

Progress Report 2005 - 2007

A background image showing the Brazilian flag and the flag of the State of São Paulo flying on tall poles against a clear sky. The image is partially obscured by a large yellow number '5' and a yellow circle.

The **Biopharmaceutical** Program is dedicated to the research of isolation, structural analysis and biological activities in different biological systems of macromolecules. Hydrogen is seen by many as a key energetic vector for the 21st century. The **Renewable Energies** Program was established in order to contribute to the national development in this area. The **Laser Technology** Program is strongly committed to the study of laser applications on nuclear area, medicine and dentistry, industry, environment and advanced research aiming not only research but diffusion and innovation. The focus of the **Materials and Nanotechnology** Program is technology development related to processing, analysis, testing and characterization of materials in general. The Program on **Environmental Science and Technology** was structured as a consequence of the continuous growth of environmental activities on areas related to nuclear programs. The mission of **Application of Ionizing Radiation** Program is 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 use of radioisotopes in medicine is certainly one of the most important social applications of Nuclear Energy and the **Radiopharmacy** Program has a special place in the history of Nuclear Medicine in Brazil. The **Nuclear Reactors and Fuel Cycle** Program is fully prepared to attend the challenges that will come with the retaking of the Brazilian nuclear program. Some of the products and services offered by **Nuclear Science and Technology** Program find their way to petroleum, aeronautical, space and semiconductor industries, medical clinics and hospitals, environmental agencies, universities and research institutions. In association with the University of São Paulo (USP), the **Education** Program, established in 1976, is organized in three areas of concentration: Nuclear Technology on Applications, Materials and Reactors.

Progress Report 2005 - 2007

A large, bold, black number "50" is centered on the page. The background of the entire page is a grayscale photograph of the IPEN nuclear reactor building, which is a tall, cylindrical structure covered in scaffolding. In the foreground, two flags are visible: the flag of Malaysia on the left and the flag of Brazil on the right. The image is framed by a stylized, light gray leafy branch that curves from the top left towards the bottom right.

Instituto de Pesquisas Energéticas e Nucleares
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Lake at IPEN location

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Foreword

The period considered in this report, from 2005 to 2007, comprehends three remarkable events to the Nuclear and Energy Research Institute - IPEN.

The first one was the celebration of the 50th anniversary, on August 2006. During this half century, many activities developed here were devoted to the improvement of the quality of life of the Brazilian society, like the production of radiopharmaceuticals for diagnostic and therapeutic procedures in nuclear medicine: the production of the FGD-18 radiopharmaceutical, used for Positron Emission Tomography PET and PET-CT diagnostics are the main ones. Due to this production, Brazil reached over 3 million nuclear medical procedures.

The nuclear reactor IEA-R1 also completed fifty years of operation, on September 2007. It was constructed during the program “Atoms for Peace” and it is considered a model of its class, being engaged in basic research and radioisotopes production since the very beginning.

The Nuclear Technology Graduate Program of IPEN, in association with the University of São Paulo USP, completed 30 years on March, 2006. It is considered a program of Excellence - an international level course - and awarded 937 master degrees and 458 doctor degrees till December 2007.

The progress and main outcomes of the research developed at the 10 R&D Centers and the Radiopharmacy directory are presented in this report, according to main activities programs: Radiopharmacy, Application of Ionizing Radiations, Nuclear Science and Technology, Nuclear Reactors and Fuel Cycle, Renewable Energies, Materials and Nanotechnology, Biotechnology and Lasers Technology.

It must be stressed that all these results were accomplished as a result of the efforts and dedication of the IPEN staff and the support from CNEN, MCT, SD/SP, IAEA, FAPESP and CNPq.

The institute was conducted by Dr. Claudio Rodrigues in the period covered in this report.

Dr. Nilson Dias Vieira Junior
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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 and Technology.

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 m². 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 458 Ph.D. degrees and 937 master degrees since then. The actual graduate enrollment is around 400 students.

The internal organization structure comprehends two levels, one for strategic and political issues and for decisions 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.

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Radiopharmacy



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Introduction

The use of radioisotopes in medicine is certainly one of the most important social applications of Nuclear Energy. IPEN, and particularly the Radiopharmacy Program, has a special place in the history of Nuclear Medicine in Brazil.

The production of radioisotopes and radiopharmaceuticals for use in Nuclear Medicine started in the late 50's at IPEN. There has been a significant and constant increase in the demand for these products over the years and nowadays more than 30 products are listed at the 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 Production area carries out the everyday production of primary radioisotopes, labeled molecules and lyophilized kits for labeling with ^{99m}Tc . Quality Assurance is responsible for the quality system management. The Quality Control executes all the tests needed to release products for human use. Research, Development and Innovation develops new radiopharmaceuticals and improves production processes and applications. The Cyclotron Accelerator Division is responsible for the operation of the Cyclotron and carries out the irradiation for obtaining the cyclotron produced radioisotopes.

The highlights of this period were:

- Development of some new radiopharmaceuticals for diagnostics and therapy: ^{111}In -DTPA-octreotide, ^{90}Y -hydroxyapatite and ^{177}Lu -DOTA-Tyr³-octreotate;
- The increase in the demand of ^{18}F -FDG that led to the modification of the law regulating the production of radioisotopes in Brazil and also to the purchase of a new Cyclotron that will be dedicated only to ^{18}F production and new possibilities for positron emission radioisotopes;
- Certification and maintenance of the ISO Quality Management System;
- A new installation project was conceived with the expectation of cGMP compliance;
- 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.

