamin E content and sensory qualities of pecan nuts (*Carya illinoensis*)" won the Panamerican Bimbo 2010, in Nutrition, Science and Technology of Food. The author is Magda Sinigallia Taipina.

Nuclear Science and Technology



Part of the antropomorphic phantom MAX06 (R. Kramer et al.) with activated glass microspheres inside the liver as simulated with the GEANT4 Monte Carlo toolkit

Nuclear Science and Technology

Introduction

The Program on Nuclear Science and Technology comprehends Nuclear and Condensed Matter Physics, Neutron Activation Analysis, Radiation Metrology, Radioprotection and Radioactive Waste Management. These activities are developed at the Research Reactor Center, the Radiation Metrology Center and the Radioactive Waste Management Laboratory. The Radioprotection activities are developed at all radioactive and nuclear facilities of IPEN.

The Research Reactor Center at IPEN is responsible for the operation and maintenance of the research reactor IEA-R1 and has a three-fold mission of promoting basic and applied research in nuclear and neutron related sciences, providing educational opportunities for students in these fields and providing services and applications resulting from the reactor utilization.

Specific research programs include nuclear structure study from beta and gamma decay of radioactive nuclei and nuclear reactions, nuclear and neutron metrology, neutron diffraction and neutron multiple-diffraction study for crystalline and magnetic structure determination, perturbed -angular correlation (PAC) using radioactive nuclear probes to study the nuclear hyperfine interactions in solids and neutron activation analysis, both instrumental as well as involving radiochemical separation applied to the fields of health, agriculture, environment, geology and industry. The research in the areas of applied physics includes neutron radiography, scientific computing and instrumentation.

During the last several years a special effort was made to refurbish the old components and systems of the reactor, particularly those related with the reactor safety improvement, in order to upgrade the reactor power. Primary objective was to modernize the IEA-R1 reactor for safe and sustainable operation to produce primary radioisotopes, such as ^{99}Mo and ^{131}I, among several others, used in nuclear medicine, by operating the reactor at 5 MW on a schedule of 120 hours/week continuous operation. During the period between 2008 and 2010 the power has been raised from 2.0 to 3.5 MW and it is planned to go up to 4.5 MW during 2011.

At the Radiation Metrology Center, all activities of research and development, services, supervision of graduate and undergraduate students and courses performed at the Center are related to the development, improvement and establishment of new methodologies or products in radiation metrology, aiming to assure the safety of IPEN workers, community and environment. Services such as personnel and environmental dosimetry, high dose and accident dosimetry, metrology in diagnostic radiology and radiotherapy, calibration of instruments and radioactivity determination in foodstuffs and food commodities (imported and exported by Brazil) are offered to internal and external communities. The financial resources for research and development are supported by scientific governmental agencies.

Study of the crystalline and magnetic structures of materials by neutron and X-ray diffraction

The IPEN Neutron Diffraction Group is, presently, involved in studies of Rietveld quantitative phase analysis employing both neutron and X-ray diffraction patterns. The X-ray diffraction patterns are measured at different laboratories, inside and outside IPEN. The neutron diffraction patterns are measured in the high-resolution neutron powder diffractometer 'Aurora' installed on the IEA-R1 research reactor at IPEN-CNEN/SP. Although the IEA-R1 is a low-flux reactor, installation of a position sensitive detector (PSD) allows a quite fast measurement of patterns with good resolution. A double-bent silicon monochromator installed in the diffractometer permits measurements with four different wavelengths, namely 1.111, 1.399, 1.667 and 2.191 Å (nominal values). Figure 1 is a photograph of the high-resolution neutron powder diffractometer 'Aurora' installed on the IEA-R1 research reactor.

Figure 1. The neutron powder diffractometer 'Aurora'

Presently, only room-temperature patterns can be measured. In near future, a helium cryostat will be installed allowing measurements close to 4 K and above. 'Aurora' has been designed mainly for crystalline and magnetic structures determination and for application of the Rietveld method in quantitative phase analysis though other different studies can be performed after an analysis of viability. Utilization of this instrument is open for cooperative studies with the latin-american scientific and technological communities.

Hyperfine interactions in solids

Experimental measurements of hyperfine interactions (interactions between the nuclear moments and magnetic field or the electric field gradient) provide a very sensitive and accurate method to investigate condensed matter phenomena in many different solids. A large variety of phenomena in solid materials, in general, originates from small differences in their electronic structure. In this perspective, it is of specific interest to investigate new material and compounds in order to understand the origin of such phenomena from an atomic view. The hyperfine interactions technique involving the measurement of Perturbed gamma-gamma Angular Correlation (PAC) is being used to investigate a series of intermetallic compounds and metal oxides which present interesting properties like superconductivity, magnetic order, phase transitions, etc. Biological materials like proteins and DNA are also a recent subject of investigation. The PAC techniques uses radioactive nuclei implanted in the solids, which can probe magnetic hyperfine field (mhf) and electric field gradient (efg) in determined sites of crystalline structure of the material and provide information about the electronic charge and spin structure around the probe. This information makes possible to investigate properties of the crystal structure and or the origin of magnetic interactions in the material. Due to the proximity of a nuclear research reactor, our laboratory can use a variety of special radioactive probe nuclei such as ^{140}La, ^{111}Ag, ^{111m}Cd which are produced by neutron irradiation in the IEA-R1 research reactor of IPEN, besides the usual ones like ^{111}In and ^{181}Hf. A 4-BaF_2-detector spectrometer setup is available in the laboratory and a 6-detectors spectrometer has been set up which incorporated improvements in the associated electronics in order to maximize the detection efficiency. A methodology using the ^{6}Li ion beam from the Pelletron accelerator in IFUSP to implant ^{111}In probe into the sample through ^{108}Pd(^{6}Li,3n)^{111}In nuclear reaction was developed and it is also available. The compounds which have been investigated are:

1) Metal Oxide: The PAC technique has been used to study the hyperfine interactions in the magnetic and paramagnetic regions of the distorted perovskites RTO_3 where R = rare-earth element and T = Cr, Fe, Co, Mn, using dilute $^{111}In \rightarrow {}^{111}Cd$ and $^{181}Hf \rightarrow {}^{181}Ta$ nuclear probes which were introduced into the samples through a chemical process. The quadrupole interaction parameters as well as the magnetic hyperfine field were obtained for each compound.

2) Diluted Magnetic semiconductors: new families of semiconductors, which are doped with magnetic materials in order to use the electron spin information , are under intensive investigation as they can be used for spintronics. ZnO, SnO_2, TiO_2 doped with Co, Mn, Fe, Ni, Cr and V are being investigated by PAC in order to understand the origin of the magnetism in these compounds.

3) Insulator oxides with large bandgap as HfO_2 and CeO_2 are promising materials to replace SiO_2 as a gate dielectric to prevent leakage current in complementary metal oxide semiconductor (CMOS) transistors. Thin films and nano-structured powders of these materials are under investigation using PAC spectroscopy in order to obtain an atomic scale characterization of their properties under different temperatures.

4) Rare-earth based compounds: series of intermetallic compounds based on rare earth elements show different magnetic behaviors and exhibit very interesting physical phenomena like Fermi liquid behavior, Kondo effect, etc. These properties are not well understood yet, and nuclear techniques are very suitable to investigate the microscopic origin of such phenomena. In our laboratory, we have studied heavy fermions compounds $CeIn_3$, and CeT_2X_2 where (T = Mn, Pd, Rh and X = Ge, Si) with PAC technique using ^{140}Ce and ^{111}Cd probe nuclei. Other families of intermetallic compounds such as RAg , RNiIn and RPdIn where R is a rare earth element are also being investigated. Ab-initio calculations: the hyperfine interaction parameters can be better understood if the electronic structure of the material is known.

5) PAC spectroscopy is also being used to investigate biomolecules of EDTA and DNA molecules of different mouse lineages (A/J, C57BL/6, B6AF1, BXA1 and BXA2) infected by the strain Y of *T. cruzi*. This parasite may cause the Chagas disease when transmitted to humans. The main objective of the present work is to investigate the neighborhood of the sites to which the ^{111}In-^{111}Cd probes are bond in the DNA molecules of the different mouse lineages by measuring nuclear quadrupole interactions in order to compare them and establish which nitrogenated base the probe are bonded to.

6) A very precise ab-inito method of electronic structure calculations based on the density functional theory using a local density approximation is being used to help in the interpretation of hyperfine interaction parameters through the WIEN2k code. The first-principles full potential linear augmented plane-wave (FP-LAPW) calculations of the electronic structure and hyperfine fields have been performed for the intermetallic compounds $CeIn_3$ and $CeMn_2Ge_2$ $CeMn_2Si_2$. A study of the changes induced by the presence of Zn or Ni impurity at Cu site in $CuAlO_2$ delafossite was also carried out by using FP-LAPW calculations. Ab-initio calculations for the series of compounds like RAg and oxides such as HfO_2 and ZnO are also being carried out.

Applied Nuclear Physics, Instrumentation and Scientific Computing

The Group of Applied Nuclear Physics, formed in 2008, performs research mainly in the areas related to development of instrumentation and methods for radiation measurements, scientific computing, measurements and analysis of nuclear data, and application of nuclear techniques in several areas.

Instrumentation

The recent technology of silicon photomultipliers brought new perspectives for compact gamma ray detectors, which can be used in X and gamma ray tomography techniques. New concepts of position sensitive detectors have been studied with the aim of reaching spatial resolution that are adequate to build a positron emission tomography equipment. These studies involve Monte Carlo simulations in parallel with experiments, in order to obtain an instrument with optimized characteristics. A fully digital multiparametric system will be used in the signal acquisition and processing of the several tomograph detectors. Also related to instrument development, a multipurpose ionization chamber was developed in collaboration with the IEE-USP. This chamber has several applications in measurements of scattered radiation from medical equipments. Moreover, a module was developed for checking the gamma-ray coincidences using the pre-amplifier signal as input to quantify the ^{18}F retained in the chimney filters during the FDG production.

Scientific Computing

Computing is a tool as important as experimentation and theory in solving the scientific challenges in the nuclear science of the twenty-first century. Our group has been working with Monte Carlo simulation softwares, mainly Geant4, applied to dosimetry, medical instrumentation and detector studies. We also developed a scientific software applied to Instrumental Neutron Activation Analysis; the software is currently undergoing a complete re-engineering to include k_0 Neutron Activation Analysis methodology. To accomplish this goal, energy efficiency calibration is being incorporated as well as all the other calculations necessary to obtain element concentrations via k_0 technique. The programming language of choice for this software was Python together with Q_t graphics library for the user interface. Two problems were analyzed with Monte Carlo simulations using the GEANT4 package: studies of dosimetry of ^{32}P activated glass microspheres used in brachytherapy, in collaboration with the Center of Materials Technology - IPEN, and simulations of gamma-ray position-sensitive detectors composed by LYSO scintillators coupled to silicon photomultipliers. A software was developed with the aim to help a nonexperienced user to compute the detection efficiency curve and to transfer the efficiency from the point source geometry to other large source geometries. This was built in the Visual Basic platform, and the efficiency transfer method was done with analytical calculus and with numerical simulations using the Monte Carlo method using the GEANT4 tollkit. It was also developed an empirical method for the efficiency determination for large sources. Studies for the implementation of a computer cluster formed by around hundred cores and some GPU units were carried on, and funds were obtained for this implementation in the next years.

Nuclear Data
Nuclear applications often require a good degree of knowledge on several parameters of the nuclei involved, both regarding the safety of the experiment and the reliability of the results. In this sense, the half lives of several radionuclides produced by neutron irradiation (^{193}Os, ^{155}Sm, ^{52}V, ^{101}Mo, ^{101}Tc, ^{127}Te) have been determined with better precision than found in the literature. Also, nuclear reaction cross sections and nuclear structure have been studied using the Pelletron heavy-ion accelerator of USP. Also, parameters related to the k_0 NAA method were studied using a rigorous statistical treatment of the uncertainties involved in the process using both covariance matrices and the least squares method. The aim of this work is to contribute to an enhancement of the international database of k_0 parameters, as well as to reduce the discrepancies found in it. These include the detection-related parameters (detection efficiency and gamma spectrum analysis), the neutron field characterization parameters (α and f), as well as the nuclide-specific ones (k_0 and Q_0).

Methods for Nuclear Radiation Measurements
With the purpose of improving experimental results on nuclear spectroscopy, a thorough analysis of nuclear peak-fitting software has been performed, as well as the development of a method to determine precise gamma transition intensity values from gamma-gamma coincidence and angular correlation experiments. On a different front, viability studies were performed aiming towards the implementation of a PGAA (Prompt Gamma Activation Analysis) facility in the IEA-R1 reactor. This technique is complementary to the regular NAA method and has several applications in the industrial, medical and environmental fields, for instance.

Neutron imaging techniques

The neutron radiography (NR) is a non - destructive testing technique commonly employed to inspect the internal structure of objects. Because of the neutron - matter interaction characteristics this technique is largely employed to inspect hydrogenous rich substances (oil, water, adhesives, rubber, etc) even wrapped by thick metal layers as well as to inspect radioactive objects. The radiography is obtained by irradiating the object in an uniform neutron beam and a converter screen transforms the transmitted neutron intensity into ionizing radiation which is able to sensitize a film forming the image. The screens consist of strongly neutron absorbing elements (gadolinium, dysprosium, lithium) and the films are the conventional for X-ray films and the track- etch foils. Alternatively, neutron scintillators are also used as converter and in this case the light emitted sensitizes either a film or the CCD of a video camera. In the last case the radiography can be obtained in real-time. For both cases the radiographs are 2-D projections of the internal structure of the object. The neutron imaging activities at IPEN - CNEN/SP began in 1988 and the primary objective of the working group was design and construct an operational facility which is installed at the 5 MW IEA-R1 nuclear research reactor. From 1992 to 1997, the group has developed several radiography techniques by employing metallic dysprosium, gadolinium and boron converter screens together with conventional X-ray films and track-etch foils. In 2001 the facility has been improved and a real - time system was installed. Furthermore two new radiography techniques, by using electrons and alpha particles, to inspect objects with thickness in the micra range were also developed. The IPEN posses also a digital system to process images with which has provided services and developed high level researches. In this period (1988-2007) four MSc and three PhD thesis have been advised. The figures 2 and 3 show examples of some typical images obtained in the present facility. Between 2009 and 2010, the working group has installed in the same facility a neutron tomography equipment able to provide 3D images and 3D digital films of the internal structure of the same objects. The figure 4 is an example of 3D images obtained by using this equipment. As has occurred in other countries, the availability of a neutron tomography equipment will spread the use of the neutron imaging techniques in Brazil since this technique reaches the industrial, technological and research fields not reached by the conventional radiography techniques.

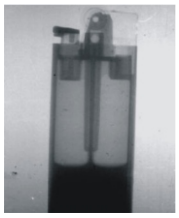


Figure 2. Lighter

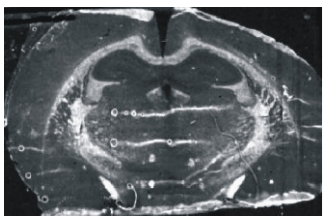


Figure 3. Biological tissue (brain)

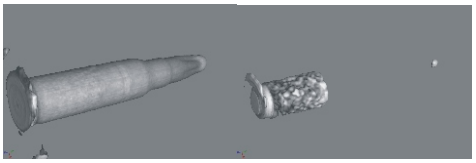


Figure 4. 3D image. Gun bullet (left); powder in detail inside the metallic housing (right)

Radiation spectroscopy and spectrometry radioactive decay

The Radiation Spectroscopy and Spectrometry laboratory (LEER) focuses its work in measurement of radiations, especially beta and gamma transitions and its scope can be divided in three main lines:

Nuclear Data
Using single beta and gamma spectroscopy coupled to coincidence and angular correlation analyses, the group have been measuring nuclear data on nuclei

produced via neutron irradiation in the IEA-R1 reactor, as gamma and beta transition energies, intensities and electromagnetic nature, beta feeding for the excited levels and measuring of thermal neutron cross activation sections and half live of beta decay.

Nuclear Instrumentation and Methodology
The group has been developing methods and methodologies to allow a better and quicker analysis of the experimental data: this includes the development of both data reduction and analysis procedures. Also, preparation of texts for teaching physics as well as didactic proposals involving gamma spectroscopy measurements for high school students.

Gamma Spectrometry Applied to Health and Environment Areas
For clinical analysis biological materials in humans and animal models and obtain reference values for use in diagnosis of different pathologies. These data are relevant to both veterinary medicine and to public health areas.

Highlights 2008-2010
- Analysis of the decay of ^{127}Te isotope using both singles and gamma-gamma coincidence for providing literature information and text nuclear models.
- Beta-decay lifetimes for ^{28}Al, ^{52}V, ^{24}Na, ^{42}K, ^{56}Mn, ^{101}Tc, ^{101}Mo, ^{127}Te, ^{140}La, ^{155}Sm and ^{193}Os (LEER, Pelletron Laboratory/IFUSP and FEI/SP).
- Development and application of software for gamma spectroscopy analyses for high school students: applied at Escola Estadual Professora Maria Aparecida Nigro Gava, São Paulo city.
- The thermal neutron cross section of ^{48}Ca and ^{81}Br using the ko-factors in NAA (LEER and UNESP/ Botucatu).
- Development of methods and software's to extract precise gamma transition properties from both beta and gamma- gamma coincidence and angular correlation data as well as for neutron flux determination (LEER, CDTN and Pelletron/IFUSP).
- Determination of reference values for humans on whole blood and serum for several elements of clinical relevance (Br, Ca, Cl, Fe, K, Mg, Na and Zn) using NAA and EDXRF techniques (LEER and CQMA at IPEN and UNISA).
- Analysis in whole blood of athletes using NAA (LEER and LABEX /UNICAMP).
- Characterization of ions in biological materials (serum, blood, urine and organs) of wistar with Acute Renal Insufficiency using gamma spectrometry (LEER and UNIFESP).
- Analysis of elements in human blood of patients with chronic kidney disease using neutron activation analysis (LEER and CRCN).
- Reference values in blood elements in crioulo breed horses, white rabbits, mice of several species and Golden hamster using NAA and EDXRF techniques (LEER and Butantan Institute).
- Reference values in blood for Elemental analyses mice (NZB , SJL , A/J, I_{max}, H_{III}, BALB/$_c$, C57BL, L_{III} , I_{min}, SJL, B_{10} and AJ) using NAA (LEER and CBT at IPEN and Instituto Butantan).
- Quantitative evaluation of blood elements by NAA in mice immunized with snake venoms: Bothrops jararaca, B. jararacussu, B. alternatus, B. moogeni and B. neuwiedi and a also venom mixture with B.atrox from Rio Negro from Amazonia (LEER and Butantan Institute).
- Plant Nutrient Distribution Analysis using NAA (LEER, FEI and IFUSP).
- Potassium incorporation in fruits of South American tropical species (LEER and LARA/UFF).
- Multielemental Nuclear Analysis of soil reference material (LEER and CDTN).
- Quality control of hyperimmune sera by Chromatography, Instrumental and k_o standardization techniques (LEER, CDTN and Butantan Institute).
- Multielemental analysis of extracts of Achornea Gandulosa, Davilla Elliptica and Davilla Nitida using TXRF and NAA techniques (LEER and UNESP/Botucatu).
- Mineral characterization of the ration managed in the diet of equines used in the antivenom production (LEER and Butantan Institute).

Activities at the Nuclear Metrology Laboratory

In recent years the Nuclear Metrology Laboratory (LMN) has been involved in developing procedures for standardization of important radionuclides applied in nuclear medicine and as reference standards for semiconductor detectors. The primary systems used by LMN for this type of standardization are two 4πβ-γ coincidence systems consisting of a proportional counter, coupled to one or two 3"x 3" NaI(Tl) crystals, and another coincidence system employing a plastic scintillator detector in 4 geometry, called 4π(PS)β-γ. The disintegration rate is obtained by the application of the efficiency extrapolation technique. During the period of 2008-2010, the following radionuclides have been standardized by this technique: ^{51}Cr, ^{57}Co, ^{111}In, ^{177}Lu, and ^{198}Au. The Nuclear Metrology Laboratory participated in the international comparison of ^{177}Lu standardization sponsored by BIPM in 2009. The Nuclear Metrology Laboratory at IPEN has also been involved in the determination of X-ray and gamma ray emission probabilities per decay of ^{51}Cr, ^{177}Lu, ^{198}Au and ^{241}Am. The measurements were carried out by means of HPGe planar and REGe spectrometers, both with a Be window. The development of radioactive water-equivalent solid sources prepared from an aqueous solution of acrylamide have been continued, by using bisacrylamide and ammonium persulphate for its polymerization. The sources have been prepared in cylindrical geometry with ^{133}Ba radioactive solutions with density similar to water as well as good uniformity. A new data acquisition electronic system for 4πβ-γ coincidence

measurements is under development which allows simultaneous recording of pulse height and time of occurrence for each nuclear event. Measurements of ^{60}Co have been performed and compared to the conventional system. As a complementary activity related to radionuclide standardization the LMN has been heavily involved in Monte Carlo simulation of the extrapolation curves obtained in the $4\pi\beta$-γ coincidence technique. For this purpose the response functions of beta and gamma detectors have been calculated by means of two radiation transport codes, namely: MCNP and PENELOPE. These response functions are used as input data for another code developed at the LMN, called ESQUEMA. This code makes use of the Monte Carlo method for simulating all detection processes involved during radionuclide decay, being able to predict the beta and gamma detection spectra, including coincidence events and secondary radiation emission such as conversion electrons, X-ray and Auger electrons. During the period of 2008-2010 this methodology has been successfully applied to achieve primary standardizations of ^{57}Co, ^{22}Na, ^{111}In, ^{177}Lu, and ^{198}Au. Another field where LMN has been involved is neutron measurements. In 2007 activities were started on covariance analysis of the k_0 Nuclear Activation Analysis (NAA) methodology. In this period parameters involved in gamma-ray spectrometry were analyzed. An additional research area has been thermal and resonance neutron cross section measurements. This work has been performed in collaboration with the Institute of Physics from the University of São Paulo. In this period, the determination of the cross section and resonance integral for the ^{165}Ho (n,γ)^{166}Ho nuclear reaction has been completed.

Nutritional Studies In Foodstuffs And Diets

Food safety is a major public concern worldwide. During the last decades, the increasing demand for food safety has been stimulating research regarding the risk associated with consumption of foodstuffs contaminated by pesticides, toxic elements and/or toxins. The necessity of healthy and good quality diets requires the ability to detect the presence of possible contaminants, as well as, nutritional composition of the diets. In terms of health and nutritional safety, to know the levels of nutrients and/or toxic elements consumed by the population through foodstuffs has become of great importance. Neutron Activation Analysis, NAA, has become an important and useful research tool due to the methodology's advantages. These include high accuracy, small quantities of samples and no chemical treatment has been successfully used on a regularly basis in several areas of nutrition and foodstuffs.

Studies carried out in the period 2008-2010:

A Brazilian Total Diet Study: evaluation of essential elements

Total Diet Study (TDS) entailing the analysis of a Market Basket (MB) means the approach adopted worldwide in estimating the daily intakes of analytes of interest by chemical analysis, for a large-scale population over a specific period of time. The MB consists of foods reflecting a defined total diet, based on amounts of food consumed, provided by representative national surveys. The MB involves preparing food in the manner in which they would be usually consumed (table-ready). This approach has been encouraged by the World Health Organization (WHO) due to the fact that dietary habits vary in every country. The MB has been the most adequate method to assess the dietary intakes of nutrients. Although there are many studies in several countries concerning Total Diet Studies, in Brazil they are still scarce. For this study, the methodology for the first Brazilian TDS for the São Paulo State population and its respective MB was developed. This MB corresponds to 72% of the daily food intake for São Paulo state population. This current study involved essential steps to establish a TDS: 1) information about food consumption obtained from the National Household Food Budget Survey "POF 2002-2003" conducted by the Brazilian Institute for Geography and Statistics from July 2002 to June 2003; 2) development of a Market Basket including 71 foods which belong to 30 food groups previously defined. The selection criteria were the foods consumed more than 2g/day/person; 3) sampling in restaurants of the University of São Paulo; 4) kitchen preparation of foods: discarding inedible portions (bone, fruit peels, etc), preparing ready-to-consume foods, individually and mixing foods of the same food group in proportions based on available consumption data. The weights of raw food consumption data were corrected for edible portions and for the ready-to-consume foods. The Instrumental Neutron Activation Analysis methodology was successfully applied to estimate the concentrations of Na, K, Ca, Fe, Zn and Cr in food groups that compose the MB. The contribution of each food group to the total daily intake of these elements was also calculated.

Essential elements in pre-term and term human colostrum

Deficiency of minor and trace elements can lead to various disorders in the early stages of child development. Trace element requirements, during early childhood are more critical due to faster growth rates. Human milk is recommended as the only source of nutrients for infants up to 6 months. In this study, human colostrum samples from two groups of newborns according to their gestational age were studied: a pre-term group and a term group. Samples were collected by manual expression from the first to the fifth day after birth. After sampling, human milk samples were frozen and freeze-dried until the analyses. In this study, Ca, Cl, Fe, K, Mg, Mn, Na, Se and Zn were determined in 15 pre-term colostrum samples and 15 colostrum term milk samples. The methodology applied was the Instrumental Neutron Activation Analysis (INAA).

Essential elements in cow milk and soy-based infant formulas

An infant formula is designed to supply nutrient requirements of neonates during the first months of life. According to Codex Allimentarius, the best substitute choices for maternal milk are infant milk formulas, and when prepared under proper hygienic conditions can be used to feed infants. Numerous infant products have been produced and formulated to meet the nutritional needs of healthy full-term infants. Due to variation in nutrient contents from the food sources used to the formula preparations, specifications of nutrient levels, including mineral elements, have been set to simulate levels of these nutrients similar to human milk. Some commercial infant formulas are deliberately fortified with essential elements such as iron, zinc and copper to ensure that they provide infants nutritional requirements for trace elements. There are three major types of infant formula: cow milk, soy-based and protein hydrolyzed formulas. Instrumental Neutron Activation Analysis (INAA) was applied to quantify the essential elements Ca, Fe, K, Na, Se and Zn in three soy-based formulas and 14 infant formulas based on cow milk, being 2 samples for newborns of high risk and 3 for special requirements.

Toxic and essential element determination in edible mushrooms

Research interest about concentrations of elements in the fruit bodies of especially edible mushrooms has started in the late nineteen sixties and continued until now. Due to its high nutritional value, mushroom cultivation has been a good alternative

to supply protein to countries where the nutritional food value is very low. Due to low sodium concentration in mushrooms, they are also a very good food source for special diets for people with hypertension. This study determined the concentrations of As, Br, Co, Cr, Cs, Fe, K, Na, Rb, Se and Zn present in edible mushrooms acquired from São Paulo city markets or directly from producers of the Mogi das Cruzes, Suzano, Juquitiba and Mirandópolis cities. The obtained results confirm that mushrooms can be considered a good source of K, Fe and Zn. The low Na level is a good nutritional benefit for the consumer.

Determination of toxic elements in octopus samples by INAA

Molluscs of the Class Cephalopoda (squid and octopus) are considered of economic interest as they are a nutritional food source. Octopuses feed up of crustaceans, mollusks, fish and other cephalopods, giving them great potential as bioindicator organisms, providing qualitative information on the contamination of the environment. In Brazil, the lack of data on the sanitary-hygienic conditions makes it essential to carry out studies in this area, in order to ensure the delivery a safe food to consumers. The objective of this study is to determine the concentration of the elements arsenic, selenium and zinc in samples of octopus acquired in different points of the production chain. In the period 2005 to 2007, 121 samples of raw octopus were obtained in the following locations: free fairs, markets/fishmongers, supermarkets, industry and fishing terminals or warehouses in 4 municipalities (Guaruja, Santos, São Vicente and Praia Grande). The samples were dried, ground and homogenized. The methodology for element determination used was Instrumental Neutron Activation Analysis (INAA). Arsenic levels were above the limit of 1.0 mg kg^{-1} (in natura) for foodstuffs established by Brazilian legislation. Se and Zn concentrations were in accordance with literature values. Statistical analyses showed no difference among samples considering “in natura” weight, city or season.

Artificial and natural radionuclides in edible and wild mushrooms

Environmental biomonitoring has demonstrated that organisms such as crustaceans, fish and mushrooms are useful to evaluate and monitor both ecosystem contamination and quality. Particularly, some mushroom species have a high capacity to retain radionuclides and toxic elements from the soil and the air. The potential of mushrooms to accumulate radionuclides in their fruit-bodies has been well recognized. The levels of ^{137}Cs varied from 1.4 to 10.6 Bq kg^{-1}, 40K levels varied from 461 to 1,535 Bq kg^{-1}, ^{232}Th levels varied from 6.2 to 54.2 Bq kg^{-1} and ^{238}U levels varied from 14 to 66 Bq kg^{-1}. The ^{137}Cs levels in Brazilian mushrooms are in accordance with the radioactive fallout in the Southern Hemisphere. The artificial and natural activities determined in this study were found to be below the maximum permissible levels as established by national legislation. Thus, these mushroom species can be normally consumed by the population without any apparent risks to human health.

Another investigation of mushroom biological assimilation of radioactive elements belonging to the natural series of uranium and thorium was conducted on the Poços de Caldas plateau, Minas Gerais State, Brazil. This region has naturally high radioactivity, due to significant anomalies of predominant mineralized uranium and thorium. The soils of this region are also enriched with uranium and thorium when compared to the world average concentration. Several studies have been conducted on the incorporation of radionuclides in agricultural products of the Poços de Caldas plateau. The objective of this study is to determine of natural and artificial radionuclides in wild mushroom samples collected from different points of the Poços de Caldas plateau region.

Assessment of the Content of Mercury, Methylmercury and Other Elements of interest in Fish and School Children Hair from Cananéia and Cubatão coastal cities, São Paulo State, Brazil

The Cananéia-Iguape estuarine-lagoon complex, located in the southern coastal region of Sao Paulo State, is part of the Biosphere Natural Reserve (UNESCO) due to its environmental-cultural importance. It is a region of overall low pollution impact in the southern part of the hydrological system (Cananeia estuary) and is an Environmental Protected Area. Located in the proximities of one of the most important industrial complexes in Sao Paulo state and the most important port of South America, the Santos Estuarine system is highly susceptible to human activities from industry, urban sewage and polluted solid waste disposal. Cubatão city, located in this estuary, was considered as one of the most polluted cities in the world in the recent past. The present study assessed total Hg content, micronutrients (Ca, Fe, K, Na, Se and Zn) and macronutrients (proteins, lipids, ash, energy, carbohydrate) in fish most consumed for the population from Cananéia and Cubatão coastal cities. Furthermore, total and MeHg levels were also determined in hair samples of children from both cities in order to verify bioaccumulation of Hg in this populational group. From these results it was possible to evaluate the nutritional content of the fish consumed and the exposure of the children to Hg and MeHg in these coastal cities, Cananeia and Cubatão.

Environmental applications of neutron activation analysis

Nowadays one of the most dangerous kinds of pollution in the Earth's ecosystem is resulting from heavy metals dumping. Its increasing use in industries and other activities considered to be

essential in modern human life, has resulted in a modification of natural geochemistry cycle of these elements, increasing their dispersion in the environment. Pollution studies require highly sensitive analytical techniques, with high precision and accuracy. Instrumental neutron activation analysis (INAA) has been used for the determination of heavy metals and other trace elements in different environmental samples.

Sediments

The study of the distribution of metals in sediments is very important from the point of view of environmental pollution. The sediment concentrates metals in aquatic systems, and represents a relevant contamination monitor. Studies of sediments from estuaries which have been polluted by heavy metals represent the comprehension of transportation phenomena in these complex ecosystems and the discovery of the pollution history. The project "Toxic metal and trace elements assessment in sediments from Guarapiranga and Rio Grande reservoirs, Metropolitan region of São Paulo," was developed. These reservoirs are important and supply water for a greater part of São Paulo metropolitan region. The contamination of Rio Grande tributary, Billings reservoir, and Guarapiranga reservoir, by determining metal concentration and other elements of interest using three analytical techniques (INAA, AAS and ICP OES) were assessed in bottom sediment samples. The content of total and organic mercury in the sediments from Rio Grande tributary was evaluated. For this purpose an analytical methodology for Hg was developed and validated. The chosen chemical parameters for sediment characterization were Al, As, Ba, Cd, Cu, Cr, Fe, Pb, Mn, Hg, Ni, Se and Zn. The concentration values obtained for the metals As, Cd, Cu, Cr, Hg, Ni, Pb and Zn were compared to the Canadian Council of Minister of the Environment (CCME) oriented values (TEL and PEL values). The contamination of two estuarine systems: a lagoon-estuary complex area of Cananéia and Santos-São Vicente, located in the coast of São Paulo State was evaluated as well. Cananéia is considered as part of Biosphere Natural Reserve due to its environmental and cultural importance and is considered not polluted. Santos - São Vicente estuary is an example of environmental degradation in coastal systems of industrial origin. The assessment concerning the distribution of some major (Fe, K and Na), trace (As, Ba, Br, Co, Cr, Cs, Hf, Hg, Rb, Sb, Sc, Ta, Tb, Th, U and Zn) and rare earth (Ce, Eu, La, Lu, Nd, Sm, Tb and Yb) elements in sediment samples was done by using INAA technique. Also AAS and ICP OES analytical techniques were applied in order to assess toxic metals in the sediment samples. Fifty bottom sediment samples were collected in each estuary in four campaigns: summer and winter of 2005 and 2006.

Soils

The urban environment quality is of vital importance as the majority of people now live in cities. Metals occur naturally in soil, but contents are generally increased in the urban environment due to anthropogenic activities. The platinum group elements Pt, Pd and Rh are the active components of car catalysts and are being spread into the environment to an as-yet incompletely known extent due to surface abrasion of the catalyst during car operation. São Paulo is a city with 19 millions of inhabitants which shows severe pollutions problems. There has been little research on metal concentration levels in soils of São Paulo. This study presents the results obtained for the concentration levels of potentially toxic elements (As, Ba, Cr, Cu, Pb, Sb and Zn) and platinum group elements in urban soils of São Paulo (green areas, public parks and soils near streets and avenues with high traffic density). The results obtained showed concentration levels of the analyzed elements higher than the values considered as reference values for soils in São Paulo, according to the Environmental Protection Agency of the State of São Paulo guidelines. These results suggest an anthropogenic source and indicate a potential damage to soil quality.

Biomonitoring of marine and atmospheric pollution

Biomonitoring of coastal areas using marine organisms is an attractive approach for the study of pollution caused by anthropic discharges. There are two main types of experiments that are generally used for this purpose: *passive biomonitoring*, in which the native organisms are collected and analyzed and *active biomonitoring*, in which organisms from a pristine area (like a mussel farm) are collected and transplanted to polluted sites. In the first phase of this work, the active biomonitoring approach was used and the marine bivalve *Perna perna*, very abundant in the coast of the State of São Paulo, Brazil, was transplanted from a mussel farm and used for biomonitoring of four sites (Itaipu and Ilha das Palmas, in Santos, and TEBAR oil terminal and Ilha Bela, in São Sebastião), situated in coastal regions close to domestic and/or industrial discharges. Hg, Cd and Pb were determined in the transplanted organisms by AAS and As, Ca, Co, Cr, Fe, Na, Se and Zn were determined by INAA. After the transplant experiments of the organisms to the sites of study, a rise in concentrations was observed for all elements, depending on the season and site of study thus indicating the applicability of the *Perna perna* mussel as biomonitor. It could be observed that the concentrations of As and Se were always above the tolerance limits of the Brazilian legislation (1.0 µg.g-1 for As and 0.30 µg.g-1 for Se), in all sites of study, including the control site. For the potentially toxic elements Cd, Hg, Pb and Zn, the concentrations obtained were always below the maximum limits established by the legislation (1.0

µg.g-1 for Cd, 2.0 µg.g-1 for Pb, 0.5 µg.g-1 for Hg and 50 µg.g-1 for Zn). In the second phase of this work, a passive biomonitoring approach is being applied, and two types of organisms have been chosen for the study: the *Perna perna* mussel, collected in a mussel farm at the Cocanha beach (clean area) and in two sites at Santos Bay (Itaipu and Palmas) and the *Crassostrea brasiliana* oyster, collected at Cananéia (oyster farm) and at Bertioga and Santos Estuary. Inorganic trace elements are being determined : As, Br, Co, Cr, Fe, K, Mg, Mn, Zn and V, by INAA and Cd, Pb and Hg by AAS. Also ecotoxicological tests with the neutral red reagent are being made, in order to evaluate the level of stress of the organisms in the polluted regions. In 2009, this work with passive biomonitoring became part of the IAEA/ARCAL Project: "Regional Programme for the biomonitoring of contaminants in molluscs and fish to ensure seafood safety in Latin America and the Caribbean using nuclear analytical techniques" was started and also the analysis of fish consumed by the population of the city of São Paulo were included in the project. Samples of some of the most consumed fish species by the population (robalo, sardinha, pescada, salmão, corvina, tainha and anchova) were acquired at the CEAGESP, the main food distributor of São Paulo and are being processed for analysis of inorganic trace elements, also by the methods of INAA and AAS. The results obtained will be compared to the values of the Brazilian legislation for food contaminants.

Figure 5. Collection of the haemolymph of oysters for the ecotoxicological test

Production and characterization of biological reference materials

A certified reference material (CRM) is a reference material accompanied by a certificate, whose values are certified by a procedure which establishes its traceability to an accurate realization in which the value is expressed and each certified value is accompanied by an uncertainty at a given level of confidence. Certified reference materials are still not widely used in Brazil and other Latin American countries. The main reason is the high cost of these materials, since most of them are imported. Reference materials are important tools in the quality assurance of analytical results as they are used in method validation, calibration of instruments and in the realization of the traceability of analytical results to stated references. The Neutron Activation Laboratory has been involved in the development of Brazilian biological reference materials, as described below:

- As a contribution to the biomonitoring programs carried out by the Institute, all the steps for the production of a Brazilian mussel reference material using the *Perna perna* species were developed. Besides being used in biomonitoring studies, this mussel species has also economical importance due to its cultivation in aquaculture farms for human consumption. The production of the reference material included sampling, sample pretreatments, freeze-drying, grinding, sieving, homogenization and gamma ray sterilization. Physical and chemical characterization following internationally agreed recommendations were performed, with emphasis on the assessment of the stability of the material, its homogeneity status, residual water content and granulometric characterization. An international collaborative interlaboratorial study was performed for assignment of certified values. Preliminary results (with associated uncertainties) for the concentration of elements such as As, Br, Co, Cr, Fe, K, Na, Se and Zn determined by INAA and Cd, Hg and determined by AAS show that the material is suitable to be used in environmental studies.

Figure 6. *Perna perna* mussel

- The International Atomic Energy Agency (IAEA) has been supporting several projects with the objective of laboratory capability improvement in Latin America. In this context, a new IAEA project ("Improvement of analytical quality through proficiency testing and certification of matrix reference materials using nuclear and related analytical techniques in the Latin American nuclear analytical techniques network"- ARCAL RLA 0214) has started in 2009. In this project, the Neutron Activation Laboratory is responsible for the preparation of a new fish reference material. Whitemouth croake (*Micropogonias furnieri*), also known as *corvina*, was chosen as it is the second fish in production in Brazil and it is widely distributed and consumed in the Latin American countries. For preparation, about 300 kg of fish was collected and only the edible parts were used. After preparation, the fish reference material will be tested for homogeneity and stability and characterized by the ARCAL participating laboratories with experience in this field and possibly by expert laboratories outside Latin America.

Figure 7. *Micropogonias furnieri*, the second fish in production in Brazil

Correlation studies of atmospheric pollution and cardio-respiratory diseases through atmospheric pollutant biomonitoring in São Paulo Metropolitan area

Atmospheric pollution is today one of the many problems facing mankind. This problem affects everything from the natural environment to human health and to climate. As result authorities of all over the world have become very preoccupied with the adverse effects derived from pollution. Health problems due to atmospheric pollution also affect São Paulo city, the capital of the State of São Paulo In order to contribute to effective public polices it is important to study the pollutants, their origins as well as to identify the spatial gradient of air pollution in order to explore its possible association with health effects. In order to evaluate pollution levels in São Paulo Metropolitan region a systematic sampling of *Canoparmelia texana* lichenized fungi species was performed in several subprefectures as well as in a reference region located at the Intervales Park, Atlantic Forest, SP considered clean region. Concentrations of elements were determined by neutron activation analysis. Elements Ca, K, Mg presented concentrations at the percentage levels, Ba, Br, Cd, Cr, Fe, Mn, Na, Rb, V, Zn, La and Ce at the levels of mg kg-1 and the elements As, Co, Cs, Hf, Sb, Sc, Se, Th, Nd, Sm, Eu, Tb, Yb and Lu presented the lowest concentrations at the levels of $\mu g\ kg^{-1}$. The occurrence of Sb can be associated to the emission of plastic material incineration. The elements Cr and Fe also presented quite similar pattern distribution and their origins can be associated to industrial emissions and terrigenous origin, derived from the deposition on lichen of solid particles stirred up by the wind. The elements Zn, Ba and V presented similar distribution in the studied areas and they may be associated to vehicular sources. The origin of Ba can be attributed to the use of diesel as a fuel and V of gasoline. Zn and Mn may be associated to industrial origin and also to brake and tire wear emissions. High concentrations of Ca found in some lichen samples can be associated to cement used in construction of buildings. Besides, high concentration of K was found in lichen from clean region of control site. K is an essential element for lichens and in polluted area this element is present in low level due to the stress caused by pollutants. The mortality rates due to the diseases of circulatory and respiratory systems (ICD I00 to I99 and J0 to J99) for two groups of individuals: children younger than 5 years and adults over 45 years of age and living in the Municipality of São Paulo were obtained from databases maintained by the municipal government on the websites:

http://ww2.prefeitura.sp.gov.br//cgi/deftohtm.exe?secretarias/saude/TABNET/SIM/obito.def
Http://ww2.prefeitura.sp.gov.br/cgi/deftohtm.exe?secretarias/saude/TABNET/POP/pop.def.

Based on slow growth of lichen and its exposure period to pollution we use mortality data for a period of 5 years from 2005 to 2009. The statistical treatment of Pearson's correlation applied to the results of lichen element concentrations and mortality rates indicated significant positive correlation for the elements Co, Mn and Zn for adults. (Partnerships: FMUSP, IBt. Financial Resources Agencies: CNPq and IAEA)

Evaluation of trace elements in human tissues to study the health of the population

With the improvement of analytical techniques and knowledge of the role of trace elements in human organism, the correlation studies between trace elements and their effects have become a challenge of many researchers. The NAA laboratory of IPEN over these years has analyzed different types of human tissues such as bone, teeth, lungs, hair and nails and interesting results have been obtained. In the period of 2008-2010 we focused in the analyses of brain samples of a normal elderly population since little is still known concerning their element level concentration and progress in understanding the role of elements in nervous system diseases has been hampered due to a lack of data of elements in different compartiments of human brains. Thus we decided to investigate trace elements in the hippocampus and frontal cortex tissues. The hippocampus is a major component of the brain and plays an important role in long term memory. It is also one of the first regions of the brain that suffers damage from Alzheimer´s disease. The frontal cortex is a sheet of neural tissue that plays a key role in memory, attention, perceptual awareness, thought, language, and consciousness. Brain samples of an over 50 year old population (mean age of 77.6 ± 8.7; range, 51-95 years) of both genders were provided by the Brain Bank of the Brazilian Aging Study Group (BBBABSG) of the São Paulo University, Medical School. Severity of cognitive impairment was assessed with the Clinical Dementia Rating scale (CDR). A CDR 0 (zero) indicates no cognitive impairment and CDRs of 0.5, 1, 2 and 3 indicate questionable, mild, moderate and severe dementia, respectively. Brain samples of a group of individuals of CDRs 0 (normal) were collected for this study from the

elderly residing in São Paulo city. Slices of brain tissue were dissected from the hippocampus and frontal cortex. Special care was taken to avoid sample contamination with metals during handling. The dissected brain tissues of each area were then homogenized, freeze-dried and analyzed by neutron activation analysis. Concentrations of Br, Fe, K, Na, Rb, Se and Zn were determined. A comparative study based on two different age groups of individuals carried out for the hippocampus and frontal cortex samples indicated that the element concentrations of hippocampus and frontal tissues from group aged 51 to 75 year group did not present significant differences from those found for the 76 to 95 years. Concentrations of results obtained in the hippocampus and frontal cortex tissues when compared in terms of genders showed that only Zn presented significantly higher concentrations in the hippocampus of males than those presented by the female group in the corresponding brain part. On the other hand, in the case of the frontal cortex no significant difference of element concentrations was verified between the genders. The results showed that the distribution of Fe, Se and Zn in brains of normal individuals is heterogeneous. Since certain cerebral diseases affect only small area of the brain it is important to select a defined area for elemental concentration analysis. Our results encourage us to continue research to obtain additional data, which can be used as base line values for normal brains and also to understand the functional activities of the specific brain region with regards to the elemental concentrations. (Partnerships: FMUSP. Financial Resources Agencies: CNPq)

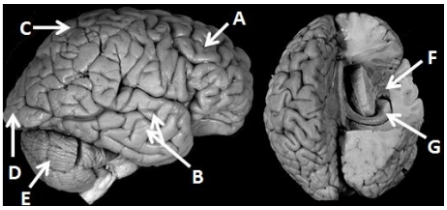


Figure 8. Photo of the brain areas for element composition determination. A. middle frontal gyrus, B. upper and middle frontal gyrus, C. pariental upper lobe, D. occipital lobe, E. cerebellar cortex; F. amygdala and G. Hippocampus

Elemental composition of herbal medicines sold over-the-counter in São Paulo city, Brazil

Medicinal plants have been used to treat diseases for thousands of years. This is true especially in third-world countries where herbal medicine has long played an important role in primary healthcare of the population. In Brazil, the use of herbal medicine is also very popular due to its immense flora, cultural aspects and to the popular belief that herbs, which are of natural origin, are safe and without undesirable side effects. Furthermore, over the last decades, public interest in natural therapies, namely drugs derived from higher plants, mainly the herbal medicines have increased expressively due the high cost of synthetic medications. Besides that they are sold over-the-counter without any prescription. Since the production of medicinal plants in Brazil is increasing and the future of this market is promising, the Regional Council of Medicine of São Paulo State approved the policy of the national medicinal plant drug and herbal medicines. It also created the municipal program for herbal medicine production. In addition, the National Health Surveillance Agency (ANVISA) published a resolution in order to orientate the use of plant based drugs. Within this scenario, it becomes important to evaluate element concentrations in medicinal plant materials since they can be active components or toxic to human health. In this study, neutron activation analysis (NAA) was applied to evaluate the element composition of herbal medicines from the plants (*Ginseng*, *Ginkgo biloba*, *Centella asiatica*, *Mulberry* and *Aloe vera*) from different origins or distributors. The variations of element concentrations obtained in the analyses of herbal medicines from the same plant species can be attributed to the environmental conditions and local soil characteristics where the plant was cultivated, to the part of plant utilized and the age of plant material utilized in its preparations. The high concentrations of Ca and Mg found in these samples can be associated to the absence of collateral effects of herbal medicines. The high concentrations of these two elements prevent stomach lesions. The high concentrations of K found in herbal medicines could be related to the diuretic actions of these products. K is present in natural diuretics, as well as, in drugs used for eliminating phlegm and to invigorate the stomach. In addition, it is known that K salts can regulate body fluids and also participate in cardiac muscle contraction. Toxic elements such as Cd, Cu and Hg were not detected in the samples analyzed and As and Sb were found in some samples but at very low concentrations. On the other hand, herbal medicines contain essential elements such as Ca, Fe, Cr, Co and Zn. Among the herbal medicines analyzed in this study, each capsule of Centella asiatica presented the highest quantities of most elements. The exceptions were found for Na in which the highest quantity of this element was found in Vera aloe. The findings of this study for over-counter herbal medicines are preliminary baseline information about inorganic constituents that can contribute to the understanding of the relationship between elemental contents and their therapeutical effects. (Financial Resources Agencies: CNPq)

Characterization of micronutrients and contaminants in plants grown in contaminated soil

In recent decades, anthropogenic activities, associated with industrial processes and mining, have been the major source of inorganic element enrichment in soils. One example is the need for wastewater treatment technology due to the increase of urban population and industrial development. In such treatment, sewage sludge is generated. This sewage sludge, after a proper microorganism reduction and physical conditioning, is usually known as biosolid. One of the alternatives to the final destination of this waste is the application in agricultural land, as fertilizer. The elements B, Cl, Cu, Fe, Mn, Mo, Ni and Zn are considered micronutrients for plants, but in high concentration in soil solution can reach levels toxic to plants and microorganisms. Other elements such as Ag, As, Be, Cd, Cr, Cu, Pb, Hg, Ni, Sb, Se, Tl and Zn are considered potentially harmful to human health depending on concentrations. Unlike organic contaminants, most inorganic elements do not undergo microbial or chemical degradation therefore their total concentrations remain in soils for a long time after their introduction. Due to the possible presence of these elements at toxic levels to plants, which can reach the food chain, the interest in the development of technologies for remediating contaminated sites has increased. The addition of substances capable of immobilizing toxic elements in the soil is a procedure that has been used for remediating contaminated sites. INAA is an efficient analytical method for monitoring several micronutrients and inorganic contaminants present in geological and biological matrices. This project is being conducted in partnership with CENA/USP. Steps carried out in 2008-2010:

- Determination of Antimony, Arsenic, Cadmium and Thorium in sugar cane juice obtained from cropland treated with sewage sludge. Elemental concentrations for As, Br, Ca, Cd, Co, Cr, Eu, Fe, La, Mg, Mn, Mo, K, Na, Sb, Sc, Sm, Th, Ti, U, V and Zn were determined in biosolid. Analysis of sugar cane juice showed no significant difference in concentration of As, Sb and Th in different doses of biosolid used, with the exception of Cd.

- INAA applied to multielement determination in a variety of lettuce grown in a contaminated soil and treated with phosphate. Super phosphate was used as a substance to immobilize toxic elements in soil. Different doses of super phosphate were added to a number of lettuce plant pots contain contaminated soil. The element concentrations absorbed in the leaves from lettuce treated with phosphate were compared with those absorbed in the leaves of a control plant. The use of 250 mg kg^{-1}(P) proved to be the most effective treatment to reduce the concentrations of Br, Ca, Cd, Cl, Co, Fe, K, Mg, Mn, Sb and Zn in lettuce leaves.

Archaeometric studies in archaeological ceramics using nuclear analytical techniques

The field archaeological chemistry dates to the 1700s, but the Archaeometric Studies Group from IPEN-CNEN/SP involvement began around 1996 with a new research program as a means of characterization of ceramics via INAA.

Studies in ceramics samples can provide information about productions centers, trade route identification, raw material, object exchange, and prehistoric people mobility patterns. This information is possible because differences in chemical composition are typically interpreted as evidence for different production locations. With the elements determined, attention is paid in establishing inter-sample similarity by means of advanced statistical methods like Mahalanobis distance, cluster analysis, principal components analysis, Kernel density, and other as Procrustes analysis, neural network to cite just a few of the statistical methods used.

A typical procedure used in our laboratory consist in cleaning the ceramic's outer surface and drilling using a tungsten carbide rotary file attached to the end of a flexible shaft, variable speed drill. After that, this material is dried in an oven at 105°C for 24h and stored in a desiccator. Approximately 100 mg of ceramic samples, the standard reference material NIST-SRM-1633b, and IAEA-Soil-7 are weighed in polyethylene bags and wrapped in aluminum foil. Groups of 8 to 10 samples and one of each reference material are packed in aluminum foil and irradiated in the research reactor swimming pool, IEA-R1, from IPEN-CNEN/SP at a thermal neutron flux of about 8.92 x 10^{12} $cm^{-2}.s^{-1}$ for 1h. Arsenic, Ba, K, La, Lu, Na, Nd, Sm, and Yb are measured after a 7-day cooling time and Ce, Cr, Cs, Eu, Fe, Hf, Rb, Sb, Sc, Tb, Th, Zn and U after 3 or 4 week's time.

During 2008 to 2010 years, the procedure was used on hundreds of archaeological ceramics specimens in sites from Manaus and Marajo island in collaboration with various archaeologists and geologist.

In Manaus the sites are in the district of Iranduba, 30 km southwest of Manaus, in the region located on the left margin of the Solimões river, next to the merging of the Negro river. The site is formed by a farm which is in a fertile valley in the central Amazon. This region contains both dry and flooded land (swamp). The dry land, which is not inundated by the annual river's flooding, is sustained by sedimentary rocks from the Alter do Chão and Novo Remanso formations from the Cretaceous and Miocene periods, respectively. The swamp area, inundated by annual floods, corresponds with the Quaternary sedimentary deposits from the Solimões and Ariaú river flood plains.

The Marajoara ceramic are from Marajó Island on the Amazon river delta area and are highly elaborated ceramics by means of a process of cultural change that occurred within communities that inhabited the area 3500 years ago. Radiocarbon dates place the period of major growth and expansion of Marajoara culture between the 5th and 14th centuries. The Marajoara style seems to be related to different regions within the Marajoara domain, as well as to different chronological periods. The archaeologists noticed that the urns were buried together with other ceramic objects, such as stools, figurines, miniatures, plates, vases, tangas (pubic covers), and a variety of ornaments, The designs had a symbolic significance of a social or religious character with highly complex ceremonial wares in form and decoration. Decorative techniques involve slip, painting, incision, excision and scraping. The ceramics are tempered with ground potsherds (grog) and two plain types that also define two different types of paste that can be found in all decorative types. Some pottery tempered with crushed ashes of a tree bark known as caraipé (*Licania scabra*) are also found. The use of caraipé as temper material was introduced in the Island AD 500. This material was used in the Amazon Basin towards the end of the first millenium and it is associated with Polychrome Tradition.

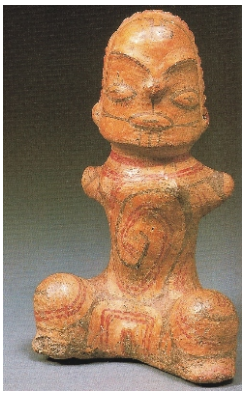


Figure 9. Female figurine

Etnoarchaeological and analytical approaches are tentatively combined in these projects to study cultural practices of clay selection and use. Emphasis to differences in raw material and characteristics resulting from cultural practice is given. One of this research's goals is to detect whether stylistic and morphological boundaries reflected at the aggregate level are also manifested at the compositional level. In the compositional studies, three main objectives guide the examination of compositional variability in raw material and in finished products: 1) to explore whether it is possible to detect chemical compositional differences between two closely situated archaeological sites in a single geological region; 2) to evaluate the closeness of fit between clay compositions from specific sources and products manufacturing; and finally 3) to seek explanation for aspects of observed compositional variability.

Neutron activation analysis and electron spin resonance for fossil samples dating

The Electron Spin Resonance (ESR) dating is based on the fact that ionizing radiation can create stable free radicals in insulating materials, like tooth enamel and bones. The concentration of these radicals - determined by ESR - is a function of the dose deposed in the sample along the years. The accumulated dose of radiation, called Archeologial Dose (AD), is produced by the exposition to environmental radiation provided by U, Th, K and cosmic rays. If the environmental dose rate (D_{an}) in the site where the fossil sample is found is known, it is possible to convert this dose into the age of the sample by the equation: age=AD/D_{an}. The annual dose rate coming from the radioactive elements present in the soil and in the sample itself can be calculated by determining the U, Th and K concentration. Therefore, the determination of the dose rate depends on the concentration of these main radioactive elements. Neutron Activation Analysis has the sensitivity and the accuracy necessary to determine U and Th with this objective. Recently our group studied some samples of pleistocene mammal teeth from Rio Grande do Sul and Stegomastodon teeth from north-eastern Brazilian megafauna.

Determination of rare earth elements, U,Th, and other trace elements in geological samples by neutron activation analysis for geochemical studies

Trace elements, including U, Th, Ba, Rb, Ta, Cs, Co, Hf and rare earth elements (REE), have been widely used in geochemical and petrogenetic studies due to the information they can provide about the formation and weathering of rocks. Instrumental neutron activation analysis (INAA) has been used as a powerful tool in these studies due to the high sensitivity, precision and accuracy in these trace elements determination. INAA provides multielemental analysis in concentrations of about mg kg^{-1} to ng kg^{-1}, without the sample chemical attack.

The Neutron Activation Analysis Laboratory at IPEN (LAN-IPEN) has been working in collaboration with the University of São Paulo (USP) and University of Campinas (UNICAMP), analyzing trace elements in different kinds of rocks by INAA.

The analytical procedure consisted of weighing aliquots of about 100 mg of the powdered rock and of the geological reference material used as standard in polyethylene bags. Samples and reference materials were submitted to a neutron flux of about 10^{12} n cm-2 s^{-1} for 8 hours at the IEA-R1 nuclear reactor at IPEN. The measurements of the induced gamma-ray activity were carried out in

a GX20190 hyperpure Ge detector (Canberra). The multi-channel analyser was a 8192 channel Canberra S-100 plug-in-card in a PC computer. The resolution (FWHM) of the system was 1.90 keV for the 1332 keV gamma-ray of ^{60}Co. The gamma-ray spectra were processed by using the VISPECT gamma-ray software which locates peak positions and calculates the energies and net areas.

Analyses of rocks of the Rio Capim belt, São Francisco Craton, were performed in collaboration with the Geoscience Institute of UNICAMP. The results obtained corroborated to support the proposition that the Rio Capim belt was a Palaeoproteorozoic intra-oceanic arc sequence that collided with a continent, of which the Mesoarchaean Uauá block is a remnant.
In order to obtain additional constraints about the Paraná Magmatic Province basalt genesis, analyses of tholeiitic rocks from flows and sills from Northern region, particularly those from Northern São Paulo, Southern Minas Gerais, and Southern Goiás States were performed in collaboration with the Astronomy, Geophysics and Atmospheric Sciences Institute of the University of São Paulo.

Radiological impact of using phosphogypsum in agriculture and as building material

Phosphogypsum is a by-product of the phosphate fertilizer industry. It is produced by precipitation during wet process of phosphate rocks, thus posing serious problems with its utilization and safe disposal. In Brazil, three main industries are responsible for the production and storage of about $5.5x10^6$ tons per year. Phosphogypsum may contain trace metals and radionuclides of U and Th series. Since, in Brazil, phosphogypsum has been used for many years as soil amendment, it is important to know their availability in the environment. The main objective of this study is to evaluate the radionuclides and metals transfer in the soil-to-plant system. To accomplish this task an experiment was carried out in green house (Figure 10), where two major crop groups (soya bean and corn) and leafy vegetables (lettuce) were grown in two types of soil (clay and sandy) amended with phosphogypsum. The transfer-factors were evaluated for the metals (As, Cd, Cu, Ni and Pb, Ba, Co, Cr, Fe, Zn and REE) and for the radionuclides U, Th, ^{226}Ra, ^{228}Ra, ^{210}Pb and ^{210}Po. The addition of PG to the two soils studied, in the dosage of 1.0 g dm^{-3} for the clayish soil and 0.4 g dm^{-3} for the sandy soil, did not alter significantly the levels of radioactivity, metals and REEs in the final mixture and consequently the TFs obtained for lettuce, soya and corn. Therefore, the impact of one single application of Brazilian PG as soil amendment does not imply in any additional risk due to the transfer of radionuclides, metals and REEs to crops. This project had financial support from CNPq and was conducted in partnership with CDTN.

Phosphogypsum can also be used as building material. In order to evaluate the radiological implications of its use, an experimental house was built, having some of its rooms entirely lined with phosphogypsum. Measurements of samples of phosphogypsum plates from different origins resulted in values of 0.2 to 2.6 for the external radiation index, thus justifying a more detailed investigation. In this study, the application of a previously developed computational model to forecast external doses indoors was described. A comprehensive radiological evaluation was performed, including measurement of the external gamma exposure and radon concentrations in one of the rooms of the house. The results show that the annual increment in the effective dose to an inhabitant of the house will remain below the 1mSv limit for every reasonable scenario. The radon measurements were carried out over a period of 18months, in order to determine the long- term average levels of the indoor radon concentrations. The results obtained are below $200Bqm^{-3}$, the recommended investigation level for radon. The radon exhalation was also evaluated from plates and bricks manufactured with phosphogypsum and from phosphogypsum piles from two fertilizers producers, Ultrafertil, located in Cubatão and Fosfertil, located in Uberaba. This project had financial support from CNPq and FAPESP and was conducted in partnership with Fosfertil.

Figure 10. Lettuce grown in the green house

Assessment of atmospheric pollution in the vicinity of a tin and lead industry using lichen species Canoparmelia texana

This study examines the viability of using Canoparmelia texana lichen species (Figure 11) as a bioindicator of air pollution by radionuclides and rare earth elements (REEs) in the vicinity of a tin and lead industry. The lichen and soil samples were analyzed for uranium, thorium and REEs by instrumental neutron activation analysis. The radionuclides ^{226}Ra, ^{228}Ra and ^{210}Pb were determined either by Gamma-ray spectrometry (GRS) (soils) or by radiochemical separation followed by gross alpha and beta counting using a gas flow proportional counter (lichens). The lichens samples concentrate radionuclides (on the average 25-fold higher than the background for this species) and REEs (on the average 10-fold higher), therefore they can be used as a fingerprint of contamination by the operation of the tin industry. This project had financial support from FAPESP.

Figure 11. Canoparmelia texana lichen

Chemical and radiological characterization of clay minerals used in pharmaceutics and cosmetics

Clays have been used for therapeutic purposes, as active ingredients or as excipient in formulations for a variety of purposes. Despite their wide use, little information is available in literature on their content of trace elements and radionuclides. The

purpose of this study was to determine the elements (As, Ba, Br, Cs, Co, Cr, Eu, Fe, Hf, Hg, La, Lu, Rb, Sb, Sc, Sm, Ta, Tb, Yb, Zn, and Zr) and the radionuclides (U-238, Th-232, Ra-226, Ra-228, Pb-210 and K-40) in Brazilian clays as well as the health and radiological implications of the use of these clays in pharmaceutical formulations.

Radioactive and stable elements' concentration in medicinal plants from Brazil

Since the early days of mankind, plants have been used as food and for medicinal purposes. Still, little information exists in literature about the activity concentration of U-238 and Th-232 decay products, as well as stable element concentrations in Brazilian plants. Activity concentrations of Ra-226, Ra-228 and Pb-210, and chemical concentrations of As, Ba, Br, Cs, Co, Cr, Cu, Eu, Fe, Hf, La, Lu, Rb, Sb, Sc, Sm, Ta, Tb, Yb, Zn and Zr were determined in ten samples commonly used in Brazilian medicinal plants.

Measurement of activity concentration of natural radionuclides for the assessment of radiological indices

The main external source of background radiation exposure of the population are the terrestrial radionuclides like the single occurring radionuclides as ^{40}K and the radionuclides of the ^{238}U and ^{232}Th decay series, with half-lives of the same order that the age of the earth.

The activity concentrations and the gamma-absorbed dose rates of the terrestrial naturally occurring radionuclides ^{226}Ra, ^{232}Th and ^{40}K were determined in commercially-used granites from Paraná state (18 samples) and from Espírito Santo State (6 samples), Brazil, and in superficial sand samples for 16 locations throughout the coast of the Great Victory, metropolitan region of the state of Espirito Santo State. All samples were measured by high resolution gamma spectrometry in triplicates after a 30-days ingrowth period.

For Paraná State granites, preliminary results show activities concentrations varying from 4 ± 1 Bq.kg^{-1} to 79 ± 3 Bq.kg^{-1} for ^{226}Ra, 7 ± 1 Bq.kg^{-1} to 142 ± 6 Bq.kg^{-1} for ^{232}Th and 214 ± 14 Bq.kg^{-1} to 1626 ± 77 Bq.kg^{-1} for ^{40}K. All results are within the range of literature values for similar rocks.

For Espírito Santo State granites, preliminary results show concentrations varying from 31 ± 10 Bq.kg^{-1} to 219 ± 29 Bq.kg^{-1} for ^{232}Th, from 17 ± 2 Bq.kg^{-1} to 270 ± 20 Bq.kg^{-1} for ^{226}Ra and from 498 ± 21 Bq.kg^{-1} to 1481 ± 60 Bq.kg^{-1} for ^{40}K. The southern region of Espírito Santo State shows the highest values for ^{226}Ra, ^{232}Th and ^{40}K. The lowest values of concentration for the same radionuclides were observed for north region.

For Espírito Santo sands, ^{226}Ra concentrations varied from 3 ± 1 Bq.kg^{-1} to 738 ± 38 Bq.kg^{-1}, with the highest values for the central locality of the Camburi beach. ^{232}Th concentrations varied from 7 ± 3 Bq.kg^{-1} to 7422 ± 526 Bq.kg^{-1}, with the highest values for Areia Preta beach. ^{40}K concentrations varied from 14 ± 6 Bq.kg^{-1} to 638 ± 232 Bq.kg^{-1}, with the highest values for Areia Preta beach. Radium equivalent and the external hazard index results showed two distinct groups. In the first one, for the majority of the beaches, the radium equivalent activities are in the range from 15 ± 6 Bq.kg^{-1} to 257 ± 18 Bq.kg^{-1}, below of the lower limit of 370 Bq.kg^{-1} recommended by OECD for the safe use of building materials and external hazard index was below the acceptable limit of 1 suggested by UNSCEAR. In the second one, for Setibinha, Curva da Jurema, South and Central locations of Camburi and Areia Preta beaches, the radium equivalent results are in the range from 818 ± 37 Bq.kg^{-1} to 11228 ± 577 Bq.kg^{-1}, exceeding 2 to 30 times the recommended minimum value for safe application in the civil construction industry and the external hazard index surpassed almost 3 to 40 times the UNSCEAR suggested limit.

Radioactivity in surface, underground and drinking waters of high natural radioactivity regions of Brazil

The levels of gross alpha and beta natural radioactivities in surface, underground and drinking waters consumed by the urban and rural population living in Lagoa Real Uranium Province of central south Bahia state, Brazil were determined in several samples collected both from the dry and rainfall season, from urban public supply of drinking water and, in the rural area, from dug and drilled wells and also from small dams and reservoirs supplied with rainfall. After pre-concentration, the samples were evaporated under an infrared lamp on inox planchets and subsequently counted on a Berthold LB 770 thin end-window low-background proportional counter. Preliminary results show natural radiation levels varying from 0.02 ± 0.001 Bq/L to 0.80 ± 0.04 Bq/L for gross alpha activity and from 0.010 ± 0.006 to 3.0 ± 0.2 Bq/L for gross beta activity. The overall results show that, for the Caetité and Livramento de Nossa Senhora regions there is no significant difference between the rain and dry seasons. Also, for these two regions, all values are within the 2004 WHO recommended levels of 0,5 Bq/L for gross alpha and 1 Bq/L for gross beta activities. Some values of Lagoa Real region exceed the 2004 WHO recommendation levels, however, as we are dealing with natural radiation in a well-known high background radiation area, more studies are needed. For a complete assessment, further uranium concentrations determinations for the same samples will be performed.

Radon and thoron dosimetry with nuclear tracks detectors

Radon, thoron and their progeny are always present in the open atmosphere, but are found in higher concentrations in the mineral waters and mud used for therapeutic and recreation purposes in spas, in confined atmospheres of underground workplaces like natural caves, where tour guides are exposed to these radionuclides, and at facilities dealing with great uranium and thorium concentrations.

Radon and thoron levels at several workplaces are determined using Solid State Nuclear Track Detectors (SSNTD), exposed at least three months over a time period of more than one year. The committed effective doses are evaluated for workers and general public according to the ICRP procedures.

At "Termas de Araxá" spa, the effective committed dose due to ^{222}Rn and ^{212}Pb inhalation was evaluated for workers and patients. Radon measurements were carried out with Makrofol E SSNTD over a period of 21 months. The ^{212}Pb air concentration was assessed through the modified Kusnetz's method. Doses received by the spa workers were below 20 mSv.y^{-1}, suggested by ICRP 60 as an annual effective dose limit for occupational exposure. The radiation doses for the patients were below the mean annual effective dose due to natural sources estimated to be 2.4 mSv.y^{-1}.

The monitoring of radon in natural caves of three touristic parks of São Paulo state, Morro Preto and Santana caves, located at PETAR (Alto Ribeira State Park), Caverna do Diabo, in Jacupiranga State Park and Caverna Colorida, in Intervales State Park. Radon measurements between October 2003 and November 2005 and further between April 2009 and June 2010 were carried out with CR-39 SSNTD. The annual effective dose was calculated considering the most realistic scenario, 52 working weeks and an equilibrium factor of 0.5. Doses received by the tourist guides were below 20 mSv.y^{-1}, suggested by ICRP 60 as an annual effective dose limit for occupational exposure.

At IPEN, São Paulo, Brazil, the nuclear materials storage site deals with considerable uranium and thorium concentrations. The occupational committed effective dose due to ^{222}Rn, received by workers was assessed through the radon activities determined with Makrofol E SSNTD, exposed over a period of 21 months. 13 measurement stations were spread inside the storage site and one was placed at the anteroom used for material transfer. The committed effective dose results varied from 4.1 to 5.4 mSv y^{-1} at the nuclear materials storage site and from 2.9 to 3.7 mSv y^{-1} at the ante-room, according to the working time. All effective committed doses received by workers were below the 20 mSv y^{-1}, suggested as an annual effective dose limit for occupational exposure by ICRP 60.

Determination of natural radionuclides from U-238 and Th-232 series, trace and major elements in sediment cores from Baixada Santista and evaluation of impacted areas

Baixada Santista is the region of higher population of the coast of São Paulo Sate, where is located the largest port and the most important industrial complex of Latin America. As a direct result of the industrial and port activities and the large population growth this region is considered highly impacted because it has received in recent years a considerable load of industrial and domestic effluents. Ten sediment cores, distributed in the ecosystems: estuary of Santos-Cubatão, estuary of São Vicente, channel of Bertioga and Santos Bay (Figure 4) were collected in order to determine the concentration of trace and major elements and natural radionuclides from the ^{238}U and ^{232}Th series using the techniques, neutron activation analysis, X-ray fluorescence and gamma spectrometry. No enrichment was found for the major elements in the ecosystems studied; the highest concentrations of ^{232}Th and ^{238}U were found in ecosystems that are under the influence of the industrial complex. In the four studied ecosystems the most critical elements found and that deserve the attention are the elements As, Br and Sb, because they are enriched in different degrees. Comparing the obtained values of As, Cr and Zn elements in the sediment cores with the values of TEL and PEL index for sediment quality, it was verified that the studied region presents As levels higher than TEL in all the sampled locations; the Cr element presented superiors values than TEL in Santos-Cubatão estuary and Bertioga channel. The Zn element presented values higher than TEL for some core slices of Santos-Cubatão estuary, for one core of São Vicente estuary, one core in Bertioga channel and Santos Bay. Taking under consideration CONAMA nº 344/04 resolution, all the sediments can be considered as class 1, and are below the reference values for the decision to manage the dredged material.

Figure 12. Sampling location in Ilha das Palmas, Santos Bay

Application of nuclear and isotopic tracer techniques to study land-ocean interactions in São Paulo State Coastal Plain and in Bransfield Strait, Antartica

The distribution of natural Ra isotopes was studied in surface, groundwater and estuarine water samples collected from dry and wet seasons (2009-2010) campaigns performed in Ribeira Valley, Southern São Paulo State. Results evidenced that there is a prevalence of Ra-228 isotope in all the set of samples analyzed, although the activity concentrations of Ra isotopes are representative of natural background levels, showing low or minimal human intervention. In the set of samples studied along Ribeira do Iguape River, Cananéia and Iguape outlets, the higher concentrations of Ra were observed in bottom waters, indicating the diffusion of Ra-228 from sediments recently deposited as a potential source of the increased concentrations of this isotope when compared with others. Fluxes of Ra for Cananéia outlet are strongly influenced by tidal oscillations, which modulate the increase and decrease of Ra concentrations in response of the respective waters salinity gradient. In Iguape outlet and in the hydrochemical stations performed along Ribeira do Iguape River it was observed a linear relationship between the amount of suspended matter and the increase of Ra-228 activity concentration. Th-234 was used as a tracer of organic carbon fluxes distribution in the Bransfield Strait in order to evaluate its influence in the CO^2 drawdown, since POC export via sinking particles is the primary mechanism of carbon sequestration in the Southern Ocean (Figure 13). Because of its very particle reactive behavior, Th-234 is removed from a parcel of water in only two ways, through decay and through particle flux. Therefore, a steady-state 1D activity balance can be used to calculate its flux. Fluxes up to 15.274 dmp m^{-2} d^{-1} were estimated, the highest value observed in Station 09 at 794 m depth. POC exported fluxes derived from the disequilibrium Th-234/U-238 model varied from 0.6 to 16,000 mmol C m^{-2} d^{-1}.

Figure 13. Water sampling in Baia do Almirantado

Dosimetric Materials

The main objective of this research area is the development of new dosimetric materials with high sensitivity, low cost and easy obtaining. Single crystals of $CaSO_4$:Tm were obtained under controlled crystal growth conditions, such as temperature, atmosphere and velocity, aiming to improve the TL sensitivity of the crystals and investigate its dosimetric properties in order to evaluate the applicability of this material to dosimetry. The preliminary results show that $CaSO_4$:Tm presents high sensitivity and reproducibility and may be applied to radiation dosimetry.

Other Rare Earth elements, namely, Ce, Nd, Eu and Tb, and the post-transition metal Tl were also assessed as $CaSO_4$ activators, with Ce and Eu being the best candidates for further studies. Both $CaSO_4$:Ce and $CaSO_4$:Eu present high sensitivity, good reproducibility, linear dose-response in the range of 0.01 to 10 Gy and energy independence to photon beams. Preliminary results suggest that $CaSO_4$:Ce response is very dependent on radiation type and energy while $CaSO_4$:Eu is nearly independent of these parameters, but both dosimetric materials present a linear dose-response for high energy electrons in the tested range of 0.01 to 0.50 Gy.

Aluminum oxide composes the modern TL and OSL radiation dosimeters. TL and OSL phenomena are related to inserted impurities on the crystalline structure of α-Al_2O_3. The studies include the dosimetric properties of alumina samples obtained by electrofusion, adsorption and coprecipitation. Electrofused alumina commercially available was used to produce pellets glass mixed and sintered. Adsorption and coprecipitation were the methods used to introduce metal ions to alumina crystal lattice. The alumina pellets produced by means of sintering at high temperature are being characterized.

DL-Alanine ($C_3H_7NO_2$) is an amino acid tissue equivalent traditionally used as standard dosimetric material in EPR dosimetry. Recently it has been studied to be applied in gel dosimetry, considering that the addition of alanine in the Fricke gel solution improves the radiation induced ferric ions production. The spectrophotomety evaluation technique can be used comparing the two spectrum wavelengths bands: 457 nm band that corresponds to ferrous ions and 588 nm band that corresponds to ferric ions concentration to evaluate the dosimetric properties of this material. The performance of the Alanine gel solution developed at IPEN was studied using spectrophotometry technique. The obtained results indicate that signal response dependence for clinical photons and electrons beams for Alanine gel dosimeter is better than 3.6 % (1σ) and the energy dependence response is better 3% (1σ) for both beams. These results indicate that the optical response is energy

independent in the studied dose range and clinical photons and electrons beams energies.

Quartz is a crystalline material abundant in the nature, found with different colors and properties according to the region where it is extracted. This material has been largely studied to be applied as dosimetric material, mainly for retrospective dose evaluation in case of accident dosimetry (Fig. 14). The TL response of green quartz powder samples from Brazil was evaluated to characterize this material to be applied in radiation dosimetry. The evaluation techniques employed were: thermoluminescence, electron spin resonance and spectrophotometry. Different heat treatments pre and post irradiation were evaluated with the purpose of to eliminate the low temperature picks and increase the TL sensitivity. Dosimetric properties such us reproducibility, dose response, dose rate and energy dependent response, TL and EPR signal stability were studied. The obtained results indicate that the green quartz is a promising dosimetric material.

Figure 14. Green quartz samples

External Dosimetry

The main research works developed in the external dosimetry area were:

Development of a passive individual gamma-neutron mixed field dosimeter calibrated to be used in ^{241}AmBe sources. The dosimeter uses the techniques of Solid State Nuclear Track Dosimetry (SSNTD) and Albedo Thermoluminescence (TL) Dosimetry.

The intrinsic efficiency of $CaSO_4$:Dy TL dosimeters, developed and commercialized by IPEN to be applied in individual, area and environmental dosimetry, was evaluated for 4, 6, 8, 12 and 16 MeV clinical electron beams at their depth of maximum ionization in polymethyl methacrylate (PMMA), Solid Water (SW) and standard liquid water phantoms aiming to use this material in personal and radiotherapy dosimetry. The reproducibility of the TL response is better than 2.4% and the TL response as a function of the dose, linear between 0.01 and 3.25 Gy. The obtained intrinsic efficiency indicates that $CaSO_4$:Dy dosimeters can be applied to the radiation protection for high energy electron beams generated by linear accelerators.

Aiming to evaluate the dose range that animals submitted to pulmonary radiographic exams are exposed and the relationship with the individual doses of owners and clinical staff, the entrance surface skin dose of dogs of different breed and sizes with cancer and with suspected pulmonary metastasis were evaluated. TL dosimeters of $CaSO_4$:Dy were used to entrance surface skin dose evaluation of 27 dogs. Simulations of dog's irradiation were also carried out using a water phantom. Each procedure was carried out by the acquisition of three chest radiographic images, two latero-lateral and one ventro-dorsal of dogs with suspect of pulmonary metastasis. The obtained results has shown to be extremely important the assessment of doses involved in veterinary diagnostic radiology procedures both to protect the occupationally exposed workers and to optimize the delivered doses to the animals.

Microdosimetry

The main objective of this research is the dosimetric evaluation of semiconductor components (surface barrier detectors and PIN photodiodes) for applications in microdosimetry and dose equivalent measurements on low dose fields (fast and thermal fluxes) using an AmBe neutron source, the IEA-R1 reactor neutrongraphy facility (epithermal and thermal fluxes) and the Critical Unit facility IPEN/MB-01 (fast fluxes).

As moderator compound to fast neutrons flux from the AmBe source was used paraffin and boron and polyethylene as converter for thermal and fast neutrons measurements. Thermal neutron measurements of AmBe source and Neutrongraphy facility were developed using 10B converter through charged particles detection and for fast neutrons measurements polyethylene converter was used as proton recoil generator. The resulting fluxes were used to the irradiation of two semiconductor components (SSB - Surface Barrier Detector and PIN photodiode).

Monte Carlo simulation methodology was employed to evaluate analytically the optimal paraffin thickness. The obtained results were similar to the experimental data and allowed the evaluation of emerging neutron flux from moderator, as well as the fast neutron flux reaching the polyethylene covering the semiconductor sensitive surface. Gamma radiation levels were evaluated covering the whole detector with cadmium foil 1 mm thick, allowing thermal neutrons blockage and gamma radiation measurements.

The obtained results were in good agreement with other studies published. Using the obtained spectra an approach to dose equivalent calculation was established.

Internal dosimetry

A cross-species extrapolation biodistribution study using ^{177}Lu-DOTATATO has been developed to aid the dose estimation due to peptide receptor radionuclide therapy (PRRT) patients. Besides, this radiopeptide uptake by the Erlich tumor (ascetic form) has been studied as a possibility for test new Somastanine analogs radiopharmaceuticals.

Whole-body retention to workers after accidental inhalation of radionuclides is studied. The counting system used for measuring high-energy gamma emitters comprises two thallium-activated sodium iodide [NaI(Tl)] detectors. The data used in this assessment include whole body and thyroid measurements over specific periods of time. The effective half-lives are obtained to both whole-body and thyroid.

The photon spectra of reference radionuclides are calculated by using the transport code Penelope. The contributions from scattered photons to the spectra fields have been determined with regard to the quantities photon fluence. The mean photon energies calculated with respect to the mentioned quantity are studied. Differences in the design of the sources and their influence on the spectra are assessment. The dependence of the scattered photon component from the energy are examined by feeding the Penelope code with monoenergetic photons of different energies.

As a part of a continuous improvement of the monitoring programme for occupationally exposed workers at IPEN, a computational code for internal dose assessment was developed. The code is an agile and efficient tool for the designing, visualization and resolution of compartmental models of any nature.

The architecture of the system is conceived containing two independent soft wares: CBT (Computer Building Tools) and SSID (Smart Software for Internal Dosimetry). The first one is responsible for the set up and manipulation of models and the SSID is responsible for the mathematical solution of the models. Four different techniques are offered for the resolution of system of equations, including semi-analytical and numerical methods, allowing comparison of precision and performance of both. The software was developed in C# programming, using Microsoft Access database and XML standards for file exchange with other applications. Compartment models for uranium, thorium and iodine were generated for the validation of the CBT software. The models were subsequently solved by SSID software and the results compared with the values published in issues of ICRP.

Metrology in Radiotherapy

In the dosimetry area related to the medical applications of the ionizing radiation were studied:

The TL performance of $CaSO_4$:Dy and LiF:Mg,Ti dosimeters to photon and electron beams applied to radiotherapy was investigated. The TL response of these dosimeters was studied for 6 and 15 MV photons and 4, 6, 9, 12 e 16 MeV electron beams using PMMA, liquid water and Solid Water (SW) phantoms. Using a Varian linear accelerator Clinac 2100C (Fig. 15). The TL dose-response of both dosimeter and three phantoms types present linear behavior on the photon and electron dose range from 0.1 to 5 Gy. The obtained results indicate that the performance of $CaSO_4$:Dy dosimeter is similar to LiF:Mg,Ti dosimeters and this material can be an alternative in clinical electron beams dosimetry.

Figure 15. Clinical electron beam irradiation set up

The complex cancer treatment techniques require rigorous quality control. The Fricke xylenol gel (FXG) dosimeter has been studied to be applied as a tridimensional dosimeter because it is possible to produce 3-D FXG phantoms of various shapes and sizes.

The optical response of the Fricke xylenol gel (FXG) dosimeter developed at IPEN using 270 Bloom gelatine from porcine skin made in Brazil, for clinical electron beams from a VARIAN® electron linear accelerator in energy range from 6 to 16 MeV to reference depth, using a water phantom and OA spectrophotometry technique was evaluated. The optical dose response is presented in Figure 16 to doses ranging from 0 to 40 Gy.

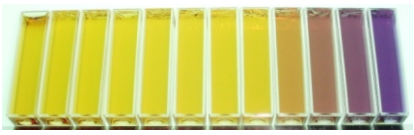


Figure 16. Optical dose responses to gamma radiation, photon and electron clinical beams of FXG solution

The performance of a head phantom filled with FXG solution was also evaluated using RMI technique to 3DCRT with multiple radiation fields and clinical photon beams. The obtained results indicates that the target volume can be clearly observed for all MR images of the FXG phantom irradiated with 6 MV clinical photon beam and, in the case of coronal image, the radiation beam projection and the overlap of different radiation fields used can also be observed. These results

encourage additional tests using complex treatment techniques and they indicate the viability of applying the studied phantom for routine quality control measurements and in 3DCRT and IMRT treatment planning.

$^{90}Sr+^{90}Y$ clinical applicators have to be periodically calibrated, according to international standards and recommendations. Four calibration methods of clinical applicators were studied, comparing the results with those of the calibration certificates. The methods included the use of a standard applicator calibrated by the National Institute of Standards and Technology, and an Amersham applicator, of the Calibration Laboratory (LCI) as reference; a mini-extrapolation chamber developed at LCI; and thermoluminescent dosimetry. The distribution of the depth dose in water, important in dosimetry of clinical applicators, was determined. The results obtained were comparable to the data of the IAEA standard. Furthermore, a dosimetry postal kit was developed for the calibration of clinical applicators using the thermoluminescent technique.

Metrology in Diagnostic Radiology

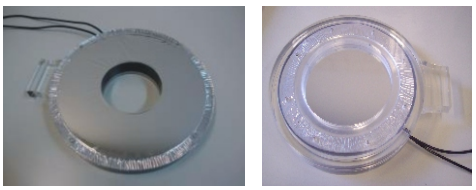
During the period of 2008-2010 new radiation qualities were characterized at the Calibration Laboratory of IPEN, São Paulo, in order to adequate its procedures to the IAEA new code of practice, TRS 457 (Dosimetry in Diagnostic Radiology: An International Code of Practice). A quality control methodology was developed for the instruments used in interventional diagnostic radiology. All X-radiation qualities were established following the recommendations of the IEC 61267 standard. Procedures to calibrate instruments used in conventional diagnostic radiology, computed tomography and mammography measurements were also improved. All these radiation qualities are available to new dosimetric materials irradiation.

The behaviour of the main X-ray equipment of the Calibration Laboratory, that operates in the range from 25 kV to 150 kV, was studied using a non invasive PTW meter, model DiavoltTM, and an ORTEC spectrometry system, model NOMAD-PLUS 92X. To complete the diagnostic radiology radiation qualities establishment, four special phantoms (two for mammography and two for conventional radiology) were developed in order to study the entrance air kerma in diagnostic radiology patients.

The influence factors of the radiopharmaceutical vial dimensions used for activimeter calibration at IPEN was studied with the objective to establish a quality control programme and to determine the correction factors for the geometry of the vials. They are used for distribution of radiopharmaceutical and activimeters calibration, using a NPL-CRC Secondary Standard Radionuclide Calibrator System with traceability to National Physical Laboratory (NPL) and calibrated with a P6 vial type with different dimensions than that of IPEN. The results showed a maximum variation of 22% for ^{201}Tl, and the minimum variation was 2.98% for ^{131}I. The correction factors must be incorporated in the routine calibration of the activimeters.

Also in this research field, some ionization chambers were developed to be used in X-ray beams, diagnostic radiology level. A ring-shaped graphite ionization chamber was designed and assembled to monitor X-ray beam intensity (Figure 17a). International organization protocols recommend the use of monitor chambers to assure the correct dose delivery. This ring-shaped ionization chamber has the advantage over commercial monitor chambers, also called transmission chambers, of not interfering in the radiation beam spectra. All characterization and response stability tests were performed with the ionization chamber, and the results were within the international recommendations. Also, a transmission chamber, with double sensitive volumes, was developed to compare to commercial transmission chambers (Figure 17b). Its response was within international recommendations.

A double faced ionization chamber was built for quality control program in mammography X-ray beams. This ionization chamber was made of PMMA and has two collecting electrodes of different materials: aluminum and graphite. Both sides of this ionization chamber have the sensitive volume of 6.0 cm^3. The double faced ionization chamber was characterized in standard mammography X-ray beams established at the Calibration Laboratory. The behavior of the developed ionization chamber was adequate when compared to international criteria.



(a) (b)

Figure 17. Monitor ionization chambers developed at IPEN

High Doses and Accident Dosimetry

A simple and inexpensive polymeric material whose optical properties can be observed, quantified and related to absorbed dose was studied. Commercial polycarbonate (PC) is a new type of film detector that suffers yellowing upon radiation exposure.The color change (Fig.18) was used as dosimetric property to quantify gamma absorbed dose. The dosimeter consists of a piece of polycarbonate film (3 x 1 cm^2) 3 mm thick and the used evaluation technique was spectrophotometry. PC films were irradiated with gamma doses between 1 and 150 kGy. The optical response was measured using a UV - VIS spectrophotometer. The

main dosimetric characteristics studied were: pre- and post-irradiation stability, dose response, environmental conditions influence and response dependence with dose rate. PC films are easy to prepare and analyze. The influence of environmental conditions was observed and must be corrected. Polycarbonate dosimeters present good stability and reproducibility and linear behavior in the dose range studied indicating that the dosimetric characteristics are suitable to determine high gamma doses.

Figure 14. Green quartz samples

Products and Services

Determination of radionuclides in environmental samples
Radioactivity is measured on a routine basis, by using alpha and gamma spectrometry, gross alpha and beta counting and neutron activation analysis, in order to determine the contents of artificial and natural radionuclides in environmental samples. The following analyses are available:

- Determination of gross alpha and beta activities;
- Determination of natural and artificial gamma emitters;
- Determination of uranium, thorium and radium isotopes;
- Determination of ^{210}Pb and ^{222}Rn;
- Determination of radionuclides in foodstuffs and food commodities imported and exported by Brazil.

High Doses Dosimetry
The High Doses Dosimetry Laboratory of Ipen developed a dosimetric system based on alanina/ESR that presents good characteristics for use in gamma fields such as: wide dose range from 10 to 10^5 Gy, low fading, low uncertainty (<3%), no dose rate dependence and non-destructive ESR signal readout. The detector is encapsulated in special polyethylene tube that reduces the humidity problems and improves the mechanical resistance. A computer program to extract signals from noise spectra based on the wavelet transform was developed in order to allow the use of the dosimetric system at radiotherapy dose ranges. The dosimetric system was validated by IDAS program from IAEA and is available to high doses measurements.

Dosimetric pellets of $CaSO_4$:Dy
The Dosimetric Materials Laboratory developed and patented the $CaSO_4$:Dy crystals growth system and the method to produce the Teflon® sintered pellets. The $CaSO_4$:Dy crystals are growth in the sealed system and they are cold pressed with the binding material to making the 6 mm diameter pellets (Fig.19). The $CaSO_4$:Dy pellets are destined to the individual, area and environmental monitoring through the thermoluminescence dosimetry. Besides they could be applied to the high doses, retrospective and to the clinical dosimetry. They are utilized to solid state dosimetry research too. The pellets of several materials such as quartz, topaz and jade could be also produced by means of the same referred method. The requests of $CaSO_4$:Dy crystals and pellets from many institutions have been attended by the laboratory.

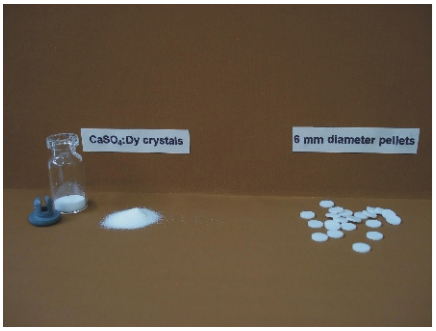


Figure 19. $CaSO_4$:Dy crystals and sintered pellets

Routine External Dosimetry
The external dosimetry applies the thermoluminescent (TL) technique and $CaSO_4$:Dy based dosimeters completely developed at IPEN to carry out individual external exposure, area and environmental monitoring. The individual monitoring service for external exposure is accredited by CNEN (Brazilian Nuclear Energy Commission) regulatory committee, CASEC (Essays and Calibration Services Evaluation Committee) and it completely satisfies CNEN's regulatory norms, besides presenting a brake in IPEN's quality control system, being responsible for the external exposure monitoring of all occupationally exposed individuals at IPEN, other autarchies, governmental offices and even some private facilities. The area and environmental monitoring, which are not under the scope of any regulatory office, are also performed in different society sectors, such as research institutes, universities and private foundations and companies. Aiming to improve individual external exposure, area and environmental monitoring services and give society a return of the budges invested in them, papers based on the results obtained by the services have been present in the most relevant scientific reunions over the triennial period of 2008-2010.

Internal dosimetry
The whole-body and thyroid measurements are routinely performed on IPEN workers, visitors, trainees, and contract workers. During 2008-2010, the Laboratory carried out occupational monitoring of workers involved in radioactive waste management, radioisotope production, research, students and visitors. Routinely the analyses of

biological samples from people occupationally exposed to radionuclides with risk of internal contamination are performed. The most frequently requested radionuclides, were: U-nat, and U isotopes in urine. ^{232}Th, ^{3}H, γ emitters and Actinides, can also be requested. In addition to bioassay measurements, the Laboratory participated in national (Brazilian Intercomparison Programs) and in international intercomparison (Promotion du Controle de Qualité des Analyses de Biologie Medicale en Radiotoxicologie) programs. The dose calculation follows the measurements of the activity in excreta or in body tissues. These calculations are based on the mathematical models recommended by the International Commission on Radiological Protection, and adopted by the Brazilian Nuclear Energy Commission, according to the type of the radionuclide and the practice. No worker received dose higher than the annual limit on intake in the period 2008-2010 for dose assessment, as part of the internal monitoring program.