Production and Quality Control of Radiopharmaceuticals

The Production of Radiopharmaceuticals is divided in 3 different areas: Radioisotopes (Tc-99m generator and Primary Radioisotopes); Labeled Compounds for diagnosis (PET and SPECT) and for therapy; and Lyophilized Kits for Labeling with Tc-99m. 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. 300 generators are delivered weekly.

Primary Radioisotopes

¹³¹I-Na - Sodium iodide solution

For oral study of thyroid gland and therapy of thyroid cancer and metastases.

¹³¹I-Na - Sodium iodide capsules

For therapy of hyperthyroidism and therapy of thyroid cancer and metastases.

⁵¹Cr - Sodium chromate

Used in nuclear medicine for study of red blood survival and spleen scintigraphy.

⁶⁷Ga - Gallium citrate

Indicated for localization and detection of soft tissue tumors and inflammatory process.

²⁰¹Tl - Thallium chloride

For cardiac function studies.

³²P - Sodium phosphate

Used in treatment of polycythaemia vera and biotechnology.

³⁵S - Sulphuric acid

Used in metabolic investigation.

Labeled Compounds

¹⁵³Sm-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.

¹³¹I-MIBG - (meta-iodobenzylguanidine)

Diagnostic and therapeutic agent of neural crest-derived tumors.

¹³¹I-Lipi - (lipiodol)

Treatment of hepatocellular carcinoma (HCC), the selective retention suggests its potential as chemotherapeutic or radiotherapeutic agents.

¹²³I-MIBG - (meta-iodobenzylguanidine)

Diagnosis of pheochromocytoma, neuroblastoma and myocardial studies.

¹³¹I-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.

¹³¹I-HSA - (human serum albumin)

For determination of plasma volume and total blood volume.

⁵¹Cr-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.

⁵¹Cr-EDTA - (ethylenediaminetetraacetic acid)

For study of glomerular filtration rate.

¹⁸F-FDG - (fluoro-2-deoxy-D-glucose)

In oncology, cardiology and neurology studies

⁵³Sm-HA - (hydroxiapatite)

⁹⁰Y-HA - (hydroxiapatite)

For synovectomy, treatment of rheumatic arthritis.

Lyophilized 'kits' for Labeling with Tc-99m

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 osteomyelitis.

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 cholecistigraphy.

Production and Quality Control of Radiopharmaceuticals

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 - Phytic acid

Indicated for imaging areas of functional reticuloendothelial cells in liver, spleen and bone marrow and in lymphoscintigraphy study.

During this period of time, the production group has validated and introduced in routine production two new radiopharmaceuticals for diagnosis and therapy of neuroendocrine tumors: ^{111}In -DTPA-Octreotide and ^{177}Lu -DOTA-Tyr³-octreotate, respectively. In 2007, it was distributed 30,784 MBq of ^{111}In -DTPA-Octreotide and 502,645 MBq of ^{177}Lu -DOTA-Tyr³-octreotate to nuclear medicine centers in São Paulo and other states. The evaluation of revenue of radiopharmaceuticals is illustrated in Figure 1.

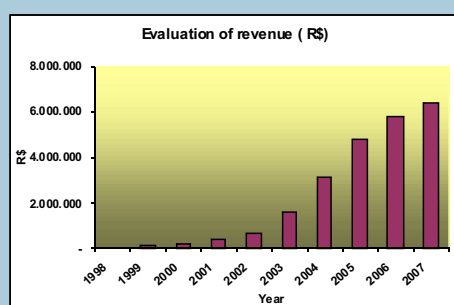


Figure 1. Evaluation of revenue (R\$) since 1998

Quality Control is that part of Good Manufacturing Practice (GMP) which is concerned with sampling, specifications and testing, and with the organization, documentation and release procedures which ensure that the necessary and relevant tests are actually carried out and the materials are not released for use, nor products released for sale or supply, until their quality has been judged to be satisfactory.

In order to evaluate if radiopharmaceuticals comply with the specifications for oral and parenteral human administration according to the GMP and pharmacopoeias, strict quality control tests must be performed. 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. Annually, about 30,000 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 2 shows the distribution of the quality control tests during the year of 2007.

The Quality Control Laboratory has adequate facilities which were improved as several modern analytical equipments were acquired. Special attention has been given to the validation of the analytical methodologies. Metal impurities can be determined by the ICP (Inductively Conducted Plasma) and UV-Vis spectrophotometer; residual solvents from the F18-FDG production in a gas chromatograph; paper and thin-layer chromatography have been used to determine radiochemical purity and the radioactivity is measured in a gamma counter or a scanner; high performance liquid chromatography has been applied to analyze lyophilized reagents labeled with Tc-99m. Sterility tests are performed according to the microbiology procedures stated in the pharmacopoeias and the Limulus amoebocyte lysate (LAL) test is used for the detection of bacterial endotoxin. The clean room environments have been monitored to assess particulate and microbial contamination for GMP purposes. Air sampling, culture medium plate exposure, surface and operator monitoring ensure the manufacturing process and quality control test conditions.

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.