Calibration of radiation monitors and dosemeters

The Calibration Laboratory has since 1980 been calibrating instruments used in radiation protection and therapy measurements and belonging to hospitals, industries, clinics and other users located in São Paulo and in other parts of Brazil. Since 2000 calibration service is being offered to users of diagnostic radiology instruments with the establishment of standard radiation quality at this level (Fig. 13). At the radiation protection level there are special set-ups with gamma (^{60}Co and ^{137}Cs), beta (^{90}Sr + ^{90}Y, ^{204}Tl and ^{147}Pm), alpha (^{241}Am, ^{233}U, ^{238}Pu, ^{244}Cm, etc.) and low energy X radiations (60 kV). Clinical dosemeters (radiotherapy level) can be calibrated, using gamma (^{60}Co) or low energy X radiation. As reference system, a secondary standard ionization chamber is used, traceable to the Physikalisch-Technische Bundesanstalt, PTB, Germany, and to the National Laboratory of Metrology of Ionizing Radiation, Brazil. Instruments used in diagnostic radiology measurements can be tested in X radiation qualities, using a Seifert X radiation system (160 kV) and a reference system with four ionization chambers for diagnostic radiology measurements (mammography, computed tomography, fluoroscopy, radiation protection and conventional diagnostic radiology ionization chamber) traceable to the PTB, Germany. The types of instruments calibrated are: several kinds of ionization chambers, pen dosemeters, survey meters (including superficial contamination detectors), alarm dosemeters, activimeters, clinical dosemeters and others. Besides this service, samples including thermoluminescent dosimeters, alanina and others, using beta, gamma and X radiation were irradiated.

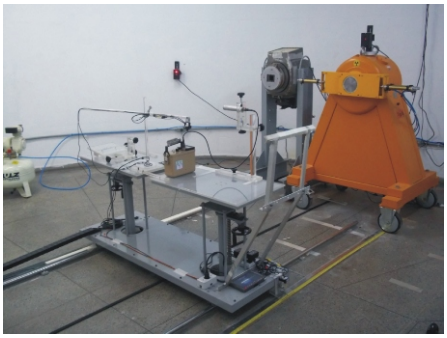


Figure 20. Gamma radiation calibration room general view

Nuclear Science and Technology

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Honor Mention and Awards

Maíra Tiemi Yoshizumi, PhD student, and Dr. Linda Caldas, supervisor, for best oral presentation at the V Congress on Applied Physics to Medicine, on September 2008, with the paper "Performance of a double-faced ionization chamber in standard beta radiation beams".

Luciana Caminha Afonso, PhD student, and Dr. Linda Caldas, supervisor, for best poster entitled "Study of the influence of scattered radiation at a gamma irradiator" presented at the International Youth Nuclear Congress, Interlaken, Switzerland, on September 2008.

Two studies developed in the LEER: "Reference values in whole blood of SJL/J mice using Neutron Activation Analysis" and "Biochemical values in whole blood of horses used for hyperimmune sera production" were awarded during the 9th International Conference on Nuclear Analytical Methods in the Life Sciences. September, 7 to 12.

Two studies developed in the LEER: "Blood Levels of Zinc in crioulo horses used in sera production" and "Sodium analysis in whole blood of athletes using NAA" were selected and awarded with financial support to be presented in the VIII Latin American Symposium on Nuclear Physics and Applications. December, 15 to 19, Santiago.

Materials and Nanotechnology

Glass matrices to contain industrial and radioactive wastes

Materials and Nanotechnology

Introduction

The focus of the Materials and Nanotechnology Program is technology development related to processing, analysis, testing and characterization of materials in general. These are achieved through execution of R&D projects in engineering and materials science, cooperative projects with private and public sector companies, universities and other research institutes. Besides technology development, this Program also fosters training and human resource development in association with the University of São Paulo and many industrial sectors. This Program is divided into sub-programs in broad areas such as ceramic, composite and metallic materials as well as characterization of physical and chemical properties of materials. The sub-programs are further divided into general topics and within each topic, R&D projects.

Ongoing work in the ceramic materials area consists of R&D projects on biomaterials and this includes bio-inert, bio-active or re-absorbable ceramics and new biomedical alloys. Glasses are being developed to immobilize nuclear wastes and to produce microspheres for radiotherapy treatment. Activities related to solid oxide fuel cells are synthesis, processing and production of appropriate materials and testing of unit cells. Advanced ceramic materials with tailored microstructures for specific applications include those based on silicon nitride, silicon carbide, zirconia and alumina. In the area of metallic materials, the activities include development of: (a) sintered components for use as cutting tools; (b) solid hydrogen storage materials based on TiFe intermetallic compounds; (c) batteries and magnets using the hydrogen decrepitation process and the powder metallurgy route. The activities related to Physical and Chemical property characterization were development of: (a) nanostructured coatings of Cr_3C_2-Ni20Cr and WC-Co for increased H.T. degradation resistance; (b) rare earth oxide gel coatings for chromia and alumina forming alloys; (c) nanocrystalline metal-organic chemical vapor deposited films of TiO_2/TiNO, CrN and CrO; (d) self-organized monolayer (SAM) coatings for steel and aluminum alloy automotive components; (e) coated stainless steels and Ti alloys for use as implants; (f) corrosion inhibitors for carbon steels for use as reinforcement in concrete structures. Efforts in terms of materials and technologies development for a sustainable environment were focused on mitigation of environmental impact of solid wastes, increase in energy efficiency, use of green technologies to reuse debris and wastes to produce glass-silicate fibers. A high-pressure cell for use at the Brazilian Synchrotron Light Laboratory and in the IPEN neutron diffractometer has been developed. A carbon and glass fiber composite pipe was constructed for use as lower limb prosthesis to optimize cost and reduce weight. Magnetics hydrogels are being developed for biomedical applications, such as treatment of burns and controlled drug delivery.

Biomedical alloys

Endosseous implant studies in the biomedical area using titanium alloy Ti13Nb13Zr has as its objective the resolution of lifetime related problems in organisms. These studies consist of production of porous metallic biomaterials using powder metallurgical techniques with additives such as gelatin, alginate, starches and albumin, either dry or as a suspension. The titanium substrate with a structurally graded interface and the molecular profile of tissue response with the implant in place were also investigated (Figure 1). The observed structures in the presence of these molecules in the tissue, improved understanding and manage setbacks faced during tissue repair involving implants.

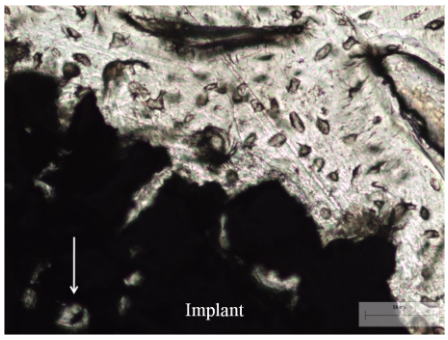
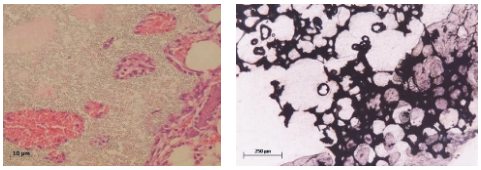


Figure 1. Bone-implant interface. Bright field shows histological slide without staining in which the dark area represents the porous metallic implant. The white arrow indicates the osteocyte inside a pore.

Bioceramics

Calcium phosphate based ceramics, especially tricalcium phosphate and hydroxyapatite, are considered promising materials for bone reconstitution and substitution due to its composition. Calcium phosphate ceramics were obtained in the macroporous form by the direct consolidation method using albumin. The filling of the macropores with new bone tissue resulted in strong interlaced bone-implant and conferred increased strength to the implant (Fig. 2a). This fact is expected to promote cell differentiations and bone formation. Fig. 2b reveals the biphasic ceramic implant (dark region) being invaded by newly formed bone (violet region).



(a) (b)

Figure 2. Histological analysis: (a) osseointegration process in decalcified slide (after 1 week); (b) general view of in-growth of bone, calcified slide (after 4 weeks). (Hematoxilin -Eosin staining)

Properties related to biocompatibility of mixed ceramics ZrO_2-TiO_2 prepared with different rates content were compared by analysis of cultured FMM1 fibroblasts. The specimens were plated on top of disks and counted in SEM micrographs and subsequently analyzed after 1 and 2 days. The cell growth on ZrO_2-TiO_2 samples was similar and significantly higher than those on TiO_2 or ZrO_2 samples. The in vitro experiments showed that sintered ZrO_2-TiO_2 ceramics were biocompatible permitting faster cell growth compared with pure oxide ceramics. Thus, the ZrO_2-TiO_2 sintered ceramics can be considered as a potential implant material.

Silicon nitride based ceramics are promising candidates for biomedical applications due to their chemical and dimensional stability along with adequate mechanical strength and relatively high fracture toughness. The sintered specimens containing La_2O_3 and Al_2O_3 additions had densities higher than 94% of the theoretical density. The bioinert characteristics of silicon nitride ceramics limit its use where the formation of chemical bonds between the material and the tissue are not essential. Samples of silicon nitride were coated with apatite using the biomimetic method. The layer of hydroxyapatite could be deposited by this method on silicon nitride samples surface.

Dental Ceramics

The development of alumina and zirconia based materials, which exhibit aesthetic properties, biocompatibility and good mechanical behavior, enables its use for dental restorations. The incorporation of a vitreous phase in these ceramics is an alternative to minimize ceramic retraction during sintering, besides improving adhesion to resinbased cements, aspects essential to bind dental frameworks to the porcelain cover. Zirconia and alumina based powders were synthesized by the hydroxide coprecipitation route and incorporation of the vitreous phase was done by impregnation of aluminum borosilicate lanthanum powder in sintered ceramics, Fig. 3. Synthesized powders resulted in ceramics with fracture toughness in the range 3.6 - 6.0 $MPa.m^{1/2}$, (comparable to commercially reinforced ceramic materials) and with no citotoxic effects. Bond durability studies between zirconia ceramic and the phosphate resin cement after surface conditioning have been also carried out.

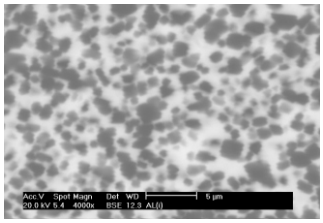


Figure 3. SEM micrograph of alumina ceramic (gray phase) after aluminum-borosilicate lanthanum glass incorporation (white phase)

Ceramic Composites

Ceramic matrix composites have been used as potential materials in load bearing aggressive environments at high temperature. In this context, NbC, TaC and TiC were used as second phase in alumina ceramics to manufacture ceramic nanocomposites for high performance cutting tools. Microcomposites with homogeneous microstructures were produced by adding 1,.5 vol.% NbC, TaC and TiC to the alumina matrix processed by pressure sintering at 1500°C/30min and 20MPa. Nanocomposites Alumina matrix composites with nanometric NbC particles were synthesized by reactive high energy milling with the addition of commercial SiC, ZrO_2 and diamond and studied. The tests simulating thermodynamic conditions of cutting a diffusion pair with gray cast iron were carried out and the composites exhibited good chemical stability upon contact with metal under high pressure and temperature. Based in these results, ceramic cutting tool samples (size 15x15 mm) were prepared and rectified to achieve the geometry of a standard cutting tool to evaluate during machining of hard metals, Figure 4.

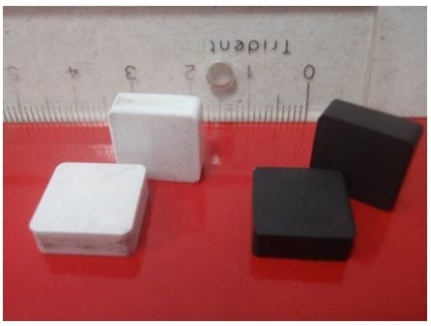


Figure 4. Ceramic cutting tool produced with alumina matrix composites

Ceramic Composites with inorganic polymers additions

Alumina and silicon carbide based composites with polymeric precursors were developed and characterized. Alumina composites were obtained with the addition of active fillers, metallic Ti with two different particle size distributions. These had a homogeneous microstructure and revealed the refractory phases, mullite and TiCN. The preparation of ceramic composites using small amounts of precursor polymers is a simple forming route, with high potential for the production of complex shaped pieces.

Covalent ceramics

Statistical mixture designs are used to systematically study the densification properties of silicon carbide (SiC) ceramics sintered with SiO_2, Dy_2O_3, and Al_2O_3. Mixture models for specific percentage theoretical density and SiC weight loss as a function of the SiO_2, Dy_2O_3, and Al_2O_3 oxide proportions have been determined and validated by analysis of variance. The results indicate a region confined by about 0-20mol% silica, 50-65 mol% dysprosia, and 40-65mol% alumina, with all samples containing 10 vol % of additives, and simultaneous maximization of densities and minimization of weight loss during SiC-based ceramic sintering. Porous structural ceramics based on covalent materials (SiC and Si_3N_4) have been developed, using gelcasting and sacrificial template with different types of starch (rice, potato and corn) as pore forming and consolidation agents. Gelcasting provided porous ceramic samples with the best mechanical properties, where the values obtained were higher than other porous ceramics used commercially, such as alumina and mullite. The porosity of the samples ranged between 25 and 37% and mechanical strength between 100MPa and 240MPa.

The ceramic research group achieved national and international recognition, as shown on the cover of the American Ceramic Bulletin, Fig. 5.

Figure 5. Page in the American Ceramic Bulletin, September 2009

The production of ceramics with special mechanical, electrical, chemical and biological properties involves control of powder characteristics such as chemical composition, crystal structure, particle size, particle morphology, specific surface area and state of agglomeration. In this context, various chemical techniques have been studied at IPEN, to synthesize powders for fuel cell components and biomaterials. Co-precipitation of metal salts followed by hydrothermal and solvothermal treatments, as well as combustion synthesis are the processing routes that were used to synthesize stabilized zirconia, alumina, titania, samaria and gadolinia doped ceria and hydroxyapatite based nanosized powders. Forming processes such as pressing, tape casting and electrophoretic deposition followed by microwave sintering were used.

One of the main outcome are the micrographs of titania nanotubes, prepared by commercial titanium dioxide treatment in sodium solution in a pressured reactor (hydrothermal synthesis), as shown in Figure 6. Among the different oxide nanotubes, worthy of mention is titania, due to its strong oxidizing power, chemical inertness, non-toxicity and unique functional properties that are relevant in terms of applications: photovoltaic cells, gas sensing, photocatalysis, selective adsorption and pollutant elimination. Quite recently, the use of titania nanotubes-based materials has been extended to drug release, based on its excellent biocompatibility and easy surface modification. Hydrothermal synthesis is also a good alternative to synthesize nanocrystalline powders at temperatures as low as 200°C, eliminating thus the calcination step. A TEM micrograph of zirconia powder prepared by this method is shown in Figure 7.

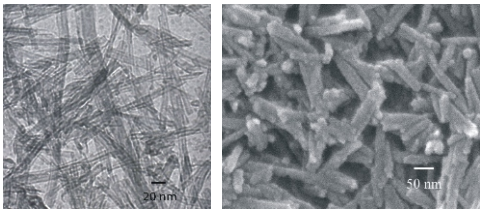


Figure 6. Micrographs obtained by transmission electron microscopy (TEM) and field-emission scanning electron microscopy (FE-SEM) of titania nanotubes

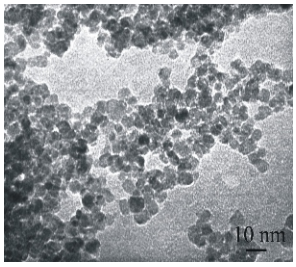


Figure 7. Micrograph obtained by transmission electron microscopy (TEM) of hydrothermally treated stabilized zirconia powders

High temperature degradation resistant coatings

A variety of single rare earth oxide coatings have been developed to improve high temperature oxidation resistance of chromium dioxide and alumina forming alloys. Further increase in high temperature oxidation resistance of these alloys was achieved through optimization of the role of specific rare earths and the use of mixed rare earth oxide coatings.

Corrosion resistant coatings

Cerium based coatings. Cerium based conversion coatings, cerium impregnated boehmite coatings and hydrotalcite coatings were developed to increase the aqueous corrosion resistance of aluminum alloys and specially to protect spent aluminum-clad research reactor fuels during long term wet storage. Hydrotalcite coatings (Fig. 8) modified with cerium was the most efficient coating.

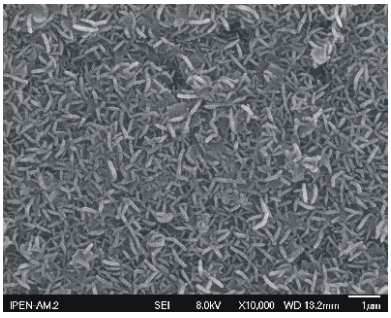
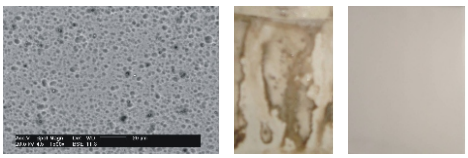


Figure 8. Hydrotalcite layer on aluminum alloy AA 6061 surface

Chromium-free coatings for aluminum alloys

The protective properties provided by various surface treatments to aluminum were evaluated with the aim of replacing coatings based on hexavalent chromium, considered harmful to the environment and to human health. Various treatments that were given to AA 1050 to increase its corrosion resistance were investigated, and included, treatment with self assembling molecules based on diphosphonates (SAM), immersion in boiling water for oxide growth (bohemitization), combination of the two treatments in the sequence (Bohemitization+SAM), passivation in trivalent chromium solution and passivation in hexavalent chromium solution. The corrosion resistance of the various surface treatments was evaluated using techniques, such as salt spray testing according to ASTM B-117, electrochemical measurements and scanning electron microscopy. The results showed that the treatment with self assembling molecules did not protect the AA1050 aluminum for long periods of exposure to NaCl. The SAM treatment after bohemitization produced a surface layer with improved corrosion resistance and showed the importance of an oxy-hydroxide surface layer for adsorption of self-assembling molecules (OS). The passivation treatment based on trivalent chromium led to a surface with corrosion resistance similar to that based on hexavalent chromium. This indicated that the former is a viable alternative to replace hexavalent chromium. Fig. 9 shows the untreated and treated AA 1050 surfaces, prior to and after the salt spray test.

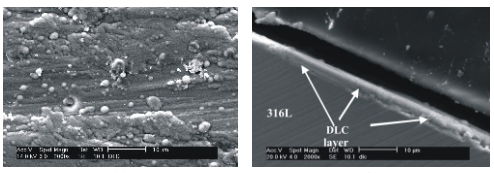


(a) (b) (c)

Figure 9. AA1050 surface (a) with bohemitization followed by SAM treatment; (b) without surface treatment after 148h of salt spray test, and (c) with bohemitization followed by SAM treatment after 148h of salt spray test

Corrosion behavior of biomaterials

Coatings produced by physical vapor deposition (PVD) methods have been applied to 316L stainless steel surfaces to increase its corrosion resistance and biocompatibility. Three thin films were tested: titanium nitride (TiN), titanium carbonitride (TiCN) and diamond-like carbon (DLC). These materials have high hardness, wear resistance and intrinsic biocompatibility, which are key features for biomedical applications. Characterization of the electrochemical behavior of stainless steel coated with the three different films revealed surface defects that decrease the corrosion resistance of the substrate. These PVD coats were non-cytotoxic and had high wear resistance. These properties along with the low cost of stainless steel make coated stainless steel a potential material for biomedical implants. However its corrosion resistance requires further improvement. Fig. 10 shows the DLC coated AISI 316 L stainless steel surface and cross section.



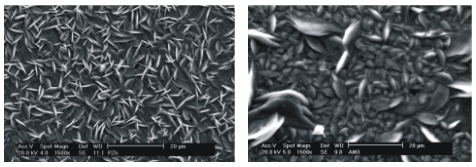
(a) (b)

Figure 10. Surface of AISI 316L stainless steel with DLC coating. (a) top and (b) cross-section.

Surface treatments to protect carbon steels

The viability of replacing nickel in zinc phosphate (PZn) baths with niobates, ammonium niobium oxalate and benzotriazole (BTAH), for phosphating of carbon steels, has been investigated. The effect of ammonium niobium oxalate as a passivant of phosphated steels was also evaluated. The phosphate layer formed in the PZn+Ni solution

consisted of needle like crystals, while that formed in PZn+Nb and PZn+Ox+BTAH solutions consisted of crystal-like grains with improved surface coverage. The phosphate layers obtained in the PZn+Nb and PZn+Ox+BTAH baths were thicker and rougher than the PZn+Ni layer favoring corrosion protection and coating adhesion. The PZn+Nb and PZn+Ox+BTAH layers were also less porous and more corrosion resistant, compared with PZn+Ni. These results helped conclude that Nb could replace Ni in phosphating baths. Fig. 11 shows the zinc phosphate coating from baths containing Ni and Nb.



(a) PZn+Ni (b) PZn+Nb

Figure 11. Zinc phosphate coating obtained in (a) Ni and (b) Nb phosphating containing baths showing different morphologies of phosphate crystals

Electroceramics

Basic research work is devoted to the study of zirconia/ceria based ionic conductors and cerate/zirconate based protonic conductors, particularly for the development of solid oxide fuel cells and oxygen sensors. The synthesis of ceramic nanopowders is carried out by several techniques: coprecipitation, polymeric precursors, combustion and polyacrylamide. The characterization of the powders is carried out by in situ high temperature x-ray diffraction, thermal analysis and Fourier transform infrared absorption spectroscopy. Ceramic pieces are prepared by pressing and sintering. Conventional as well as fast firing and two-step sintering procedures are performed to obtain electroceramics with improved electrical behavior. Electrical measurements consist of impedance spectroscopy measurements in a wide temperature range (LNT-1500K) and oxygen partial pressures (10 ppm - 1 atm). Microstructure evaluations were carried out using x-ray diffraction analysis, scanning and transmission electron microscopy, scanning probe microscopy and Raman spectroscopy.

Soft chemistry techniques have been used to synthesize several oxygen-ion conductors with the aim of improving their microstructure and electrical properties. Pure and Gd-doped $La_2Mo_2O_9$ powders and films were prepared by the polymeric precursor technique. Gadolinium doping was efficient to stabilize the β-$La_2Mo_2O_9$ phase. Homogeneous and crack-free films were obtained using glass, alumina and silicon substrates. Nanostructured and single-phase tetragonal zirconia was prepared by the spray pyrolysis technique (Fig. 12).

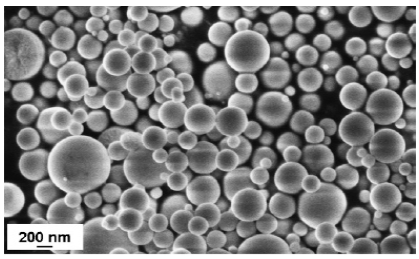


Figure 12. Scanning electron micrograph of tetragonal zirconia powder synthesized by spray pyrolysis

Thermal aging studies revealed that degradation is mainly related to chemical modifications at electrode/electrolyte interfaces and near the grain boundaries. Samaria-doped ceria nanoparticles were synthesized by homogeneous precipitation with hexamethylenetetramine. The as-prepared nanoparticles were crystalline and single-phase with fluorite-type structure (Fig. 13), and consisted of a mixture of anhydrous and hydrated cerium oxide with average size of 5-8 nm.

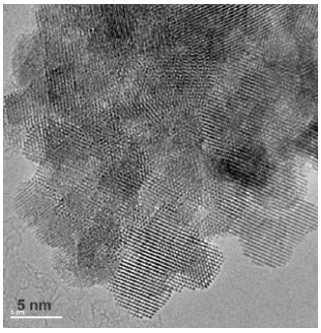


Figure 13. HRTEM micrograph of as-prepared samaria-doped ceria nanoparticles

Technological research work consists of operation of unitary solid oxide fuel cells with proton and oxide ion conductors in hydrogen, methane and ethanol atmospheres as well as the testing of oxygen lambda sensors. The main results were:

- Development and testing of anode-supported solid oxide fuel cells with slurry-coated electrolyte and cathode under ethanol and hydrogen;
- Characterization of sintered zirconia-yttria ceramics by impedance spectroscopy;
- Development of zirconia:magnesia-zirconia:yttria composites for high temperature oxygen sensors;
- Determination of the effect of additives on electrical performance of ceria-based materials;
- Synthesis of nanostructured zirconia and ceria-based solid electrolytes.

Glasses are being developed to immobilize industrial and nuclear wastes, to produce microspheres for radiotherapy treatments, and for optical, electrical, and mechanical applications. A mathematical model using the Monte Carlo method is being developed to determine the dose distribution in human liver when neutron active glass microspheres are directly injected in the organ through and artery. This is one option to eliminate tumors in the liver.

High Purity Rare Earths

High purity rare earth oxides for use in research and development

A simple and economical procedure was developed to obtain high purity rare earths oxides. The raw material in the form of mixed rare earths carbonate is delivered after industrial separation of rare earths, thorium and uranium from Brazil' monazite. Using both ammonical precipitation and cationic ion exchange techniques, with EDTA solution as eluent, high purity rare earths oxides (99-99,9%) were obtained with impurity levels identical to those imported. The yield of this combined technique was reasonably high ≥ 70%. Molecular absorption spectrophotometry was used to monitor the rare earths content during the process and mass spectrometry to determine purity. Typical praseodymium oxide presents the following contaminants in micrograms per gram - Sc(18.20); Y (20.20); La (6.75); Ce (26.10); Nd (3.31); Sm (18.30); Eu (17.00); Gd (19.40); Tb (16.30); Dy (16.90), Ho(17.90); Er (18.40); Tm (16.90); Yb (17.60); Lu (17.70). Typical neodymium oxide presents the following contaminants in micrograms per gram - Sc(4.2); Y (1.40); La (7,75); Ce (6.21); Pr (5,31); Sm (12.3); Eu (11.2); Gd (10.4); Tb (15.3); Dy (6.92), Ho(7.49); Er (6.4); Tm (3.0); Yb (4.53); Lu (15.7).

High purity rare earths' acetate

Praseodymium acetate was prepared from praseodymium oxide Pr_6O_{11} ≥ 99.9%, treated in acetic acid medium. Sc(15); Y (15); La (5); Ce (20); Nd (2); Sm (15); Eu (14); Gd (17); Tb (15); Dy (14), Ho(13); Er (15); Tm (14); Yb (16); Lu (15).

Solubility behavior of rare earths with ammonium carbonate and ammonium carbonate plus ammonium hydroxide: Precipitation of their peroxicarbonates

The purpose here is to report the significant behavior of the rare earths when treated with ammonium carbonate and with a binary mixture of ammonium carbonate plus ammonium hydroxide. The carbonates of some rare earths are completely soluble in ammonium carbonate or in ammonium carbonate plus ammonium hydroxide, while others are only partially soluble and finally some are completely insoluble. Addition of hydrogen peroxide to the soluble complex rare earth carbonates results in the precipitation of a series of new compounds described as rare earth peroxicarbonates. The rare earths have a different precipitation behavior in the carbonateperoxide system. Some are completely and immediately precipitated, others are completely precipitated after an aging period, and some are not precipitated at all. These differing behavior opens up new possibilities in separation chemistry of rare earths. Sm, Gd, Dy, Y, Yb and Tm dissolve quickly and are completely soluble in ammonium carbonate. Ho, Eu and Tb are completely soluble in ammonium carbonate but dissolve slowly. La, Ce, Pr and Nd are only partially soluble in ammonium carbonate. While Ce, Pr, Nd, Sm, Eu and Dy are completely and easily soluble in the ammonium carbonate plus ammonium hydroxide mixture, La is only partially soluble and Tb is completely insoluble in the same mixture. Among the peroxicarbonates, La, Ce, Pr, Nd, Sm, Eu, Gd, Dy and Ho are quantitatively precipitated. The precipitation of the Er peroxicarbonate is quantitative, but after an aging period of 24 h. Y is not precipitated at all. The process is very easy, simple and economically attractive. Eventhough the process has been demonstrated on a bench scale, scale-up is considered to be feasible (Figure 14).

Figure 14. Assembly made to prepare high purity rare earth' oxides by ion exchange

Magnetic and Electric Materials

Significant progress has been made in R&D activities related to these materials over the past three years and consisted mainly of production of hydrogen processed magnetic and electric materials. Magnets and batteries have been produced and investigated and the results have been published in international journals. The microstructure and properties of magnetic materials based on Pr-Fe-Co-B alloy have been studied. Electric materials based on La-Al-Mg-Mn-Co-Ni alloys have also been studied and promising results were obtained. Batteries and magnets have been produced using the hydrogen decrepitation process and standard powder metallurgy route. Due to the extremely high prices of Dy and Co, alternate elements are being introduced in the alloys to overcome this problem.

In the last three years the main R&D activities were: (a) development of technologies and processes as well as management to mitigate environmental impact caused by solid wastes; (b) support efforts to increase energy efficiency, by designing components for fuel cells and porous burners for biogas. Besides, this group used green technologies to make use of debris (from civil construction) and wastes (from ornamental stones) to produce glass-silicate fibers known as e-glass, that is widely used as reinforcement in polymeric or metallic structures (Fig. 15).

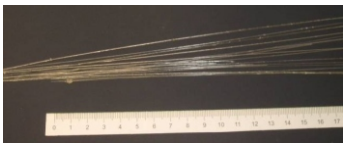


Figure 15. Glass fibers from SiO_2-NaO-CaO-B_2O_3-R_2O_3 glasses

The optimization of processes based on thermodynamic concepts derived from phase diagrams that enabled incorporation and inertization of galvanic solid wastes with heavy metals (up to 40wt%) in soda-lime-borosilicate glass. Based on these results, glasses with higher hydrolytic attack strength than commercial glasses were produced. High color frits with similar compositions were obtained for ceramic enamel. For crystalline glass ceramics, color shifts from green to brown with metallic brightness (as aventurine), has shown increased commercial application (Figure 16). In partnership with almost 27 galvanic/electroplating factories and the São Paulo State Institute for Technological Research (IPT/SP) a database of the volume of wastes generated from these activities in the eastern part of São Paulo was prepared. Based on this, technological options were provided to store, reduce and reuse galvanic mud produced in that region.

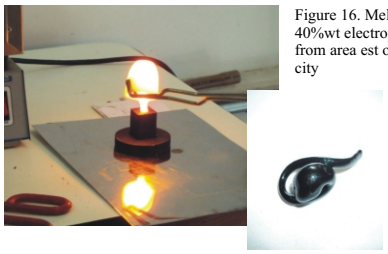


Figure 16. Melting glass with 40%wt electroplating waste from area est of São Paulo city

International environmental directives such as "Waste Electrical and Electronic Equipment" (WEEE) and "Restriction of Use of Certain Substances in Electrical and Electronic Equipment" (RhoS), that are operative in the European community (EU) and North America, were applied to evaluate Brazilian electro medical devices. These actions, without high cost processes, showed that Brazilian products are competitive. In the context of increased product upgrades and discard of electronic equipment, this group evaluated and developed technologies for re-qualifying, recovering and recycling e-waste from mainly, IPCs. To prevent discard of wastes in emerging technological areas this group is involved in inertization in a vitreous matrix of toxic metal nano-wastes produced from the use of nano-catalysts in direct ethanol polymeric fuel cells.

Other activities carried out by this group include: synthesis of materials like lanthanum chromite for applications in ceramic interconnectors; in lighting transmission from doped yttria and doped yttrium dissilicate for biogas burners; colloidal conformation involving rheology, such as tape casting and slip casting, to conform solid oxide fuel cell components; development of a replica method using natural fibers as template to produce porous ceramic membranes for use as mantles in biogas lighters. (Figure 17).

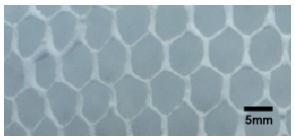


Figure 17. Sintered ceramic yttria based membrane for biogas burning

The development of high temperature glass matrix from BaO-SiO_2-Al_2O_3 for sealants has been studied. This composition is considered to have the right parameters in terms of mechanical strength and thermal expansion coefficient for use in solid oxide fuel cells.

A process based on mechanical alloying has been developed to enable direct low temperature sintering of nickel-zirconia (Ni-YSZ) cermets. The milled powder morphologies were pod-like or wafer-like, where the round fine ceramic particles - the pies were plated with metallic lamellae the pod. Alloying this powder with specific additives and oxygen partial pressures lead to the development of a new low-temperature consolidation process: Sintering by Activated Surface (SAS). Ni-YSZ plus Cu powders can be readily sintered to good densities by SAS rendering nano-sized constituents with good dispersion and pore structure (Fig. 18). The same process was also used for YSZ electrolyte to enable sintering around 1200°C.

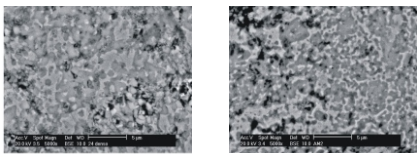


Figure 18. Sintered cermets: Cu-Ni-YSZ (left) and Cu-Mo-Ni-YSZ (righ)

The TG/DTA data coupled with mass spectrometric analysis validated the above mentioned approach suggesting that the process to dry reform methane can be optimised. The last result is the main contribution to state-of-art research.

High energy milling has been used to obtain nanocrystalline TiFe intermetallic compound by mechanically alloying the two elements in powder form (Figure 19). Powders were handled in an argon-filled glove box with automatic control of the humidity and oxygen contents. A PCT (pressure-composition-temperature) system was built to characterize hydriding/dehydriding behavior of hydrogen storage materials. The operation of the system was made entirely automatic by a PLC, solenoid valves, and electronic pressure as well as flow controllers. PCT curves can be measured in two modes: static (as a Sievert apparatus) and dynamic (hydrogen flow maintained constant).

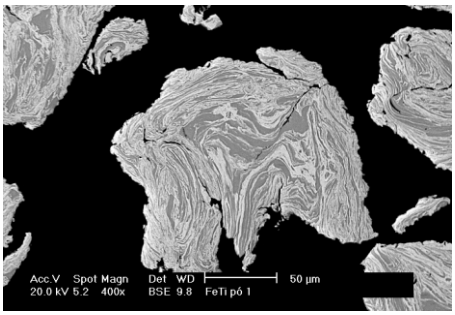


Figure 19. Partial mechanical alloying of a Ti+Fe mixture showing particles with lamellae microstructure (white and gray phases are Fe and Ti, respectively)

In the development of metallic components using powder metallurgy techniques, high speed steel powders with niobium were used. The main objective was to use of the high wear resistance of austenitic stainless steel in the manufacture of filters. Exploratory TIG welding was performed to increase the height of the filters (Figure 20 and 21).

Figure 20. TIG welding on stainless steel filters

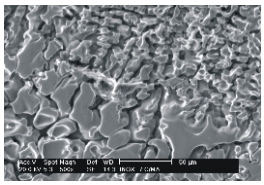


Figure 21. Interface of the weld metal and heat affected zone in filters (SEM)

High-pressure cell for neutron diffraction experiments

The high-pressure cell body consists of a zircaloy cylinder encapsulated in a carbon fiber composite and this device has innovative applications. Use of the high-pressure cell will permit neutron diffraction experiments of polycrystalline materials under high hydrostatic pressures to study “in situ”, high pressure phase crystal structure transformations, among other pressure induced transitions. This device, one of its kind, will be available to scientists in the field of neutron diffraction using the IPEN neutron diffractometer, which is also one of its kind in Brazil. Similar cells, also innovative, were developed by this group for high pressure x-ray diffraction and absorption studies, and were made available to users of the National Synchrotron Light Laboratory - LNLS / CNPq in Campinas.

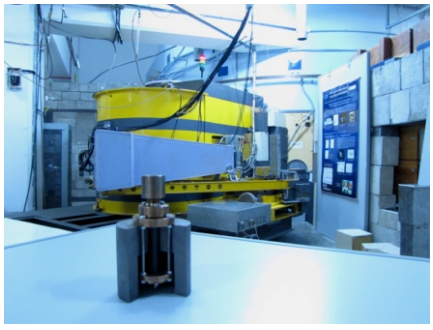


Figure 22. High-pressure cell (front) and the neutron diffractometer in the background

Standard Reference Materials for Powder Diffraction

The development of reference standard materials for calibration and determination of instrumental parameters of equipments used in diffraction (conventional x-rays, synchrotron and neutrons) is of paramount importance. The following standard high quality materials for diffraction were obtained: silicon (Si), yttria (Y_2O_3), lanthanum hexaboride (LaB_6), α-alumina (Al_2O_3), ceria (CeO_2), hydroxyapatite, silicon nitride and others. Other materials for SAXS, XAFS and for in situ measurements under high hydrostatic pressures are being prepared. The first samples have been sent to other Brazilian institutions and abroad for testing and the feedback has been encouraging.

Copper alloy wires for use in electrical transmission lines

Copper is a ductile metal that has high electrical and thermal conductivity. However, adequate mechanical properties are required for it to be used as power supply lines. Consequently, alloying elements are added to copper to improve its mechanical properties, without significantly altering its electrical conductivity. The objective of this project was to develop copper alloys for use as power transmission wire. The steps involved in the manufacture of copper wires were studied, starting with melting of copper and magnesium in an arc furnace, dilution of the melts in a resistance furnace and casting in a copper die. The billets were then forged and drawn into wires. The wires were characterized in terms of its mechanical properties and electric conductivity. The yield and the ultimate tensile strengths increased with increase in the alloying element content, while the electrical conductivity decreased to approximately 60% of IACS.

The Microscopy Laboratory of IPEN is well equipped, enabling examination and analysis of a variety of materials (ceramics, metals, polymers and composites) using multiple techniques. The scanning electron microscope (SEM) with a conventional tungsten filament has been used to observe particulate samples (fig. 23), fracture surfaces (fig. 24), dense polished and etched samples (fig. 25), glass microspheres sintered to different extent and bio-mimetic coatings on metals (fig. 26). The scanning electron microscope with a field emission gun (FEG-SEM) allows examination and analyses similar to that in a conventional SEM, but at higher resolution, as shown in fig. 27. Due to the high intensity of the beam in the FEG-SEM, it is possible to obtain x-ray maps of chemical constituents in samples. The transmission electron microscope (TEM) has been used extensively. Fig. 28 reveals the pores in a mesoporous silica. Thin foils of materials can be observed and the TEM allows determination of grain size, morphology, analysis of sub grain and precipitates, as well as EDS (energy dispersive spectroscopy) analysis of regions, or line scans, which reveals variation in concentration of different elements (Al, Si, Mn, Cu). High-resolution images can be obtained, and the crystal structure of specific regions can be determined, fig. 29.

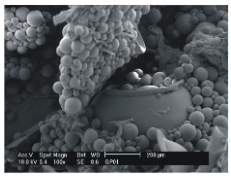


Figure 23. SEM micrograph of polymer microspheres

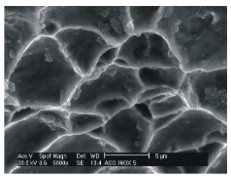


Figure 24. SEM micrograph of fracture surface on stainless steel

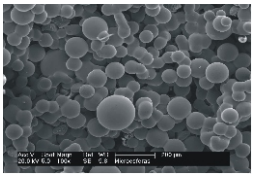


Figure 25. SEM micrograph of sintered glass spheres

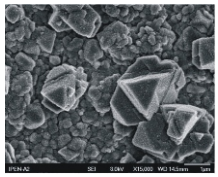


Figure 26. SEM micrograph of a bio-mimetic coating on metal

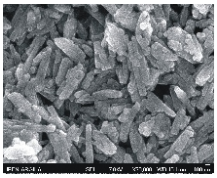


Figure 27. Image of Nanometric clays, obtained in the FEG-SEM

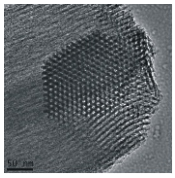


Figure 28. TEM of mesoporous silica

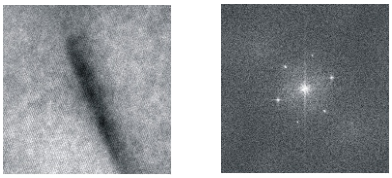


Figure 29. HTEM showing crystalline structures (left) and Fourier Transformed (FT) analysis (right) of the same field

Nanotechnology has been pointed as a high innovative technology allowing a deep and wide change in the materials production and application. The molecular design in an atomic scale is a new concept starting in technology that allows a new functionality of the matter. This leads to the control of the physical, optical, electronic, surface and magnetic properties and reactivity of the nanostructured functional materials. The nanotechnology is today one of the main points of the research activities, development and innovation in all of the industrialized countries. There are some nanotechnological products in the current market, but there's a trend to increase production in a short time. That new technology concept has been at development at IPEN, by the study of nanostructured functional materials. The following activities are in development:

Nanostructured coatings

Chromium carbide

Mechanically milled nanocrystalline feedstock powders of Cr_3C_2-Ni20Cr were used to develop thermally sprayed nanostructured coatings. These coatings were more uniform, exhibited higher hardness, high temperature erosion-oxidation resistance, increased fracture toughness and thermal stability compared to conventional coatings of the same materials.

Chromium dioxide

Thin film chromium dioxide coatings on steel substrates were prepared by MOCVD for increased high temperature oxidation resistance of the substrate. (Fig. 30)

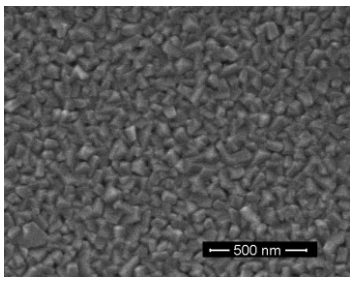


Figure 30. Surface of chromium dioxide thin film prepared by MOCVD

Titanium dioxide

Titanium dioxide (TiO_2) is an important commercial material and is used extensively in applications such as cosmetics, paint pigments, building materials, antibacterial surfaces, biomaterials and solar cells. Thin films of TiO_2, shown in Fig. 31, were grown under different conditions using the MOCVD technique.

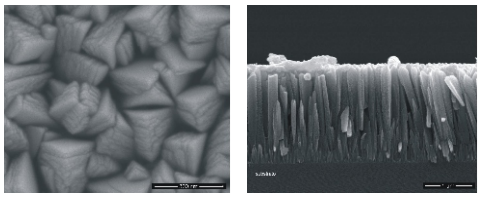


Figure 31. TiO_2 film grown at 500ºC on patterned Si. (a) surface; (b) cross-section

Indium Oxide nanoparticle and application

Indium oxide, a wide band gap (~3.6 eV) transparent semiconductor is of great interest for many device applications due to the unusual combination of high transparence in the visible region and high electrical conductivity. Current applications of indium oxide are in electro optic modulators, in the electronic field as window heater, electrochromic mirrors, solar cells, and flat-panel displays. Moreover, nanostructures based on indium oxide are promising device materials for chemical sensors and in the upcoming nanoelectronic building blocks. Indium oxide nanoparticles were synthesized by a surfactant-free room-temperature soft chemistry route. After thermal treatment of the gels at 400ºC indium oxide nanoparticles of 8 nm of average size were obtained. A small population of nanoparticles of 2.8 nm in size was evidenced by small-angle X-ray diffraction. The single-crystalline nature of the produced nanoparticles was confirmed by transmission electron microscopy. The synthesized material exhibits a weak and broad photoluminescence emission in the blue-UV region due to a quantum size effect.

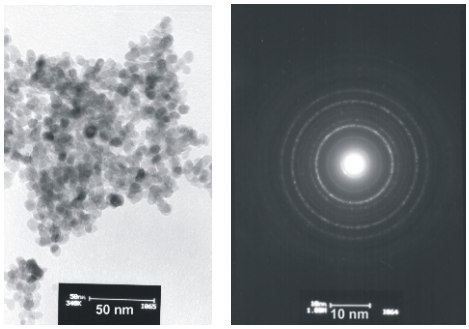


Figure 32. Bright-field TEM micrograph and selected area diffraction of In_2O_3 nanoparticles

Titanium dioxide nanostructured films: characterization and nanotechnological applications

Nanoscience and Nanotechnology is a scientific-technological field committed to understanding how the control of matter at the molecular level may lead to the development of new materials with unique and exclusive properties. Among these characteristics, the nanomaterials have got an extraordinarily high ratio of surface area to volume

and their mechanical and electrical properties are considerably improved. The numerous potential applications of nanomaterials have led researchers to consider them as the "philosopher's stone" of modern science that among the major challenges is the question of environmental sustainability. As solar energy is an abundant resource there, it is of great interest to find ways and means to collect this energy as to create an ecologically and healthy environment. Nanoporous and nanostructured films and surfaces have been successfully exploited for these spectacular effects. So a very promising material within nanotechnology applications for the use of solar energy is titanium dioxide nanostructured films which are able to absorb solar energy by converting it into chemical energy. Our studies seek mainly the improvement of the photocatalytic activity of titanium dioxide nanostructured films for the development of green technologies related to wastewater treatment and solar cells with high efficiency conversion of solar energy in the production of electricity.

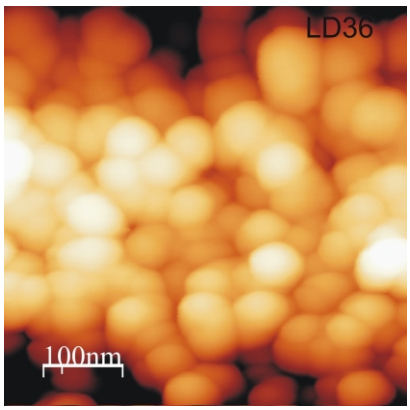


Figure 33. AFM image of nanostructured TiO_2 film demonstrating control of morphology in the molecular level

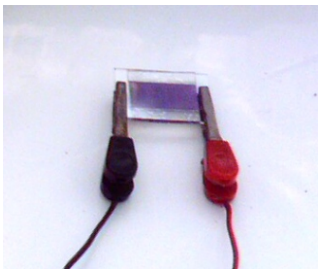


Figure 34. Biosolar cells with TiO_2 nanostructured films and natural dye

Electro-oxidation of ethanol using PtSnRh/C electrocatalysts prepared by an alcohol-reduction process

Pt/C, PtRh/C (90:10 and 50:50), (Pt:Sn 50:50) and PtSnRh/C (Pt:Sn:Rh 50:40:10) electrocatalysts were prepared in a single step (co-reduction of mixed metal ions) using ethylene glycol as solvent and reducing agent in the presence of Vulcan XC72. The X-ray diffractograms of Pt/C, PtRh/C, PtSn/C and PtSnRh/C electrocatalysts are shown in Fig. 35. All diffractograms showed a broad peak at about 25° that was associated with the Vulcan XC72 support material and four peaks at approximately 2 = 40°, 47°, 67°and 82°, which are associated with the (111), (200), (220) and (311) planes, respectively, of the face-centered cubic (fcc) structure characteristic of platinum and platinum alloys. In the diffractograms of PtSn/C and PtSnRh/C it was also observed two peaks at approximately 2θ = 34°and 52° that were identified as a SnO_2 phase. The (220) reflections of Pt(fcc) crystalline structure were used to calculate the average crystallite sizes using the Scherrer equation and the calculated values were in the range of 22.5 nm. It was observed that the (220) diffraction peak of PtRh/C (50:50) was dislocated to higher angle compared to Pt/C and PtRh/C (90:10) indicating an alloy formation between Pt and Rh. Similarly, the (220) diffraction peak of PtSnRh/C electrocatalyst was also dislocated to higher angle compared to PtSn/C electrocatalyst. TEM micrographs of PRh/C(90:10) (Fig. 36a), PtRh/C (50:50) (Fig. 36b), PtSn/C (Fig. 36c) and PtSnRh/C (50:40:10) (Fig. 36d) electrocatalysts showed a good distribution of the Pt particles on the carbon support with particle sizes of 3.0±1.0 nm, which is in agreement with XRD results.

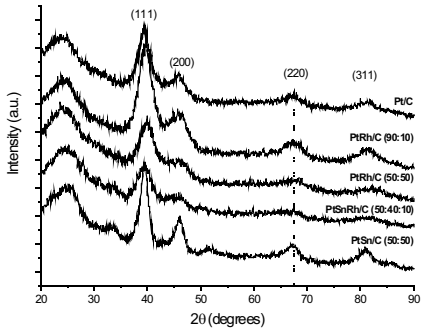


Figure 35. X-ray diffractograms of Pt/C, PtRh/C, PtSn/C and PtSnRh/C electrocatalysts

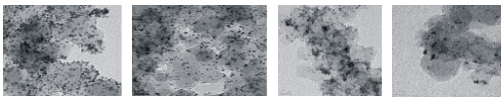


Figure 36. Transmission electron micrographs (20 nm) of PtRh/C (90:10) (fig 36a), PtRh/C (50:50) (fig 36b), PtSn/C (50:50) (fig 36c) and PtSnRh/C (50:40:10) (Fig 36d) electrocatalysts

Magnetite nanoparticles and magnetic carrier technology as adsorbent of metallic ions and dyes

Synthetic magnetite nanoparticles have been prepared by precipitation process of the Fe^{2+} and Fe^{3+} ions with a basic solution and from precipitation of Fe^{2+} ions and were subjected to heat treatment in water bath and microwave irradiation. Characterization studies were carried out for

various heating times and the two types of synthetic magnetite were compared. The techniques used were X-ray powder diffraction (DRX), transmission electronic microscopy (TEM), scanning electronic microscopy (SEM), thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR), differential scanning calorimetry (DSC) and magnetization curves (VSM). Fig. 37 shows the prepared magnetite nanoparticles from Fe^{2+} ions and subjected to microwave irradiation. They are good adsorbents of metallic ions and dyes and may be easily removed from wastewater using a magnet due to their magnetic properties. Synthetic magnetite nanoparticles have been studied as adsorbent of U ions from nitric solutions and have shown great perspective as an alternative adsorbent to conventional adsorbent.

The magnetite nanoparticles have been conjugated with adsorbent materials for obtaining magnetic carrier and its effect on enhancement adsorption capacity has been investigated. The technology of magnetic carrier for the wastewater treatment combines contaminant separation by sorption and magnetic recovery into a simple and compact process. Also called magnetic adsorbent it provides a simple way to remove contaminants from solutions under a wide range of chemical conditions. Adsorbents as chitosan, sugarcane bagasse and synthetic zeolite, from mineral coal fly ash, were combined with the magnetite nanoparticles for obtaining magnetic adsorbents. Characterization and adsorption studies of U and Mo ions, Reactive Orange 16, Indigo Carmine and Congo Red dyes and proteins were carried out. The performance of the magnetic adsorbents was investigated varying the pH, dose of adsorbent, agitation time and adsorption temperature using the batch system. The equilibrium data were analyzed by the Langmuir and Freundlich isotherm models. The magnetic adsorbent of chitosan presented the best results of adsorption of the U ions, and moreover showed that trypsin, BSA and collagen can be adsorbed. The Langmuir model was found to best describe the equilibrium isotherm data and the pseudo second-order model was found to explain the rapid kinetic of sorption. For all studied adsorption processes, the Gibbs free energy indicated the spontaneous nature. The magnetic zeolite exhibited a potential application in treatment of textile wastewater as an efficient and low cost adsorbent for dye removal. In 2008-2010, this research has produced three patents pending and one Master completed in 2010.

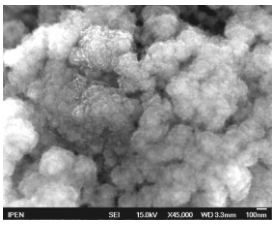


Figure 37. Magnetite nanoparticles prepared from Fe^{2+} ions and subjected to irradiation in a domestic microwave oven by Holland H

Preparation and characterization of smart magnetic hydrogels and its use for drug release

We have synthesized magnetic hydrogels based on chitosan that were successfully fabricated by chemically cross-linking of glutaraldehyde and $MnFe_2O_4$ nanoparticles (ca. 10-50 nm) to be used in biotechnology applications (drug release).
Those hydrogels were cured by two sources of energy γ-radiation (cobalt source with 3, 5, 10 KGy and 60 Watts UV-radiation lamp). For characterization, the hydrogels were dried until constant weight and analyzed by infrared spectra (IR), thermal analyses and UV absorption spectra The IR spectra of dried materials showed characteristic bands of chitosan, attributed to ν_{OH}= and ν_{NH} centered at 3446 cm^{-1}, Amide I band corresponding to $\nu_{C=O}$ vibration (1650 cm^{-1}) of acetyl groups in chitosan. The band Amide III at 1332 cm^{-1}, due to combination of NH deformation and the ν_{CN} stretching vibration and the band due to ν_{C-O} at 1089 cm^{-1}.The magnetic particles showed bands at ca. 600 and 400 cm^{-1} attributed to the stretching ν1 and ν2 from octahedral and tetrahedral sites of crystalline structure respectively. Thermal analyses (TGA/DTGA) showed three events of loss. Molecular absorption spectra in UV-vis showed large bands in visible line of spectra. All swelling behavior is plotted on the average of three trials. The cross-sectional SEM observation demonstrates that the $MnFe_2O_4$ nanoparticles were fairly uniformly distributed in the gel matrix.
The swelling kinetics and time dependent-swelling behaviors of chitosan/glutaraldehyde/ $MnFe_2O_4$ hydrogels was obtained in deionised water (pH 7) and serum solution.
Moreover, in vitro release data reveal that drug release profile of the resulting hydrogels is controllable by switching on or off mode of a given magnetic field. While applying magnetic fields to the magnetic hydrogels, the release rate of vitamin A of the hydrogels was considerably decreased as compared to those when the field was turned off, suggesting a close configuration of the hydrogels as a result of the aggregation of $MnFe_2O_4$ nanoparticles. Based on this on-&-off mechanism, the smart magnetic hydrogels based on the hydrogels ferrite hybrid composites can be potentially developed for application in novel drug delivery systems.

Development of luminescent and magnetic membranes based on chitosan and crosslinked with exudate of cashew

Natural polysaccharides such as chitosan in acidic medium, present the amine groups protonated, allowing ionic interactions between their cationic groups and anionic groups present in polysaccharides exudates. Covalent interactions and formation of imine groups (-C = N-), cannot be

disregarded. As a result of these kind of interactions membranes with good mechanical strength and able to function as drug-releasing, with no toxic agents in their formation are achieved.
The incorporation of manganese ferrite (Ni-Zn-Mn), a ceramic material with super paramagnetic behavior and β-diketonate complexes of rare earths, into the chitosan membranes, allow study the functionalization of the drug release matrix by the magnetic field and could follow the drug release monitoring the luminescence of the complex. The swelling of chitosan membranes crosslinked with exudate of cashew, shows that when the percentage of crosslinking increases, the swelling capacity of the membrane decrease.
The micrographs show the surface of chitosan membranes, enlarged 25 times. For membranes without crosslinking the surface appears smooth and without pores. The membranes with 1% and 3% of crosslinking present a crystalline structure and could be observed the pore formation. The membrane with 5% crosslinker has an amorphous structure with no crystallinity and pore.

Potential applications for the use of rare earth complexes and nanoparticles as luminescent biolabels

The ever growing interest in rare earth coordination compounds and materials with optical properties stems from applications in multidisciplinary fields such as nanosensors, materials for telecommunications, lighting devices, and luminescent probes for bioanalyses and live cell imaging and sensing. Most of these applications use the unique ability of the rare earth ions to emit well-defined narrow bands in different spectral ranges, from visible to near-infrared. Due to the low absorption cross-section of the f-f transitions, efficient population of the excited 4f states has to rely on energy transfer from the surroundings of the metal ion (antenna effect or luminescence sensitization). Another point desirable is the use of RE^{3+} ions with emission in the near-infrared (NIR) region reducing the interference from biological materials in the analyses.
At this point, special attention was given and reported to the preparation and characterization of rare earth coordination compounds and nanoparticles based on rare earth ions. RE^{3+} β-diketonate complexes with macrocyclic ligands, tetracycline, piroxican and carboxilate have been synthesized and characterized and are being used to mark hemoglobin S (sickle cells) *Leishmania* (tropical desease) and PSA (Prostate-specific antigen that is a protein produced by cells of the prostate gland. The PSA test measures the level of PSA in the blood). Several nanoparticles based on Nd^{3+}, Eu^{3+} and Tb^{3+} ions doped into the Y_2O_3 matrix, are also being synthesized and evaluated for labeling anti-oxLDL (anti-oxidized low density lipoproteins), one of the principal causes of cardiovascular diseases and responsible for around 30% of deaths worldwide. Information about structure and average grain size of these nanoparticles were obtained by x-ray diffraction (XRD), transmission electron micrograph (TEM) and infrared absorption spectroscopy.

Biolabeling with detection in the near-infrared luminescence based on $Y_2O_3:Nd^{3+}$ nanoparticles

Rare earth ions (RE^{3+}) are excellent luminescent probes for biological systems because their narrow emission bands are easily recognizable and well separated from the broad band fluorescence emission, which results in a high color purity of the emitted light. Moreover, applications in medical diagnosis make use of immunoassay detected by time-resolved luminescence in order to allow the separation of the RE^{3+} ions phosphorescence from the fluorescence of the biological samples. A number of important technological advances have been made by applying nanotechnology for biomolecular detection using luminescent RE^{3+} nanomaterials. Furthermore, nanoparticles are unique because their nanometer size gives rise to a high reactivity and beneficial physical properties (e.g. electrical, electrochemical, optical and magnetic) that are chemically inert.
The Nd^{3+} ion doped in Y_2O_3 matrix used in immunoassay can provided the benefit of being absorbed in visible and infrared irradiations (500 to 900 nm) with emission in the NIR region.

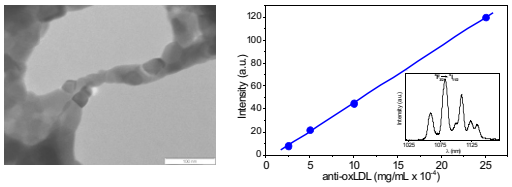


Figure 38. TEM image of Y_2O_3:2%Nd^{3+} nanoparticles and integrate emission intensity of $^4F_{3/2} \rightarrow {}^4I_{11/2}$ as function of functionalized nanoparticles bound to the LDL-antibody in the sensibilized plate with oxLDL-antigen. The insert figure shows the emission spectrum obtained from the sensitized well in the 96-plate containing the anti-oxLDL-labeled Y_2O_3:2%Nd^{3+} functionalized nanoparticles.

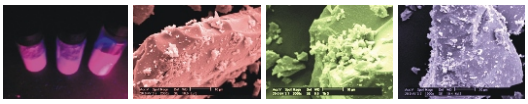
Preparation and photoluminescence properties of functionalized silica material incorporating europium complexes

The development of functionalized silica particles containing luminescent material has received special attention because of its biological applications such as optical markers *in vitro* and *in vivo*, clinical diagnosis and drug delivery.
New optical markers are still being developed, and rare earth materials are proposed as very attractive candidates. RE^{3+}-complexes containing carboxylate ligands are one the most largely investigated kind of coordination compounds due to higher thermal stability.
This work presents the development of a new process to obtain silica particles incorporating Eu^{3+}-complexes using APTES as a source of silica. The

synthesis was adapted from the literature, where the Y_2O_3:Eu^{3+} sample was used as label.
In summary, a kind of material incorporating Eu-BTC complexes has been prepared by a new method and has been characterized. The materials showed characteristic emission of Eu^{3+} ions. According to our results the most efficient luminescent system is the Eu-TMA-Si material, presenting the highest quantum efficiency $\eta = 27\%$. The outstanding aspect is clearly the transparency of the material based on modified silica, allowing the full detection of the Eu^{3+}-complex luminescence. Therefore, it is a promising candidate for molecular conjugation in clinical diagnosis.

Synthesis and characterization of SnO_2 and TiO_2 nanoparticles doped with lanthanide for biological labeling

Fluoroimmunoassay is an ultrasensitive technique for investigation of enzymes, antibodies, cells, hormones and others. The demand for highly sensitive systems brought the nanomaterials for biomedical and biotechnological field. Semiconductor nanocrystals (quantum dots) doped with lanthanide ions, when functionalized with biomolecules, can be used as luminescent biomarkers. Aiming this application, nanoparticles of titanium and tin mixed oxide doped with europium, terbium and neodymium have been synthesized and characterized. The synthesis was made by the co-precipitation method and characterized by SEM, IR, XRD, TGA and luminescence spectroscopy.



(a) (b) (c) (d)

Figure 39. (a) Nanoparticles of SnO_2/TiO_2:Eu^{3+}, (b) Mev SnO_2/TiO_2:Eu^{3+}, (c) Mev SnO_2/TiO_2:Tb^{3+} and (d) Mev SnO_2/TiO_2:Nd^{3+}

Europium Betha-Diketonate complexes with Tetracycline (Tc)

Luminescent materials containing trivalent rare earth (RE^{3+}) complexes with β-diketonate ligands have been intensively studied in recent years. The RE^{3+} compounds present characteristic narrow emission bands in the UV-Vis region, large Stokes shift and the antenna effect that enhance the overall quantum efficiency. As a result, these complexes have found wide applications as luminescent markers, photoluminescent sensors, electroluminescent devices, and multicolor display. In the field of biomarker these compounds have linked with biological parts.
At this point in time, four new complexes of europium β-diketonate with tetracycline as ligand were synthesized. IR spectra of the Eu(III) complexes show two strong absorption bands at ~1597 and ~1566 cm^{-1} attributed to ν_s(C=O) and ν_{as}(C=O) vibrational stretching modes, suggesting that the β-diketonate ligand acts as chelate ligand. SEM image showed particles rounded with grain size lower than 10 nm (Fig 40). The emission spectrum of europium complexes, in the solid state, recorded in the range of 420 to 720 nm at liquid nitrogen temperature, under excitation at β-diketonate transitions (~350 nm) is shown in Fig. 41. This emission spectrum exhibits characteristic narrow emission bands that are assigned to the $4f^6$-$4f^6$ transitions of Eu(III) ion, emanating from the emitting $^5D_0$ level to the ^7F_J (J = 0, 1, 2, or 4) levels, where the most intense corresponds to $^5D_0 \rightarrow {}^7F_2$ transition taking place around 613 nm. An important feature to be observed is the nonexistence of broaden bands arising from the β-diketonate centered transitions, indicating that intramolecular energy transfer from the β-diketonate ligands to the Eu(III) ion is operative.

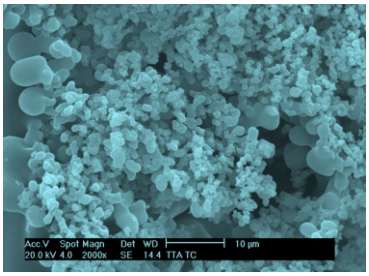


Figure 40. Scanning Electron Microscopy of the $Eu(TTA)_3(TC)_2$

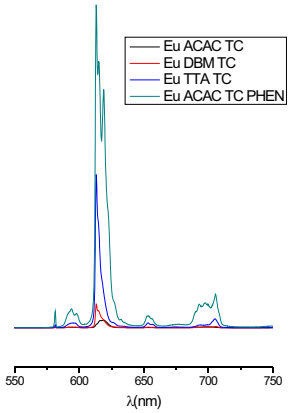


Figure 41. Emission Spectra of Eu(β-diketone)$_3$ TC_2

Development of nanobiomarkers for use in sickle cell anemia

Luminescent materials, such as the complex of rare earth, can be used as markers in cytology and immunology, being also used as luminescent biomarkers, once the development of these nanomaterials create new possibilities to many fields, particularly in diagnostic medicine. Besides, it establishes one kind of fluorescent probes, for which there are no equivalent organic molecules.

Due to its potential in market application, the objective of this work was to develop luminescent materials, allowing the use of these supermolecules of lanthanides as markers for the detection of Sickle Cell Disease (HbS). Six luminescent markers were developed based on rare earth compounds. The main methodology used for the detection of HbS was fluoroimunoassay, which is already used in investigation of enzymes, antibodies, cells, hormones, and so on. During this work, absorption spectrum in the infrared by Fourier's Transform (FTIR) was also used to detect the HbS.
The studied methods were applied for the diagnosis of this disease, which has genetic origin, very typical of the hemoglobin-pathology group and considered to be a public health problem in Brazil (ANVISA). When early diagnosed, Sickle Cell Disease (SCD) has a significant decrease in morbidity and mortality. Comparing the obtained results to the already known methodologies, it was possible to conclude that they are viable methods to detect HbS. Besides, when totally developed, these methods will contribute to the production of Sickle Cell Anemia's diagnostic, and they will have impact in São Paulo state public measures, as well as nationwide in Brazil.
We are extending our tools to validate a safe, sensitive and inexpensive methodology. Therefore, the Maldi-Toff method is being used in order to have a better understanding of the peptide profile on the target of biological materials. A database of most peptides present in the sickle cell disease will also be made in order to come up with the biomarking of HbS with Iodine-131 for comparative analysis of these methods.

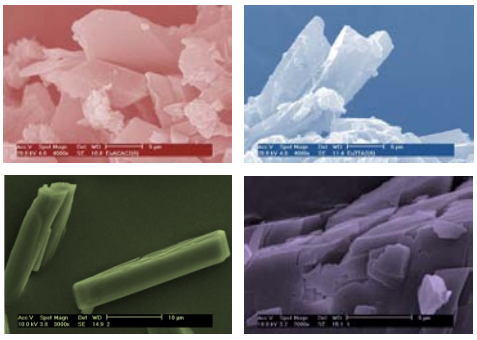


Figure 42. SEM of the Europium and terbium supermolecules

Persistent luminescent materials. Thermoluminescence and synchrotron radiation studies on the persistent luminescence of $Baal_2O_4:Eu^{2+},Dy^{3+}$

Since 1995, the research on persistent luminescence materials has increased substantially. This is due to the progress in the properties of these materials. Nowadays they can emit in the visible range up to 24+ hours, after ceasing the irradiation. Because of the long emitting time, these phosphors can be exploited commercially in emergency signs, road signalization, wall painting, watches, micro defect sensing, optoelectronics for image storage and detectors of high energy radiation. The persistent luminescence is not anymore just a scientific curiosity.
According to the literature, the $BaAl_2O_4(:Eu^{2+},R^{3+})$ materials are prepared via a solid state route, usually by heating $BaCO_3$ with Al_2O_3 (or their precursors) at elevated temperatures. However, low temperature routes as combustion and sol-gel syntheses are not uncommon. In the present work, the $BaAl_2O_4:Eu^{2+},Dy^{3+}$ materials were prepared with different synthesis methods and with different Eu^{2+} and Dy^{3+} concentrations. As usual, the combustion synthesis produces crystals with smaller size, evidently due to higher local temperature during the spontaneous explosion. Since the thermoluminescence analyses suggested the presence of one and three traps for the combustion and solid state prepared materials, respectively, the method of preparation has a significant effect on the defect structure of the materials. The mismatch between the band gap (E_g) value obtained from the synchrotron radiation excitation spectra and the DFT calculation was deduced to result from the covalent bonding in the $BaAl_2O_4$ host. The XANES spectroscopy showed a predominance of Eu^{3+} which can be present as a result of the *in situ* conditions of persistent luminescence during the X-ray irradiation. A systematic study of the effect of other R^{3+} co-dopants than Dy^{3+} is needed to a better understanding of the persistent luminescence mechanism of $BaAl_2O_4:Eu^{2+},Dy^{3+}$.

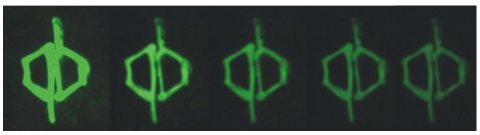


Figure 43. Afterglow of persistent material

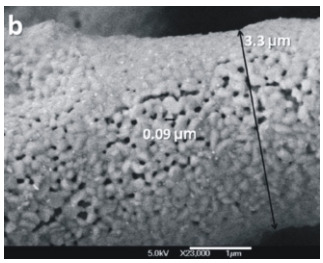


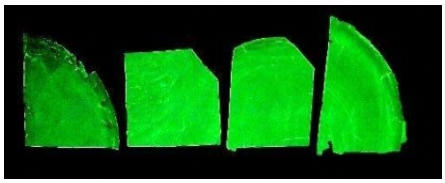
Figure 44. SEM Images of the $BaAl_2O_4:Eu^{2+},Dy^{3+}$ phosphors prepared by the combustion method

Luminescence-Tuneable multicolour PMMA films doped with Lanthanide B-Diketonate complexes

Interest in luminescent materials containing trivalent lanthanide ions (Ln^{3+}) as emitting centres has grown significantly in recent years. However, the Ln^{3+}-complexes generally present low thermal

stability, limited photostability and poor mechanical properties. Another parallel challenge is that most of these compounds are usually achieved as hydrates, consequently the luminescence intensity is suppressed due to the activation of non-radiative channels. In order to overcome simultaneously these deficiencies and improve the characteristics of light emission (*e.g.* quantum yield, lifetimes), Ln^{3+}-complexes have been incorporated into organic polymers, liquid crystals and sol-gel derived organic-inorganic hybrids. Polymers offer several advantages for the development of materials, such as: flexibility, versatility, optical quality and moderate processing conditions. By incorporating luminescent Ln^{3+}-complexes within the polymer matrix, the resulting product represents not only the sum of individual contributions of both organic and inorganic phases, but also novel properties for a new class of materials. In the present work, diaquatris(thenoyltrifluoroacetonate)-europium(III), $[Eu(tta)_3(H_2O)_2]$, ditriphenylphosphine oxide (thenoyltrifluoroacetonate)-europium(III), $[Eu(tta)_3(TPPO)_2]$, triaquatris(acetyl-acetonate)Terbium(III), $[Tb(acac)_3(H_2O)_3]$ and triphenylphosphine oxide (acetyl-acetonate)Terbium(III), $[Tb(acac)_3(TPPO)_2]$, complexes were co-doped into the PMMA polymer in order to obtain multicolor light-emitting devices due to their strong luminescence and relatively simple and inexpensive preparations.

λ_{exc} = 254



PMMA: x%$Tb(acxac)_3(H_2O)_2$

Figure 45. Samples of PMMA films under and without UV irradiation excitation

Nanomaterials for organic light emitting devices

Complexes containing Rare Earth ions are of great interest in the manufacture of electroluminescent devices as organic light emitting devices (OLED). OLEDs are regarded as the next generation flexible flat panel display technology and a new platform for low-cost illumination source. These devices, using rare earth trivalent ions (TR^{3+}) as emitting centers, show high luminescence with extremely fine spectral bands due to structure of its levels of energy, a long lifetime and high quantum efficiency. The preparation was made of rare earth β-diketonate complexes (tta = tenoyltrifluoroacetonate and acac = acetylacetonate) (Tb^{3+}, Eu^{3+} e Gd^{3+}) contend crown ether macrocyclic ligand (DB18C6 = dibenzo18crown6). The thermal analyses of the $Eu(tta)_3(DB18C6)_2$ and $Tb(acac)_3(DB18C6)_2$ and morphology are important in the manufacture of OLED devices using PVD technique. This Physical Vapor Deposition used in this work have been done with lab thin film which belongs to Departamento de Física da Pontifícia Universidade Católica do Rio de Janeiro - PUC - Rio and Lab-*f* from Chemistry Institute-São Paulo University.

Formation of chitosan-cashew gun hydrogel with UV radiation

Chitin, obtained from lobster, shrimp and crab shell waste, is the second most abundant polysaccharide found in nature. In the 21st century, chitin and its derivative (chitosan) face new opportunities to contribute to functional materials and environmentally friendly materials as to meet the diverse needs of today society because of their nontoxic, biodegradable, biocompatible, antibacterial, etc.
UV Radiation processed chitosan cashew gun hydrogels have been observed to be suitable for producing transparent, flexible, mechanically strong, biocompatible, effective and economical hydrogel. No additives were used and different formulations containing chitosan and cashew gun selected from combinations, also with agar, were used to make the hydrogels. Chitosan formulations containing the cashew gun and agar show significantly different pre-gel viscosities behavior. By increasing the concentration of agar in the formulation, it converts the sheet gel to paste gel useful for filling wound cavities. For characterization, the hydrogels were dried until constant weight and analyzed by infrared spectra (IR) thermal analyses, scanning electron microscopy and UV absorption spectra. The results indicate that the pre-irradiation network structure of the formulation plays an important role in properties of the irradiated gel dressing. Scanning electron micrographs show highly porous structure of the gel. To observe swelling response of the chitosan/Cashew gum hydrogels when exposed to different pH conditions, the hydrogels were emerged until equilibrium in an aqueous medium of pH 2, 4, 7 and 9 at 25°C. The hydrogels presented higher swelling content in acid medium. The hydrogels will be observed to be useful in treating burns, non-healing ulcers of diabetes, drug controlled deliver and other external wounds.

Synthesis, characterization and cytotoxicity of polymeric hydrogels for use to immobilization and drug release on Leishmaniasis treatment

Hydrogels were obtained with poly(N-vinyl-2-pyrrolidone) (PVP), poly(vinyl alcohol) (PVAl) and poly(ethylene glycol) (PEG). The process of obtaining was: 1) irradiation process using ^{60}Co gamma source, 2) Crystallizing process of freezing/thawing thermal cycles, and 3) crosslink by chemical reaction with citric acid in chloride acid as catalyses. The hydrogels obtained were characterized by Thermogravimetry Analysis (TGA), differential scanning calorimetric (DSC), gel content, swelling, scanning electronic microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and cytotoxicity by neutral red test.
Insertion of nanoclays in PVAl/PVP hydrogels performs distinct characteristics and the presence of PEG is essential to the membranes properties. Cytotoxicity behavior was similar to negative control curves indicating that matrices have no toxicity when formulated with laponite nanoclay. Higher thermal stability was observed in membranes obtained by gamma and chemical process was showed in displacement of PVAl decomposition temperature associated to higher crosslink stability in presence of nanocompound studied. Glucantime immobilization was done in membranes that showed higher crosslinking obtained by different processes. Drug delivery was observed in period of 24 hours and similar results were achieved. The different processes of PVAl/PVP hydrogels have performed different release kinetics. The characteristics of homogeneity, integrity, no cytotoxicity and swelling of the systemized membranes are qualities of suitable hydrogels for drug delivery and biomedical applications.

Development of sulfonated multifunctional fluoroelastomer based on nanocomposites

Nanomaterials are new research areas that have been attracting a lot of attention due to their potential applications in several areas such as: electronics, optics, catalysis, ceramics, magnetic data storage, and polymer nanocomposites. These nanomaterials are incorporated in polymer matrices which makes it possible to improve the performance and physical-chemical properties for the modified material. These researches reveal that it is necessary to incorporate a small quantity of inorganic material in order to obtain a polymer with better properties than the original, for example: nanoparticles of POSS and clay incorporated in a polymer matrix are used to improve mechanical and barrier properties. The objective of this project is the synthesis and characterization of ion exchange membranes, from nanostructered fluoroelastomer. This process of nanostructering will be realized by nanoparticle of POSS and clay incorporated in the polymer matrix.

Development of HMSPP with nanoparticles of silver with biocide activity

HMSPP is a long chain branched PP as a result of radiation modification of PP in presence of acetylene monomer to promote the grafting of long chain branches. This study started in the doctorate project of Washington L Oliani, based on the study of microgel structures in HMSPP and silver inclusion.
Among the wide variety of plastics, polypropylene of high melt strength (HMSPP) offers overall balanced properties (mechanical, thermal, chemical, drawability, etc. Characterization of thermal stability and degradation processes at temperatures useful ranges of HMSPP-Ag compounds with different formulations and processing conditions was carried out by differential scanning calorimetry (DSC). Presence of silver nanoparticles also slightly improves thermal stability of HMSPP-Ag compounds thus enabling easy processing; this could be done to the interaction between polypropylene chains and surfactant-coated silver nanoparticles. These interactions lead to a decrease in motion of polymer chains and also, they act as nucleating sites for crystallization. XRD diffraction patterns of HMSPP-Ag compounds show presence of silver since the main diffraction peak of silver is located at typical 2η angle. Future tests will be done for biocide activity evaluation.

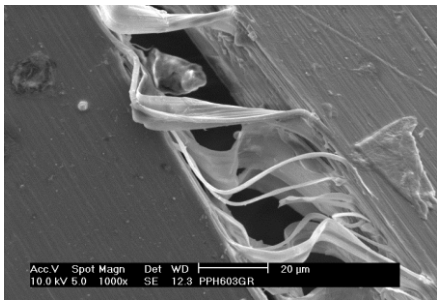


Figure 46. PP nanostrucure

Development of thermoplastic starch films and packing products from renewable sources

Starch is a natural, renewable, biodegradable polysaccharide produced by many plants as a storage polymer. It usually has two major components, a largely linear amylose of molecular weight between one thousand and one million and amylopectin, having the same backbone as amylose but with a myriad of α-(1→6)-linked branch points. The most commercially important starch for the development of thermoplastic starch is corn in most countries, however in tropical countries starch from tapioca appear to be a much more interesting source. Native starch occurs in the form of discrete and partially crystalline microscopic granules that are held together by an extended micellar network of associated molecules. A small amount of water is always associated with this structure, therefore the use of this bound water as expansion agent is an opportunity to developed foam with very low impact to the environment.
Starch is not truly thermoplastic as most synthetic polymers. However, it can be melted and made to flow at high temperatures under pressure and shear. If the mechanical shear becomes too high, then starch will degrade to form products with low molecular weight. Addition of water or other plasticizers enables starch to flow under milder conditions and reduces degradation considerably. However, the thermomechanical stability is strongly reduced by the addition of plasticizers. This project has developed a mixture of starch and nanoparticles for production of active and smart films and it was also developed very resistant packing with the addition of natural fibers.

Figure 48. Foams of Starch and Sugar cane bagasse for packing application

New hydrogels and silicone matrix as polymeric biomaterials for applications in the heath and cosmetic industry

New hydrogel dressings were developed from PVP and PVA. Hydrogels were synthetized by radiation induced crosslinking and freeze and thawing method, without the use of extra chemicals, usually very toxic. Multiple drugs were incorporated to these hydrogels and due to its characteristics, special formulations were developed. For instance:

- Hydrogels with Assai oil as powerful radical scavenger for anti-aging properties;
- Hydrogels with resveratrol (from grapes) as powerful radical scavenger for anti-aging properties;
- Hydrogels with algae extract for new skin formation and skin quality;
- Hydrogel with nanosilver incorporation to avoid contamination of wounds.

Silicone matrix were also used to encapsulate essence oils with repellency activity.

Figure 49. Silicone matrix with natural oils for repellency

Microwave and ionizing radiation assisted degradation of rubber and heavy oil

The saturated polymer systems (rubber) and butyl rubber (BR) used in the manufacture of tires and rubber chlorobutyl and bromobutyl used to make inner-air in the tires, electric wires and cables and has high resistance to the action of oxygen, ozone, solar radiation and bacteria, thus contaminate the environment for long periods of time. The re-use of these polymers also reduces the emission of substances give (aliphatic) in the atmosphere. The objective is to develop processes of controlled degradation (devulcanization) of butyl rubber and halobutyl rubbers (chlorine and bromine) using radiation of Co source. The development of combined methodologies as thermal degradation and ionizing radiation produce recycled material with characterized properties to be applied in new formulations.
Several types of petroleum hydrocracking and hydrotreating processes can benefit from microwave technique. It has recently been studied all over the world to identify (qualitatively and quantitatively) and define the mechanism of microwave-material interaction. Rubbers devulcanization is one of the main stream of this line. The application of this process in our country is a very recent field and has been studied as a new tool in materials processing for rubber devulcanization, which uses high temperatures and shear and also for heavy oil degradation as well as desulfurization of oil and diesel. The knowledge of this technology is important to begin the development process in industrial scale and consequently in reducing the environmental pollution caused by these kinds of residues.
Microwaves are a form of electromagnetic energy in the frequency band from 300MHz to 300GHz (not ionizing radiation). Industrial microwave processing is usually accomplished at a frequency of 2.45GHz (which corresponds to a wavelength of 12.24cm) to avoid interference with telecommunication and cellular phone frequencies. Microwave processing offers numerous advantages in relation to conventional heating methods (convection or conduction), where the material surface heats first and then the heat moves inward. One of the most important characteristics is saving energy, because the material absorbs microwaves readily (the heat is generated from the inner parts to the surface of the material) reducing the processing time. Also the selective energy absorption allows heating in specific points of the material. This process is environmentally clean because it reduces pollutant emission. Finally, the microwave heating requires no appreciable amount of time to effect temperature changes such as conventional methods and when the microwave device is turned off the effect of these electromagnetic waves are instantaneously stopped.

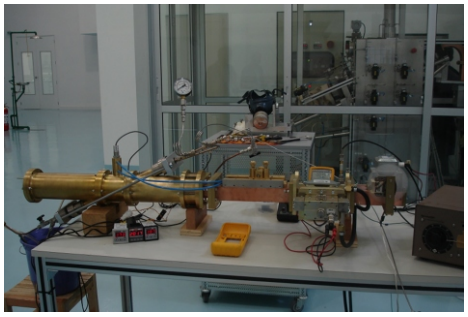


Figure 50. Microwave system

Development of nanohybrid materials and shrinkage evaluation

Dental filling materials used to repair decayed tooth structure are dental amalgam and composite materials based on acrylics. Photo-activated composites started to be used in the 60's, and are nowadays widely applied for dental restorative procedures. Polymerization of composite filling is considered to be an important factor in achieving longevity of the restorative treatment. Contraction induces certain amount of stress, which is transferred to surrounding dental structures (dentine and enamel).
The objective of the Phd work of Luiza M.P. Campos, oriented by Dr. Duclerc F. Parra, is the study of the dimensional changes in tooth and resin, produced by light-induced polymerization of dental composite filling. The first part in the study of shrinkage of different formulations was done with evaluation of thermo-mechanical analysis (TMA). The second part of the project was conducted in the University of Porto-Portugal using Digital Holography (DH) and ESPI. Both techniques present some characteristics that make them well adapted to this study; a high resolution can be achieved in non-contact displacement field measurements with small objects. All the preparations were placed in a digital holography or ESPI set up to obtain the holographic recordings. A blue led lamp (420nm - 480nm) is used to induce composite polymerization, and the real time deformation during resin cure was recorded. Experimental data was post processed for the deformation assessment. The study won the price Francisco Degni in the Scientific Forum of the CIOSP Congress-SP - Brazil and was presented at International Congress of ADA - Orlando, USA in 2010.

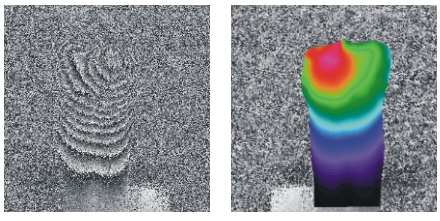


Figure 51. Toth deformation by digital holography

Materials and Nanotechnology

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Honor Mentions and Awards

The Brazilian Society of Science in Laboratory Animals (SBCAL) honored the work "Influence of ionizing radiation on well being of animals producing anti ophidic serum", realized by Nanci do Nascimento, Miriam C. Guarnieri, Pedro C.L. Oliveira and Roberto Rogero, during the XI Brazilian Congress of Science in Laboratory Animals and the II Forum of Ethic Committee on Animal Use.

Environmental Science and Technology

Ipen's water quality monitoring

Environmental Science and Technology

Introduction

The Program on Environmental Science and Technology developed at the Chemical and Environmental Technology Center comprehends environmental chemistry (water, soil and atmospheric chemistry), clean technologies (desulfurization of diesel and oil, biodegradable polymers and structural modification of polymers, recycling, pyrolysis of dangerous chemicals by molten salt technology), nanotechnology (magnetic nanoparticles, dendrimers, nanobiomarkers, catalysers) and chemical characterization of nuclear fuel and nuclear fuel cycle waste (chemical and isotopic characterization).
The Chemical and Environmental Technology Center was established in 1995, as an evolution of the former Department of Chemistry Engineering (1970). The program on environment science and technology was structured as consequence of the continuous growth of environmental activities on areas related to nuclear programs of IPEN. Moreover, it was an answer towards the society concerning the climate changes and biodiversity preservation.
All activities of research and development, services, supervision of graduate and under graduated students and courses performance at the center were related to the development, improvement and establishment of new technologies.
The highlights of this period (2008-2010) were:

- Development and use of modern analytical technology for the characterization of persistent pollutants and endocrine disrupters (metals, PAHA's, PCBs, Pesticides, hormones, surfactants and plasticizer) in order to evaluate water quality;
- Atmospheric chemistry & Greenhouse gases: Evaluating an estimation of surface trace gas fluxes from aircraft measurements above the Amazon;
- Cooperation with SABESP (Water and Sewage Company) and CETESB (State Environment Agency) in program for the development of public policies;
- Studies and development in biodegradable polymers, polyolefins and advanced methods for polymer and rubber recycling and re-use;
- Studies and development of recycling technology re-use of materials, waste storage and decontamination;
- Clean Technologies: Safe decomposition of organohalogenated pesticides by molten salt oxidation;
- Synthesis and development of magnetic nanoparticles, biosorbents, nanobiomarkers, zeolites and ceramics applied to environmental monitoring and wastewater treatment;
- Establishment of procedures and techniques for nuclear forensic investigations;
- Recovery of thorium and rare earth processing technologies;
- Certification and maintenance of the Quality Management System - ISO 17 025;
- Environmental Monitoring program (EMP-Q) to assist the nonradioactive chemical at IPEN;
- Non-Radioactive Chemical Waste Disposal Program

These achievements were conducted with support from national and international funding agencies - FAPESP, CNPq, FINEP/MCT, CAPES, IAEA, NOAA, NERC, NASA as well as cooperation with partners and clients, such as SABESP, CETESB, INPE, INPA, USP; Braskem, Biolab Sanus and Petrobras.

Environmental chemistry and water science

The global importance and vulnerability of our water supply, both in terms of quantity and quality has been well documented and, although water is a renewable resource, it is also a finite resource. Water, vital to both human health and ecosystem sustainability, is under increasing pressure as urbanization and agricultural intensification increase and, as such, it is essential that we improve our understanding of the types, and complexity and potential impacts of chemicals that are increasingly being released into the environment.

It is recognized that the pollution influences living organisms, humans included, both directly (by affecting their health) and indirectly (via contamination of food and abiotic compartments). Heavy metals and organic compounds, such as polyaromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), volatile organic compounds (VOCs) and pesticides, have been the center of attention for a long time.

Environmental and Analytical Chemistry focus activities in these areas by bringing together academic staff with a common research interest in understanding a variety of aspects of natural and polluted present-day environments and ancient environments. Since these are complex systems, studies of both organic and inorganic species of natural and anthropogenic origin rely heavily on the use and development of modern analytical methods. Scientific Cooperation programs supported by CNPq and FAPESP in partnership with Environmental Agency State (CETESB) and SABESP were developed.

Historical reconstruction of the pollution Guarapiranga Reservoir and diagnostic of sediment quality of São Paulo Metropolitan Region (SPMR) water supply reservoirs facing their management (thematic project set with Botany Institute)

Freshwater is a natural resource that has been under strong pressure, to the point of becoming one of the greatest XXI Century threatens. Under this water crisis context, the São Paulo Metropolitan Region (SPMR) is emblematic. This research will adopt innovative approaches that consist of the paleolimnology-neolimnology integration, as well as of the use of antropogenic impact multi-proxies. It is based on three main approaches: (a) evaluation of the eutrophication process evolution and the historical antropogenic impacts of Guarapiranga Reservoir (1906/2009-present); (b) characterization of multi-proxies present in recent sediments of 30 reservoirs circumscribed in the SPMR water supply area; and (c) integration of approaches (a) and (b) to elaborate a diatom-phosphorus inference model that will allow estimation of the past phosphorus levels of the Guarapiranga Reservoir water, as well as the positioning of the other reservoirs along the trophic gradient reconstructed. Paleolimnology will include the reference levels (before impact) of Guarapiranga Reservoir, and will be evaluated by ^{210}Pb dated sediment core. Neolimnology will include variables collected from the water column and superficial sediments of 30 reservoirs (20 water supplying ones). Multi-proxies will include eutrophication bioindicators (diatoms), granulometry, organic geochemistry (TOC, TP, TN, ^{13}C, ^{15}N) and sterols, geochemistry of persistent pollutants (metals, PAHs, PCBs, pesticides, hormones, surfactants and a plasticizer). Elaboration of the inferential model will be accomplished by using quantitative diatom species distribution in water and recent sediments. Watersheds of reservoirs will also be analyzed concerning physical environment (geology and geomorphology) and also land use and occupation. Above information will contribute towards the proposition of targets to support recovery and/or conservancy, and the elaboration of environmental scenarios, providing innovative subsides for the SPMR water supply reservoir management. The Laboratories of Chemistry and Environmental Diagnosis Center will conduct the analysis of metals and organic compounds evaluating endocrine disrupters in the samples colleted.

Water quality evaluation of the Taquaruçu Grande sub-basin and its interference area in the reservoir of the Luís Eduardo Magalhães hydroelectric power station - TO, Brazil

This work aimed at verifying the physical, chemical and biological contribution of the Taquaruçu Grande sub-basin to the reservoir of the Luís Eduardo Magalhães hydroelectric power station, in the medium Tocantins - TO, Brazil. Data were collected quarterly in the rainy and dry periods from January 2007 to October 2008, in eight points located in the sub-basin and in the reservoir. Limnologic variables were colleted and analyzed in accordance with CETESB (2006) and APHA (2005), as well as metals and trace elements analyzed by inductively coupled argonium plasma (ICP-OES) atomic absorption spectrometry (AAS) with graphite furnace or hydride generation. Biologic samples were collected with 20 µm plankton net, fixed in Transeau's solution (proportion 1:1) for qualitative analysis and in acetic Lugol's solution for the quantitative analysis made in a Olympus BX41 microscope. Electric conductivity, water temperature, total dissolved solids, phosphorus and pH showed higher concentration in the lentic points than in the sub-basin, with higher parameters in the rainy period, which is attributed to the amount of allochthonous matter carried by the rain to the reservoir. Nitrate and fixed solids show higher concentration in the reservoir in the dry period. Eutrophycation conditions have become more intense with the rise of the dry weather and can be a consequence of organic matter decomposition processes, which release nitrogen compounds. The higher concentration of fixed solids in the dry period is probably related to the low depth of the points,

which favors the re-suspension of sediments by the action of winds and local hydrodynamics. Ba, Na, Si, K, Al, Mn, Zn, Li, Mg, P, Ca, Fe and Ag were present in all points in most of the sample period. Samples from October 2007 presented high concentrations of B, Al, Cr, Ni, Cu, Zn, Mo, P, Co and Ag mainly in the sample points of the sub-basin, associated to the low depth of the streamlets, which in this period shown shallow water favoring weathering. Al, Ag, Be, Co, Cr, Cu, Fe, Mn Mo, Ni, P and Zn presented samples in discordance with CONAMA 357. The phytoplankton was characterized by presenting 227 taxons, 95 genus, distributed in 13 taxonomic classes. It was observed a spatial variation of species richness, with a lower number of species in the microbasins due to lotic conditions of these places, and a higher number of taxons in the reservoir. High stability of the water column and elevated nutrient availability propitiated an expressive phytoplankton development in the lentic points similar in composition and density.

Hydrobiological aspects of Paraibuna and Paraitinga dams, São Paulo, with emphasis to phytoplankton community

The inland waters are less than those of the Earth's surface and are extremely important for the survival of living things, and that´s why the concern for quality increases daily. The dams Paraibuna and Paraitinga, located in Paraibuna city, state of São Paulo, total 224 km^2 of extension of water surface, with the current function of generating electricity. In order to analyze the water quality of these dams, this work presents a diagnostic physical, chemical, and biological, which was used in the phytoplankton community and calculation of quality indexes. Four samples were taken at nine sampling points distributed along the dams. The physical and chemical analysis of water showed low concentrations of nutrients such as nitrate and phosphate. Most metals and trace elements analyzed were within the limit established by federal law. The element phosphorus is presented above the values established by CONAMA Resolution 357/05 on all sampling points in the month of May, but this fact seemed not to influence the phytoplankton amount. High levels of dissolved oxygen and transparency, low levels of turbidity, conductivity and dissolved solids, pH close to neutrality, together with high diversity and low dominance of phytoplankton, with a predominance of green algae, showed that the dams still retain features of aquatic environment preserved, which suffer little influence of anthropogenic factors. However, the presence, even at low densities, of cyanobacteria and the existence of plantations and livestock activities on the borders of these dams deserve special attention in terms of their correct management not to become harmful factors to the quality of those waters.