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

- A research project sponsored by IAEA (CRP) is under way and it has the objective of developing production methods for the ^{188}W - ^{188}Re and ^{90}Sr - ^{90}Y generators.
- A research project was developed aiming the purification of high activity ^{99}Mo - $^{99\text{m}}\text{Tc}$ generators. The use of an alumina SepPak cartridge has given very good results, and now it is being used in the routine production of the generators.
- A concentration unity is being studied for increasing the radioactive concentration of $^{99\text{m}}\text{Tc}$ eluted from gel type generators.

Primary radioisotopes

- The optimization of the parameters for the production of ^{131}I by the irradiation of TeO_2 in the reactor and the dry distillation method of ^{131}I separation were completed. Two hot cells were assembled and are currently in use for the routine production of ^{131}I .
- The project for the nationalization of the production of ^{201}Tl was completed and this radioisotope can now be fully produced at IPEN.
- A project aiming the evaluation of the routine production of ^{177}Lu is being developed, using both the direct and indirect reactions for the production at IEA-R1 Nuclear Reactor.
- The recovery of enriched water used in the production of ^{18}F is being studied in order to minimize the costs of production.

Labeling of molecules for diagnosis (PET and SPECT) and therapy

Research projects have been developed aiming the preparation of the following radiopharmaceuticals:

^{177}Lu -DOTA-Tyr³-octreotate - optimization of labeling conditions of the peptide DOTA-Tyr³-octreotate (somatostatin analog) with ^{177}Lu to produce a radiopharmaceutical applied in the therapy of

neuroendocrine tumors. The work is part of a project sponsored by IAEA (Coordinated Research Programme) started in 2002.

^{131}I -DOTA-Tyr³-octreotate - optimization of labeling conditions of the peptide DOTA-Tyr³-octreotate (somatostatin analog) with ^{131}I to produce a radiopharmaceutical applied in the therapy of neuroendocrine tumors. The work is part of a project sponsored by IAEA (Coordinated Research Programme) started in 2002.

^{131}I -Anti-CD20 antibody - optimization of labeling conditions, quality control and purification procedures to produce radioiodinated anti-CD20 antibody applied in the therapy of non-Hodgkin lymphoma.

^{188}Re -HEDP - optimization of labeling conditions, quality control and purification procedures to produce a phosphonate labeled with ^{188}Re for bone pain palliation.

^{90}Y - citrate and hydroxiapatite - preparation of radiopharmaceuticals for therapeutic applications in radiosynovitis.

^{166}Ho -microspheres - development of methods of preparation of resin and polymer based microspheres for therapy of liver cancer.

^{188}Re -Anti CD20 antibody - optimization of labeling conditions, quality control and purification procedures to produce anti-CD20 antibody labeled with ^{188}Re for therapy of non-Hodgkin lymphoma.

^{177}Lu -bombesin - development of bombesin analogs labeled with ^{177}Lu for therapy of prostate cancer.

Development of therapeutic radiopharmaceuticals based on ^{177}Lu for radionuclide therapy - International Atomic Energy Agency (IAEA) Coordinated Research Programme.

Lyophilized kits

The synthesis of compounds not commercially available and the development of new lyophilized kits are necessary to introduce new $^{99\text{m}}\text{Tc}$ -based radiopharmaceuticals. Lyophilized kits allow the instant preparation of the labeled molecules with $^{99\text{m}}\text{Tc}$ without purification steps prior to administration.

Development of ^{99m}Tc based small bio-molecules using novel ^{99m}Tc cores - International Atomic Energy Agency (IAEA) Coordinated Research Programme - New ligands as peptide with sequence RGD, annexine, glucose analogue, and quinazoline analogue will be labeled with the Tc cores and evaluated for tumor uptake *in vivo* (2003-2006).

^{99m}Tc -**anexin-V** - labeling of anexin-V for apoptosis studies.

^{99m}Tc -**glucoheptonate** - preparation of kits for *in vivo* labeling of leukocytes with ^{99m}Tc .

^{99m}Tc -**antimyosin** - labeling of fragments of the antimyosin antibody for cardiac evaluation.

^{99m}Tc -**DMSA-V** - preparation of a direct kit for labeling of DMSA-V for tumor localization.

^{99m}Tc -**BFCA-HYNIC-[Tyr³]-Octreotide** - labeling of octreotide with ^{99m}Tc for diagnosis of neuroendocrine tumors.

^{99m}Tc -**thymidine** - labeling of thymidine with ^{99m}Tc for tumor localization.

Quality control analytical methodologies

Determination of Sn(II) using polarography - application in the routine quality control of lyophilized kits.

HPLC technique for quality control of ^{99m}Tc -radiopharmaceuticals - application in the routine quality control of lyophilized kits.

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 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 210 of 04 August, 2003 of the National Sanitary Agency (ANVISA) of the Health Ministry. This Resolution does not include any particular reference to radiopharmaceuticals. A specific regulation for radiopharmaceuticals is in course in Brazil. A "Radiopharmaceutical Group" is being coordinated by ANVISA in order to establish the rules governing radiopharmaceutical products in Brazil. Also recently, the Brazilian Pharmacopoeia constituted the "Sub-commission of Radiopharmaceuticals" to prepare the radiopharmaceuticals monographs to integrate the Brazilian Pharmacopoeia. IPEN has participated in these work groups which reflect the importance of the radiopharmaceuticals in the context of pharmaceutical production in Brazil.

In the Radiopharmacy Directory of IPEN, the Quality Assurance Management is responsible for maintenance and improvement of the Quality Management System (according to ISO-9001-2000) 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 checks the production and quality control operations to ensure that a radiopharmaceutical of sufficient quality is produced. 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:

Personnel

To ensure the safe manufacture of radiopharmaceuticals, personnel was trained in GMP, safe handling of radioactive materials and radiation safety procedures. Periodic courses and training have kept abreast of the latest developments in the field, according to a previous and annual training plan.

Production, quality control and research equipments

A calibration program has been continuously developed at regular intervals and equipments are checked daily or before production is started. A specific plan for qualification of production and quality control equipments was elaborated and has been implemented.

Contamination control

Clean areas should be maintained in an appropriate cleanliness standard and supplied with air, which has passed through filters of an appropriate efficiency. This condition protects the product from microbiological contamination by the environment. A planning to improve the conditions of the hot cell was started and an “Environmental Monitoring Program” was introduced in the production and quality control installations, which included elaboration of specific procedures, personnel training and acquisition of equipments and materials.

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. The Project of a new installation for the Radiopharmacy Center was concluded and sent to the sanitary authority for appreciation. In parallel, some rebuilding is in course to adapt the actual clean areas of the production to GMP conditions.

Regularization of the radiopharmaceutical in Health Ministry

All the directions of some radiopharmaceuticals (lyophilized reagents for labeling with technetium-99m) were reformulated to attend the regulatory instructions. Technical report for the register of the products is in course.

Cyclotron Accelerator

The cyclotron, Cyclone 30 model, manufactured by Ion Beam Applications-Belgium, is a compact, fixed-field, fixed-frequency, that can accelerate H⁺ ions with energies between 15 and 30 MeV. This energy range and its high external beam current available (350 μ A) 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 . The ions are produced by a multicusp external source, located above the cyclotron. They are axially injected and inflected in the medium plane of the cyclotron and are subjected to a fixed magnetic field and gain energy due to a high voltage alternating electric field induced on the Dees. The beam is extracted by means of a carbon foil, which can be adjusted at different radii. The stripping foils are mounted on two stripping probes, one for each beam line and can be radially and azimuthally adjusted, allowing the extraction of two different beams at different energies or current simultaneously. That means, two radioisotopes can be produced at the same time. Figure 2 shows the Cyclotron Cyclone 30.



Figure 2. 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. These target facilities are described as follows:

Solid Target

This system was manufactured by Ion Beam Applications-Belgium, and it uses a target at 6° with respect to the beam axis, resulting in an enlargement of the beam by a factor of 10. A cylindrical steering magnet rotates the beam by an adjustable AC field. The target material (^{68}Zn or ^{203}Tl) is electrodeposited on an elliptical area measuring 10mm x 100mm,

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.

Liquid Target

At IPEN, ^{18}F is produced by the $^{18}\text{O}(\text{p},\text{n})^{18}\text{F}$ reaction using enriched water as target material. The liquid target system (Figure 3) 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 of 5 mL 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 .

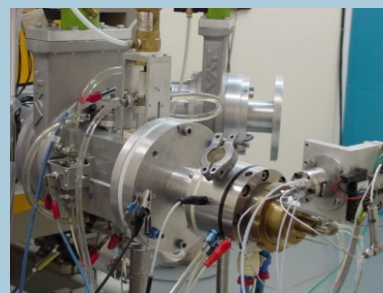


Figure 3. Liquid target system

Gas Target

Due to the high cost of acquisition, IPEN has decided to develop its own system to produce ^{123}I via ^{124}Xe irradiation. This system (Figure 4) 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. In the

unlikely event of a dual window failure, the Xe-124 gas will be expanded in the beam alignment system and will be trapped in a safety volume cooled with liquid nitrogen and the beam gate automatically will be closed. 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.

Typical values:

target volume: 75 mL;

$P_{Xe} = 2$ bar (without beam);

$P_{Xe} = 4.5$ bar (during irradiation);

$E = 30$ MeV; $i = 50\mu A$;

Yield (EOB) = 8mCi/ μAh

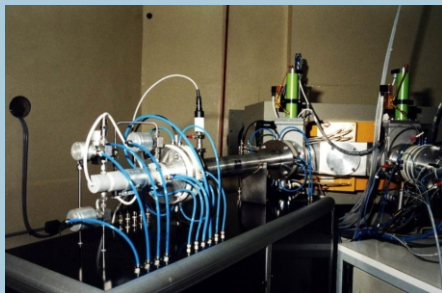


Figure 4. ¹²⁴Xe target system

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Honor Mention and Awards

Menção Honrosa ao Centro de Radiofarmácia do IPEN por relevantes serviços com a Rádio Sinoviortese no tratamento de Sinovite Crônica hemofílica. Federação Brasileira de Hemofilia - Cuiabá (MT) - 02/01/2007.

Application of Ionizing Radiations



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Introduction

The main subject of the Application of Ionizing Radiations Program is to disseminate and consolidate techniques leading to the use of the radiation technology and radioisotopes applications in Industry, Human Health, Agriculture and Environmental Preservation. This Program is divided into four subprograms:

- Food and Agricultural Products Irradiation;
- Radiation and Radioisotopes Applications in Industry and Environment;
- Radioactive Sources and Radiation Applications in Human Health;
- Radioactive Facilities and Equipments for Nuclear Techniques Applications.