The monitoring process of the surface water with dense pollution in the Pirajuçara Stream, metropolitan area of São Paulo, Brazil

The water quality is important in any location, is an indispensable condition for the cities development considering the social and economic aspect and also to keep the life quality. The main aim of this project was the development of a non-conventional biomonitoring process which shows the environmental diagnosis using physical, chemical and biological measurements of the Pirajuçara Stream water, located on the Metropolitan Area of São Paulo. This stream presents high levels of environmental degradation due to raw sewage discharge and the pressure of unplanned urbanization. A ceramic sampler have been developed to monitor the surface water quality and it has been capable of adsorbing toxic compounds dissolved in water column and it also acts as an artificial substrate for the establishment of diatom biocenosis. The physical and chemical analyses of the water quality shows high levels of contamination, by typical domestic sewage compounds and the suspended solid particles enhancement, resulting of soil leaching process. As expected it was measured the high content of nutrient enrichment - nitrogen and phosphorus, as an evidence of the eutrofization state. The diatom biocenose was represented by 67 taxa, in specific level, divided in 27 genera with 20 families. An important representative genus, as for the number of taxa was: Nitzschia with 9 taxa, followed for Eunotia with 8 taxa and Navicula with 6 taxa. Nitzschia and Navicula are widely cited in literature as important representative genera indicator for water nutrient enrichment with low dissolved oxygen concentrations. It was possible to observe in dry season the presence of 49 taxa and 39 were exclusive ones. Most of the species were identified in dry season due to the reduction of the flow rate and velocity of the water and the effect allows better development and fixation of the algae community. The biological water quality index was calculated for all monitoring samples and shows the characteristic values for polluted waters. The levels of pollution were confirmed by the presence and identification of the species with high percentages of relative abundance and high or very high tolerance to eutrofization and organic pollution, characterizing strong and very strong polluted waters. The results obtained in this project allow to conclude that the diatomite has favorable properties to be used in water quality monitoring processes of urban stream showing information about soluble toxic compounds dissolved in water column and it allows the development of the indicator algae community which confirms the high pollution levels in the Pirajuçara Stream.

Determination of selenium in underground water using Atomic Absorption Spectrometry with Graphite Furnace (GFAAS) and Hydride Generation (HGAAS)

Report carried out for CETESB in 2006 showed that 80% of the 645 cities are totally or partially supplied by underground water, and for 13 river basins of São Paulo State, the underground water is the priority source for potable water supply. The use of underground water in different sectors reinforces its importance and highlights the concern about their protection and quality. The northwest region of São Paulo state is mainly supplied by underground water of the Guarani aquifer extracted by tubular wells, which presents some occurrence of Selenium. In the present study, Selenium was observed and evaluated through the comparison of high sensitivity and selectivity methods as the atomic absorption spectrometry using electrothermal atomizaton (GFAAS) and the hydride generation (HGAAS). Underground water samples of four deep tubular wells of Guarani aquifer in the northwest of São Paulo State were collected for measuring the selenium element. In addition to selenium other metals were analyzed (B, Al, V, Cr, Mn, Ni, Cu, Zn, Mo, Sn, Li, Be, Mg, P, Ca, Fe, Ba, Co, Na, Si, K, Ag, Cd, Pb, Sb, Hg and As). The characterization of selenium was carried out with quarterly sampling regularity, in the period of March of 2006 to March of 2007, totalizing five campaigns. The metal analyzed in this study was selected according to the requirements of the legislation for potable water quality evaluation, establishing permissible maximum limits for metal concentration. The results had shown the concentrations of Se are above the maximum permissible limits and the techniques electrothermal atomization (GFAAS) and hydride generation (HGAAS) demonstrated equivalence in their results. The results also demonstrated that the presence of Se can be associated to mineralogical factors (as geologic formation of the region) and the applied methodologies were efficient with similar answers for different operational factors and expenses.

Policyclyc aromatic hidrocarbons study at São Vicente and Santos Estuaries SP using diatomite as adsorbent material

Diatomite spheres were used as an adsorbent material for policyclyc aromatic hidrocarbons (PAH) adsorption at water column for the identification of the main contamination sources at two estuaries of Baixada Santista. The samples were held at Santos Estuary (site 1) and São Vicente Estuary (site 2) during the year 2006, at two sites, and in 2007, only at site 2. Together with PAH analyses, some parameters of water quality were measured. This methodology is unique with unprecedented published study, and some tests were conducted focusing on the development of PAH extraction from diatomite and the recovery tests. Between the tested methodologies, the PAH extraction was more efficient using mechanic shaking and ultrasonic process. The recovery tests indicated better results for PAH with high molecular weight and the adsorption efficiency occured in a period of 15 to 30 days. A high influence of the environmental factors was observed at PAH adsorption and the results showed different magnitude when conducted at laboratory and at the natural environment. The water quality result showed that the site 2 was more influenced by a river system in comparison to site 1, located at estuary mouth, with higher salinity values. The dissolved oxygen was lower at site 2 indicating higher organic matter contribution. The turbidity analyses also showed higher values at site 2 probably due to the diffuse pollution release and the reduced natural water flow in this place. The powder diatomite characterization showed the main composition of well preserved diatoms frustules. On the other hand, the diatomite characterization made after the diatomite going through the process to be used at samples sites, showed the frustules with unpreserved structure, condensed and with high loss of integrity. The PAH concentrations were higher at site 1 than in site 2, probably due to biggest environmental contamination from the harbor (Porto de Santos). Both of the samples sites showed the pyrolytic as the main PAH sources which is probably the result of the combustion process and atmospheric deposition in water column. These sources can be associated to the atmospheric emissions from Cubatão Complex and refineries. The lower PAH concentrations with low molecular weight at site 2 indicate light contamination from petrogenic source, like those related to harbor activities. The results obtained for PAH analysis with the diatomite spheres placed in different depth on water column indicate higher concentrations near the surface, probably due the atmospheric deposition. The measurements performed at water column were kept almost constant and near the sediment, the concentration started to increase again, indicating the presence of sediment movement and re-suspension.

Pesticides residues in water treatment plant sludge: Validation of analytical methodology using liquid chromatography coupled to Tandem mass spectrometry (LC-MS/MS)

Tine evolving scenario of Brazilian agriculture brings benefits to the population and demands technological advances to this field. Constantly, new pesticides are introduced encouraging scientific studies with the aim to determine and evaluate impacts on the population and on environment. In this work, the evaluated sample was the sludge resulted from water treatment plant located in the Vale do Ribeira, São Paulo, Brazil. The technique used was the reversed phase liquid chromatography coupled to electrospray ionization tandem mass spectrometry. Compounds were previously liquid extracted from the matrix. The development of the methodology demanded data processing in order to be transformed into reliable information. The processes involved concepts of validation of chemical analysis. The evaluated parameters were selectivity, linearity, range,

sensitivity, accuracy, precision, limit of detection, limit of quantification and robustness. The obtained qualitative and quantitative results were statistically treated and presented. The developed and validated methodology is simple. As results, even exploring the sensitivity of the analytical technique, the work compounds were not detected in the sludge of the WTP. One can explain that these compounds can be present in a very low concentration, can be degraded under the conditions of the water treatment process or are not completely retained by the WTP.

Environmental and occupational exposure assessment levels to emerging pollutants, metals, endocrine disruption and organic compounds

Endocrine disruptors are chemicals substances, synthetic or natural, which can interfere in endocrine system from humans and animals. Emerging contaminants have received much attention in recent years, principally organic compounds. Pharmaceuticals, personal care products, and other potential endocrine disrupters are part of a large and diverse group of organic compounds, labeled as emerging contaminants. Many of these contaminants are introduced in the environment by way of wastewater systems and their fate. The consequent environmental risks are being considered through ecotoxicological studies. Considering that re-use of wastewater is becoming more common worldwide, driven by pressure on other sources of fresh water, it is important to assured good water quality for future generations. Several chemical substances suspected of acting as endocrine disruption are currently being used in industrial and agricultural activities in Brazil. The effects of the endocrine disruptors related to human health are not totally known, and then the need to implement a national program to evaluate such impacts. Scientific Cooperation programs supported by CNPq and FAPESP in partnership with SABESP were developed focusing:

Evaluation of endocrine disruptors in water for public supply on Paraíba do Sul River region, Brazil. Development and validation of analytical methodology

This research line focuses on investigating the presence of some endocrine disrupters agents as pharmaceuticals, personal care and industrial products which may affect the quality of public water supply on Paraíba do Sul River Basin. The study aimed to develop and validate analytical methodologies to determine hormones, estrogens, plasticizers and other compounds considered endocrine disruptors, in drinking and surface water beyond effluents of a municipal sewage treatment plant at region of Paraíba do Sul River. The hormones stand out as endocrine disruptors because they are potentially active compounds in biological systems and are related to the origin of several types of cancers. The population served by the units of SABESP is around 11 million inhabitants.

Applying the validated analytical procedures for the samples, some compounds were detected at raw and drinking water, principally due to drought season. In this work it was used the technique of solid phase extraction (SPE) for extraction and concentration of samples, followed by gas chromatography coupled to mass spectrometry (GC-MS) for identification and quantification of compounds.

Figure 1. Paraíba do Sul River

Environmental impact evaluation of Pedroso Reservoir, Santo André / Brazil, by the construction of the beltway. Geochemical distribution of Polycyclic Aromatic Hydrocarbons (PAHs) in the region sediments

The focus of this project was to evaluate the environmental impact on the study region, characterizing water and sediments of the reservoir before and after the beltway construction, to compare the actual and initial health of this water resource. It was analyzed the actual and potential risk of contamination by polycyclic aromatic hydrocarbons (PAHs) in sediments and water, examining the main compounds from air pollution caused by motor vehicles that circulate in the region. The basic aspects of meteorology, hydrology and soil characteristics were related and evaluated the soil use and occupation around the Pedroso Park. An environmental diagnostic identifying the presence of PAHs in sediment and surface water was carried out. The investigation of impact agent presence which can contribute to Reservoir Pedroso impoverishment quality was realized, aiding the management plan for water resources of the Santo André city. The “Park Pedroso” reservoir is an area of Integral Protection Conservation, situated in the area of Billings basin, main potability water source of the MRSP. The area is rich in water resources, but has poor infrastructure. The Park Pedroso reservoir provides about 7% of drinking water in the city (54,3 million liters of water).

Adsorbent studies based on zeolites and ceramics applied to environmental monitoring and wastewater treatment

Measurements of physical, chemical and biological aspects for water quality evaluation of urban stream and the development of adsorbent material to monitor and remove phenol and phosphorous from surface water under eutrophic condition as rivers, lakes and reservoirs with dense pollution located in the Metropolitan area of São Paulo are studies under development.

Figure 2. Macrophytes collected at Guarapiranga water reservoir located in metropolitan area of Sao Paulo

The combustion of high ash content coals promotes a serious environmental problem in southern Brazil. It is in the south, in the States of Rio Grande do Sul, Santa Catarina and Paraná, that the coal mines coal-fired power plants are located. The States of Rio Grande do Sul and Santa Catarina show areas that are already environmentally degraded with the resulting contamination of both surface and ground waters.
Brazilian coals are characterized, among others things, for very high ash content ranging between 45 and 60%. This represents 4.0 x 10^6 tons/year of ashes produced in 2005. Most of these ashes are deposited randomly in landfills and has contributed to the deterioration of the surrounding environment. Since just 30% of that total is commercialized for the production of building materials (bricks, blocks, cement), it is necessary to search for new alternative uses for this abundant residues and give a high added-value to coal ash.
The Brazilian coal ashes consist, basically, of aluminosilicate with high silicon and aluminium oxide contents. Depending on its origin, the iron oxide contents can vary over a wide range. Since coal ashes are composed of a large amount of silica and alumina and also due to a low ratio SiO_2/Al_2O_3, they can be converted into zeolite by alkaline hydrothermal activation. Various types of zeolites can be obtained by changing the source of ashes or activation parameters. The zeolitic material obtained contains a non-converted part of coal ash and the zeolite content in the conversion product varies as a function of the coal ash properties and the conditions selected. Figure 3 shows the SEM (Scanning Electron Micrographs) images of zeolitic material synthesized from fly ash.

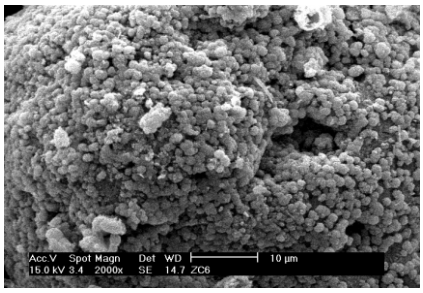


Figure 3. SEM photographs of zeolitic material synthesized from fly ash

Zeolites have uniform pore sizes and large surface area that make them very useful materials for a wide range of applications such as ion exchange, molecular sieves, adsorbents and catalysts.
The coal ash samples were obtained from a coal-fired power plant located in Figueira county, in the North of Paraná State, Brazil. The utilization of synthetic zeolites as adsorbent for the removal of metal ions and removal dyes from aqueous solution has been evaluated.
The results obtained in the project showed a great reduction in the pollutant concentration in treated wastewaters and demonstrated the high potential of the zeolites synthesized from Brazilian coal ashes as low-cost adsorbent material. The production of synthetic zeolites from coal ashes constitutes an alternative and noble use for a residue that has historically contributed for the degradation of large areas located in Brazil. The environmentally-friendly use of coal ash is important from the viewpoints of energy, economy and environmental strategy in order to realize the concept of sustainable development.

Toxic elements mobility in soils columns with coal fly ash

Coal ashes are a residue generated of the combustion of coal in power plant. This residue is composed of toxic elements and if it is inadequately placed on soil surface these elements can be leached and transported to underground water or absorbed by plants and animals, representing a risk to human health.
Studies of toxic elements mobility in soils are important because they can assist in the remediation and prevention of contaminated soils. The soils were collected in Estiva Gerbi (Figure 4) and Piracicaba cities in the State of São Paulo, in an area under native vegetation or ancient reforestation. Experiments of coal fly ash added on soils columns (Figure 5) were conducted to simulate soils contamination by acid leaching. Toxic elements have been determined in the coal fly ash, soils and column percolated samples to evaluate mobility of elements. This study has been financed by São Paulo Research Foundation (FAPESP).

Figure 4. Soil collect in Estiva Gerbi City in the State of São Paulo

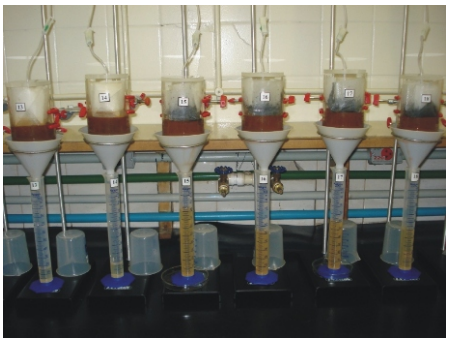


Figure 5. Soil columns and soil with coal fly ash columns

Environmental diagnosis of soil vulnerability to toxic metals contamination from foundry industries of Loanda, Paraná

Foundries and electroplating industries have been investigated to improve industrial processes, reducing the external environment impact and promoting human health and welfare. There are 23 industrial plants located in the municipality of Loanda, Paraná, Brazil. The main goal of the project was the evaluation of the soil contamination by toxic metals in the vicinities of their most profitable unit. The soil characterization was performed by physical-chemical measurements and their extractable toxic metals (Ag, Al, Cd, Cr, Co, Cu, Fe, Mg, Mn, Ni, Pb, V, Zn) content. They were analyzed after the HNO_3 extraction, and also after EDTA extraction. This procedure was adopted to provide additional information about the toxic metals mobility on soil samples. According to results, the soil is composed of sand with siliceous acid properties, with low content of clay, organic matter and cationic exchange capability. The higher extractable toxic metals concentrations were found for Cu, Zn, Cr, Ni and Pb in the boundary of the industrial plant and their vicinity. The extraction results indicate that the soil toxic metals contamination can be considered stabilized with an exception for high mobility elements as Cr and Ni. It was an indication of the need of continuous monitoring and control for Cr and Ni content including periodically groundwater evaluation. Production process must be improved in order to avoid further contamination.

Green House Gas studies in Amazon Basin. Vertical profiles of carbon dioxide and other trace gas species over the Amazon Basin using small aircraft

This project is part of the LBA project (Large-Scale Biosphere-Atmosphere Experiment in Amazônia). Since December 2000 vertical profiles of CO_2, CH_4, CO, H_2, N_2O and SF_6 have been measured above central Amazônia (Over Tapajós National Forest, a primary forest in Para State - SAN). In January of 2010 it was add vertical profiles over more 3 places in Amazon Basin: Rio Branco, Acre state (RBA), Tabatinga, Amazonas state (TAB) and Alta Floresta, Mato Grosso state (ALF). In the coast, more 3 sites were started: Salinopolis, Para state and Natal, Rio Grande do Norte state and Arembepe, Bahia state.
Samples are collected aboard light aircraft between the surface (300m) and either 4.4 km (SAN, RBA, TAB and ALF) using the NOAA/CMDL semi-automatic portable flask package (PFP). The PFP consists of 17 (SAN) and 12 (RBA, TAB and ALF) glass flasks with 750 mL volume that are pressurized to about 3 bar to enable measurements of all the gases mentioned above. Until the end of 2003 the PFP's were sent from Boulder, Colorado to Brazil, where they are filled, and then sent back to Colorado for analysis. The strategy was changed to increase the frequency of measurements.
In order to accomplish this, a replica of the NOAA/CMDL trace gas analysis system was constructed and installed in IPEN/LQA starting in May 2004. The equipment set up in Brazil is capable of high-accuracy and high-precision measurements of CO_2, CH_4, CO, N_2O and SF_6 in the flask and PFP samples. All measurements are calibrated with standards from NOAA to internationally accepted scales. The fluxes are determinate for each profile by Integration Column Technique using atmospheric measurements from aircraft profiles over the 4 sites (SAN, RBA, ALF and TAB) subtracted with background concentration of air when cross the Brazil coast. The CO_2 background was determinates using a co-measured SF_6 as a transport tracer. Two NOAA/CMDL background sites, Ascension Island (ASC) located in the Atlantic Ocean (8'S, 14'W) and Barbados (RPB) located in the Atlantic Ocean (12'S, 59'W) were used to calculate the fractions of air arriving at the sites studied. The fluxes are related with the time that air mass take between coast until profile sites. Back trajectories from HYSPLIT model were calculated for every profile each 500m height to determinate the time of the air mass between coast and the sites.

Ozone precursors in São Paulo City

The pollution in the urban area of São Paulo city is mainly related with vehicular emissions and secondary with industrial emissions. The metropolitan area of São Paulo suffers from heavy air pollution, for both gases and aerosol particles. The vehicular emissions are responsible about 98% of CO, 97% of HC, 96% of NOX, 50% of aerosols, and 55% of SOX. Volatile organic compounds speciation and the contribution to the ozone formation have been investigated. The research were made with different ways to determinate vehicles emissions like tunnels campaigns, dynamometer studies with different kind of fuels (ethanol, gasoline, diesel) and vehicles (light and heavy). OZIPR mathematical model was used to determinate the principals VOCS in ozone formation. Financing by NASA, NOAA, CNPq, FAPESP and NERC.

X-Ray Fluorescence techniques applied to environmental, biological and geological studies. Chemical characterization of nuclear and non-nuclear materials

The X-Ray Fluorescence Laboratory has worked in environmental, biological, geological and nuclear areas establishing new analytical methodologies for different matrices, such as those used in lubricating oils, domestic dust, stainless steels, nuclear fuel, whole blood and serum, treated woods, and others. The R&D activities have been supported by the IAEA, CNPq and FINEP organisms. The main R&D activities, carried out and ongoing, are listed below:

Human resource
The X-Ray Fluorescence Laboratory has offered, annually, graduate (TNM 5813, TNM 5767) and undergraduate (IPN0014) courses, besides many extension courses for universities: (UFCG: Universidade Federal de Campina Grande, UFRN: Universidade Federal de Rio Grande do Norte, UFBa: Universidade Federal de Bahia, USP/Riberão Preto), research institutes (CDTN/CNEN: Centro de Desenvolvimento de Tecnologia Nuclear, IC/SP: Instituto de Criminalistica/SP, CEA: Centro Experimental de Aramar, Iperó/SP) and industries (Gerdau Aços Especiais Piratini, Grupo Labmat, Denso do Brasil Ltda., MMX: Corumbá Mineração e Metalurgia Ltda., Invensys Controls Ltda., NSK Brasil Ltda.).

Residue recovery
Brazil produces circa 1bi L/year of lubricating oils, and around 650 mi L/year are consumed by the automotive sector. Only 20 - 30% of used oils are treated by conventional processes, and their re-using and recovery treatments are important for the environmental policy and economic aspects. The efficiency and technical feasibility of the Advanced Oxidation Process (AOP) using gamma radiation (^{60}Co source) were evaluated. The X-Ray fluorescence analysis showed inorganic elements removal, mainly of sulphur, calcium, iron and nickel, at acceptable levels by environmental protection law for oils re-use. The different absorbed doses (100, 200 and 500 kGy) showed the organic compounds degradation. The compounds, such as tridecane, 2-methyl-naphthalene and trietilamina-tretramethyl urea, which have important industrial applications, were identified by GC/MS analysis, showing that the AOP process is a viable and promising treatment for used lubricating oil recovery.

Indoor pollution
House dust has been identified as an important exposure source of inorganic and organic substances, potentially toxic for children and adults. House dust pollution in Pirituba, Freguesia do Ó, Jaraguá and Perus neighborhoods of São Paulo metropolitan region was studied. The X-Ray fluorescence analysis showed the presence of elements, such as Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, Zn, Br, Rb, Sr, Zr and Pb. Natural and anthropogenic contributions were identified. The elements P, S, Cr, Ni, Cu, Zn and Pb were classified as being significant and extremely enriched in the dust. Cr, Ni, Cu, Zn and Pb were present in significantly elevated concentrations, in relation to the total exposure values (ingestion, inhalation and skin contact). The phthalates are classified by WHO (Word Healthy Organization) as toxic compounds, due to their correlation with respiratory diseases, dermal toxicity and endocrine disturbance. The compounds BBP (benzyl butyl phthalate), DnBP (di-n-buyl phthalate) DEHP (2-ethylhexyl phthalate), DEP (diethyl phthalate) and DMP (dimethyl phthalate) were found in house dusts, showing the release of these compounds from plastic materials used in house stuffs such as domestic utensils, computer, furniture and toys.

Material sciences
A continuous development has been outlined to improve API (American Petroleum Institute) grade of alloy steels, due to the increase of gas and petroleum demanding in the world. A comparison between X-Ray fluorescence (WDXRF) and Optical Emission (OES-spark) spectrometries, for the analysis of API grade steel plant samples, was outlined. The main influence source in the chemical analysis was evaluated for designing experiments. Major and minor elements (Al, Si, P, S, Ti, V, Cr, Mn, Co, Ni, Cu, As, Nb, Mo and Sn) were determined and parameters, such as precision, uncertainty, accuracy, repeatability and reproducibility, were evaluated.

Medicinal and Veterinary Clinic
Interval reference values in whole blood are used for human healthy control and the diagnosis of several diseases. Na, Mg, P, S, Cl, K, Ca, Fe, Cu and Zn interval reference values, in Brazilian healthy population, were determined by EDXRF spectrometry. A comparative study was outlined with NAA (Neutron Activation Analysis) data. Interval reference values for laboratory animals are

important for their healthy control during the experiments. Na, Mg, P, S, Cl, K, Ca, Fe, Cu and Zn internal reference values were determined in gold hamster (Mesocricetus auratus). The production of hyper-immune serum (snake antivenom immunogloblulin) is considered imperative to prevent death and amputation in envenomation accidents, demanding a strict biochemical and metabolic control. The interval reference values, for Na, Mg, P, S, Cl, K, Ca, Fe, Cu and Zn, were determined in Brazilian Crioula breed horses (*Equus caballus*) and the results were compared to NAA data. The comparative study presented results in agreement with the two techniques, showing the applicability of EDXRF technique in Medicinal and Veterinary Clinic analyses (CNPq financial support).

Wood preservation
Treated woods are requirements in the construction of houses and in the manufacturing of stakes, pillars and trails. The CCA treatment (cupper and chromium arseniate) is one of the most used processes for wood preservation. A new methodology using PXRF (Portable X-Ray Fluorescence), is being established for the determination of Cu, Cr and As in Brazilian treated eucalyptus (Eucalipts spp). This work is under development in co-partner study with IPT (Instituto de Pesquisas Tecnológicas, SP).

Metrology on Chemistry
The major elements determination (Na, Mg, K, Ca and P), using X-Ray fluorescence spectrometries, in water quality control is being established. (MCT/FINEP financial support).

Quality assurance
During the period, the annual audit by SGI/IPEN was carried out. U_{total} and Si determination assay in U_3Si_2 nuclear fuel was audited. The Laboratory participated in the "Proficiency test for X-Ray fluorescence laboratories" programs (PTXRFIAEA-05: marine sediment and PTXRFIAEA-06: grass sample) and "IAEA-CU-2009-02 Regional proficiency test on the determination of trace elements in algae and water" program (IAEA financial support).

Others
The X-Ray Fluorescence Laboratory has established different methodologies, using X-Ray fluorescence techniques, to support several research projects (internal and external co-partners). In the nuclear area, the chemical characterization of IEA-R1 nuclear fuel and Pb determination for radiogenic recovery was performed. In the air pollution study, several species of plants were chemically characterized (FM/USP and CENA/USP co-partners) and sulphur determination in diesel is ongoing (CENPES, RJ co-partner).

Chemical and isotopic characterization
The activities at the Grupo de Caracterização Química e Isotópica (GCI) emphasizes interdisciplinary research dealing with chemical and isotopic analysis providing isotopic, qualitative and quantitative analytical data in different fields of knowledge. Core competencies include analytical atomic spectrometry and inorganic mass spectrometry, gas and liquid chromatography, organic mass spectrometry, and chemical separations for both organic and inorganic trace analysis.

Environmental monitoring
The determination of metals and organic compounds in environmental matrices is performed routinely by the laboratory. During the period of 2008-2010 two projects were carried out in GCI's laboratory.
The first one has the objective to establish the environmental quality of the sediments from Laje de Santos Marine State Park (PEMLS) and in its surroundings. The main objective of this project is to support the management plans of the park and the public policies for its use. Sediment samples were collected from Santos-São Vicente Estuary as far as the Laje. Metals and organic compounds were analyzed. High contents of metals and organic compounds were identified in sediments of the Estuary. The influence of the estuary on the Laje is still under evaluation.
The second one was a collaboration with the Instituto de Pesca do Estado de São Paulo, and had the objective to verify the environmental quality and the quality of fish captured in Billing's Dam, São Paulo, aiming the health of the local fishermen. Samples of most consumed fish species in the region, water and sediments were collected monthly during one year.
Preliminary data demonstrated that despite the periodic discharges of contaminated water from the Pinheiros-Tietê rivers systems as well as domestic and industrial effluents, the levels of metals in the analyzed were within the limits established by the World Health Organization (WHO) and the Brazilian National Health Surveillance Agency being, in both criterium, suitable for human consumption.

Environmental biomonitoring
The identification of pollutants in the environment by means of selected organisms consists of biomonitoring the focus. Many different species have been used as bioindicator as plants, soft crab and fish.
Some plants can be used to evaluate the air pollution and, in this context, the *Tibouchina Aubl* - "quaresmeira" - has been proposed as bioindicator species for metals indication in the air. Therefore, one of the goals of this work is to evaluate seasonally the dry deposition of metals in the leaves of *Tibouchina Aubl* collected in the metropolitan São Paulo State, Brazil.
Beyond leaves of *Tibouchina Aubl*, crab and fish have also been considered as biomonitor of areas

such as the Santos Estuarine System. This contaminated area has already been studied with the purpose of environmental diagnosis and monitoring, mainly for metal concentration and predominantly analyzing sediment samples. In a new perspective, *Callinectes danae*, commonly known as blue crab, has been studied as a biomonitor of the presence of metals in this estuary. Moreover, it has been study the probable fingerprint that *Callinectes danae* may present due to this exposure, which may provide the possibility of characterization of the contamination dynamic of this estuary.
The Ariidae catfish *Cathorops spixii* and *Genidens genidens* also have been used in biomonitoring studies because previous works have demonstrated that these fish are efficient bioindicator for metal contamination in Santos-São Vicente estuary. Crabs and Ariidae catfish are distributed along the Santos Estuarine System and are exposed to sewage discharges, to effluents produced by industrial activities and by the harbor of Santos that may interfere in the bent habitat.

Environmental metallomics
Monitoring environmental pollution using biomarkers requires detailed knowledge about the markers, and many only allow a partial assessment of pollution. Another focus is connected to the presence of metals bound to biomolecules, which adds an additional dimension to metal-biomolecule and metalloprotein characterization - the field of metallomics. The metallomic approach considers the metallome: a whole individual metal or metalloid species within a cell or tissue. Metallomics studies initiate on 2009 Ariidae catfish from polluted and non-polluted areas in the Brazilian coastal. Several metalloproteins have been proposed as biological indicators of xenobiotics exposure. Among these, the metallothioneins (MT) are taken into consideration due to its important detoxification function of toxic metals. MT is a protein with 6-7 kDa and is great as exposure biomarkers to environmental contamination by metals.

Chemical metrology
Chemical Metrology program focuses on understanding the basic principles and science that support the identification and quantification of chemical species using the core analytical techniques in a wide variety of materials. The main activities include development of reliable methodologies for inorganic and organic trace analysis as well as the provision of certified reference materials (CRM's) used in the validation of analytical methodologies as well as in laboratorial inter-comparison programs. The chemical metrology program at GCI started more than twenty years ago with the beginning of the Quality Assurance Program including all activities associated to insuring that all measurements were made properly, interpreted correct, and reported with appropriate estimates of error and confidence levels. During the decade of 1990, a Good Laboratory Practice program was started, focusing on the organization, on processes and conditions under which laboratory activities are planned, performed, monitored, recorded and reported. The ABNT NBR ISO IEC 17025:2005 protocols were implemented. This norm was developed with the objective of demonstrating the capability of laboratories to produce accurate, repeatable, verifiable, cost effective, timely, and believable measurements analytical results. As a consequence of all these efforts, in 2007, the laboratory obtained the accreditation by the National Institute of Metrology, Standardization and Industrial Quality (Inmetro). Laboratory accreditation is a formal recognition by a third-party (national) authority of a laboratory's capability to perform testing, measurement and/or calibration activities. Since then, the accreditation has been renewed periodically. During the period of 2008-2010 the following activities were developed:

- Evaluation of uncertainty for stable nuclides isotopic measurements;
- Evaluation of uncertainty for the determination of metals in environmental, biological, forensics and nuclear samples;
- Participation in several inter-comparison programs involving biological, nuclear and foods samples;
- Development of in-house control samples provide day-to-day check of variability of the measurements due to various factors, e.g. storage effects, different operators, blanks, instrumental effects, environmental effects etc.;
- Participation on the project: Preparation, Characterization and Certification of Uranium Isotope Reference Materials;
- Preparation and certification of fish tissue certified reference material.

Clinical chemistry
Many metals play critical roles in maintaining life. Many metals have no known biological function and some of these are capable of disrupting essential physiological processes. Some are important for the structure of biological materials. Other stabilizes proteins in unique and active conformations, or structures. Metals also serve a chemically important role as essential components of many enzymes. These metalloenzymes are involved in the synthesis, repair and degradation of biological molecules, the release and recognition of certain biological signaling molecules, and the transfer of small molecules and electrons in crucial process such as photosynthesis and respiration. The main objective for this program is to gain a better understanding of how each metal causes human disease. During the period of 2008-2010 several collaborative studies with the Medicine Faculty, USP were performed involving individuals with diabetes Mellitus, Bipolarity, Alzheimer disease and Down Syndrome. The imbalance among essential metals in serum and blood samples were identified and assessed. GCI has established a partnership with São Paulo's Scientific Police to

develop an innovative research with the objective to evaluate the occupational exposure to metals and the worker health conditions in the Ballistic laboratory of the Criminalist Institute.
New methodologies based on finger stick protocol were developed in order to measure metals in blood samples. These new protocols were based on a single drop of the sample deposited in a Schleicher & Schuel nitrocellulose membrane allowing an easy collection and transport of blood samples, from distant locations, without the loss of integrity and sanitation restrictions.

Forensics science: Classical and Nuclear
Forensic sciences or forensics is the application of various scientific methods and principles to investigate criminal and civil actions which are of interest to the legal systems. This may be in relation to a crime or a civil action. GCI's activities in this field can be divided in two branches: Classical forensic and nuclear forensic.

- Classical forensic
CNEN, represented by GCI, has since the beginning of 2000 decade, a collaboration agreement with Superintendence of Scientific Police of the São Paulo's State, with the objective to develop public policies and new protocols and methods for forensic investigations. Since then, several collaborative studies, and investigations, were performed involving narcotics, adulteration of alcoholic beverages, crimes scene and gunshot residues (GSR). New protocols were developed allowing the identification of GSRs in fabrics and accessories, i.e. glasses, rings, earrings, watches etc. Based on the chemical signatures of the GSRs in the hands of shooters and on the fabrics, it was also possible to establish evidences for attribution related with the use of a firearm.

- Nuclear forensic
What would happen if a nuclear or radioactive explosive device were left in the center of a large city? Or whether extremists groups took possession of nuclear material? To whom would interest the smuggling of tons of geological radioactive materials? What would the consequences of a criminal discharge of radioactive water in a reservoir or a growing food? These and other related questions have been discussed since the first seizures of nuclear and other radioactive material were reported in the beginning of the 1990's, with the breakdown of the Soviet Union. Since then, nuclear and radioactive materials seizures have become a considerable concern due to the associated hazard to the public health, as well as to the environment. As a result, a new branch of science was recognized for the nuclear and forensic community: nuclear forensics. The first initiative in Brazil in this field was launched by GCI with the project: Establishment of Procedures and Techniques for Nuclear Forensic Investigations, sponsored by the International Atomic Energy Agency (Research Contract 1426-2009). The main objective of this project was to establish a permanent program in nuclear forensic science in order to enhance the national and international ongoing efforts to combat illicit trafficking of nuclear and other radioactive materials. In particular, it was expected to strengthen human technical resources in the main concepts and analytical procedures used in this field harmonized with the IAEA recommendations. The main achievement obtained with this project was the launching of the Brazilian Network Laboratories in Nuclear Forensic Science with the participation of 6 IPEN's laboratories, Federal Police and the Scientific Police of São Paulo's State. Using the capabilities of this network the first exercise was launched with the objective of creating a nuclear forensic databank of the main Brazilian nuclear materials.

Risk evaluation methodology and eco-efficiency analysis of techniques

Proposal of risk evaluation methodology for hazardous materials transportation
The increasing concern with the level of risk associated to the transportation of hazardous materials took some international institutions to pledge efforts in the evaluation of risk in regional level. Following this trend, the objective of this work was to analyze the most recent processes of analysis of risks from road transportation of hazardous materials. In the present work 21 methodologies of analysis of risks, developed by some authors and for diverse localities have been evaluated. Two of them, in special, have been reviewed and discussed: a method recently developed by the Swiss Federal Institute of Technology (Nicolet-Monnier and Gheorghe, 1996) and the strategy delineated by the Center for Chemical Process Safety CCPS (1995), taking into consideration the estimate of the individual and social risk. Also, the models of Harwood *et al*. (1990) and of Ramos (1997), adapted by Hartman (2003) have been applied to the reality of the roads of the state of São Paulo. The extension of these methodologies was explored, in order to find its advantages and disadvantages. As a study case the present work considered the ammonia transportation throughout two routes evaluating the reality of the roads of the state of São Paulo, including a significant parcel of evaluation in a densely populated area, getting the results using risk, at least, one of the methodologies mentioned above. The innovation proposed by this work was the research, the development and the introduction of two variables to the model considered by Harwood *et al*. (1990). These variables that influence in the value of the risk are: the age of the driver of truck and the zone of impact that is function type of product, period of the day where the transport was carried and the volume that has been transported. The aim of the proposed modifications is to let the value of the risk more sensible in relation to the type of the product carried and the age of the truck driver. The main related procedural stages with the quantitative

analysis of the risks for transportation systems are supported by the preceding methods for fixed installations. Special attention was given on to how to collect local information and estimate those coefficients that represent the actual conditions of the region considered in the presented study case.

Eco-efficiency analysis of techniques for disposal of urban solid waste

Municipal solid waste is one of the major problems of modern society. In this sense it is necessary to discuss solutions to the fate of waste generated daily, increasingly in larger amounts, being imperative to advance the search for alternatives, not only in search of new technologies and solutions, as well as the improvement of methodologies currently employed to solve this relevant issue, especially in large urban centers. This paper presents the results of a comparative economic-environmental assessment performed by a method developed by BASF, two potential technologies: landfill and incineration with energy recovery. This ecoefficiency analysis tool covers the methodology of Life Cycle Assessment (LCA), which is a technique to evaluate the inputs and outputs of matter and energy and the potential environmental impacts associated to all stages of resource extraction and their transformation, and the use and final disposal of the product. The data in the environmental assessment are presented according to major categories, using a weighting method developed by BASF and understand the consumption of natural resources, consumption of energy resources, air emissions, water and soil, potential for human toxicity, accidents work, occupational diseases and land use. Economic evaluation in this work took into account the costs involved in operation and maintenance of disposal techniques or treatment of waste. The comparative study found the treatment or disposal of 7.324109 billion kilograms of waste. As a result, the array of eco-efficiency indicates that the incinerator is the alternative that stands out, considering the environmental and economic profile, within the assumptions made in this study and taking into account which is a technique with energy recovery.

Validation of an analytical method for the determination of total mercury in urine samples using cold vapor atomic absorption spectrometry (CV-AAS)

Mercury (Hg) is a toxic metal applied to a variety of products and processes, representing a risk to the health of occupationally or accidentally exposed subjects. Dental amalgam is a restorative material composed of metallic mercury, which use has been widely debated in the last decades. Due to the dubiety of the studies concerning dental amalgam, many efforts concerning this issue have been conducted. The Tropical Medicine Foundation (Tocantins, Brazil) has recently initiated a study to evaluate the environmental and occupational levels of exposure to mercury in dentistry attendants at public consulting rooms in the city of Araguaína (TO). In collaboration with this study, the laboratory of analysis at IPEN's Chemistry and Environment Center is undertaking the analysis of mercury levels in exposed subjects' urine samples using cold vapor atomic absorption spectrometry. This analysis requires the definition of a methodology capable of generating reliable results. Such methodology can only be implemented after a rigorous validation procedure. As part of this work, a series of tests were conducted in order to confirm the suitability of the selected methodology and to assert that the laboratory addresses all requirements needed for a successful implementation of the methodology. The following parameters were considered in order to test the method performance: detection and quantitation limits, selectivity, sensitivity, linearity, accuracy and precision. The assays were carried out with certified reference material, which assures the traceability of the results. Taking into account the estimated parameters, the method can be considered suitable for the afore mentioned purpose. The mercury concentration found for the reference material was of (95,12 ± 11,70)μg.L^{-1} with a recovery rate of 97%. The method was also applied to 39 urine samples, six of which (15%) showing urinary mercury levels above the normal limit of 10μg.L^{-1}. The obtained results fall into a range of concentration from 1,02 to 23,36 μg.L^{-1}.

Evaluation of the levels of occupational exposure to mercury in the public dental offices in Araguaína, To, Brazil

The aim of this work was to evaluate the occupational risks of exposure to mercury utilized in dentistry, to which dental auxiliaries from the public health system of the city of Araguaína, Tocantins, Brazil are exposed. The dentistry process from the city health system was rigorously studied based on an assessment, in the DATASUS database, of the number of amalgam restorations conducted in the period from January 2003 to December 2009. In this study, we evaluated the storage conditions of mercury residuals in the public dental offices in the studied municipality. Different barrier solutions for the storage of mercury residuals were studied regarding their efficacy in controlling the emission of mercury vapor in the environment. The solution containing ammonia presented the best time of storage of residuals (19 days at 37°C), followed by the solution containing photographic fixative (12 days) and the sodium bicarbonate solution (7 days). Based on such information, a profile of the dental auxiliaries actuating in the public dental offices of the city of Araguaína was characterized according to age, sex, level of exposure to mercury and conducted biosecurity procedures. The study allowed the elaboration of a database regarding handling and discard of amalgam residuals, with the purpose to assist public health policies to modify risk situations to which the professionals and/or their patients are exposed. It was also conducted a biological monitoring program, using as marker the presence of mercury in urine samples (HgU) from members of the dentistry team of the

Araguaína public health system and from students from the Araguaína unit of the health care technical school who actuated as dental auxiliaries in other municipalities of the north region of the state (n=91). For comparison, a group of "not exposed" to mercury was evaluated (n=43), determining their HgU values. The research allowed to conclude that the HgU values from the dentistry team were within the limits set by Regulatory Norm nº 7 by the Brazilian Ministry of Labor (up to 35µgHg.g^{-1}CR). Regarding mercury contamination, the average HgU concentration for the "exposed" group (5,61µgHg.g^{-1}CR) was approximately 8 times higher when compared to the values from the "not exposed" group (0,65µgHg.g^{-1}CR), making evident the need for a professional training and residuals management program, as well as for a more effective actuation in the realization of periodical biological monitoring in the professionals from the dentistry team.

Evaluation of zinc in plasma samples collected from aged pacients admitted in the Regional Hospital of Araguaína City, Tocantins

Zinc is an important micronutrient that plays a role in many enzymatic and immune system activities in human body. Considering this, the goal of this work is to evaluate zinc deficiency in elderly patients. A total of 88 volunteers with more than 65 years old, admitted at ER from Regional Hospital, were monitored and zinc concentration was dosed in their plasma. The sampling volunteers were evaluated according to: race, gender, anthropometric measurements, hemoglobin, albumin, total lymphocyte count, C-reactive protein dosage and length of stay. The results suggest that zinc deficiency is more pronounced in females and patients with zinc deficiency also show lower values of body mass index (BMI), hemoglobin, albumin and lymphocyte count. There was no increased incidence of infection in the group with zinc deficiency, but the group had a longer hospital stay.

Environmental Monitoring Program (EMP-Q) to assist the non radioactive chemicals at IPEN

IPEN has implemented an Environmental Monitoring Program since 2007, which has as main objective the life and property safety in its plant of 478,000 m^2. As a research and production facility, many chemical, biological and radioactive products are manipulated in its laboratories. To assure that no hazardous substances were released to the environment, the program analyses stable chemical compounds in the groundwater and wastewater generated by the institute.
Annually, the program performed more than eight hundred analyses, including pH, temperature, total and dissolved solids, metals (Al, Sb, Ba, Cd, Pb, Co, Cu, Cr, Hg, Mo, Ni, Ag, Na, Zn, Ca, Mg, Be, Sn, Li, K, Sr, Ti and V), semimetals (As, B, Se and Si) and anions (chloride, nitrate, sulfate and fluoride).

Wastewater

Wastewater aliquots were collected daily at a Monitoring Station installed in the north entrance of the institute, according to nationally recognized sampling references (CETESB, 1987). The obtained results were compared to the values established by Brazilian laws for releasing of effluents in domestic sewer system. Currently the effluent released from Ipen is compliant with legal guidelines values established by São Paulo 8468 state law (article 19-A).

Ground water

In order to assure that IPEN's activities do not lead to groundwater resources contamination, the environmental program includes chemical quality evaluation of six monitoring wells installed in different areas of the institute. The program so far showed there is no evidence of contamination by the evaluated compounds in IPEN's ground waters. Currently the environmental monitoring program has the following aims:
-To assess the environmental impact from activities of IPEN;
-To keep systematically records from natural levels of stable chemical compounds, in the influence area of IPEN;
-To improve the current sampling, preparation and analysis protocols;
-To detect any potential flaw on environmental safety and plan corrective measures;
-To provide information to the general public.

Non-radioactive chemical waste disposal program

Environmental issues are a major concern for the Institution. Despite the Brazilian laws, IPEN has implemented a chemical waste disposal program dealing with the non-radioactive chemical wastes following the ideas stated in its Mission.
Residuals are yielded in the several processes even running routinely or in the periods of a variety of researches, or also due to expired reagents. These materials are identified, classified and kept in temporary vessels until final disposal.
Proper training is provided to internal technical personnel in order to equalize procedures and actions. The management of the information is carried out using an internal computer network to input data and visualize details and exchange items (Fig. 6).

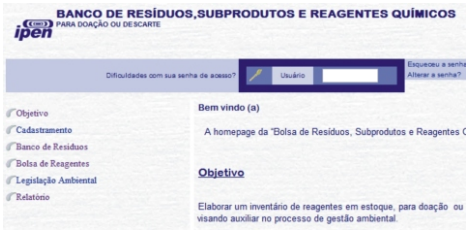


Figure 6. : Information of chemical residues and reagents can be viewed in an internal network

The benefits of this policy are: cleaner and safer areas, organized work place, lower environmental risks, reduced budget for management of remaining items, and environmental laws and regulations compliance. All these wastes are sent to disposal in accordance to technical norms and Brazilian and local laws and regulations.
One of the most significant goals was the understanding by the internal community that the residual is a consequence of our acts and a proper behavior can make living and working easier and safer.

Analytical chemistry for environmental diagnosis

The Laboratories of Chemistry and Environmental Diagnosis Center, have established methodologies for evaluation of physical-chemical, chemical and toxicological parameters to support several research projects in development for environmental diagnosis. All methodologies adopted are established in the standard methods (ASTM, EPA) or specified by the clients. The classic methods and instrumental techniques analysis such as atomic absorption spectrometry (AAS), inductively coupled plasma emission spectrometry (ICP-OES), ion chromatography (IC), gas chromatography (GC), gas chromatography mass spectrometry (GCMS), high performance liquid chromatography (HPLC), X-ray fluorescence (WD-XRFS), differential pulse anodic stripping voltammetry (DP-ASV) have been used.