After the modernization and implantation of new irradiation systems for the Electron Beam Accelerators 1,200 km of wire and electric cable, 35 km of polyethylene foam and 107,000 powered diodes were processed per year. In this period, two patents were issued, scientific prizes were awarded and the radio sterilization of medical, pharmaceutical and biological products increased around 50%. Furthermore, the production of 240 ¹⁹²Ir and ⁶⁰Co sealed sources, the ⁷⁵Se sources loading services and several inspections in irradiators, command cables and guide pipes have covered all the national demand. Since 2005, the Multipurpose Irradiator has been used as a demonstration facility for manufacturers, who need an economic and logistic in house irradiation alternative system and as support to the local scientific community on the development of process and products using gamma radiation, assisting the traditional and potential users on processes validation, training and qualification of operators and radioprotection officers.

The improvement of the activities of radioisotope technology application in the petrochemical and chemical industries for processing control and sanitation, the distribution of 36,000 ¹²⁵I seeds per year and activities in the RadTech South America Conference can also be highlighted.

All realizations and achievements were only possible due to governmental financial support, standing out six projects by FAPESP, six by CNPq, two by FINEP and six international projects (ARCAL, TC and RC) supported by the IAEA, as well as, national and international partnership and cooperation with industries, universities and other institutions.

The research and development activities concerning this program are developed at the Radiation Technology Center - CTR. A technical staff of 59 professionals including 20 Ph.D., 8 M.Sc., 5 engineers and 26 technicians are engaged in this Program. Besides that, 15 undergraduate students and 49 graduate students also contributed for the activities development. In the last 36 years, US\$ 26 millions were invested in the Center responsible for the Program. During its trajectory of success and achievement, it can be highlighted, in the last three years, the FAPESP, CNPq and IAEA projects for construction and implantation of the ⁶⁰Co Multipurpose Irradiator, for technological domain of ¹²⁵I seeds production for prostate cancer treatment; and also, the assembling of industrial tomography equipment for multiphase flow system analyses.

Ionizing radiation application in food and agricultural products

Irradiated food detection laboratory located at the Radiation Technology Center (Nuclear and Energy Research Institute) 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, our laboratory research also included the detection of genetically modified food, irradiated and unirradiated. 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.

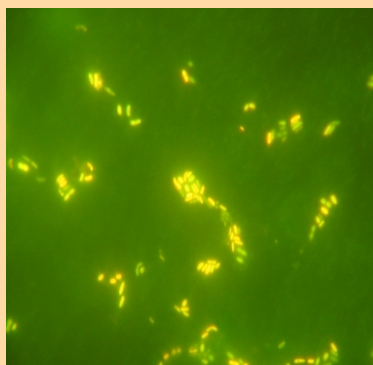


Figure 1. Biological method of food irradiation detection (DEFT/APC), a microbiological screening method based on the use of epifluorescent filter technique (DEFT) and the aerobic plate count (APC). Bacterial cells recovery from irradiated ready-to-eat chicory sample (0,5kGy)

Brazil started the use of radiation technology in food processing in the sixties, with research led to disinfestations and extension of shelf life of fruits. The regulation on food irradiation started in 1969, establishing dose limits for the products; a new version was updated in 2001 without limits and similar to recent modifications introduced by the Codex Alimentarius. Ionizing radiation may be used as a treatment for pest risk management. The adoption of irradiation treatments requires that the efficacy of the treatment be scientifically demonstrated. Application of the treatment

requires dosimetry and dose mapping to ensure that the treatment is effective in particular facilities and with specific commodity configurations. The availability of methods for detection of irradiated food contributes to increase consumer's confidence. Tommy Atkins mangoes were from Petrolina, northeastern region of Brazil, harvested at degree 3, on October 7th and sent immediately to the Instituto de Pesquisas Energéticas e Nucleares-IPEN (São Paulo, Brazil) where they were treated. The total quantity was around 630 fruits, which were divided in three batches. One of them was submitted to a hot water dip treatment (46°C during 110 minutes), that consisted in a control group. The two other groups were irradiated in a Multipurpose Gamma Source (IPEN, São Paulo, Brazil). The delivered doses were 0.4 and 1.0 kGy. After treatment, the mangoes were stored in a conditioned room (11°C $\pm$ 1 and 66,6% RH) until the trip to Canada (October, 17th). Fruits were sent to the Canadian Irradiation Center (CIC, Laval, Canada) by plane at environmental conditions. The storage at CIC was done under environmental conditions (20°C $\pm$ 2; 40% $\pm$ 2 RH). The experiment consisted in: Visual observations (mold, rot end or defect occurrence); Mass loss evaluation; Physical and chemical analysis (pH, titrable acidity, total soluble solids, ratio (solids/acidity), texture, color and water activity); Sensory evaluation (color, odor, taste and texture acceptance and overall appearance of whole fruits). Physico chemical analysis showed that gamma irradiation or thermal treatment resulted in similar performances and the main results indicated that irradiation could be a potential disinfestation treatment comparable to thermal treatment, for international purposes.



Figure 2. Irradiated mangoes

Modification and preparation of polymeric materials and composites by ionizing radiation

Electron beam curing using epoxy resin system

The electron beam (EB) curing technology allows the use at room temperature and reduced curing times and this is one of the main advantages over thermal technology. The aim of this work was to investigate electron beam curable epoxy formulations to use in filament winding processes to produce composite material with similar or better properties than thermal curable composites.

Utilization of rice husk ash as reinforcement filler for polyamide 6 irradiated by electron beam

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 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 composites producers.