The Center has been participating in several international interlaboratory programs sponsored by: CETAMA (Etablissement Comission des Analyse Méthods, France), IAEA (International Atomic Energy Agency), INTI (Instituto Nacional de Tecnologia Industrial, Argentina), SENAC (Brazil), ABACC (The Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials), SABESP (Basic Sanitation Company of the State of São Paulo, Brazil) and Rede Metrológica Rio Grande do Sul (Brazil). The Quality Handbook according to ISO GUIDE 17025 has been elaborated. The Laboratory of Chemical and Isotopic Characterization - LCQ has been accorded the prestigious INMETRO Certificate of Accreditation in accordance with ISO/IEO 17025: 2005. Several methodologies were established to support research projects in development:

Environmental
- The specificity and sensitivity in the pesticides analyses were enhanced by a new methodology using solid-phase extraction followed by high performance liquid and gas chromatography mass spectrometry (GCMS). The impact of pesticides use in agriculture was verified.
- Metal evaluation in top water and drinking water.
- Metal evaluation for industry wastewater.
- Metal evaluation in sediments and soils.

IEA-R1 Reactor
Evaluation of IEA-R1 water quality.

Quality System Based on ABNT ISO/IEC 17025 Norm

In order to align to the requirements of Nuclear Fuel Cycle, a Quality System based on the ABNT ISO/IEC 17025 norm is being implemented in the analytical laboratories.
As required, laws and rules compliances, personnel training, documentation, processes and environment monitoring and controlling are examples of what issues are to be done. Existing analytical procedures are being configured and new ones are in course according to that norm.
As the system will be implemented, many benefits are expected not only about documentation or organization, but also in the quality of results. Procedure validation and the estimative of measurement uncertainties allowed increasing the knowledge of what is being done.
In a broader view, this local system is part of the institutional integrated quality system management policy which includes other norms of conformity, such as the ABNT NBR ISO 9001.
As the system is implemented, higher levels of standards are met showing the way for total conformity assessment.

Polymers & environment

Development of thermoplastic starch and products from renewable sources
Starch is a natural, renewable, biodegradable polysaccharide produced by many plants as a storage polymer. It usually has two major components, a largely linear amylose of molecular weight between one thousand and one million and amylopectin, having the same backbone as amylose but with a myriad of α-(1→6)-linked branch points. The most commercially important starch for the development of thermoplastic starch is corn in the vast majority of countries, however in tropical countries starch from tapioca appear to be a much more interesting source. Native starch occurs in the form of discrete and partially crystalline microscopic granules that are held together by an extended micellar network of associated molecules. A small amount of water is always associated to this structure, therefore the use of this bound water as expansion agent is an opportunity to developed foam with very low impact to the environment. Starch is not truly thermoplastic as most synthetic polymers. However, it can be melted and made to flow at high temperatures under pressure and shear. If the mechanical shear becomes too high, then starch will degrade to form products with low molecular weight. Addition of water or other plasticizers enables starch to flow under milder conditions and reduces degradation considerably. However, the thermomechanical stability is strongly reduced by the addition of plasticizers. This project has developed a mixture of starch and nanoparticles for production of active and smart

films and it was also developed very resistant packing with the addition of natural fibers. PVA as biodegradable polymer was mixed with starch and irradiated.

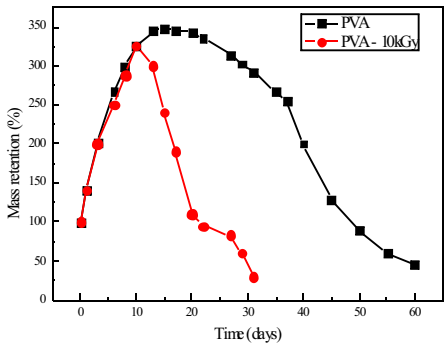


Figure 7. Biodegradation curves of PVA films (0 and 10kGy) in simulated soil

Figure 8. PVA films, prepared by casting process, were examined for biodegradation by composting over 60 days

Recycling of rubber and degradation of heavy oil by means of microwave and ionizing radiation assisted processing

The saturated polymer systems (rubber) and butyl rubber (BR) used in the manufacture of tires and rubber chlorobutyl and bromobutyl used to make inner-air in the tires, electric wires and cables and has high resistance to the action of oxygen, ozone, solar radiation and bacteria, thus contaminate the environment for long periods of time. The re-use of these polymers also reduces the emission of substances given (aliphatic) in the atmosphere. The objective is to develop processes of controlled degradation (devulcanization) of butyl rubber and halobutyl rubbers (chlorine and bromine) using radiation of Co source. The development of combined methodologies as thermal degradation and ionizing radiation produce recycled material with characterized properties to be applied in new formulations.
Several types of petroleum hydrocracking and hydrotreating processes can benefit from microwave technique. It has recently been studied all over the world to identify (qualitatively and quantitatively) and define the mechanism of microwave-material interaction. Rubbers devulcanization is one of the main stream of this line. The application of this process in our country is a very recent field and has been studied as a new tool in materials processing for rubber devulcanization, which uses high temperatures and shear and also for heavy oil degradation as well as desulfurization of oil and diesel. The knowledge of this technology is important to begin the development process in industrial scale and consequently in reducing the environmental pollution caused by these kinds of residues. Microwaves are a form of electromagnetic energy in the frequency band from 300MHz to 300GHz (not ionizing radiation). Industrial microwave processing is usually accomplished at a frequency of 2.45GHz (which corresponds to a wavelength of 12.24cm) to avoid interference with telecommunication and cellular phone frequencies. Microwave processing offers numerous advantages in relation to conventional heating methods (convection or conduction), where the material surface heats first and then the heat moves inward. One of the most important characteristics is saving energy, because the material absorbs microwaves readily (the heat is generated from the inner parts to the surface of the material) reducing the processing time. Also the selective energy absorption allows heating in specific points of the material. This process is environmentally clean because it reduces pollutant emissions. Finally, the microwave heating requires no appreciable amount of time to effect temperature changes such as conventional methods and when the microwave device is turned off the effect of these electromagnetic waves are instantaneously stopped.

Figure 9. Microwave system

The mankind has faced challenges from energy needs and prices, resource shortages and global environmental problems. Therefore, there are new needs such as knowledge-based products or services that improve operational performance, productivity, or efficiency while reducing costs, inputs, energy consumption, waste or pollution. Nowadays, products, services or processes should use limited or zero non-renewable resources and creates significantly less waste. Such technologies are named Clean Technologies that use energy, water and raw materials more efficiently, create less waste or toxicity, deliver equal or superior performance, and promote cost reduction and/or increased revenues. Given the environmental benefits these technologies confer, Clean Technology is an intrinsic part of a Sustainable Economy. Some major clean technology sectors are energy, water, manufacturing, advanced materials and transportation. The pollution control and waste reduction are also some important fields, consequence of the public perception of problems like global warming and the impact from the burning of fossil fuels, besides the introduction of contaminants into the environment, as a result of industrial activities. Clean technologies are seen to be the next engine of economic growth and the IPEN has dedicated attention and research initiatives in accordance with this approach.

Biosorbents for the wastewater treatment

Nowadays, some biomass types have been used for the removal of heavy metal ions and dyes at trace quantities from wastewater. They have shown different levels of contaminant uptake and are available in large quantities so have been considered low cost biosorbents.
Biosorbents are biological resources, include by-products or waste materials from agriculture, food processing industries, are abundant in nature and classified as non-conventional adsorbents. In our research, sugarcane bagasse, coir pith, banana pith, chitosan and fish scales have been investigated as alternative biosorbents to commercial adsorbents for the removal of contaminants from wastewater. These biosorbents were characterized by scanning electronic microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and themogravimetric analysis (TGA); as illustration, the Fig. 10 shows an image of a fish scale by SEM.

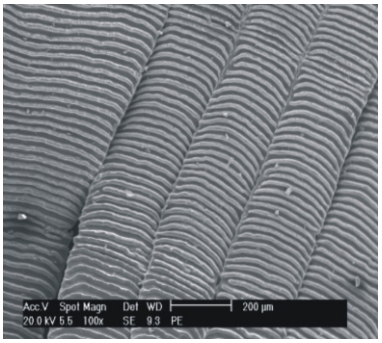


Figure 10. A fish scale image with magnitude 100x

The biosorption has been evaluated for U, Th, Pb, Ni, Zn, Cr ions and dyes such as Reactive Orange 16 and Congo Red. Influence of stirring speed, stirring time, dosage of biosorbent and pH on contaminant removal by adsorption process was investigated. Freundlich and Langmuir isotherm models were used to evaluate the data of equilibrium isotherm in the range of concentration from 5 mg L^{-1} to 1000 mg L^{-1}. In the greater number of cases, the Langmuir model was found to best represent the equilibrium isotherm. In all cases, the pseudo second-order model was found to explain the biosorption kinetics and Gibbs free energy indicated the spontaneous nature of the adsorption processes.
The experiments conducted in this research have shown the sugarcane bagasse, banana pith, coir pith, fish scales and chitosan were potential biosorbents for removal of U, Th, Pb, Zn, Ni, Cr ions, reactive orange 16 dye and Congo Red dye from wastewater. Figure 11 shows high performance of adsorption kinetics of the fish scales for removal of U ions.

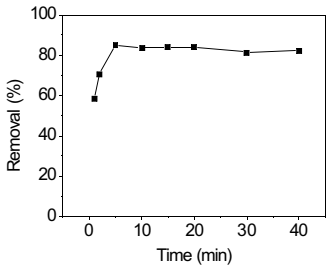


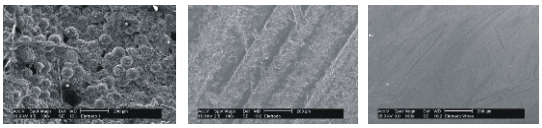
Figure 11. Adsorption kinetics of U ions on fish scales in the batch process, at room temperature

The investigated biosorbents are wastes or subproducts available in large quantities that represent pollutants and hence their application to the treatment of wastewater is considered a clean and economic sustainable technology. Two Masters were completed and one CNPq project (nº 472746) was concluded for period 2008-2010. The research project of sugarcane bagasse was awarded the 1º Prize DOW of Sustainability, DOW Chemical Company, in 2008 and one patent pending in 2010.

Use of cyclic voltammetry and electrochemical impedance spectroscopy techniques as quantitative/qualitative combined approach for determination of active surface area of modified carbon-based electrodes

Carbon-based electrodes as well the ion exchange electrodes among others, have been applied mainly in the treatment of industrial effluents and radioactive waste. Its use has been proven in the treatment of effluents containing different metals and radioactive wastes from chemical laboratories in the nuclear field. The carbon-based electrodes, which are also widely used in fuel cells as substrate

for the electrocatalyst, have high surface area which surpasses its geometric area. The knowledge of the total active area is important in determination of the operating conditions of an electrochemical cell with respect to the currents to be applied (current density). In this study it was used two techniques to determine the electrochemical active surface area of glassy carbon electrodes, porous electrodes and ion exchange: cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). The experiments were carried out with solutions of KNO_3 0,1 mol.L^{-1} in electrochemical cell with three electrodes: working electrode based on carbon, auxiliary electrode of the platinum and reference electrode of the Ag / AgCl. The glassy carbon electrode used had an area of 3,14 x 10^{-2} cm^2. The porous carbon electrode consisted of a rod with the geometric area of 2,83 x $10^{-1}cm^2$. The electrode of the ion exchange was prepared by mixing graphite, carbon, ion exchange resin and a binder, and this mixture applied in three layers on carbon felt in experiments using a geometric area of 1.0 cm^2. The capacitance (C_d) of the materials was determined by EIS directly using Bode diagrams. The value of 172,0 µF.cm^{-2} found for the glassy carbon is consistent with the literature data (200 µF.cm^{-2}). For VC, varying the scan rate from 0.2 to 2 mV.s^{-1}, the capacitance C_dS (S = active surface area) in the region of the electric double layer (EDL) for each material was determined. For EIS, the values of C_d 3,0 x 10^{-5} µF.cm^{-2} and 11 x 10^3 µF.cm^{-2} was found for the porous carbon electrode and ion exchange, respectively, which allowed the determination of surface areas of these active electrodes as 3,73 x 10^6 cm^2 and 4,72 cm^2. To sum up, the combined use of EIS and CV techniques is a valuable tool for the calculation of surface areas of active carbon-based electrodes.



(a) (b) (c)

Figure 12. Micrographs of (a) ion exchange, (b) porous carbon and (c) glassy carbon electrodes

Recovery and recycling of sulfuric and nitric acids and molybdenum from liquid waste of lamp industries

The waste treatment of certain industrial processes is becoming more important, either by the economic impact of simple disposal in the environment, or by the economic value of materials and substances that can eventually be recovered and recycled. The rapid depletion of mineral reserves and increasing primary energy demand are problems that deserve special attention. In this context, the recovery of metals present in waste of some manufacturing processes assumes a great importance. The recovery of molybdenum present in nitro-sulfur solutions in the form of liquid waste in the manufacturing process of incandescent and fluorescent lamps, is no exception with regard to the importance of recycling. The tailing from the dissolution of the molybdenum mandrel wires used in the conformation of the tungsten filament of electric lamps, has values that can be recovered and recycled to the process itself. That's the case of sulfuric and nitric acids. Molybdenum, present in concentrations around 40 to 90 g.L^{-1}, can be recovered and used in the manufacture of special stainless steel, pigments, lubricants, fertilizer, etc. In this work two processes for the recovery of this waste were proposed. At the first one, the waste is diluted and molybdenum is recovered by ion-exchange chromatography. The effluent acid from this process was distilled to extract water used in the dilution step. In the second case, the waste goes through a distillation while the molybdenum is precipitated. In both cases, the acids are recovered back to the lamp factory for the dissolution of the molybdenum mandrel wires and molybdenum finds other different applications, as well as having significant value in the market.

(a) (b) (c)

Figure 13. Distillation process for recovering waste generated in lamp industries: (a) distillation of nitric and sulfuric acids with release of nitrous vapors, (b) precipitation of molybdenum during distillation, and (c) molybdenum product after filtration and calcination

Chemical characterization of environmental contaminants by gas chromatography and mass spectrometry techniques

The scope of this research is to characterize chemically, contaminants and constituents on environmental samples using gas chromatography and mass spectrometry techniques. Air, water and soil are the matrix that has been studied in this research. These activities are concerns to Brazilian government institutes within mission to improve the health and protection of the Brazilian population before environmental surrounding. The work is divided into three activities, one is the specific scientific research plan, second is students training to do research in the laboratories, including science lectures and the last one has the target to publish the research and apply the methodology developed into the environmental agency besides the analytical work routine. The first task has been the concern about projects on gas chromatography and mass spectrometry link to characterization of environmental contaminants. The second task, concerns about gas chromatography and mass

spectrometry courses, training students: undergraduate and graduate. The third task concern about publications, introducing the developed analytical methodology before the Brazilian environmental agency and routine analysis to the IPEN nuclear community. The proliferation on hybrids analytical techniques develops parallels analytical techniques, such as Gas Chromatography, Liquid Chromatography and Infrared absorption techniques that are explored in this research as well. The following list shows each research work done and ongoing in the laboratory the years from 2008 to 2010.

- Evaluation of the occupational risks of organic volatile compounds VOCs in airport environments: Implementation of methodology.
- Characterization and analytical optimization in the determination of trihalomethanes in drinking water by purge and trap coupled to a gas chromatography.
- Development of a multi-residue method for the determination of benzimidazolic pesticides, carbamates and triazines in corn by liquid chromatography coupled to tandem mass spectrometry and its certification.
- Study of the acceleration of ammonia generation process from poultry residues aiming at hydrogen production.
- Levoglucosan and water soluble ions in atmospheric particulate matter PM10 and PM2.5. Characterization of South American sites.
- Assessment of gamma radiolytic degradation in waste lubricating oil by GC/MS and UV/VIS.
- Quality control in the carbon/sulfur analysis for the nuclear materials.

Safe decomposition of organohalogenated pesticides by molten salt oxidation

Banished or discarded pesticides constitute a serious risk, because they can leak from corroded metallic drums or other packages, discarded in an inadequate way, with contamination of the environment. The wastes infiltrate in the soil and ground water and they can affect human beings, domestic animals and fauna. It is necessary to identify the stocks and the storage places of the POPs (persistent organic pollutants), to collect and to destroy them appropriately, stanching their migration for the environment. Therefore, there is interest in the development of advanced technologies for POPs safe and complete decomposition. An alternative to the incineration, for treatment of these wastes, is the molten salt oxidation. Molten salt oxidation is a thermal means of oxidizing (destroying) the organic constituents of hazardous wastes while retaining inorganic and radioactive constituents in the salt. In this process, organic wastes are injected with a stoichiometric excess of air (oxidant) beneath the surface of a pool of molten sodium carbonate at temperatures between 900-1000°C. Therefore, the waste and the oxidizer are mixed in a turbulent bed of molten salt. The large thermal mass of the molten salt provides a stable heat-transfer medium and ensures temperature uniformity. Flameless oxidation takes place within the salt bath converting the organic components of the waste into CO_2, and water. It is inherently safe, especially as the emissions of organohalogenated compounds, because the reaction of chlorine with sodium (from the molten sodium carbonate), forming an innocuous compound sodium chloride. Therefore, molten salt oxidation of hazardous wastes is considered a promising alternative to incineration for the treatment of a variety of organic wastes. IPEN has constructed pilot-scale molten salt oxidation equipment in which tests are being performed under carefully experimental conditions. In the figure 14 it is presented a schematic drawing of the molten salt oxidation process and in the figure 15 the molten salt, the heated reactor vessel and the molten salt equipment prototype.
The emphasis in recent years has been the study of the decomposition of organochloride pesticides. With the support of a FAPESP PIPE project (Project 07/51603-6) IPEN and the company Vitex developed an advanced prototype of molten salt reactor and the decomposition parameters three pesticides: Heptachlor, DDVP and Fenil Pirazol were evaluated. This project was concluded in the period 2008-10. Another CNPQ project (Project 567892/2008-5) was approved in the period, for the development of a more advanced prototype, constituted by two reactors in a tandem system. Besides the construction of the prototype, this project is also dedicated to the study of the safe decomposition parameters for BHC hexachlorcyclohexane. The conclusion of this project will occur in November, 2011.

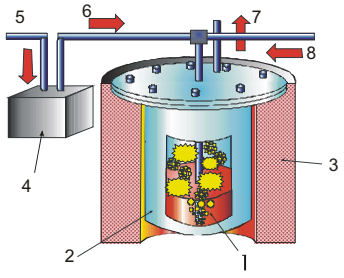


Figure 14. Schematic drawing of the molten salt oxidation process where 1- molten salt, 2- reactor vessel, 3- heating system, 4- wastes pressurized reservoir, 5- compressed air, 6- waste feeding piping, 7- off gas, 8- air/oxygen injection

Figure 15. Molten salt (left), heated reactor vessel (center) and molten salt equipment (right)

Decontamination of carbon steel structures by molten salt stripping

One problem detected during the nuclear fuel cycle dismantling activities already performed in the IPEN was the difficulty of treatment of contaminated painted carbon steel structures. During nuclear fuel cycle dismantling and decommissioning activities several tons of contaminated steel structures used to be generated. The operational conditions of the fuel cycle pilot plants were deleterious to structural materials, submitting them either to a chemically aggressive environment or to radioactive liquids and dust that form deposits in the structure surfaces. The chemicals employed, like acids, caused accelerated corrosion of the carbon steel. To protect the structures and to increase their useful lives, it was employed painting as a protection agent. During the facilities operational life, several layers of paint were applied in an attempt to stop or reduce the corrosion rate, since the structures were submitted to aggressive environments. Besides this, it was found that the most contaminated regions, in the steel structures, were the oxidized or corroded areas. The presence of several layers of paint combined with corroded regions makes the decontamination process much more difficult. Several methods were previously attempted with the objective of superficial contamination removal and the consequent reduction of the contaminated waste volume, such as: decontamination by rinsing methods employing acid and alkali solutions (chemical method), combination of rinsing methods and ultrasonic device and abrasive removal (physical method). However, the results employing the methods above mentioned were not satisfactory. The combination of painting and carbon steel corrosion creates serious problems in terms of decontamination efficacy and workers exposition. It was proposed to adapt the molten salt system, developed for the thermal decomposition of contaminated organic solutions, to evaluate the possibility of using the salt bath as a stripping and descaling system. *A priori* the organic compounds of the paints would be decomposed and the steel corrosion products mixed with uranium or thorium compounds would be retained in the molten salt as oxides. After dissolution of the salt, filtration to separate the radioactive wastes (insoluble in the salt when in the oxide form), the salt can be recrystallized and re-used in the process. In the figure 8 a flow sheet of the developed process is presented. One of the goals of the project is to determine the most suitable salt mixture composition to achieve optimization between the minimal possible bath temperature and an efficient removal of the paint and the corrosion products to make possible the superficial decontamination and to release the metallic structures as common scrap. A patent privilege request was submitted to analysis in the Brazilian Industrial Property Institute - INPI: related to the Process for Radioactive Decontamination of Parts Components and Metallic Structures in Molten Salt Baths (Processo de Descontaminação Radioativa de Peças, Componentes e Estruturas Metálicas em Banho de Sais Fundidos). This patent was registered in the INPI in 04/07/2008 (PI0803710-8). In this development IPEN had the support of the IAEA Co-ordinated Research Project: CRP on Innovative and Adaptive Technologies in Decommissioning of Nuclear Facilities (2004-2008). This development is part of the document IAEA-TECDOC-1602, Innovative and Adaptive Technologies in Decommissioning of Nuclear Facilities, 978920110085, Vienna, Austria, IAEA - International Atomic Energy Agency, 2008, p. 57 - 84.

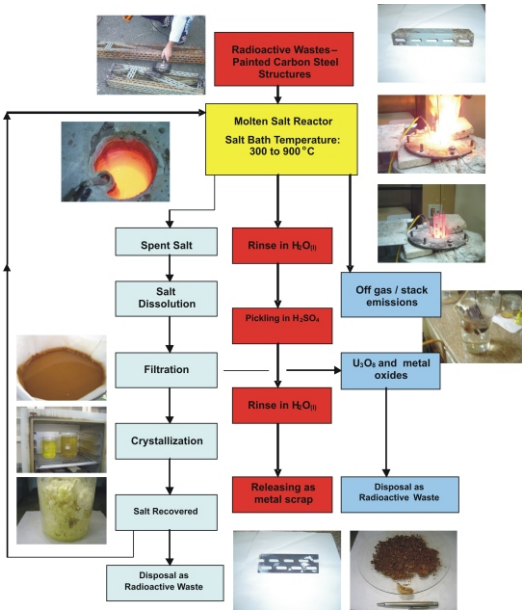


Figure 16. Flow sheet of molten salt stripping for superficial decontamination of radioactive wastes.

Thorium

Two major international projects, namely the IAEA initiated INPRO, and the US-led GIF, are presently underway for innovations in nuclear reactors and fuel cycles in order to meet the global energy needs in the 21st century and beyond. Some of the common objectives of INPRO and GIF are long term sustainability and favorable economics of nuclear power and addressing associated public concerns in the areas of safety; nuclear waste storage, disposal, environmental protection and actinide management; and proliferation-resistance in nuclear fuel cycles. The Generation IV International Forum (GIF) was initiated in 2000. After some two years' deliberation, GIF (then representing ten countries - Argentine, Brazil and UK are considered inactive members) announced the selection of six reactor technologies which they believe represent the future of nuclear energy. These were selected on the basis of being clean, safe and cost-effective means of meeting increased energy demands on a sustainable basis, while being

resistant to diversion of materials for weapons proliferation and secure from terrorist attacks. Three of the six are fast neutron reactors (FNR) and one can be built as a fast reactor, one is described as epithermal, and only two operate with slow neutrons like today's plants. Only one is cooled by light water, two are helium-cooled and the others have lead-bismuth, sodium or fluoride salt coolant. The latter three operate at low pressure, with significant safety advantage. The last has the uranium fuel dissolved in the circulating coolant. Temperatures range from 510°C to 1000°C, compared with less than 330°C for today's light water reactors, and this means that four of them can be used for thermochemical hydrogen production.

Uranium, as well as thorium, can be used as a nuclear fuel. During the pioneering years of nuclear energy, from the mid 1950s to mid 1970s, there was considerable interest worldwide to develop thorium fuels and fuel cycles in order to supplement uranium reserves, since it is more abundant in the Earth's crust than uranium. Starting around the end of the 50's, a great number of prototypes based on thorium were built. The feasibility of thorium utilization in several reactor concepts has already been demonstrated. Could be mentioned the following types of reactors: high temperature gas cooled reactors (HTGR), light water reactors (LWR), pressurized heavy water reactors (PHWRs), liquid metal cooled fast breeder reactors (LMFBR) and molten salt breeder reactors (MSBR). Nevertheless, the great success of the Light Water Reactors, with good availability of uranium and the reliability in the UO_2 fuels, leads to abandon in some extent the interest devoted to thorium cycle.

Thorium is 3 to 4 times more abundant than uranium and is widely distributed in nature as an easily exploitable resource in many countries. Fuel cycles based on uranium in a thermal spectrum cannot be considered sustainable. Preserving natural resources imposes fissile regeneration by capture on a fertile support of thorium or uranium. Although not fissile itself, Th-232 will absorb slow neutrons to produce uranium-233, which is fissile. Besides this, thorium fuels produce much less minor actinides than uranium fuels, so that induced radiotoxicities are lower by more than two orders of magnitude. Then, thorium with thermal neutrons is a very promising option for scenarios with a significant nuclear contribution.

The Molten Salt Reactor - MSR was studied in depth in the 1960s, but is now being revived because of the availability of advanced technology for the materials and components. There is now renewed interest in the MSR concept in Japan, Russia, France, USA and China and one of the six Generation IV Advanced Reactors designs selected for further development is the MSR. The molten salt reactor (MSR) is an advanced breeder concept, in which the coolant is a molten salt, usually a fluoride salt mixture. The salt is hot, but not under pressure, and does not boil below about 1400°C. Much research has focused on lithium and beryllium additions to the salt mixture. The fuel can be dissolved enriched uranium, thorium or U-233 fluorides, and recent discussion has been on the Liquid Fluoride Thorium Reactor, utilizing U-233 which has been bred in a liquid thorium salt blanket and continuously removed to be added to the core.

The Brazilian's interest in the nuclear utilization of thorium has started in the 50's as a consequence of the abundant occurrence of monazite sands. Nevertheless, as the worldwide fuel industry and the reactor technology have been developed predominantly in the uranium field, the lack of interest in the thorium affected the prospecting and the reserve's evaluation. The reasonably assured reserves and the estimated additional resources in Brazil can reach 1.3 million metric ton of ThO_2.

Since the sixties, IPEN has performed some activities and developments related to the solvent extraction purification process, thorium tetrafluoride preparation and its reduction to metallic thorium, studies of some properties of the UO_2-ThO_2 solid solutions. Besides those activities, IPEN carried out for about 18 years the production and purification of thorium compounds, supplying Universities, Research Institutes and Industry. During this period, the main product sold was the thorium nitrate (mantle grade) for the Welsbach mantle Brazilian industry, used for portable gas lamps, having been produced over 170 metric tons of this material in the period, obtained through solvent extraction. The raw materials were some thorium concentrates obtained from the industrialization of monazite sands, a process carried out in S. Paulo between 1948 and 1994 on an industrial scale by the company ORQUIMA, later NUCLEMON. IPEN's facility for thorium nitrate production and purification was unique in Brazil and in all South America.

Nowadays, predicting the importance that the thorium will have on the future of nuclear energy and mainly due to the size of the Brazilian reserves, IPEN initiated an activity to preserve the experience and knowledge associated with thorium, ranging from processing methods to the analytical procedures developed during the time. In 2010, it was initiated a cooperation with CYTED/IAEA involving thorium with a presentation about the IPEN's Experience with Thorium Processing Activities. The MINIFORUM Iberoeka - Thorium occurred on 09 December 2010 in Rio de Janeiro and was promoted by the Center for Mineral Technology (CETEM) and the Ibero-American Program of Science and Technology for Development (CYTED). The activity is part of the area Promoting Industrial Development - CYTED, whose manager is the Professor Robert C. Villas-Boas. The event, held in the auditorium of CETEM, had financial support from FINEP and CNPq.

Participation in the RMB Design - Subsystem 19300 - Decommissioning

Among several tasks, an important point that is already being considered in the design phase of the new reactor is its future decommissioning. The main concern in this phase is to take into account the lessons learned and the worldwide accumulated experience in the decommissioning of research reactors avoiding mistakes and reducing the amount of problematic wastes in the future. In spite of the currently lack of specific regulatory legislation in Brazil, with respect to reactors decommissioning, the activities in the design phase of the new reactor are concentrated in rising the international regulatory licensing procedures and recommendations that can be useful for the RMB's case.

Another approach adopted is the definition of specifications and standards, facilitating the future decommissioning. The decommissioning of nuclear facilities, especially research reactors, can be facilitated, with significant reduction of the costs and amount of wastes, if suitable choice of the employed materials and their chemical composition is made during the design phase. It deserves special attention the reactor regions where activation of materials is possible, since the presence of determined elements in the composition of alloys and structural materials can affect significantly the future decommissioning. Then, the proper selection of materials and their chemical composition, besides respective allowances, will reduce the impact in the future decommissioning costs, in the amount of high level wastes generated during the process and in the safety of the personnel involved in the operations.

Environmental Science and Technology

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Honor Mentions and Awards

The researcher Maria Beatriz Bohrer-Morel received the Copesul Brasken Award for her contribution to ecotoxicology in Brazil. The prize was awarded by the first time in the X Brazilian Congress of Ecotoxicology promoted by the Brazilian Society of Ecotoxicology in Rio Grande do Sul, 2008.

The Project "Ecomaterial: Development and application of zeolite synthesized from coal ashes in the remediation of contaminated soils and effluents" of the authors Dr. Denise Alves Fungaro and Anderson Oliveira de Andrade won the "Prize of The International Fair of Technology for the Environment - FIEMA" in the category Environmental Technology. FIEMA took place in Bento Gonçalves, RS, from 29/Oct to 1/Nov of 2008.

Honor Mentions and Awards

The Dow Sustainability1st Prize was awarded to Amanda Pongeluppe Gualberto Yamamura, MSc student at IPEN. The prize was awarded to the academic community for its significant contribution to sustainable development. The award was announced in July 7 in the categories of undergraduate, master's and doctoral degrees. The work was guided by the researcher Mitiko Yamaura. The magnetic nanoparticles, particles infinitely small, were combined with sugarcane bagasse to obtain a product adsorbent of metals. The sugar cane bagasse is a residue from the sugarcane agroindustry. The work used an agroindustrial biomass waste to obtain a material capable of decontaminating wastewater, 2008.

During the third edition of the Werner von Siemens Technological Innovation on November 15th, 2008, was announced the first place in the category Industry for the project of the master's degree of IPEN Thais de Oliveira. The winning project is entitled "Recovery and recycling of sulfuric and nitric acids and molybdenum from liquid waste of lamp industries", advised by the researcher Christina Aparecida Leão Guedes de Oliveira Forbicini.

Study conducted for the Masters of Patricia Cunico entitled "Adsorption of reactive black 5 dye of aqueous solution using zeolite of the coal fly ash" received special honors in "2nd International Workshop Advances in Cleaner Production" in São Paulo, SP, from 20 to 22/May of 2009. The study had the guidance of Dr. Denise Alves Fungaro and co-authored by Carina P. Magdalena and Terezinha E.M.de Carvalho.

Dr. Duclerc F. Parra and undergraduate student Luiza Mello de Paiva Campos received the "Francisco Degni" Prize for the winner work: "New methodology for resin contraction measurements", carried through at 28 International Congress of Odontology of São Paulo - CIOSP, 2010.

Radiopharmacy

The radiopharmaceuticals allow the diagnosis and treatment of a several diseases, and the products go through a series of quality control procedures before being sent to the hospitals and clinics all over the country

Radiopharmacy

Introduction

The production of radioisotopes and radiopharmaceuticals for use in Nuclear Medicine started in the late 50's at IPEN. There has been a significant increase in the demand for these products over the years and nowadays more than 30 products are listed at IPEN catalogue.

The Radiopharmacy Program is organized in six activities areas: Production; Quality Assurance; Quality Control; Research, Development and Innovation; Infrastructure and Maintenance Support; and Cyclotron Accelerator.

The highlights of this period were:

- All the efforts related to overcome the ^{99}Mo supply crisis that begun in 2009. A fast solution was achieved by a two countries agreement with Argentine and the implementation of a new schedule of production of ^{99m}Tc generators, three times a week.
- A new scheme of importation of ^{99}Mo was initiated, with the possibility of receiving the radioisotope from 3 different suppliers.
- The project of nationalizing the production of ^{99}Mo by the fission of LEU targets was started, together with the new reactor (RMB) project.
- The reform of the installations in on the way with financial resources from CNEN and FINEP in order to comply with the needs arising from the regulatory agencies, CNEN and ANVISA.
- ANVISA published the regulations for registration of radiopharmaceutical products and for acquiring the GMP in radiopharmacy. The actions required to comply with these regulations were implemented.
- The increase in the demand of ^{18}F-FDG that lead to the modification in the law that regulates the production of radioisotopes in Brazil and also to the purchasing of a new Cyclotron, dedicated only to ^{18}F production and new possibilities for positron emission radioisotopes.
- The effects of the modification in the law that regulates the production of radioisotopes in Brazil lead to the assembling of new producers and subsequently a decrease in the demand of ^{18}F-FDG produced by IPEN. Anyway IPEN acts as a back up for the producers and continues its role of transferring technologies of producing new PET radiopharmaceuticals.
- The research and development projects shifted with time to new products for therapy (with ^{177}Lu, ^{90}Y and ^{166}Ho) and for PET (^{18}F, ^{68}Ga, ^{64}Cu).
- Certification and maintenance of the ISO Quality Management System.
- An environmental monitoring plan was established to evaluate clean areas.
- A validation master plan was prepared considering the whole production process, personnel and material flow procedures were implemented and new equipments have permitted the introduction of modern analytical methods in the quality control.

Radiopharmacy

Production

The Production of Radiopharmaceuticals is divided in 3 different areas: Radioisotopes (^{99m}Tc generator and Primary Radioisotopes); Labeled Compounds for diagnosis (PET and SPECT) and for therapy; and Lyophilized Kits for labeling with ^{99m}Tc. The Commercial Department (SAC) is responsible for receiving the product order from the clients weekly or by demand. The main product specifications are described as follows:

Radioisotopes

Generator

^{99m}Tc Generator - IPEN-TEC
The ^{99m}Tc - Generator is a system which produces Technetium-99m for labeling lyophilized "kits" and it is used in nuclear medicine for thyroid and salivary glands scintigraphy. More than 300 generators are delivered weekly.

Primary Radioisotopes

^{131}I-Na - Sodium iodide solution
For oral study of thyroid gland and therapy of thyroid cancer and metastases.

^{131}I-Na - Sodium iodide capsules
For therapy of hyperthyroidism and therapy of thyroid cancer and metastases.

^{123}I-Na - Sodium iodide solution
For oral study of thyroid gland.

^{51}Cr - Sodium chromate
Used in nuclear medicine for study of red blood survival and spleen scintigraphy.

^{67}Ga - Gallium citrate
Indicated for localization and detection of soft tissue tumors and inflammatory process.

^{201}Tl - Thallium chloride
For cardiac function studies.

^{32}P - Sodium phosphate
Used in treatment of polycythaemiavera and biotechnology.

^{35}S - Sulphuric acid
Used in metabolic investigation.

^{18}F - Sodium fluoride
Used in bone image in PET and PET-CT.

Labeled compounds

^{153}Sm-EDTMP (ethylenediamine-tetramethylene-phosphonic acid)
Therapeutic agent indicated for relief of pain in patients with confirmed osteoblastic metastatic bone lesions in breast and prostate cancer.

^{131}I-MIBG (meta-iodobenzylguanidine)
Diagnostic and therapeutic agent of neural crest-derived tumors.

^{177}Lu-DOTATATE (DOTA-Octreotate)
Therapeutic agent for neuroendocrine tumors.

^{111}In-DTPA-TOC (DTPA-Octreotide)
Diagnostic agent for neuroendocrine tumors.

^{131}I-Lipi (lipiodol)
Treatment of hepatocellular carcinoma (HCC), the selective retention suggests its potential as chemotherapeutic or radiotherapeutic agents.

^{123}I-MIBG (meta-iodobenzylguanidine)
Diagnosis of pheochromocytoma, neuroblastoma and myocardial studies.

^{131}I-Hipp (o-iodo-hippurate)
Used for the investigation of kidney function, gives information about the renal blood flow, urinary tract potency and urinary flow in nuclear medicine.

^{131}I-HSA (human serum albumin)
For determination of plasma volume and total blood volume.

^{51}Cr-HSA (human serum albumin)
For the measurement of proteins lost by gastro intestinal tract, it is an ideal radionuclide for long time studies in nuclear medicine.

^{51}Cr -EDTA (ethylendiaminotetracetic acid)
For study of glomerular filtration rate.

^{18}F-FDG (fluoro-2-deoxy-D-glucose)
In oncology, cardiology and neurology studies.

^{53}Sm-HA (hydroxiapatite)
^{90}Y-HA (hydroxiapatite)
For synovectomy, treatment of rheumatic arthritis.

Lyophilized "kits" for labeling with ^{99m}Tc

DTPA - Diethylenetriaminepentaacetic Acid
For brain imaging, renal flow study and glomerular filtration rate measurement.

MDP - Methylene Diphosphonate
To demonstrate areas of altered orthogenesis as seen, in metastatic bone disease and osteomyelitys.

DMSA (III)- Dimercaptosuccinic Acid
For renal cortical imaging.

DISIDA - Diisopropyliminodiacetic Acid
Commonly used as hepatobiliary agent to evaluate hepatic and biliary duct function, also in cholescingraphy.

PYRO - Pyrophosphate
For localization of primary bone tumors, metastatic tumors and metabolic bone diseases, also in myocardial infarct.

Dextran70 and Dextran500
Used in sentinel node scintigraphy.

EC - Ethylene dicysteine
For renal function study.

ECD - Ethylene dicysteine diethyl ester
Used for cerebral perfusion studies and detection of intra-cerebral inflammatory conditions; detection of an abnormal focus in patients with head trauma and cerebral-vascular accidents; differentiation of Alzheimer's disease from multi-infarct dementia.

Sn-colloid - Stannous colloid
Indicated for imaging, localization and evaluation of liver and spleen pathology.

Fitato - Fitic acid
Indicated for imaging areas of functional reticuloendothelial cells in liver, spleen and bone marrow and in lynphoscintilography study.

During this period of time, the demand for therapeutic radiopharmaceuticals increase, while the total activity of ^{99}Mo used in the preparation of generators decreases (Figure 1). Otherwise, the total number of generators sent to clinical an hospitals was increased. The production of ^{18}F-FDG was decreased (Figure 2) due to the assembling of new PET production centers.

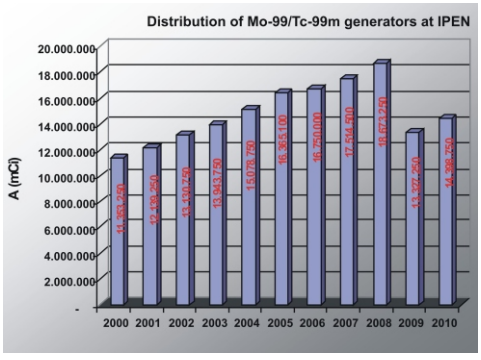

Figure 1. Radioactivity of ^{99}Mo-^{99m}Tc generators

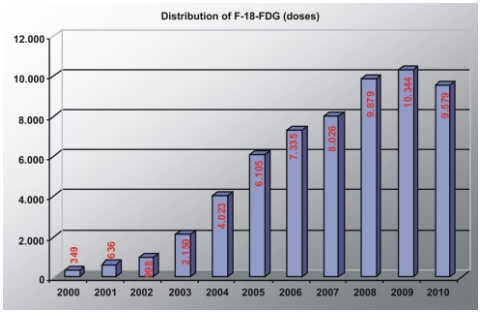

Figure 2. Distribution of ^{18}F-FDG doses

Quality Control of Radiopharmaceuticals

Annually, about 40,000 assays are executed in primary radioisotopes, labeled molecules, lyophilized reagents, starting materials, packaging materials and intermediate products at the Radiopharmacy Directory of IPEN-CNEN/SP. Figure 3 shows the distribution of the quality control tests during the year of 2010.

Specific tests that ensure the purity, potency, product identity, biologic safety and efficacy include physicochemical and biological tests: physical appearance of the sample, pH, humidity and particle size measurements, determination of radionuclidic and radiochemical purities, chemical impurities, dissolution test, sterility, bacterial endotoxin test, biodistribution and toxicity.

The quality control staff improves analytical methods for new products together with the research and development group and participates actively in the maintenance of the ISO 9001-2000 Certification.

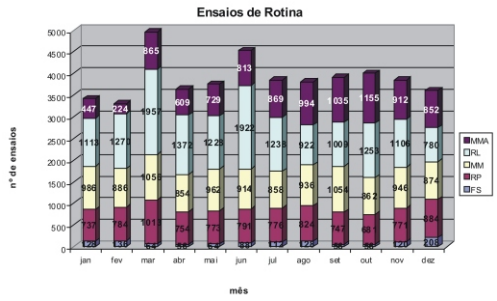

Figure 3. Number of quality control tests (2010)

Radiopharmacy

Research and Development related to Radiopharmacy

The area of Research and Development applied to Radiopharmacy at IPEN is divided into 6 different fields: Radionuclide generators; Primary radioisotopes; Labeling of molecules for diagnosis (PET and SPECT) and therapy; lyophilized kits and quality control analytical methodologies. The main achievements are described as follows:

Radionuclide generators

- Research project are under way with the objective of development of ^{68}Ge-^{68}Ga and ^{90}Sr-^{90}Y generators.
- Study of high activity generators.

Primary radioisotopes

- Development of a production method for ^{64}Cu.
- A project is under way aiming a new production method for ^{67}Ga.
- Project aiming the production of ^{99}Mo through the fission of LEU targets with the assistance of IAEA (CRP).
- Purification of ^{123}I and ^{131}I.
- Improvements in the gas target for the production of ^{123}I.

Labeling of molecules for diagnosis (PET and SPECT) and therapy

Research projects have been developed aiming the preparation of the following radiopharmaceuticals:

- A research project sponsored by IAEA (CRP) is under way and it has the objective of developing labelling methods for the Therapeutic Radiopharmaceuticals based on ^{188}Re and ^{90}Y.
- Labelling of octreotide with ^{68}Ga.
- Labelling of antibodies with radiometals and lantanides: studies concerning the derivation of the antibody.
- Labelling of substance P with ^{177}Lu.
- Labelling of DMSA with ^{188}Re.

Lyophilized kits

The synthesis of compounds not commercially available and the development of new lyophilized kits are necessary to introduce new ^{99m}Tc-based radiopharmaceuticals. Lyophilized kits allow the instant preparation of the labeled molecules with ^{99m}Tc without purification steps prior to administration.

- Labelling of the peptide ubiquicidine with ^{99m}Tc for oncology.
- Development of ^{99m}Tc radiopharmaceuticals for Sentinel Node detection and Cancer diagnosis (CRP-IAEA).

Quality control analytical methodologies

- Gram tests for classification of microorganisms.
- Radiochemical quality control of labeled kits: comparison of methodologies.
- Evaluation of total organic carbon in the water used in the Radiopharmacy.

Radiopharmacy

Quality Assurance in Radiopharmaceutical Production

Preparation of radiopharmaceuticals for injection involves adherence to regulations in radiation protection as well as to appropriate rules of working under aseptic conditions that should follow the regulations on current Good Manufacturing Practices (cGMP). Good Manufacturing Practices (GMP) is a system designed to ensure that pharmaceuticals are consistently produced and controlled according to quality standards, with a view to eliminating the risks involved in drug production. The compliance of GMP is directed to minimize the risks presented in the pharmaceutical production that can not be detected in the analysis of the final product: cross-contamination, contamination with particulate material and change or mixture of products.

Quality Assurance is a wide ranging concept which covers all matters that individually or collectively influence the quality of a product. It is the total sum of the organized arrangements made with the object of ensuring that medicinal products have the required quality for their intended use. Quality assurance therefore incorporates GMP and thus Quality Control. Because of their short half-lives, many radiopharmaceuticals are released and administered to patients shortly after their production, so that quality control (e.g. tests for sterility, endotoxin, radionuclidic purity, etc) may sometimes be retrospective. The implementation of and compliance with the quality assurance program are therefore essential.

Manufacturing practices are the methods, facilities, and controls used in the preparation, processing, packaging, or holding of a drug. The GMP in Brazil is published in the Resolution RDC 17 of 16 April, 2010 of the National Sanitary Agency (ANVISA) of the Health Ministry. Specific regulations and registration of radiopharmaceuticals were published by ANVISA (Resolution RDC 63 and 64 of 18 December, 2009).

The Brazilian Pharmacopoeia constituted the "Technical Comission of Radiopharmaceuticals" to prepare the radiopharmaceuticals monographs to integrate the Brazilian Pharmacopoeia. IPEN has participate in these work groups which reflect the importance of the radiopharmaceuticals in the context of pharmaceutical production in Brazil.

In the Radiopharmacy, the Quality Assurance Management is responsible for maintenance and improvement of the Quality Management System (according to ISO-9001-2008) and the implementation of all the aspects related to cGMP in production and quality control of radiopharmaceuticals. There is a group responsible for control, maintenance and improvement of data generated in the production and quality control process and all documents of the Quality Management System. The accompaniment of non-conformities generated in the System and the attention to the fulfillment of ISO 9001 are also attributions of this group. The Quality Assurance Management coordinates the Instrument Calibration, Equipment Qualification, Process Validation and also the implementation of other GMP requirements.

The Quality Assurance Management can oversee the production and quality control operations to ensure that a radiopharmaceutical is produced according the specifications. It is the responsible for approving or rejecting components, in-process materials and finished product to ensure compliance with procedures and specifications affecting the identity, concentration, quality and purity of the radiopharmaceutical.

In the last years, the maintenance of the ISO 9001 Quality Management System Certification was very important and contributed to the introduction of the GMP concepts. Some aspects of the GMP applied to the Quality Assurance Program are of special interest and have been discussed and introduced in the radiopharmaceutical production context at IPEN, including:

Validation
It was elaborated the "Validation Master Plan", including process validation, analytical procedures, cleaning procedures and personnel training. Validation program is in course for utilities (water and air) and attention has been given to process validation, including validation of sterilization process, process control and the monitoring of the established parameters, especially from the environment, particularly when the product should be released before the conclusion of all the quality control assays.