Ionizing radiation effect by electron beam on ultra high molecular weight polyethylene virgin and recycled industrial

Ultra High Molecular Weight Polyethylene (UHMWPE) is an engineering plastic which has several applications, chiefly, in specific areas of the industry and medicine. The goal of this was to recycle the UHMWPE UTEC 3041 and study the properties of this recycled material and of the virgin UHMWPE and compare the results between both with the materials undergone to different radiation doses.

Characterization of crosslinking polyethylene foam by irradiation process with electron beam

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 polyethylene of low density 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.

Ionizing radiation effect on the properties of polyamide 6 with glass fiber reinforcement

The aim of this work was to study the effect of ionizing radiation on properties of polyamide 6 with glass fiber reinforcement, undergone to different radiation doses. The properties of the non-irradiated and irradiated polyamide 6 with glass fiber reinforcement were evaluated.

Ionizing radiation effect on composites of wood flour in polypropylene matrix using barium titanate as coupling agent

The composites of wood-polymer are materials that have the combination of a polymeric matrix and a reinforced cellulosic load. These materials are characterized for their versatility, lightness, better resistance to humidity and environmental deterioration, besides their economical advantage in using as reinforcement a cellulosic residual as the wood fiber. The aim of this work was to study the ionizing radiation influence in the properties of the composite of wood fiber reinforced polypropylene using barium titanate with coupling agent to improve the interaction between the interfaces.

Degradation of cellulose from sugarcane bagasse by ionizing radiation to obtain ethanol and natural polymers

In recent years, there has been an increasing trend towards more efficient utilization of agro-industrial residues, such as sugarcane bagasse, as raw materials for industrial applications. Several processes and products have been reported that utilize sugarcane bagasse as a raw material. These include electricity generation, pulp and paper production, and products based on fermentation. Sugarcane bagasse generally contain up to 45% glucose polymer cellulose, 38% hemicelluloses, an amorphous polymer usually composed of xylose, arabinose, galactose, glucose, and mannose and 20% lignin. The main obstacle to produce ethanol bio-fuel from cellulose is how to accelerate the hydrolysis reaction that breaks it down into starches and sugars suitable for fermentation. The major cellulose hydrolysis processes, as chemical or enzymatic reactions, are so harsh that toxic degradation products are produced and can interfere with fermentation. The radiation processing is a powerful technology to accelerate this hydrolysis reaction. The main benefit of ethanol production from sugarcane bagasse is the environmental protection and recovery, reducing greenhouse gas emissions compared to oil derivatives and the increase

Industrial and environmental application of ionizing radiations

of ethanol production per planted hectare. In addition, as a large source of lignocelluloses biomass, sugarcane bagasse is a cheap and annually renewable resource suitable for producing natural cellulose fibers. The preparation and properties of new polymers from hemicelluloses and the use of cellulose and its derivatives in a diverse array of other applications, such as films, plastics, coatings, suspension agents, and composites are another important part of this research program. This project is being realized together with Sugarcane Technology Center, located at Piracicaba, Sao Paulo, Brazil, CTC and has about 200 associated sugarcane farms.



Figure 3. Sugarcane bagasse processed by ionizing radiation

Food polymeric packaging processed by radiation

In this study, the mechanical properties (tensile strength and percentage elongation at break and penetration resistance), optical properties, gas oxygen and water vapor permeability, the overall migration tests into aqueous food simulants (3% aqueous acetic acid) and fatty food simulants (n-heptane), as well as the formation of volatile radiation products tests were used to evaluate the effects of ionizing radiation (gamma irradiation or electron-beam irradiation) on commercial monolayer and multilayer flexible plastics packaging materials. These films are two typical materials produced in Brazil for industrial meat packaging, one of them is a monolayer low-density polyethylene (LDPE) and other is a multilayer coextruded low-density polyethylene (LDPE), ethylene vinyl alcohol (EVOH), polyamide (PA) based film (LDPE/EVOH/PA). Film samples were irradiated with doses up to 30 kGy, at room temperature and in the presence of air with gamma rays using a ^{60}Co facility and electron beam from 1.5 MeV electrostatic accelerator. Alterations of these properties were detected according to the dose applied initially eight days after irradiation took place and new alterations of these values when the properties were evaluated two to three months after irradiation process. The results showed that scission reactions are higher than cross-linking process for both studied films, irradiated with gamma rays and electron beam. The evaluated properties of the irradiated films were not affected significantly with the dose range and period studied. The monolayer Unipac PE-60 and the multilayer Lovaflex CH 130 films can be used as food packaging materials for food pasteurization

and in the sterilization process of by ionizing radiation using gamma facilities and electron beam accelerators in commercial scale.

Radiation-induced grafting in polymeric film

On Radiation-induced Grafting research, the Radiation Technology Center at IPEN, CTR-IPEN, approved the project "Development and Application of Chlorinated, Fluorinated and Technological Polymer Films Modified by Grafting Process Using Electron Beam and Gamma Radiation", which begins in October, 2007. This is a Coordinated Research Project from the International Atomic Energy Agency, CRP-IAEA, which includes participants from fourteen countries, and the scope is "Development of Novel Adsorbents and Membranes by Radiation-induced Grafting for Environmental and Industrial Applications". From 19 to 23 November of 2007, the CTR researcher J.E. Manzoli, coordinator of the project, was in Vienna, Austria, for the first meeting of the participant countries. Preliminary results on grafting of styrene on polytetrafluoroethylene (PTFE), polyvinyl difluoride (PVDF) and polypropylene (PP) were reported. Part of the heating chamber of the project was finished at 2007.

Disinfection and 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 disinfections of works of art and the preservation of artifacts in their original form or in the state in which they are found, by impregnation with gamma radiation curable resins, avoiding their reinfestation. The effects of gamma radiation on Brazilian paper and wood based cultural heritage have been investigated concerning some specific characteristics.

Biological assays for effluent control

Several effluents are submitted to electron beam irradiation in order to reduce color, surfactants and whole toxicity. In order to measure the efficacy of radiation as a technology for hard effluents, luminescent *Vibrio fischeri* bacteria have been used, called as biological assays, also applied for toxicity effluent evaluation. Other biological indicators are also studied. As radiation is used for biological control on contaminated paper and materials the radiation doses and its effects on material integrity are being determined.

Gemstones enhancement using Gamma Radiation

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



Figure 4. Green quartz

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/CTR 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 97,5kW and 37,5kW) facilities of the Center in ordinary services and to develop new products and services by radiation processing. The dosimetry procedures for radiation processing in our laboratory 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, we have 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, irradiation and data procedures. The dosimetry systems that we use 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

system: Red Perspex 4034, Amber 3042, Gamachrome YR, CTA, Gafchromic, FWT and TL dosimeters. The response of routine dosimeters are often influenced by changing dose rates, irradiation temperature, post irradiation time and also environmental conditions, then, studies and evaluations of these parameters are carried out so that the radiation absorbed dose by products may be precisely and accurately measured.

Ionizing radiation application on the treatment of liquid and solids residues and soil remediation

Since 1992 this group has been studying the feasibility to use the Advanced Oxidation Process by ionizing radiation on removing toxic and refractory pollutants (organic compounds) in industrial effluent, drinking water, solid wastes, and on destroying pathogenic microorganisms in wastewater, sludge and in historic documents. These studies are focused to become economically feasible the use of this technology in real industrial effluents that are recalcitrant when treated by conventional methods. As a consequence of pesticides used in agriculture, the human population is constantly exposed to numerous chemical species present in the environment. The Brazilian agriculture activities have consumed about 288,000 tons of pesticides per year conditioned in about 107,000,000 packing with a weight of approximately 23,000 tons. Brazilian Federal law states that the disposal responsibility of the pesticide packing is by attributed to the industry that has search for new technologies to recover and recycle the material. In order to evaluate the efficiency of radiation processing on removal of the pesticides contamination; high-density polyethylene (HDPE) packing were irradiated using Radiation Dynamics Electron Beam Accelerator with 1,5MeV energy and 37kW power and gamma rays. The radiation processing was efficient on removing the pesticides: methomyl, dimethoate, carbofuran, and methyldathion, triazine, thiophos, atrazyme, endosulfan, chlorpyrifos, thiazophos), and trifluralin.



Figure 5. Irradiation of pesticides HDPE packing

Industrial and environmental application of ionizing radiations

In the Radiation Technology Center (CTR) there are two Industrial Electron Beam Accelerators of 97.5 kW (1.5MeV-65mA) and 37.5 kW (1.5MeV-25mA), supplied by IBA Industrial Inc. and two Cobalt-60 Irradiators - Gammacell (11,000Ci) and Panoramic (5,000Ci) designs. The Electron Beam Accelerator and Cobalt-60 Irradiators are mainly applied for research, development and services of preservation and disinfestation 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 2007, 25,600 medical, pharmaceutical and biological products were radiosterilized and 107,000 semiconductors (diodes) were processed by ionizing radiation in these radioactive facilities. Annually, 1,200 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.

Radiation processing applying industrial electron beam accelerators and gamma irradiators

In the Radiation Technology Center (CTR) there are two Industrial Electron Beam Accelerators of 97.5 kW (1.5MeV-65mA) and 37.5 kW (1.5MeV-25mA), supplied by IBA Industrial Inc. and two Cobalt-60 Irradiators - Gammacell (11,000Ci) and Panoramic (5,000Ci) designs. The Electron Beam Accelerator and Cobalt-60 Irradiators are mainly applied for research, development and services of preservation and disinfestation 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

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Sealed source production for gammagraphy 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. The non-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 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, the Radiation Technology Center (CTR) has supplied industrial gamma sealed sources to more than 20 customers in Brazil and other countries in Latin America and Caribbean. Annually, the laboratory produces 240 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 several inspections in irradiators, command cables and guide pipes and also selenium sources loading services. These supplies allow taking more than 100,000 radiography per year. The principal CTR's customers are Arctest, ASNDT, Brasitest, CBC, Confab, End Test, Gamatron, Qualitec, Voigth, Tricom and Sertech.

Industrial and environmental application of ionizing radiations

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

Radioactive tracer, bromine-82, and dye tracer, rhodamine WT, applications in grounded pipe flow rate measurements. Flow rate order of magnitude: 0,1 m³/s up to 3,5 m³/s. Radioactive tracer, iodine-131, applications for mean residence time determinations in tanks and digestors of domestic and industrial waste water treatment plants. Volume order of magnitude: 7,000 m³ up to 12,000 m³. This tracer application group took part in the practical activity on radioactive tracer application during the AIEA-ARCAL course held in Santos, S.P. Brazil, with the presence of 12 countries representatives under RLA 8/042 project. Period: October 1 to 5, 2007.