Installations
As a general principle of GMP, buildings must be located, designed, constructed, adapted and maintained to suit the operations to be carried out within them. Laboratories for the handling of radioactive materials must be especially designed to take into consideration aspects of radiation protection in addition to cleanliness and sterility. Some projects for improving the Radiopharmacy Instalation are in course in order to attend the GMP requirements for radiopharmaceutical production.

Regularization of the radiopharmaceutical in Health Ministry
Considering the new ANVISA Resolution for radiopharmaceuticals registration (RDC 64), the Quality Assurance group works in the development of final dossies to be submitted to the regulatory organ in order to obtain the register of the radiopharmaceutical in the Health Ministery.

To produce specific radioisotopes, in IPEN are installed two cyclotrons:

Cyclone 30

The cyclotron Cyclone 30 model, manufactured by Ion Beam Applications - Belgium, is a compact, fixed-field, fixed-frequence, that can accelerate H- ions with energies between 15 and 30 MeV. This energy range and its high external beam current available (350 μA) is optimum for production of the most important SPECT and PET cyclotron radioisotopes used in nuclear medicine: ^{18}F, ^{11}C, ^{13}N, ^{15}O, ^{67}Ga, ^{201}Tl, ^{123}I, ^{111}In, ^{124}I and ^{64}Cu.

Figure 4. Cyclotron Cyclone 30

The Cyclone 30 cyclotron has two external beam lines. One is dedicated to irradiation of solid target where ^{67}Ga and ^{201}Tl can be produced. At the end of the other beam line, a switching magnet with five exit ports is installed. In two of these positions liquid targets are installed and in another exit is a gas target, which allows the production of ^{18}F and ^{123}I, respectively.

The target system for production of ^{67}Ga and ^{201}Tl was manufactured by Ion Beam Applications - Belgium, and it uses a target at 6º with respect to the beam axis, resulting in an enlargement of the beam by a factor of 10. The target material (^{68}Zn or ^{203}Tl) is electrodeposited on an elliptical area measuring 10 mm x 100 mm, giving a typical thickness of 150 - 170 μm. On the back of the target there are fins to increase the water cooling efficiency. Irradiation with current up to 250 μA is possible.

At IPEN, ^{18}F is produced by the ^{18}O(p,n)^{18}F reaction using enriched water as target material. The liquid target system was manufactured by Ion Beam Applications - Belgium and it basically consists of four main parts: a conical collimator of 10 mm diameter, a window holder with two windows cooled by helium gas, one for the vacuum side and one for the target side (Havar of 25 and 50 μm respectively), a water cooled semi hemispherical niobium body and a high pressure valve for remote-controlled filling, unloading and purging of the target. In front of the target there is a four sector collimator, which helps the optimization of the cyclotron parameters. The production is made with protons of 18 MeV and current of 50 μA.

For ^{123}I production, due to the high coast of acquisition, IPEN has decided to develop its own system to produce ^{123}I *via* ^{124}Xe irradiation. This system includes a water cooled target ^{124}Xe chamber, a double Mo window (50 μm) cooled by helium gas, an alignment system, which consists of a pair of four sectors collimators and a safety volume cooled with liquid nitrogen and a valve manifold for vacuum and transference of the ^{124}Xe gas from the storage vessel to the irradiation chamber and recovery. The ^{124}Xe transfer from the storage bottle to the target and the recovery of the gas after irradiation to the bottle is made cryogenically with liquid nitrogen, through stainless steel pipes. If occasionally there is a rupture in the first window, the ^{124}Xe gas will be trapped in the helium cooling system and the mixture can be transferred to the storage bottle. The control system uses a PC and a PLC with a Siemens SIMATIC S5. A friendly software permits to control the process in manual mode selecting the desired action (valve open/off, pump on/off, and so on) by pointing the appropriate icon on the screen. The fully automated operation mode can be selected via keyboard and makes the process flexible.

Cyclone 18

The increase in the demand of ^{18}F-FDG that lead to the modification in the law that regulates the production of radioisotopes in Brazil and also to the purchasing of a new Cyclotron, dedicated only to ^{18}F production and new possibilities for positron emission radioisotopes.

Cyclone 18 is a fixed-energy cyclotron, accelerating H^- ions up to 18 MeV. The beam intensity is 150 μA. It includes eight independent exit port allowing eight targets to be simultaneously mounted on the cyclotron. Figure 5 shows the Cyclone 18.

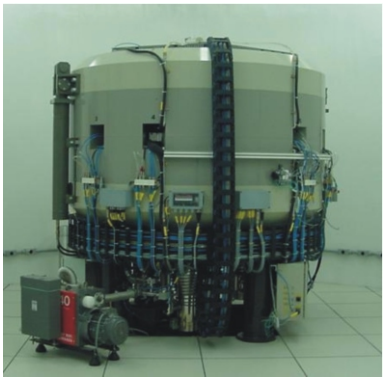

Figure 5. Cyclotron Cyclone 18

Nuclear Safety

Radioprotection supervisors care for the radiological safety of workers, public and environment at nuclear and radioactive facilities

Nuclear Safety

Introduction

The Program on Nuclear Safety comprehends Radioprotection, Radioactive Waste Management and Nuclear Material Control. These activities are developed at the Nuclear Safety Directory.

The Radioprotection Service cares for the radiological safety of IPEN workers and general public, through radiation protection programs in accordance with national and international standards. Research related to the main activities is also performed.

The Radioactive Waste Management Department was formally created in 1983, to promote research and development, teaching and service activities in the field of radioactive waste. Its mission is to develop and employ technologies to manage safely the radioactive wastes generated at the Nuclear and Energy Research Institute (IPEN) and at its customer's facilities all over the country, in order to protect the health and the environment of today's and future generations.

The Nuclear Material Control has been performed by the Safeguard Service team, which manages the accountability and the control of nuclear material at IPEN facilities and provides information related to these activities to ABACC and IAEA.

The IPEN Safeguard Service team acts in collaboration with several operators from different Material Balance Areas (MBAs/IPEN). The team makes the annual planning schedule and pre-inventory procedures to accomplish the Physical Inventory Taking (PIT), checks the Design Information Questionnaire (DIQ) and carries out the Physical Inventory Verification (PIV) in order to realize the inspections of the Brazilian National Authority (National Nuclear Energy Commission / CNEN), Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials (ABACC) and International Atomic Energy Agency (IAEA) in compliance with the Quadripartite Safeguards Agreement (INFCIRC 435). This agreement was signed by Argentina, Brazil, ABACC and IAEA, and it adopts the Common System of Accounting for the Control of Nuclear Materials SCCC for Brazil / Argentine. During the period 2008-2010, the Safeguards Service team managed the nuclear material control of the areas under the INFCIRC 435 agreement and carried out records as the General Accountability Book and the Notifications for Movement of Nuclear Material to support the Inventory Variation Report issued by CNEN. The Safeguard Service team has been following the inspections of CNEN, ABACC and IAEA.

Environmental risk assessment

Evaluation of the contamination risk caused by lightning rods disposed at uncontrolled garbage dumps

Americium-241 (^{241}Am) migration experiments were performed in order to evaluate the risk of contamination caused by disposition of radioactive lightning rods as common solid waste. Sources removed from lightning rods were placed inside lysimeters filled with organic waste, and the generated leachate was periodically analyzed to determine its characteristics, such as pH, redox potential, solid content and the concentration of the radioactive material. Besides the risk evaluation, the mechanism of the ^{241}Am release or retention in waste as well as its influence in the waste decomposition processes was investigated. Leachate samples collected in a lysimeter were periodically analyzed for bacterial growth, under both aerobic and anaerobic conditions. Results revealed that ^{241}Am inhibited bacterial growth, and the degradation of organic matter was delayed in comparison with the control. Figure 1 shows the inhibition of bacterial growth by americium.

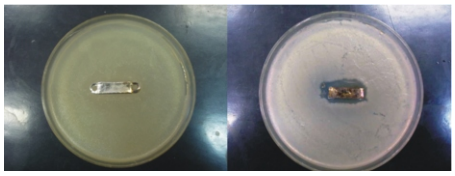

Figure 1. Culture plates showing antimicrobial action of americium: a) steel base without ^{241}Am; b) bacterial growth inhibition halo around the ^{241}Am strip

Public perception of risks associated with radioactive waste

It is well known that one of the major concerns regarding the peaceful use of nuclear energy is related to the safe management of the radioactive wastes. Nuclear energy has always been a polemic issue, and it is usually more intensely associated, by the general public, with the risks than with the benefits from its use. The aim of this study is to try to understand how local public will face to the construction of a nuclear waste repository and what role the mass media plays in the formation of public opinion. The coverage of the nuclear issue in the press, identifying the amount of negative and positive views on the subject, as well as the people involved in the process, were analyzed comprising the period from February 2007 to February 2008. From that sample, 172 texts published in widely known newspapers and magazines, most of them from Sao Paulo State, were identified. The analyzed speeches were classified according to five information sources: specialists, users, authorities, protagonists and entrepreneurs. It was noted the predominance of positive articles, mainly due to the weight of the favorable speeches by authorities, cited as the most frequent in this paper.
Another ongoing study deals with the assessment of the public perception of risks associated with the construction of the national radioactive waste repository, aiming at supporting project managers to establish an effective communication program with the society. A questionnaire to be applied in an opinion pool have been developed, in order to evaluate the following aspects: attitudes toward radioactive waste and nuclear power, risk denial, beliefs, stigmata, social and epistemic trust, antagonism, expressed preferences, technological risk, emotional reactions and the precautionary principle. The questionnaire is being applied for validation and adjustment.

Radioactive waste characterization, treatment and disposal

Characterization of ion exchange resins and activated charcoal

The radioactive waste characterization program of GRR follows the guidelines of "IAEA-TEC-1537 - Strategy and methodology for radioactive waste characterization" in order to complete, for each waste stream and each waste package, the waste form required by CNEN-NN 6.09 - Acceptance criteria for disposal of low - and intermediate- level radioactive wastes. The main development goals are to set up routine radioanalytical methods and the determination of scaling factors and correlation functions that allow calculate the radioactive inventory of difficult to measure radionuclides (DMR) from the activity of key radionuclides (KR), present in the wastes. DMR include alpha and pure beta emitters, and low energy, low yield gamma emitters that require radiochemical treatment of waste samples to be quantified. KR include gamma emitters that can be detected and quantified by simple gamma spectroscopy or calibrated gamma scanning of waste packages, like Cobalt-60 and Caesium-137. This method is being applied for characterization of ion-exchange resins and activated charcoal beds replaced from the water treatment system of the IEA-R1 research reactor. Radioanalytical methods for determination of twenty one fission and activation products as well actinides were developed and/or implemented to measure the radioactivity concentration of forty nine samples taken from twenty one waste drums containing that kind of waste.

Optimization of the radioactive waste storage

IPEN is optimizing the radioactive waste storage capacity, taking into account that a fraction of the stored treated wastes has decayed to a very low level and considering that "retrieval for disposal as very low level radioactive waste" is one of the actions suggested to radioactive waste managers. The optimization study evaluated two main options: either to maintain the present situation or to open old packages and segregate the wastes that may be subject to clearance levels. The results showed that maintaining the present situation is not the best option, even though some parameters of each scenario may be reevaluated. In 2010, a new storage facility was constructed and all treated waste packages were transferred to it. All the

packages that may be subject to clearance levels were stored separately for further segregation.

Isotopic characterization of radioactive waste drum

An automated system for isotopic characterization of radioactive waste by gamma scanning of waste drums was developed (Fig. 2). The detection system is composed of an HPGe detector and associated electronics. The drive system of drum is automated and controlled by a PLC (Programmable Logic Controller). This system allows controlling the elevation and rotation of a base where the radioactive waste drum is positioned. The system operates in continuous and in programmable mode, in which the number of measurements, operation time and the axial positioning of the detector in the drum can be preset. This system associated with the mathematical techniques such as Monte Carlo Method and Artificial Neural Networks are efficient in isotopic characterization of radioactive waste drums.

Figure 2. Automated system for isotopic characterization of radioactive waste drums

Treatment and disposal of disused sealed sources

The R&D work undertaken in respect to the management of lightning rods, smoke detectors and other disused sealed radioactive sources (SRS) is divided into designing the facilities to handle the sources, decontaminate lightning rod scrap metal and into developing the concept of a deep, fully-dedicated repository to dispose of SRS.

During the reported period, the hot cell designed to handle the sealed sources was further assembled, with wiring, pneumatic lines and accessories being partially installed. The methodology of SRS characterization was developed either for simple certification of the data supplied by owner or for identification of the radionuclide and quantification of its activity in the case of orphan sources.

The contribution of the IPEN for the establishment of a feasible alternative for final disposal of SRS continued in the period, with further development the concept of repository in a deep borehole. The assessment of the durability of cementitious material under the conditions deemed to prevail in the deep environment continued by accelerated tests in laboratory. Specimens of Portland cement paste undergone irradiation and high temperature storage and the effects on the paste properties were analyzed by X-ray diffraction, electron microscopy and other techniques (Fig. 3). The results of this work showed that exposure of cement paste specimens to the stressing factors induced changes in cement properties that could affect the performance of this engineered barrier in the long term. Results also showed that some analytical methods were capable of detecting those changes in the cement properties, for instance, mineralogy changes detected by X-ray diffraction and by scanning electron microscopy while others must be improved, e.g. reduction of mechanical strength as measured by axial compression of exposed specimens.

The methods for assessing the safety of such a facility were investigated in two lines of research: one looking for qualitative and quantitative safety indicators, which could be applied to the licensing of a disposal facility, and the other integrating different lines of evidence to build confidence in the acceptability of the disposal concept.

Aspects of site analysis and selection for a disposal facility were approached by searching literature with description of the geological features of a model sites.

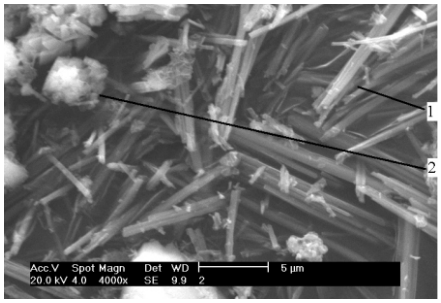

Figure 3. Scanning electron micrograph of precipitates on the cement paste specimen surface showing needles of ettringite (1) and grains of Porlandite (2)

Treatment of radioactive liquid wastes using different biomasses

In order to reduce the volume of radioactive liquid wastes stored at the IPEN, the method based on the capacity of biopolymers to remove heavy metals from wastewater has been developed. Among the biopolymers, alginate beads, agricultural waste (sugar cane bagasse, coconuts fiber and rice husks) and microbial (Saccharomyces cerevisae, Ochrobactrum sp, C. metallidurans and B subtilis) biomasses have been studied to remove Americium-241 from aqueous solution. The following parameters were evaluated: contact time, pH and radionuclide concentration. The removal of plutonium and cesium from radioactive liquid

wastes were also assessed using agricultural wastes as vegetal biomasses. The bioremoval technique has been so far considered efficient (removal >90%), low cost and sustainable. Figure 4 shows the reduction of waste volume obtained with alginate beads.

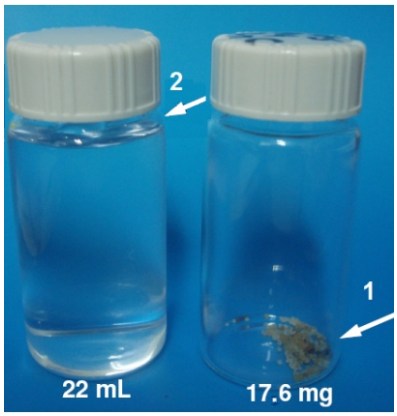

Figure 4. Reduction of the volume using alginate beads: 1- aqueous solution with Americium-241; 2- alginate beads after the biossorption process

Occupational epidemiology

The main evidence for the presence or absence of various health outcomes is provided by epidemiological investigations. The main objectives of the research group are:

- To get a solid introduction and a detailed study of the basic epidemiologic methods including the special features of occupational epidemiology;
- To assess the different types of epidemiological study, the applications, advantages, and limitations of the major types of observational and experimental studies, emphasizing the many possibilities for errors in epidemiological for a clear understanding;
- To use epidemiological principles and methods to the practice application of data derived from epidemiologic research, in particular for the radiation epidemiology;
- To determine the possible health consequences of the workplace exposures (exposure standard setting) and to recommend remedial efforts, when applicable.

Presently, with the emphasis in studies about quality of life (QOL) involving workers with potential risk to ionizing radiation has been carried out, in order to identify the social-demographic factors related to these workers and its influence on QOL, as well to establish the profile of workers handling radioactive material.

Radioactive waste management

The Radioactive Waste Management Department is responsible for reception, treatment and interim storage of the radioactive waste generated at IPEN, as well as those generated at many other radioactive facilities all over the country. IPEN has units for: waste reception and segregation; decontamination of small pieces; liquid waste immobilization and conditioning; in-drum compaction; disassembling of radioactive lightning rods; storage of disused sealed sources; storage of untreated and treated wastes; characterization of primary wastes and waste forms. The existing storage for treated waste is being restructured to receive 850 m^2 of extra area, divided into two sheds. The first shed was concluded in 2010 and treated waste was transferred from the old building to the new one (Fig 5). The second shed will be concluded by July, 2011 to receive untreated wastes. It is noteworthy that in the reported period 113 neutron sources were repatriated to Los Alamos National Laboratory (USA), as part of cooperation program between CNEN and IAEA.

Figure 5. New radioactive waste storage building (outside and inside view)

Nuclear Safety

Radioprotection Service

The main task of the Radioprotection Service of IPEN is to provide for IPEN workers and for general public an adequate protection against ionizing radiation. The Radioprotection Service implements appropriate procedures and monitoring techniques according to the national and international standards.

The team helps the employer to comply with the requirements specified by the National Regulatory Authority. The Radioprotection Service provides support to general obligations for any practices which involve or could involve exposure to radiation or radioactive substances in compliance with the standards that include:

- Preparation of local rules and procedures;
- Designation of radiological areas;
- Control and accounting of radioactive material;
- Restriction of exposure;
- Optimization of radioprotection for practices;
- Individual dosimetry (internal and external) and dose assessment;
- Ocupational and environmental control and contamination monitoring;
- Contingency planning and radiological risk assessment;
- Training in radiological protection.

In additional, when required, the Radioprotection Service can provide the following services:

- Preparation and review of radiological protection aspects of safety documents;
- Advice and assistance on radiological aspects of categorization of plant and modifications;
- Participation in safety audits;
- Support to engineering projects;
- Analysis of transport packages and waste contents, including assistance with waste characterization;
- Investigation of abnormal dosimetry results;
- Routine reports on personal dose statistics;
- Provision of appropriate radiological information for reports;
- Personal protective equipment including respiratory protection;
- Preparedness and emergency response involving radioactive material.

The Radioprotection Service is available to the customers 24 hours per day. During 2008-2010, the Radioprotection Service in the field of emergency response and preparedness in Brazil has been carried out according to the expected work program.

Concerning the program for the improvement of infrastructures for protection and safety at IPEN, the Radioprotection is the authority responsible for managing the radiological activities survey of access areas under the direction and instructions of Radioprotection staff. The Radioprotection Service is updating in a continuous way its procedures in order to fulfill the new legal requirements derived from the Standards.

Preparedness and response to nuclear and radiological emergencies

IPEN is an operational unit of the Protection System for the Brazilian Nuclear Program (SIPRON) that is a group of organizations with the objectives of the integrated planning, the combined action and the continuous execution of measures in order to assure the nuclear safety in the country and to respond to radiological and nuclear accidents in Brazil. IPEN also takes part in the implementation of the Emergency Situation plan that was developed by the National Commission of Nuclear Energy (CNEN) to respond to nuclear or radiological emergencies, as loss of radioactive sources and accidents during the transport of radioactive material.

The Nuclear and Radiological Emergency Response Team (NRERT) of the Radioprotection Service is responsible for the evaluation and first response to situations of nuclear or radiological emergencies in São Paulo state. NRERT works with other federal and local agencies to monitor, contain, and clean up the release of radioactive material for protecting people and the environment from harmful exposure to radiation.

Training in radiation protection at IPEN

The Radioprotection Service is responsible for the development and implementation of training in radiation protection for a range of users and applications of ionizing radiation. This activity has been established to attend: the training requirements for IPEN workers for any levels; to emergency response personnel, such us fire fighters, civil defense personnel; and to provide and disseminate information in radiation protection education for students and community.

Workers who are occupationally exposed to ionizing radiation need more extensive and deeper training to ensure that radiation is used safely. The training of the principles of radiation protection is based on the Standards of the CNEN and IAEA (International Atomic Energy Agency). The competences are acquired, developed and maintained through a programme of regular training. The courses are offered, such as basic training, refresher training and on the job training. The content and level of the courses offered are established for each category of persons to be trained.

For the 2008-2010 period, the courses were offered periodically. Upon completion of the course, an examination is given to authenticate the program requirements. A Certificate of Achievement is provided to those who have successfully completed the course, and a permanent record of training is kept in the Radioprotection office. The basic course covers the general principles of occupational

radiation protection in the following subject areas: basic radiation physics, definitions and units of radioactivity, principles of radiation protection against external and internal exposures, biological effects of radiation, the risk and assessment of such exposures, instrumentation, inventory and contamination control, emergency response, requirements of the National and International Standards and IPEN procedures. After this basic course all workers must be trained in this specific practice in each work area. The Radioprotection Service provided training to workers as shown in Figure 6.

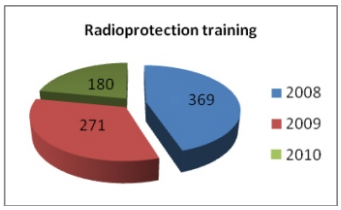

Figure 6. Number the workers trained on protection and safety over the years

In addition, the Radioprotection Service has participated in graduate course to obtain Master Science and Ph.D. Title in the Nuclear Technology-Application Area.

Nuclear Safety

Program Team

Research Staff

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Graduate Students

Alex Barbosa; Amanda Juliene da Silva; Bianca Geraldo; Cristiane Teixeira Manetti; Eliana Rodrigues de Leite; Felipe Ramos Correa; Ivani M. Fernandes; Kátia Suemi Tanimoto; Leandro Goulart de Araujo; Maurício Guimaraes Sabbag; Paula Perrucho Nou Silva; Rafael Vicente de Pádua Ferreira; Rosana L'agua de Oliveira; Tânia Regina de Borba.

Undergraduate Students

Aline Sayuri Takara; Alvaro Wei Chiang Wang; Ana Carolina Martins; André de Oliveira; Carina Machado do Nascimento; Catia Kim; Daiane Cristini Barbosa de Souza; Daniel Terio Kanashiro; Denise Minematsu; Eduardo Gurzoni Alvares Ferreira; Érico Sant'anna Perrella; Fernando Nogueira da Silva; Guilherme Altomari Geribola; Heverton Cardan Oda Fonseca; Jacqueline dos Santos Rodrigues Silva; Jaime Souza Borges; Josenilson Barbosa de Lima; Juliana Meira Matavelli; Lucyana Martin Falcon Gomes; Ludmila Cabreira Vieira; Luis Henrique Florêncio; Marcos Paulo Pianissola de Cerqueira; Maximiliano Bruno da Silva Guimarães; Mirella Curto Gomes; Nadia Suemi Nobre Ota; Paulo Henrique Senise; Priscila Batalha de Lima; Reginaldo Vitorino dos Santos; Renato Camargo; Rozilene Elaine da Silva; Zenaide Aparecida Figueiredo.

Co-Workers

Dr. Gian-Maria A. A. Sordi; MSc. Adelia Sahyun; MSc. Maria Helena Tirollo Taddei.

Honor Mentions and Awards

Catia Kim, an undergraduate student supervised by Dr. Júlio Takehiro Marumo of Waste Management Department, received an award for the third best junior poster presented at INAC - International Nuclear Conference, held in Rio de Janeiro from September 27 to October 2, 2009. The title of work is "Application of advanced oxidative process in treatment radioactive waste".

Education

The Education Program at IPEN aims to develop human resources and to provide and disseminate scientific information in nuclear and correlated areas

Education

Introduction

The Education Program at IPEN aims to develop human resources through scientific training programs and to provide and disseminate scientific information in nuclear and correlated areas.

IPEN is responsible for the graduate program in the nuclear area at University of São Paulo, the Nuclear Technology Program IPEN/USP. Since its creation, in 1976, the Program was evaluated with grade A by the Federal Government Evaluation (CAPES), the highest in this classification. In 2003 CAPES changed the evaluation criteria; since then, it has been considered a program of Excellence, with grade 6. Levels 6 and 7 are granted only to those programs having internationally recognized expertise. Level 6 was maintained in the last evaluation considering the period 2007-2009. Along its 34 years the Nuclear Technology Program awarded 1813 titles: 1238 master's degrees and 575 doctor's degrees.

The institution is also responsible for the Professional Master Degree - Lasers in Dentistry, in partnership with the School of Dentistry from University of São Paulo.

IPEN has a Scientific Initiation Program for undergraduate students aiming to stimulate young people to enter the scientific research career. This program allows the student to enter scientific world under the guidance of a supervisor to develop a specially assigned study. CNEN and CNPq are the main funding agencies supporting this Program. The institute also offers, since 2000, undergraduate disciplines for students of University of São Paulo. A total of 21 disciplines have been approved by the University.

There is also a Scholarship Program for graduate students, funded by CNPq, CAPES and Fapesp. Since 2006, CNEN and IPEN started a scholarship program, a long lasting demand from the IPEN community.

The postdoctoral program regulating the activity at the institution was approved in 2005. Since then, around 100 professionals were enrolled in the program.

To accomplish all the activities mentioned above, IPEN has a Board of Education and a dedicated building, having facilities such as: one hall for events, two auditoriums (70 people approximately), two computer labs and 10 classrooms.

Concerning scientific information support, there is available a central specialized library, which offers, beyond traditional collections and services, a comfortable study place for the students and the IPEN community, nicely equipped with wireless and stations for search support and consult to all the information resources.

The education page on the IPEN website provides information about the structure and application procedures for the graduate program on nuclear technology, the professional master degree program on lasers in dentistry and the Scientific Initiation program for undergraduate students. Other information like the disciplines to be offered, the advisors for the graduate program, and the scholarships offered and conditions and period for submitting applications are also available.

Nuclear Technology Graduate Program

IPEN is responsible for the graduate program in the nuclear area at University of São Paulo, the Nuclear Technology Program IPEN/USP. This Program started at 1976 and presents nowadays three concentration areas: Applications, Materials and Reactors. The Federal Government Evaluation (CAPES) granted grade 6, considering it a program of Excellence, for the period comprehending 2007-2009. IPEN has maintained this grade since 2003. Until 2010 the program awarded 1813 titles: 1238 master's degrees and 575 doctor's degrees. The performance during this period is shown in Table 1.

Table 1. Nuclear Technology Graduate Program

	2008	2009	2010
Master's degrees awarded	109	101	93
Doctor's degrees awarded	39	46	31
Offered disciplines	87	89	89
Total graduate enrollment	592	598	565

Professional Master Degree

IPEN is also responsible for the Professional Master Degree - Lasers in Dentistry, in partnership with the School of Dentistry from University of São Paulo. This program began in 1999 and, till now, 131 master's degrees were conceded. This program was evaluated with the highest degree by CAPES. It is considered a model program. The numbers for the period are presented in Table 2.

Table 2. Professional Master Program

	2008	2009	2010
Master's degrees awarded	1	9	13
Offered disciplines	8	8	8

Undergraduate disciplines

The institute also offers, since 2000, undergraduate disciplines for students of the University of São Paulo. A total of 21 disciplines have been approved by the University (Table 3).

Table 3. Undergraduate Program

	2008	2009	2010
Offered disciplines	15	18	16
Enrolled students	284	325	333

Scientific Initiation Program

The institute has a Scientific Initiation Program for undergraduate students aiming to stimulate young people to enter the scientific research career. This program allows the student to enter scientific world under the guidance of a supervisor to develop a specially assigned study. CNEN and CNPq are the main funding agencies for a Scientific Initiation Scholarship Program supporting these activities. The number of scholarships offered in the period is shown in Table 4.

Table 4. Undergraduate Scholarship Program

Funding Agency	2008	2009	2010
CNPq	45	50	60
FAPESP	9	6	7
CNEN	25	25	25
TOTAL	79	81	92

Graduate Scholarship Program

There is also a Scholarship Program for graduate students, funded by CNPq, CAPES, Fapesp. Since 2006, CNEN and IPEN started a scholarship program, a long lasting demand from the IPEN community. The total number of scholarships awarded during the period considered is presented in Table 5.

Table 5. Scholarship Programs

Funding agency	DOCTOR DEGREE		
	2008	2009	2010
CNPq	36	35	36
CAPES	17	20	29
FAPESP	14	15	24
CNEN	4	9	4
IPEN	3	7	7
TOTAL	**69**	**79**	**89**

Funding agency	MASTER DEGREE		
	2008	2009	2010
CNPq	24	24	24
CAPES	15	20	29
FAPESP	18	10	10
CNEN	17	15	11
IPEN	14	16	17
TOTAL	**76**	**80**	**83**

Postdoctoral Program

The postdoctoral program regulating the activity at the institution was approved in 2005. During the period considered in this report, around 80 professionals were enrolled in the program and 30 were supported by postdoctoral scholarships.

Education

Facilities and support

To accomplish all the activities mentioned above, IPEN has a Board of Education and a dedicated building, having facilities such as: one hall for events, two auditoriums (70 people approximately), two computer labs and 10 classrooms.

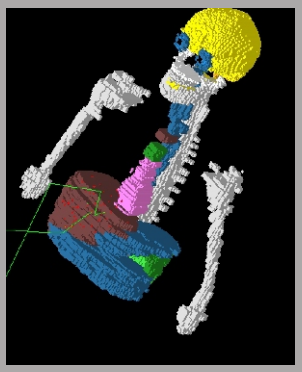
Concerning scientific information support, there is available a central specialized library, which offers, beyond traditional collections and services, a comfortable study place for the students and the IPEN community, nicely equipped with wireless and stations for search support and consult to all the information resources.

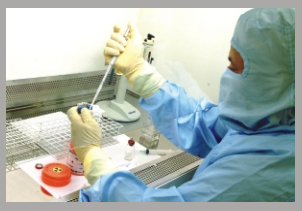
There is a technical and reference information desk provided by the staff. Access to all the main electronic information resources in the country, like electronic journals provided by CAPES and USP data bases are assured and managed through collaborative arrangements.

The library also manages the institutional repository with 65% of the full text records authored by the IPEN scientific community.

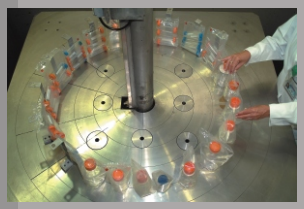
The education page on the IPEN website provides information about the structure and application procedures for the graduate program on nuclear technology, the professional master degree program on lasers in dentistry and the Scientific Initiation program for undergraduate students. Other information like the disciplines to be offered, the advisors for the graduate program, and the scholarships offered and conditions and period for submitting applications are also available.

Technical and Scientific Production






Patents Pending	180
Journal Articles	181
Thesis and Dissertations	232

Technical Production

Patents Pending

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MACHADO, ADRIANE; LIMA, EVANDRO, F. de; CHEMALE JUNIOR, FARID; ALEXANDRE, FELIPE M.; SOMMER, CARLOS A.; FIGUEIREDO, ANA M.G.; ALMEIDA, DELLA DEL P.M. de. Mineral chemistry of volcanic rocks of South Shetland Archipelago, Antarctica. ***International Geology Review***, v. 50, p. 154-162, 2008.

MARUMO, JULIO T.; ISIKI, VERA L.K.; MIYAMOTO, HISSAE; FERREIRA, RAFAEL V.F.; MARUMO, MARIA H.B.; LIMA, LUIS F.C.P. de. Investigation of the radiation risk due to environmental contamination by sup(241) Am from lightning rods disposed at uncontrolled garbage dumps. ***Radiation and Environmental Biophysics***, v. 47, p. 131-137, 2008.

VENTURINI, LUZIA; SORDI, GIAN M.A.A.; FERNANDES, FERNANDA. Anatomical data and development of geometric models for the knee region bones. ***Radiation Protection Dosimetry***, v. 129, n. 4, p. 486-490, 2008.

CALVO, FERNANDA B.; SANTOS JUNIOR, DARIO; RODRIGUES, CONSUELO J.; KRUG, FRANCISCO J.; MARUMO, JULIO T.; SCHOR, NESTOR; BELLINI, MARIA H. Variation in the distribution of trace elements in renal cell carcinoma. ***Biological Trace Element Research***, v. 130, n. 2, p. 107-113, 2009.

FERREIRA, RAFAEL V. de P.; SAKATA, SOLANGE K.; ISIKI, VERA L.K.; MIYAMOTO, HISSAE; BELLINI, MARIA H.; LIMA, LUIS F.C.P. de; MARUMO, JULIO T. Influence of americium-241 on the microbial population and biodegradation of organic waste. ***Environmental Chemistry Letters***, on line, 2009.

CARNEIRO, JANETE C.G.; BETTI, FLAVIO; SANCHES, MATIAS P.; SORDI, GIAN M.A.A.; PECEQUILO, BRIGITTE R.S. Assessment of the environmental outdoor gamma radiation levels in Sao Paulo city. ***International Journal of Low Radiation***, v. 7, n. 5, p. 359-365, 2010.

SANCHES, MATIAS P.; CARNEIRO, JANETE C.G.; SORDI, GIAN M.A.A. Social-demographic profile and dose evaluation of the radiopharmaceutical facility workers. ***International Journal of Low Radiation***, v. 7, n. 6, p. 467-472, 2010.

PALETTA, FRANCISCO C.; VIEIRA JUNIOR, NILSON D. Information technology and communication and best practices in it lifecycle management. ***Journal of Technology Management Innovation***, v. 3, n. 4, p. 80-94, 2008.

ZOUIAN, DESIREE; DAMIAO, DEVANILDO; CATHARINO, MAURO; GARGIONE**,** LUIS A. Proposta de uma metodologia orientada para o uso em incubadoras de base tecnológica. ***Revista Ciências Administrativas, Fortaleza***, v. 14, n. 1, p. 43-53, 2008.

MIORANZA, CLAUDIO; AQUINO, AFONSO R. de. Modelo multidimensional de avaliação da qualidade educacional - MULTQUALED. ***Revista Iberoamericana de Educacion***, v. 49, n. 6, p. 1-20, 2009.

SOUSA, WILLY H. de; IGAMI, MERY P.Z.; BIDO, DIOGENES de S. RD management and the Stokes Diagram: an exploratory study. ***Journal of Technology Management Innovation***, v. 4, n. 4, p. 95-109, 2009.

ZOUAIN, DESIREE M.; D'ELIA, MARCO A.G. "Pseudo technical barriers" to medical devices exportation. ***Espacios***, v. 30, n. 2, p. 25-30, 2009.

ALMEIDA, MARIA L.O.D.; ZOUAIN, DESIREE M. Programas interlaboratoriais - Parte I - Importância no setor de celulose e papel. ***O Papel***, n. 5, p. 38-51, 2010.

ALMEIDA, MARIA L.O.D.; ZOUAIN, DESIREE M.; KOGA, MARIZA E.T.; YOUN, DANIELA. Programas interlaboratoriais - Parte II - Estudo de caso para o ensaio de gramatura de papel. ***O Papel***, n. 5, p. 53-62, 2010.

CAMARGO, ROSANA; BARROSO, ANTONIO C. de O. A educação na era do conhecimento. ***Sinergia***, São Paulo, v. 11, n. 1, p. 79-85, 2010.

PINTO, ADILSON L.; IGAMI, MERY P.Z.; BRESSIANI, JOSE C. Visibilidade e monitoramento científico na área nuclear e ciências relacionadas: uma perspectiva a partir da produtividade do IPEN-CNEN/SP. ***Perspectivas em Ciência da Informação***, v. 15, n. 2, p. 198-218, 2010.

SATO, RENATO C.; ZOUAIN, DESIREE M. Markov models in health care. ***Revista Einstein***, v. 8, n. 3, p. 376-379, 2010.

SOUSA, WILLY H. de; GIARDINO, ADRIANO; MAGALHAES, ADRIANA B.V.B.; FEHER, ANA C.M.; PASCHOAL, JOSE O.A. The construction of scenarios: lessons learned at a brazilian nuclear and energy research institute. ***International Journal of Nuclear Knowledge Management***, v. 4, n. 2, p. 114-131, 2010.

Scientific Production

Thesis

2008

ANDRADE, MARCOS L.G. ***Neutron induced electron radiography***.

BANCZEK, EVERSON do P. ***Development and characterization of zinc/nickel and zinc/niobium phosphate layers***.

CARMO, MARCELO do ***Preparation, characterization and evaluation of electrocatalysts supported on functionalized carbon black for polymer exchange membrane fuel cell applications***.

CECILIO, PAULO J. ***Commissioning and implantation of the dynamic intensity modulated radiation therapy using secondary multi-leaf collimator (MLC)***.

COSTA, GUSTAVO C.C. da ***Synthesis, structural, thermochemical and electrical characterization of ceramic materials for solid oxide fuel cells***.

CRUZ, SIMONE F. de A. ***Single crystals growth and characterization of BaY_2F_8:TR (TR = Nd^{3+}; Pr^{3+}; Er^{3+}; Tb^{3+}; Dy^{3+})***.

DEANA, ALESSANDRO M. ***Study, development and optimization of a diode pumped Er^{3+}:YLF laser emitting in the 3 'micron'm region***.

EMILIO, CLAUDIA R. ***Comparison of efficacies of ALA-PDT and methyl aminolevulinate-PDT in the treatment of feline squamous cell carcinoma***.

GALEGO, EGUIBERTO ***Study of alloys and magnets prepared by the hydrogenation, disproportionation, desorption and recombination process (HDDR) based on the Pr-Fe-B system with dopants and alloying additions***.

GALISTEO JÚNIOR, ANDRES J. ***Toxoplasma gondii vs ionizing radiation: Cell and humoral immunity in spleen and gut of isogenic mice immunized with ^{60}Co irradiated tachyzoites***.

GERALDES, ADRIANA N. ***Polymer fluorinated membranes for fuel cells***.

GHOBRIL, CARLOS N. ***Technology management for the installation of a food irradiator in the Ribeira Valley***.

GONCALVES, VALERIA de S. ***Influence of process control agents on the mechanically activated combustion synthesis of $NbAl_3$.***

GOUVEIA, DEIBY S. ***Obtention of hydroxyapatite nanometric powders synthesized with magnesium using ultrasound***.

LIMA, EMMANUEL P.R. ***Development and performance analysis of sintered high speed steels tools***.

MACEDO, LUIZ A. ***Performance of a passive emergency heat removal system of advanced reactors in two-phase flow and with high concentration of non-condensables***.

MALAFRONTE, ALEXANDRE A. ***Project and implementation of the control system for the microtron accelerator***.

OLIVEIRA, FÁBIO B.V. de ***Development of a high density fuel based on uranium-molybdenum alloys with high compatibility in high temperatures***.

OLIVEIRA, LAURA C. de ***Study of the distribution of ions and metals in blood using nuclear methodology***.

PALETTA, FRANCISCO C. ***Information technology, innovation and entrepreneurship: Success critical agents using management tools on based-technology incubators firms***.

PEREIRA, MARCO A.S. ***Neutron-Induced alpha radiography***.

PRADO, ULISSES S. do ***New alternative for reuse hazardous waste from the primary aluminum production process (SPL): Production of the opal glass and frits***.

PUGLIESI, FÁBIO ***Characterization of the polymer Durolon as a solid state nuclear track detector***.

QUEIROZ, FERNANDA M. ***Investigation on the corrosion behavior of the intermetallics in the AA2024-T3 alloy by microscopy associated to electrochemical techniques***.

RIBEIRO, CHRISTIANE ***Obtention and characterization of porous bioceramics based calcium phosphate processing by using albumin***.

ROCHA, CLÁUDIO J. da ***Mechanical activation mechanisms of niobium and aluminum mixtures for the reaction synthesis of $NbAl_3$.***

SANTOS, IVAN ***Uranium hexafluoride production plant decommissioning***.

SCAPIN, MARCOS A. ***Inorganic elements and organic compounds degradation studies by gamma irradiation in used lubricating oils***.

SERNA, MARILENE M. ***Quantification and Identification of the Carbides in Tool Steel AISI/SAE M2***.

SILVA, ANGELICA M. da ***Environmental quality in area of influence of mud of stations for treatment of water and sewage***.

SILVA, ANTONIO C. da ***Glass and glass-ceramics with high metal concentration of from industrial wastes***.

SILVA, MURILO C. da ***Interaction study of water radiolysis products with crotalus durissus terrificus miotoxin***.

SOARES, EUFEMIA P. ***A study on the radiometric method for evaluating element migration from plastic packaging to its contents***.

SOUZA, EDUARDO C.C. de ***Microstructure-property relationship in $Ce_{1-x}Sm_xO_{2-}$ solid electrolyte prepared from nanoparticles***.

TERADA, MAYSA ***Corrosion of advanced stainless steel in physiological solutions***.

TORRES, ANI S. ***Development of a methodology for an independent water vapor Raman LIDAR calibration to study water vapor atmospheric profiles***.

TORRES, WALMIR M. ***Development of a technique for level measurement in pressure vessels using thermal probes and artificial neural networks***.

VIEIRA, ANDRÉ M. de M. ***Dosimetry systems for stereotactic radiosurgery with linear accelerators equipped with micro multileaf collimators***.

ZEITUNI, CARLOS A. ***Dosimetric study in iodine-125 seeds for brachytherapy application***.

2009

ALEXANDRIA, JOAO C.S. de. ***Information security management - a proposal to improve the effectiveness of information security in the scientific research environment***.

ALVES, JANAINA B. ***Immune response against the irradiated Bothropstoxin-1: Identification of main cytokines involved and the participation of scavengers substances***.

ARONNE, IVAN D. ***Development of a transient identification and classification system to an integral pressurized water reactor***.

AVEGLIANO, ROSEANE P. ***Total diet study in São Paulo state: estimation of dietary intakes of toxic (arsenic and cadmium) and essential elements (calcium, chromium, iron, selenium, sodium, potassium and zinc)***.

BARBOSA, JOSE A.M. ***Contribution to the Brazilian legislation in nuclear energy field***.

BERNARDI JUNIOR, PAULO. ***Alternatives for the hydrogen production in the Brazilian regions aiming at to generation of distributed electric energy***.

BERNARDI, CATERINA V. ***Perspectives of the generation of carbon credits on the basis of the attainment of a fertilizer - exploitation of residues of biomass of Brazilian agriculture***.

CALDEIRA FILHO, JOSE de S. ***Synthesis, analysis, purification and biodistribution in an animal model of radiopharmaceutical $^{177}Lu^{3+}$ - dotatato for diagnostic and therapeutic use in neuroendocrine tumors***.

CALY, JOSE P. ***Study and evaluation of radiometry in phototherapeutic treatment of the neonatal hiperbilirubinemia***.

CAMARGO, FABIO de ***Development of dosimeters with rad-hard silicon diodes for high dose dosimetry***.

CARDOSO, SERGIO G. ***Study of mechanical properties and fracture mechanisms of synthetic fibers like nylon and polyester in engineering fabrics***.

CAVALCANTE, FABIO H. de M. ***Hyperfine interaction study in $RCoO_3$ (R = Gd and Tb) and HfO_2 thin film oxides by perturbed angular correlation technique***.

CUNHA, EDGAR F. da ***Evaluation and application of PEMFC fuel cell's technologies developed at IPEN applied to a 500 We fuel cell stack***.

FANARO, LEDA C.C.B. ***Experimental determination of spectral indices by scanning of fuel rod in the IPEN/MB-01 reactor***.

FERRO, WALDIR P. ***Utilization of rice husk ash as filler for polyamide 6 and ionizing radiation effect studies on this composite***.

MELO, ROBERTO C. de ***Study of cognitive and technological prerequisites for virtual laboratories and collaborative virtual environments for radiopharmacy***.

MIORANZA, CLAUDIO ***Development and application of multidimensional model for the quality evaluation of graduate-level education in program of the IPEN***.

MORCELLI, APARECIDO E. ***Study of deformation and fracture micro mechanisms of titanium alloy Ti-6Al-4V using electron microscopy and X-ray diffraction techniques***.

OLIVEIRA, HENRIQUE B. de ***Analytical models for development of high performance metal targets irradiated in IPEN-CNEN/SP cyclone 30 and cyclone 18 cyclotrons***.

OLIVEIRA, MARA C.L. de ***Preparation of Pr-Fe-Co-B-Nb-M (M=Al, P, Cu, Ga and/or Gd) HDDR magnets and alloys and characterization of their magnetic properties and corrosion resistance***.

OLIVEIRA, MARCOS R.R. de ***Evaluation by EPR of potential antioxidant components of ^{60}Co-irradiated varieties of soybean***.

PEREIRA, MARCO A. dos S. ***Study of the action of ^{60}Co gamma radiation on Salmonella poona, Escherichia coli and Alicyclobacillus acidoterrestris in mango pulp***.

PERIGO, ELIO A. ***Relations microstructure - magnetic properties - squareness factor of PrFeB and NdFeB sintered magnets prepared with hydrogen***.

RIBEIRO, MARIA A.M. ***Contribution to environmental impact study of fuel cells of low and medium temperature using the Delphi methodology***.

RICCIARDI, RITA I. ***Strategic knowledge management a methodology for structuring and analyzing knowledge resources***.

Scientific Production

Thesis

ROBALINHO, ERIC ***Development of a computational model applied to a unitary 144 cm^2 proton exchange membrane fuel cell***.

RODRIGUES, DEMERVAL L. ***Radiological impact from the transport of radiopharmaceuticals IPEN-CNEN/SP***.

ROMANATO, LUIZ S. ***Study of a Brazilian cask and its installation for PWR spent nuclear fuel dry storage***.

SALVADOR, PABLO A.V. ***Monoenergetic and polyenergetic gamma ray computer tomography for multiphase systems analysis***.

SANTORO, THAIS A. de B. ***Preparation and characterization of Pt-rare earth/C electrocatalysts for PEM fuel cells.***

SANTOS, JOSEFINA da S. ***Evaluation of radiolabeling of annexing A5 with technetium-99 m: influence of the labeling methods on physico-chemical and biological properties of the compounds.***

SANTOS, LAURO R. dos ***UO_2-7%Gd_2O_3 fuel process development by mechanical blending with reprocessing of waste products and usage of densification additive***.

SCAPIN, VALDIRENE de O. ***Assessment of contamination for inorganic elements and phthalate esters in household dust from the metropolitan region of São Paulo***.

SETZ, LUIZ F.G. ***Lanthanum chromite colloidal processing***.

SILVA, DEVANILDO D. da ***Articulation of the innovation system in Sorocaba city: a study based on the Brazilian experience of innovation habitats and French competitive clusters***.

SILVA, DIONISIO F. da ***Preparation of PtRu/C e PtSn/C eletrocatalysts using electron beam irradiation for direct methanol and ethanol fuel cell***.

SILVA, JOSE A.A. da ***Protease activated receptors (PARs) mediation in gyroxin biological activity***.

SUZUKI, KATIA N. ***Studies of techniques for the post-elution concentration of ^{99m}Tc obtained from gel type ^{99}Mo/^{99m}Tc generators***.

TAIPINA, MAGDA S. ***Gamma radiation effect on tocopherol content in natural and industrialized products and sensory evaluation***.

TALLARICO, LENITA de F. ***Evaluation of toxic and mutagenic effects of environmental samples of Tiete River in the region of Suzano in Biomphalaria glabrata (Say, 1818)***.

2010

BERA, ALEXANDRE ***Effects on physicochemical, microbiological and sensory characteristics of honey bee samples submitted to gamma radiation***.

BORBOREMA, SAMANTA E.T. ***Development and pharmacokinetic of antimony encapsulated in liposomes of phosphatidylserine using radioisotopes in experimental leishmaniasis***.

CAMARGO, FABIOLA de A. ***Single frequency Nd:YLF and Nd:YVO4 laser in the red emission***.

CHIBA, RUBENS ***Synthesis, processing and characterization of the solid oxide half-cells cathode/electrolyte of strontium-doped lanthanum manganite/yttria-stabilized zirconium***.

DAMATTO, SANDRA R. ***Determination of natural radionuclides from ^{238}U and ^{232}Th series, trace and major elements in sediment cores from Baixada Santista and evaluation of impacted areas***.

DIAS, CARLA R. de B.R. ***Comparative studies of antibody anti-CD20 labeled with ^{188}Re***.

FELIPPE, MONICA T.S.D. ***Regional nitrous oxide flux in Amazon basin***.

FERREIRA, JOAO C. ***Removal of ammonia generated from farm poultry and their use in the fuel cells and as fertilizer***.

HASHIMOTO, MARIA C.E. ***Photodynamic therapy in induced infection by multi-resistant Pseudomonas aeruginosa. In vivo study***.

LEONARDO, LUCIO ***The use of lichens as bioindicators of atmospheric contamination by natural radionuclides and metals in a region impacted by tenorm***.

LOPES, VALDIR M. ***Incidents in nuclear research reactor examined by deterministic probability and probabilistic safety analysis***.

MADUAR, MARCELO F. ***Development of an open source software of quantitative analysis for radionuclide determination by gamma-ray spectrometry using semiconductor detectors***.

MENEGUZZO, DAIANE T. ***Phototherapy with low intensity laser in carrageenan-induced acute inflammatory process in mice paw - dosimetry studies***.

MONTOYA, CYNARA V. ***Development of a computational system (software) of risk management in the process of radio-sterilization of biological tissues***.

MOREIRA, EDSON G. ***Preparation and characterization of a Perna perna (Linnaeus, 1758) mussel reference material***.

NAHUEL CARDENAS, JOSE P. ***Dosimetric evaluation of semiconductor detectors for application in neutron dosimetry and microdosimetry in nuclear reactor and radio surgical facilities***.

NAKAEMA, WALTER M. ***Time resolved supercontinuum cavity ring-down spectroscopy for multicomponent gas detection***.

OLIVEIRA, NELIO A. de J. ***Development of animal models of growth hormone gene therapy using transduced keratinocytes and direct injection of naked DNA***.

PARTATA, ANETTE K. ***Epidemiology of human visceral Leishmaniasis in Araguaina (TO) and serological diagnosis of this disease***.

PINTO, TERESA C.N.O. ***Dosimetric methodology for extremities of individuals occupationally exposed to beta radiation using the optically stimulated luminescence technique***.

PRATES, RENATO A. ***Evaluation of the photodynamic antimicrobial therapy on pathogenic yeasts***.

RAIA, FABIO ***Experimental assessment of the temperature effects on instrumented concrete pavement***.

SATO, RENATO C. ***Nuclear technology and chronic diseases: an exploratory study evolving the clinical physician perception***.

SOARES, JOSE R. ***Development of system for product tracking and data acquisition of data irradiation process in large gamma irradiators***.

TEODORO, RODRIGO ***Preclinical evaluation of neurotensin (8-13) analog radiolabeled with ^{99m}Tc: In vitro and in vivo characterization***.

TUSI, MARCELO M. ***Preparation of hybrids metal/carbon by hydrothermal carbonization for application in the electro-oxidation of methanol***.

WIENDL, TONI A. ***Effect of low doses of gamma radiation of Co-60 (Radio-hormesis) in tomato seeds***.
YOSHIZUMI, MAIRA T. ***Design, construction and characterization of special ionization chambers for X radiation beams monitoring***.

ZINI, JOSIANE ***Uses of extraction and ion exchange chromatography in the thorium and rare earths separation from industrial residue generated in thorium purification unity at IPEN. Application of rare earths as catalysts for generation of hydrogen***.

Scientific Production

Dissertations

2008

ALENCAR, MARCOS M. de ***Determination of uranium and thorium isotopes in lichens samples Canoparmelia texana***.

AMARAL, MARCELLO M. ***Development of laser induced breakdown spectroscopy of on "Cruzeiro" from 1945 and thousand "Reis" from 1939 for applications on cultural heritage analysis: study of coins***.

ANDRADE, ALEXANDRE B. de ***Development of electrode-membrane-electrode assemblies for proton exchange membrane fuel cells (PEMFC) by sieve printing***.

ANDRADE, DEBORA C. de ***Study of herbicide ametryne degradation in HDPE packaging using the advanced oxidation process by ionizing radiation***.

ANDREASSA, WAGNER L. ***The consumerism as a relevance component in global environmental degradation - current situation and analysis of possible action of mitigation***.

ANTUNES, ALBERI ***A study of physics of sub critical multiplicatives systems driven by sources and the utilization of deterministics codes in calculation of this systems***.

ARAÚJO, MICHEL M. ***Application of the microbiological method DEFT/APC and DNA comet assay to detect ionizing radiation processing of minimally processed vegetables***.

BEDIA, CARLOS E.M. de ***Organizational knowledge management through the identification and analysis of core competences: USEXA, a case study***.

BEIM, KIRA F. ***Comparative study of the interlaminar shear stress in a carbon fiber reinforced polymeric composite using numerical and experimental methods***.

BIANCHI, PAULO H. ***Nuclear reactors transients identification and classification system***.

BOGHI, CLÁUDIO ***Procedures development and methodology of control for application of good manufacture practices (GMP) on human blood irradiation***.

BONIOLO, MILENA R. ***Biosorption of uranium on banana pith***.

BRUNO, MARIZA ***Utilization of zeolites synthesized from fly ash on the removal of dye from aqueous solution***.

CARDOSO, VANESSA M. ***Electron beam application as pre treatment of sugar cane bagasse to enzymatic hydrolysis of cellulose***.

CARNEIRO JUNIOR, VALDECI ***Neutron field characterization in the installation for BNCT study in the IEA-R1 reactor***.

CASTAGNET, MARIANO ***Compressibility evaluation of mechanically activated niobium and aluminum powders mixtures***.

CASTRO, ALEX T.P. de ***The viability of PVC/Al blister reuse and PVC property studies after ionizing radiation processing***.

CASTRO, LILIAN P. de ***Natural radionuclides and ^{137}Cs in commercialized edible mushrooms in São Paulo - Brazil***.

CECCHI, CLAUDIA R. ***Secretion of mouse growth hormone by transduced primary human keratinocytes: prospects for an animal model of cutaneous gene therapy.***

COSTA, RENATA F. ***Development of methods of preparation of polymer-based and resin-based microspheres labeled with holmium-166***.

DOURADO, BENJAMIN T. ***Evaluation of the production process - dimensional study of sintered part***.

FERRARI, MARCO A. ***Nd:YLF Laser for LIDAR applications***.

FERRARI, RICARDO B. ***Characterization of aluminum - magnesium base metallic alloy obtained due conventional after thermomechanical treatments aiming microcrystalline structures***.

FERREIRA, ÉLINER A. ***Microstructure and magnetic properties of Pr-Fe-B-Co-Nb sintered magnets produced from HD and HDDR powder***.

FERREIRA, RAFAEL V. de P. ***Biodegradation of radioactive organic liquid waste from spent fuel reprocessing***.

FRAZAO, SELMA V. ***Trace element determination study in human hair by neutron activation analysis***.

FREITAS, ANTÔNIO A. de ***Recovery of thorium and rare earths by their peroxides precipitation from a residue produced in the thorium purification facility***.

FUKUMORI, NEUZA T.O. ***Determination of bacterial endotoxin (pyrogen) in radiopharmaceuticals by the gel clot method validation***.

FURLAN, NATALIE S. ***Recenticity study of documents using gas chromatography/mass spectrometry (GC-MS)***.

GARDUSI, CLAUDIA M. ***Guidelines for an environmental code of ethics for research institutions***.

GODOI, EVELYN L. de ***The monitoring process of the surface water with dense pollution the Pirajuçara stream, metropolitan area of São Paulo, Brazil***.

GOIA, TAMIYE S. ***In vitro and in vivo evaluation of porous Ti-13Nb-13Zr alloys obtained by powder metallurgy***.

Dissertations

GOMES JUNIOR, ALCIDES ***Determination of selenium in underground water using atomic absorption spectrometry with graphite furnace (GFAAS) and hydride generation (HGAAS)***.

GOMES NETO, JOSÉ ***Relational database hybrid model, of high performance and storaging capacity, for nuclear engineering applications***.

GONÇALVES, LUCAS B. ***Calibration of the nuclear power channels of the IPEN/MB-01 reactor obtained from the measurements of the spatial thermal neutron flux distribution in the reactor core through the irradiation of infinitely diluted gold foils***.

HAMADA, NATALIA ***Toxicity assays applied for the evaluation of the effects of Suzano wastewater treatment system, ETE Suzano, and its surroundings, using aquatic organisms***.

HELLER, SUSANA da R. ***Utilization of CHO cells cultivated in presence of cycloheximide for obtainment and characterization of recombinant human glycosylated prolactin (G-hPRL)***.

HIGA, MARCELA C. ***Toxicity assays applied for evaluation of ionizing radiation and zeolites adsorption as treatment technologies for colored effluent***.

INAMURA, PATRÍCIA Y. ***Effect of gamma irradiation in the viscosity of gelatin and pectin solutions used in food industry***.

IZIDORO, JULIANA de C. ***Studies on removal of metal ions in water using zeolites synthesized from coal ashes***.

JAKUTIS NETO, JONAS ***Crystalline fluoride fiber laser and characterization of rare earth doped fibers***.

KIBRIT, EDUARDO ***Analysis of normative requirements for the development and implementation of a quality management system in Brazilian nuclear installations and activities***.

KOMATSU, CINTIA N. ***Guidelines for evaluation of the environmental expense in the nuclear fuel cycle***.

LAMEIRA, VANESSA ***Study of sublethal and lethal effects (reproduction and teratogenisis) of the pharmaceutical compound triclosan to Daphnia similis, Ceriodaphnia dubia, and Ceriodaphnia silvestrii (cladocera, crustacea)***.

Lucia, silvio R. de ***Development of a gamma ray spectrometry software for neutron activation analysis using the open source concep***.

LUGON, MARCELO di M.V. ***Polarographic determination of Sn(II): application in the quality control of radiopharmaceutical reagents***.

MAGEIKA, CRISTIANE M. de C. ***Preparation and characterization of alpha and beta subunits of recombinant human glycoprotein hormones: folliclestimulating hormone, luteotropin, thyrotrophin and their comparison with pituitary glycoprotein hormones***.

MARCHESINI, PAULO R. de A. ***Diagnosis and suggestions for the knowledge management applied to a nuclear installation: the uranium hexafluoride production unit***.

MARTINHO JUNIOR, ANTONIO C. ***Study of physical, chemical and structural effects caused by ionizing radiation and preservation on human costal cartilage***.

MARTINS, MARIA da P. S. ***Study of human factors, and its basic aspects, focusing the IEA-R1 research reactor operators, aiming at the prevention of accidents caused by human failures***.

MATOS, BRUNO R. de ***Fabrication and characterization of Nafion-TiOsub(2) composite electrolytes for proton exchange membrane fuel cells***.

MELERO, LAURA T.U.H. ***Preparation and standardization of an in vitro labeling methodology and study of [^{99m}Tc]-EDDA/TRICINE/HYNIC-[Tyr^3]-octreotide biodistribution***.

MELLO, DJALMA de ***Characterization of solid deposit formed in automotive gasoline engine by the analytical techniques***.

MELO, IVANI B. ***Preparation of lyophilized kit of hynic-[Tyr^3]-octreotate and labeling studies with 99m-technetium***.

MONTE, MARCIA V.R. da C. ***Engobes evaluation in the water stains ocurrence in ceramics coatings***.

MORACCI, LUIZ F.S. ***Pesticides residues in water treatment plant sludge: validation of analytical methodology using liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS)***.

MORAES, GILBERTO de O. ***Polyethylene (PE) based proton exchange membrane for use in fuel cell***.

MORAES, LUIZ A.G. de ***Contribution to the values study, perspectives and effectiveness of the clean development mechanism of the Kyoto protocol***.

MOSCA, RODRIGO C. ***Growth inhibition of oral microflora by snake venoms***.

MOURA, LUIZ A.A. de ***Proposal of implementation of environmental management system the laboratory of radioactive waste in IPEN-SP***.

MOURA, PATRICIA L. da C. ***Essential and toxic element determination in edible mushrooms by neutron activation analysis***.

MUELLER, RENATA M.L. ***Can metals be used in to diagnose of type 2 diabetes?***

NAKAMURA, GERSON H. de G. ***Thermal analysis and phase equilibria in the BaF_2-YF_3 system***.

NETO DE JESUS, ANTONIO C. ***Study of the parameters: NaCl concentration and surface finish on the pitting corrosion resistance of copper tubes***.

Scientific Production

Dissertations

NISHITSUJI, DELMO A. ***Development of epoxies matrices and study of process parameters on electron beam curing of polymeric composites***.

NUNES, MAÍRA G. ***Assessment of $CaSO_4$:Dy and LiF:Mg, Ti thermoluminescent dosimeters performance in the dosimetry of clinical electron beam***.

OLIANI, WASHINGTON L. ***Study of HMS-PP (high melt strength polypropylene) behaviour under conditions of environmental and accelerated degradation***.

OLIVEIRA, DIRCEU P. de ***Correlation study among the International Atomic Energy Agency standards and markets standards on management system applicable to a UF_6 conversion plant operation***.

OLIVEIRA, FERNANDO L. de ***Analytical solution of spatial kinetics of the diffusion model for subcritical homogeneous systems driven by external source***.

OLIVEIRA, MARIA J.A. de ***Synthesis, characterization and citoxicity of polymeric hydrogels for use to immobilization and drug release on Leishmaniose treatment***.

OLIVEIRA, TAIS L. de ***Development of the fermentation process in bioreactor for the production of human prolactin secreted in the periplasmic space of Escherichia coli***.

ORLANDO, JOÃO P. ***A study about the ozone precursors in the São Paulo city using computational simulation***.

PASTORELLO, NILCE A.H. ***Evaluation of the occupational risks of organic volatile compounds-VOCs in airport environments: implementation of methodology***.

PEREIRA, MARIA C. ***Best practices study of institutional performance indicators systems in research, development and innovation (R&D&I) public organizations, with focus on military organizations***.

PERINI, RINALDO ***The use of X-ray diffraction to determine the residual stress induced by the cold rolling process in a cylinder liner of a diesel engine***.

PIRES, CARLOS A. ***Standardization of ^{201}Ti and ^{55}Fe radionuclides in a 4π (PC)-NaI(Ti) coincidence system***.

POLAKIEWICZ, LILIAN ***Polycyclic aromatic hydrocarbons study at São Vicente and Santos Estuaries SP using diatomite as adsorbent material***.

POVEDA, PATRÍCIA N.S. ***Study of the electron beam irradiation effect on some properties of aromatic aliphatic copolyester films***.

PUTVINSKIS, RODRIGO ***Study of the superconducting compound Hg, Re-1223 by x-ray diffraction with application of Rietveld method***.

QUEIROZ, PAULO C.B. de ***Cooperative research: The carbon fiber development for uranium centrifuges project***.

RIBEIRO JUNIOR, MARCOS A. ***Comparative study of Bothrops jararaca venom from mainland and specimens of the island of São Sebastião***.

RIBEIRO, THATIANA G.D. ***Synthesis and characterization of magnetic nanoparticles of mixing oxides of $MnFe_2O_4$ recovered with chitosan. Studies of the dopade influence with Gd3+ in properties structural and magnetic***.

RIBEIRO, VILMARIA A. ***Preparation of PtRuNi/C eletrocatalysts prepared by an alcohol reduction process for methanol electro-oxidation in direct methanol fuel cell***.

RODRIGUES, DANIELLE B. ***Antiangiogenic therapy using endostatin producer cells encapsulated in immunoisolation devices***.

ROSA, FAENA M.L. ***Simulation study of ε-caprolactam monomer and metallic elements migration from irradiated polymeric packaging into food simulants***.

ROSA, GUSTAVO A.B. ***Study of the effects of the pharmaceutical compound propranolol to ceriodaphnia silvestrii (Cladocera, crustacea) with emphasis on the effects on populations***.

SALUM, DEBORA C. ***Determination of volatiles produced during radiation processing in food and medicinal herbs***.

SANTANA, LEONARDO de P. ***Study of tape casting of yttria stabilized zirconia for apply in solid oxide fuel cell***.

SANTOS, ANTONIO C. dos ***Study of poli (ethylene terephthalate) pet postconsumption and its properties when it is undergone ionizing radiation***.

SANTOS, IVANILDO A. dos ***Study of $LiGd_{1-x}Lu_xF_4$ solid solutions aiming the crystal growth***.

SILVA NETO, JOAO B. da ***Dry uranium tetrafluoride process preparation using the uranium hexafluoride reconversion process effluents***.

SILVA, DOUGLAS R. da ***Analysis of gas shielding influence in the properties of Ti6Al4V laser welding***.

SILVA, ELIEZER A. da ***Determination of the disintegration rate and gamma emission probabilities per decay of ^{182}Ta***.

SILVA, FERNANDO R. da ***Single fiber crystal growth of Nd^{3+} and Er^{3+}- doped $LiYF^4$ for solid state laser applications***.

SILVA, GIOVANA P. da ***Study of the viability of the production of lutetium 177 in the Nuclear Reactor IEA-R1 at IPEN/CNEN-SP***.

SILVA, GRAZIELA C.T. da ***Effect of cobalt oxide on sintering and electrical conductivity of yttria stabilized zirconia***.

SILVA, ROBERTO W.R.V. da ***Preparation and characterization of PtRu/C and PtSn/C electrocatalysts using the citric acidic chemical reduction process for direct alcohol fuel cell (DAFC)***.

SOUSA, EDUARDO C. ***TEM00 mode efficiency enhancement in high power diode-sidepumped Nd:YLF lasers***.

SOUSA, KEILA C.P.M. de ***Fluvial fluxes of natural radium isotopes and dissolved barium for Ubatuba embayments, São Paulo***.

SOUZA, WANDER B. de ***Study of red mud addition in the poli (methyl metacrylate) by mass polymerization and casting process molding***.

TETZNER, GUARACIABA de C. ***He industrial computerized tomography applied on the rock analysis***.

TOFFOLI, DANIEL J. ***Spectroscopic characterization of hypocrellin B: lanthanide complexes for use in photodynamic therapy***.

VALGODE, FLAVIA G.S. ***Evaluation of the radioinduced damage, repair capacity and cell death on human tumorigenic (T-47D and MCF-7)and nontumorigenic (MCF-10) cell lines of breast***.

VALLEJO, NATÁLIA M. ***Obtaining high serum levels of murine endostatin in mice using recombinant chinese hamster ovary cells secreting endostatin transplanted in imunoisolation devices***.

VANNI, SILVIA R. ***Economic viability of alternative energy sources for a typical community of the northeast region of Brazil***.

VIEIRA, HEVELINE. ***Study of the surface crystallization and resistance to dissolution of niobium phosphate glasses for nuclear waste***.

VILLAVERDE, FREDDY L. ***External exposure assessment in dwelling built with phosphogypsum***.

ZEN, HELOÍSA A. ***Polypropylene (PP) based proton exchange membrane for use in fuel cell***.

ZERLIM, ANELISA ***Evaluation of the dissolution rate of niobium phosphate glasses in water and simulated body fluid solution***.

2009

ABEL, JOAO L. ***Synthesis of silicon carbide by carbothermal reduction of silica***.

AGUIAR, RODRIGO O. de ***Determination of elements in blood of golden hamster by NAA***.

ALBERO, FELIPE G. ***Thyroid lesions diagnosis by Fourier transformed infrared absorption spectroscopy (FTIR)***.

ALCANTARA, MARCELA C. ***Evaluation of the image quality criteria and study of doses in a mammography department***.

ALEGRE, GABRIEL F. ***Ecotoxicological assessment of sediments from Tiete River between Salesopolis and Suzano, SP***.

ALMEIDA, BEATRIZ E. de ***Analysis of recombinant and native human lutropin and human chorionic gonadotropin by reversed-phase high performance liquid chromatography***.

ALMEIDA, ERIKA V. de ***Development and validation of methodology for technetium-99m radiopharmaceuticals using high performance liquid chromatography (HPLC)***.

ALMEIDA, TIAGO L. de ***Development of a PVAI/chitosan composite membrane compatible with the dermo-epidermic system***.

AMARAL, RENATA H. ***Immobilization and release study of a red alga extract in hydrogel membranes***.

ANTONIO, PATRICIA de L. ***Comparative study among calibration methods of clinical applicators of beta radiation***.

BALDUINO, KELI N. ***Refolding in high hydrostatic pressure of recombinant proteins from inclusion bodies in Escherichia Coli***.

BARBOZA, ALEX ***Radioactive waste management of the nuclear medicine services***.

BARROS, GUSTAVO A.S.J. ***Project of an integrated calibration laboratory of instruments at IPEN***.

BEOLCHI, RAFAEL da S. ***Bone regeneration effect after the addition of a vitamin complex in two bioceramics***.

BOMFIM JUNIOR, FRANCISCO A. ***Production and characterization of rare-earth and metallic nanoparticle doped heavy metal oxide glasses***.

BRAMBILLA, TANIA de P. ***Development of methods of labeling pentavalent DMSA with ^{99m}Tc and ^{188}Re***.

BRITO, CARLOS F. de ***Development and validation of analytical methodology for determination of polycyclic aromatic hydrocarbons (PAHS) in sediments. Assessment of Pedroso Park dam, Santo Andre, SP***.

CALICCHIO, LEONARDO ***The effect of shot peening on the gas nitriding of iron components produced by powder metallurgy***.

CALVO, FERNANDA B. ***Construction and characterization in vitro of a bicistronic retroviral vector coding endostatin and interleukin-2 for use in gene therapy***.

CAPRONI, PRISCILA ***Effects of irradiated Bothropstoxin-1 and Bothrops jararacussu crude venom on the immune system***.

Scientific Production

Dissertations

CARDOSO, ELISABETH C.L. ***Development of foams from linear polypropylene (PP) and high melt strength polypropylene (HMSPP) polymeric blends***.

CARUSO, MARCEL W. ***Analysis of the irradiation and thermal treatment combined effect in the quality of mangoes for exportation***.

CAVINATO, CHRISTIANNE C. ***Standardization of the fricke gel dosimetry method and tridimensional dose evaluation using the magnetic resonance imaging technique***.

CONCEICAO JUNIOR, OSMAR ***Application of the failure modes and effects analysis technique to the emergency cooling system of an experimental nuclear power plant***.

CORDEIRO, THIAGO da S. ***Study of Laser pulses propagation through an ultrashort pulse amplifying systems for the development of an Öffner temporal stretcher***.

COSTA, OSVALDO L. da ***Load and unload system optimization on H2 ^{18}O irradiation target used for ^{18}F - production at the cyclotron cyclone 30 from IPEN-CNEN/SP***.

COUTO, RENATA M. ***Development of radiopharmaceutical for radiosinovectomy***.

CURCHO, MICHEL R. da S.M. ***Assessment of micro, macro, toxic elements (Cd, Hg, Pb) and fatty acids profile in consumed fish commercially available in Cananéia and Cubatão, São Paulo State***.

DAMIANI, RENATA ***Stable expression of human thyrotropin (hTSH) in mammalian cells (CHO) expressing 2,6 sialyltransferase***.

DIAS, RICARDO R. ***Preparation of PtSn/C, PtRu/C, PtRh/C, PtRuRh/C and PtSnRh/C electrocatalysts using an alcohol-reduction process for methanol and ethanol oxidation***.

DRESCH, MAURO A. ***Synthesis and electrochemical characterization of Nafion-SiO_2 hybrid membranes for application as polymeric electrolyte in PEM fuel cells***.

DUARTE, DANIEL G. ***Synthesis and ceramic processing of alumina and zirconia based composites infiltrated with glass phase for dental applications***.

DUARTE, RENATO C. ***Comet assay as a cold chain control tool***.

DUVAIZEM, JOSE H. ***Study of the microstructural and mechanical properties of titanium-niobium-zirconium based alloys processed with hydrogen and powder metallurgy for use in dental implants***.

FABBRI, ADRIANA D.T. ***Study of radiation in fresh tomatoes (Lycopersicon esculentum mill) and in the levels of sauce lycopene***.

FANARO, GUSTAVO B. ***Ionizing radiation effects on volatiles formation in Camellia sinensis (L) teas***.

FARINA, LUIS C. ***Viscoelastic characterization of carbon fiber-epoxy composites by creep and creep rupture tests***.

FEITOSA, MARCOS A.F. ***Compatibilization of polyamide 6.6 and low density polyethylene polymeric blend using electron beam ionizing radiation***.

FERREIRA, DANILO C. ***Gamma radiation processing dosimetry with commercial silicon diodes***.

FONTES, TALITA F. ***Alpha prime effect on mechanical properties and corrosion resistance of UR 52N+ Duplex Stainless Steel***.

FRANCISCATTO, PRISCILA C. ***X-radiation qualities characterization following the standard IEC 61267 recommendations at the calibration laboratory of IPEN***.

FURGERI, CAMILO ***Effect of ^{60}Co radiation processing in mate (Ilex paraguariensis)***.

GARCIA, MARCIA A.A. ***Radiochemical characterization and environmental radiological impact in tin and lead processing from casiterite***.

GIMENES, CELSO H. ***Identifying and building consensus about the essential competences for R&D managers - case study at IPEN***.

GUILHEN, SABINE N. ***Validation of an analytical method for the determination of total mercury in urine samples using cold vapor atomic absorption spectrometry (CV-AAS)***.

HIRATA, DANIEL M. ***Estimative of core damage frequency in IPEN's IEA-R1 research reactor (PSA level 1) due to the initiating event of loss of coolant caused by large rupture in the pipe of the primary circuit***.

JESUS, WELLINGTON C. de ***Study of inverse methods in remote sensing with laser***.

LAINETTI, ELIZABETH B. de F. ***Critical analysis for physical adaptation and implementation of new procedures in the IPEN's laboratory animal division***.

LEE, SEUNG M. ***A study of calculation methodology and experimental measurements of the kinetic parameters for source driven systems***.

LINHARES, HORACIO M. da S.M.D. ***Synthesis and characterization of KY_3F_{10} and Ky_3F_{10}:Yb:Nd:Tm crystals for optical applications***.

LOPES, PAULA R.C. ***Study of different adsorbent materials for the preparation of generator systems of ^{99}Mo - ^{99m}Tc and ^{188}W-^{188}Re***.

MACHADO, GLAUSON A.F. ***Development of ceramic composites from mixture of alumina and ceramic precursor polymer poly (silsesquioxane)***.

MAIO, MIREIA F. ***Evaluation of effects of ionizing radiation on materials user in dental restorations***.

Scientific Production

Dissertations

MANETTI, CRISTIANE T. ***The press midia and the risk perception***.

MANGUEIRA, THYAGO F. ***Dosimetric evaluation of the fricke gel dosimeter using the spectrophotometric technique for application in electron and neutron dosimetry***.

MARQUES, ROGERIO A. ***Alternative materials study for dental magnetics attachments applications***.

MARTINS, EMERSON A. ***Assessment of the effect of surface treatments on the corrosion resistance of Nd-Fe-B Magnets***.

MARTINS, RENATA de S.L. ***Evaluation of toxicity of rainwater on aquatic organisms***.

MERCURIO, MARCIO E. ***Investigation of hyperfine interactions in ZnO and $Zn_{1-x}Co_xO$ by means of perturbed angular gamma-gamma correlation technique***.

MESQUITA, RODRIGO M. ***Development of porous silicon nitride-based ceramics***.

MIRANDA, JUREMA A. de ***Development of a calibration methodology for instruments used to interventional radiology quality control***.

MONTEIRO, RAQUEL A. ***Evaluation of potential of adsorption of U, Th, Pb, Zn and Ni by the coir pith***.

MORAES, JAIR R. de ***Alkali rare earth double tungstates single crystal fibers: A growth study by the micro-pulling down method***.

MORENO, CAROLINA dos S. ***Study of radioprotective effect of the resveratrol***.

MOURA, JOAO A. ***Study and methodology development for quality control in the production process of iodine-125 radioactive sealed sources applied to brachytherapy***.

NAKANO, MILTON S. ***Technological forecasting at medium and long term about hydrogen generation process, considering probabilistics future scenarios and taking into account fuel cells development***.

NISTI, MARCELO B. ***Environmental system applied to radioactive liquid effluent release***.

NOGUEIRA NETO, ANTONIO C. ***Acute and chronic toxicity evaluation at Jundiaí river, influent and effluent from Novo Horizonte Wastewater Treatment Plant (NHWWP), Jundiaí, São Paulo***.

NUNES, ILIENE R. ***Evaluation of life cycle as a tool for environmental education: the use of waste reduction and increase productivity as indicators***.

NUNES, KELLY P. ***Archeometric studies on the Hatahara archaeological site***.

NUNES, THAISE C.F. ***Evaluation of the effects of gamma radiation on the vegetables of Brassica oleracea species minimally processed***.

OLIVEIRA, OLAVO R. de ***Development of ceramic protonic conductors for solid oxide fuel cells operation under methane and hydrogen fuels***.

OLIVEIRA, THAIS de ***Recovery and recycling of sulfuric and nitric acids and molybdenum from liquid waste of lamp industries***.

PEREIRA, LARISSA M. ***Cloning, periplasmic expression, purification and structural characterization of human ribosomal protein L10 recombinant***.

PEREIRA, THIAGO M. ***Thermal diffusivity measurement of dental hard tissue as function of temperature obtained by infrared thermography***.

PUJATTI, PRISCILLA B. ***Development of lutetium-labeled bombesin derivatives: Relationship between structure and diagnostic-therapeutic activity for prostate tumor***.

RABELO NETO, JOSE da S. ***Nanostructured synthetic hydroxyapatite and dental enamel heated and irradiated by Er,Cr:YSGG. Characterized by FTIR and XRD***.

RAELE, MARCUS P. ***Development of an polarization sensitive Fourier domain optical coherence tomography and it utilization on the Mueller matrix determination***.

RAMOS, AMANDA C.O. ***Application of gamma radiation on disinfestation feed grain based food for domestic animals***.

ROGOVSCHI, VLADIMIR D. ***Radiation degradation of biological waste (aflatoxins) produced in food laboratory***.

RUBY, ELAINE C. ***Environmental quality of agricultural soils within the Jaguari River basin - São Paulo***.

SANCHEZ, ARETHA ***Human activities and climate and environment changes: an inevitable relation***.

SANTOS, ELEN G. dos ***Development of nanobiomarkers for use in sickle cell anemia***.

SANTOS, JANILSON S. ***Remediation of soil contaminated with pesticides by treatment with gamma radiation***.

SANTOS, PAOLA de S. ***Evaluation of essential trace elements in preterm and full term milk and infantile formulas by neutron activation analysis***.

SILVA, CARLOS E. da ***Validation of methods for WDXRF and OES-SPARK techniques in steel analysis. Determination of the uncertainty of measurements for API steel plant sample***.

SILVA, CLEBER F. ***Dosimetric evaluation of spectrophotometric response of alanine gel solution for gamma, photons, electrons and thermal neutrons radiations***.

Scientific Production

Dissertations

SILVA, PRISCILA V. da ***Evaluation of peach palm (Bactris gasipaes Kunth) processed by radiation***.

SILVA, RENILSON A. da ***Hyperfine interaction studies of the perovskite oxides of the type $RCrO_3$ (R = Gd, Tb e Dy)***.

SUZUKI, LUIS C. ***Candida albicans biofilm development in vitro for photodynamic therapy study***.

SZURKALO, MARGARIDA ***Investigation on the of effect of self assembling molecules on the corrosion resistance of the 1050 aluminum alloy***.

TOLEDO, FABIO de ***Design of an electronic system with simultaneous registering of pulse amplitude and event time applied to the 4'pi''beta'-'gama' coincidence method***.

TONDIN, JOSE E.M. ***Prospection of implementation of distance learning for the course on fundamentals of nuclear physics at the graduate course of IPEN using free software infra-structure***.

TONELLO, KAROLINA P. dos S. ***Composites based on Al_20_3, with additions of NbC and MgO***.

TONGU, MARGARETH L.O. ***Application of Monte Carlo simulation to the standardization of positron emitting radionuclides***.

TORETI, DALILA L. ***Acceptance, commissioning and quality control in radiosurgery***.

TOYOTA, ROSIMEIRI G. ***Chemical characterization of Marajoara ceramics***.

UEHARA, SANDRO T. ***Data analysis of a backscattering LIDAR system correlated with meteorological data***.

YAMAMURA, AMANDA P.G. ***Environmental nanotechnology application: Magnetic biosorbent for uranium removal***.

2010

ALFARO, EDUARDO de F. ***Study of the rice husk ash utilization as filler in polypropylene matrix and ionization radiation effect on this composite***.

ANTUNES, PAULA C.G. ***Reconstruction of voxel phantoms for skin dosimetry***.

AQUINO, REGINALDO R. de ***Assessment of natural radiation of beachs sands in Great Vitoria, Espirito Santo***.

ARAKAKI, ALEXANDER R. ***Obtaining of ceria - samaria - gadolinia ceramics for application as solid oxide fuel cell (SOFC) electrolyte***.

BAPTISTA, TATYANA S. ***Reference values in blood elements in crioula breed horses by nuclear methodology***.

BARRIO, GRACIELA ***Development of technology for the preparation of $^{90}Sr/^{90}Y$ generators at the radiopharmacy directory of IPEN/CNEN-SP***.

BATISTA, RAFAEL M. ***Effects of NiO addition on the densification, microstructure and electrical conductivity of yttria fully-stabilized zirconia***.

BENETTI, CAROLINA ***Study in vitro of Er, Cr:YSGG laser effects in bone tissue by ATR-FTIR spectroscopy***.

BONIFACIO, RAFAEL N. ***Development of a membrane electrode assembly production process for proton exchange membrane fuel cell (PEMFC) by sieve printing***.

BORBA, TANIA R. de ***A study on application of biosorbents for treatment of radioactive liquid waste containing americium-241***.

BORGES, WESDEN de A. ***Computational simulation studies of the reduction process of UFsub(4) to metallic uranium***.

BOURLEGAT, FERNANDA M. LE ***Availability of metals in samples of phosphogypsum and phosphate fertilizers used in agriculture***.

BRANDALISE, MICHELE ***Preparation and characterization of PtRu/C, PtBi/C, PtRuBi/C electrocatalysts for direct elector-oxidation of ethanol in PEM fuel cells using the method of reduction by sodium borohydride***.

BUENO, LILIAN de O. ***Climate change as seen by science and scientific dissemination***.

CAMPOS, FABIO E. de ***Development of a pattern hot cell for production of injectable radiopharmaceuticals***.

CARVALHO, CLAUDIA A.Z. de ***Integrated management system best practices in radioecological laboratories***.

CARVALHO, TEREZINHA E.M. de ***Adsorption of anionic dyes from aqueous solutions onto coal fly ash and zeolite synthesized from coal fly ash***.

CINTRA, FELIPE B. de ***Evaluation of the methodology for dose calculation in microdosimetry with electrons sources using the MCNP5 code***.

COELHO, TALITA S. ***Dosimetry system development for $^{90}Sr+^{90}Y$ betatherapy applicators***.
CORREA, EDUARDO de L. ***Quality control methodology and implementation of X-radiation standards beams, mammography level, following the standard IEC 61267***.

CORREA, VALERIA R. ***Analysis and epidemiology of chagas disease in Araguaina - Tocantins***.

COSTA JUNIOR, NELSON V. da ***Analytical characterization and optimization in the determination of trihalomethanes on drinking water by purge and trap coupled to a gas chromatography***.

Scientific Production

Dissertations

COSTA, ANA E.B. da ***Effects of gamma radiation immunogenicity of ribonucleoprotein (RNPS) of rabies virus and purification of anti-RNPs antibodies for diagnosis***.

COSTA, RENATA F. da ***Aerosol optical property studies in Sao Paulo State with Raman LIDAR technique***.

CRUZ, JULIANA N. da ***Study of quarantine treatment in mango (mangifera indica L.) for export***.

CUNHA FILHO, SILVIO C. da ***Epidemiology and study of the factors responsible for spongiosis ocular in the city of Araguatins - TO***.

DIAS, DANIEL M. ***Establishment of a new calibration method of pencil ionization chamber for dosimetry in computed tomography***.

DIAS, VIVIANE L. ***Aspects of resistance to experimental infection with Trypanosoma cruzi***.

DMITRIJEVAS, CIBELE ***Eco-efficiency analysis of techniques for disposal of urban solid waste***.

DOMINGOS, DOUGLAS B. ***Neutronic, thermal-hydraulic and safety analysis calculations for a miniplate irradiation device (MID) of dispersion fuel elements***.

EGUTE, NAYARA dos S. ***Study of the acceleration of ammonia generation process from poultry residues aiming at hydrogen production***.

ESTEVES, JOSELFINA M. da S. ***Comparative evaluation of activity-based costing and variable costing: a case study at IPEN***.

FERREIRA, ROBSON de J. ***Methodology development for sealed radioactive sources characterization***.

FONSECA, GABRIEL P. ***Project and construction of energy degrading and scattering plates for electron beam radiotherapy for skin diseases***.

FONSECA, LIZANDRA P. de S. ***Contribution to optimization of individual doses of workers in shipment of generator technetium-99m***.

FORSTER, PEDRO L. ***Photoluminescent study of Polycarbonate (PC) and Poly (9-vinylcarbazole) (PVK) doped films with europium complex***.

FRANKLIN, ROBSON L. ***Determination of total organic mercury and evaluation of methylation and demethylation processes in sediments of the Rio Grande reservoir, State of São Paulo***.

GAUGLITZ, ERICA ***Study and survey of assembling parameters to a radioactive source production laboratory used to verify equipments***.

GIBELLI, EDISON B. ***Preparation and characterization of electroluminescent devices based on complexes of 'beta'-diketonates of Tb^{3+}, Eu^{3+}, Gd^{3+} ions with macrocyclic ligands and UO^{2+}_2 films***.

GONCALVES, ANDRE ***Obtention and characterization of TiO_2 / TiN multilayers coatings***.

GRASSO, ANDREA N. ***Spectroscopic studies of europium-tetracyclines complexes and their applications in detection of hydrogen peroxide and urea peroxide***.

HABITZREUTER, ANGELA B. ***Implementation of total body irradiation in radiotherapy***.

IRINEU, ROSA M. da S. ***Comparison of the properties polyamide 6.6 surfaces treated by plasma and by ionizing radiation***.

ISIDORO, ROBERTA A. ***Performance of Nafion-TiO_2 hybrid membrane and PtSn/C electrocatalysts in PEMFC fed with ethanol and H_2/CO at high temperature***.

JESUS, SUELI C. de ***Assessment of natural radioactivity levels in waters from Higher Ribeira Valley until the Southern São Paulo state Coastal plain***.

LEITE, DOUGLAS W. ***Cermet Ni-ZrO_2 by mechanical alloying***.

LIMA, IARA B. de ***Measurements of gaseous multiplication coefficient in pure isobutene***.

LUCENA, RODRIGO F. de ***Establishment of an X radiation equipment quality control programme using non invasive meters***.

MACHADO, ANA C.H.R. ***Açaí oil development and evaluation of immobilization and release in poly (N-vinyl-pyrrolidone) hydrogels***.

MAGDALENA, CARINA P. ***Adsorption of reactive remazol red RB dye of aqueous solution using zeolite of the coal ash and evaluation of acute toxicity with Daphnia similis***.

MARTINS, ELAINE W. ***Study and determination of the influence factors of the radiopharmaceutical vials dimensions used for activimeter calibration at IPEN***.

MARTINS, ILSON C. ***Microstructural studies on chemical interactions in U-Mo alloy with Al***.

MARTINS, LUIZ ***Analysis of technical feasibility, economic and environmental operation of the lift system Tietê-Billings in the supply of water and electricity in the metropolitan region of São Paulo***.

MASSICANO, FELIPE ***Quantification of tomography images for dose calculation for diagnosis and therapy in nuclear medicine***.

MATOS, JANARA de C. ***Hydrobiological aspects of Paraibuna and Paraitinga dams, São Paulo, with emphasis on phytoplankton community***.

MATSUSHIMA, LUCIANA C. ***Response evaluation of thermoluminescent detectors in clinical beams dosimetry using different phantoms***.

Scientific Production

Dissertations

MOMESSO, ROBERTA G.R.A.P. ***Resveratrol immobilization and release in polymeric hydrogels***.

MONTEZANI, EDMILA ***A study on elemental composition in epiphytic lichen samples used as bioindicator of air pollution in São Paulo city***.

MUNIZ, RAFAEL O.R. ***Development of an anthropomorfic simulator for simulation and measurements of neutron dose and flux in the facility for BNCT studies***.

MURA, LUIS F.L. ***Measurements of nuclear reaction rates and spectral indices along of the radius of fuel pellets at IPEN/MB-01 reactor***.

NAIME, NATALIA ***Active packaging from renewable source***.

NASCIMENTO, CLAUDIO S. do ***Human error probability quantification using fuzzy methodology in nuclear plants***.

NEGRINI, AGUINALDO D. ***Automation of cells of radiopharmaceuticals production***.

NOGUEIRA, GUSTAVO B. ***Development of blue lasers, from second harmonic generation using a Nd:YAG laser emitting at 946 nm***.

NONATO, FERNANDA B.C. ***Study of radiation detectors response in standard X, gamma and beta radiation beams***.

OLIVEIRA, FLAVIO S. de ***Profit environmental and energy use of biomass with residual of dairy cattle in the Lagoa da Prata - MG***.

OLIVEIRA, KARINA C. de ***Structural aspects of crotalic venom proteins modified by ionizing radiation***.

OTOMO, JULIANA I. ***Development and validation of analytical methodology for endocrine disruptors hormones determination in water for public supply at region of Paraiba do Sul River, SP***.

PASCOALINO, KELLY C. da S. ***Comparative study of Si diodes response for gamma radiation dosimetry***.

PAULA, RUTH C.M.F. de ***Epidemiological profile cases of snakebites treated at the hospital for tropical diseases in Araguaina - TO (years 2007-2009)***.

PUSCEDDU, FABIO H. ***Ecotoxicological assessment of the pharmaceutical compound Triclosan to freshwater invertebrates with emphasis to spiked sediment tests***.

RAMOS, GUILHERME da F. ***Determination of uranium and thorium isotopes and polonium in sediment profiles from Baixada Santista, SP***.

REIS, SHIRLEY L. dos ***Grain growth and electrical conductivity of samaria-doped ceria sintered by the two-step method***.

RIBEIRO, ROSANGELA do S.P. ***Incidence and epidemiology visceral Leishmaniasis in norte Tocantins - Brazil.***

ROGERIO, ADEMILSON ***Characterization of niobophosphate glasses for solid oxide fuel cell (SOFC) sealing***.

RUBBIA, WALDIR M. LA ***Rehabilitation program of the central area of São Paulo (Procentro) and its influence on the formation of heat island***.

SALGADO, ANDREIA R. ***Ovarian transfer as an alternative to restore the reproductive functions in mice females irradiated with gamma radiation from ^{60}Co***.

SANTOS, BRIANNA B. dos ***Investigation of local magnetism in RZn (R = Ce, Gd, Tb, Dy) and GdCu intermetalic compounds using perturbed angular correlation gamma-gamma spectroscopy***.

SANTOS, ROBERTO C. dos ***Time response of temperature sensors using neural networks***.

SANTOS, SILAS C. dos ***Colloidal processing of ceramic components for gas burners***.

SEVERIANO, LUCIO C. ***Influence of gamma radiation on some physical-mechanical properties of wood used in Brazilian cultural heritage***.

SOMESSARI, SAMIR L. ***Development of an automation system for iodine-125 brachytherapy seed production by (Nd:YAG) laser welding***.

SOUZA, ELISEU W. de ***Study for manufacturing automobile reflectors using a thermoset composite material and a thermoplastic material***.

TADA, ARIANE ***Dosimetric analysis of radiation sources for use dermatological lesions***.

TEIXEIRA, LUCIANE dos S. ***Studies of optical properties of complexes europium tetracycline and its applications in detection of lipoproteins***.

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