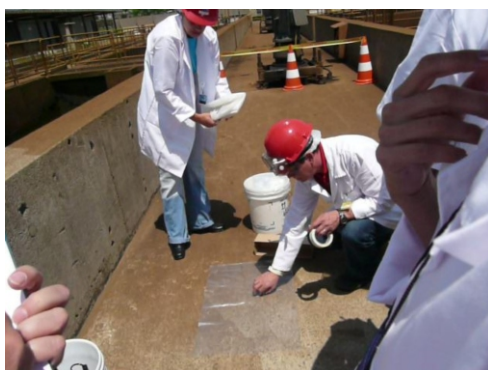


Figure 6. Procedures for radiotracer injection

Health application of ionizing radiation and radioactive sources

Radiosterilization for tissue banks

In Latin America, the industrial level ionizing radiation sterilization is used for more than three decades, ever since, foods and medical, pharmaceutical and cosmetics products are treated. Later, this activity extended to the sterilization of human tissues for graft and this activity was reinforced in some countries by the technical cooperation and financial support of the International Agency of Atomic Energy - IAEA. Brazil was incorporated to this project in 1998 through the "Hospital das Clinicas" of Sao Paulo City, where the Tissue Bank was installed and the Institute for Energetic and Nuclear Researches, where the tissues are being irradiated. Most of the tissues transplanted, such as, skin, bone, amnion and other not viable tissues, can be treated with ionizing radiation to minimize the immunogenicity, to kill bacteria and to reduce the contagious diseases transferring risk. Besides implanting the irradiation services routine to the tissue banks of the country, the researchers developed irradiation devices for human tissues, implant dosimetry procedures for irradiation processes control, implant the quality warranty program for tissue irradiation, optimize type and dose to be supplied according to the preservation process of which the tissue was submitted, collaborate with the implementation of quality systems of the Tissue Banks and experimental and clinical applications of irradiated tissues. In the last few years, preserved tissue allograft, such as bone and skin, have been used in reconstructive surgery in many clinical disciplines, like orthopedic and plastic surgery. The risk of transmission of infectious diseases by allograft, however, is a constant concern. To this end, many steps should be taken, including tissue sterilization. Of the available sterilization techniques, the application of ionizing radiation deserves to be considered for its efficiency. Its deployment, nevertheless, is still contested since there is few data on its effects upon the tissue allograft. The skin glycerol preservation has a bacteriostatic effect after certain time. On the other hand, skin sterilization by ionizing radiation may reduces the quarantine period for transplantation in patients and their safety is considered excellent. At the CTR department, we established procedures using two sources of ionizing radiation for sterilization of human skin allograft, and to evaluate the skin after gamma and electron beam irradiation. Skin samples were submitted to doses of 25 kGy and 50 kGy. We evaluated the impact of the irradiation on the mechanical properties through the analysis of stress-strain and they were also accomplished by morphology and ultra-structure studies. Also in the current work, we have started de standardization of the tests that will be used for the characterization of irradiated bones. Initially, two types of analysis have been chosen: biomechanical and histological.

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 (diameter = 0.8mm e length = 4.5mm) 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.

Facilities and devices for application of nuclear techniques

Irradiation facilities

Focusing the development of new irradiators and support the activities on radiation processing the following activities were performed:

- Conception design of a under water gamma irradiator dedicated for gemstones processing.
- Conception design of gamma irradiator and methodology of control for application of good manufacturing practices (GMP) on human blood processing to avoid the transfusion- associated graft-versus-host-disease (TA-GVHD).
- Gamma irradiation activities on samples for food preservation supporting 8 Research Institutions.
- Experiments on gamma processing for validation and routine control.
- Workshop in Petrolina : technical and economical aspects of mango irradiation (commercial scale).
- Training of fellowships from South America on radiation processing and design of irradiation facilities.
- Training of Radiation Protection Officer (06 students).
- Sterilization services (500m^3).
- Gemstone irradiation studies for dose definition.
- Art collection irradiation for decontamination of xylograph wood dye printings and manuscripts from cordel literature (6000 pieces).

Radiation detectors and industrial computed tomography

The evolution of the fundamental and applied research in the nuclear field is entailed to the development of new types of radiation detectors, which have determined different technological applications in several areas, such as, Nuclear Medicine, Industry, Agriculture, Environmental Science and Radiological Protection. Aiming to reach these goals, the following activities were carried out in the period covering the years 2005-2007:

Development of radioguided surgery probe

Intraoperative gamma probes have been widely used in surgery oriented to locate the

sentinel lymph nodes in breast cancer and malignant melanoma during the surgery. Two surgical gamma probes were developed, being one with a CsI(Tl) crystal coupled to a PIN photodiode and another with a TlBr crystal as radiation detectors. The TlBr developed at IPEN was used, since the production of this crystal is in development stage and it is not yet commercially available. Both probes, TlBr and CsI(Tl), presented similar performance and met the necessary requirements to be used in radioguided surgery. At present, the probe prototypes are being evaluated concerning their clinical use by Escola Paulista de Medicina - UNIFESP and Hospital Albert Einstein.



Figure 7. Radioguided surgery probe

Study of the long term stability of the TlBr Semiconductor Detector

Thallium bromide is a very attractive compound semiconductor for applications as a room temperature radiation semiconductor detector. However, it is known that the lack of long term stability has limited the use of this kind of semiconductor detectors. The TlBr crystal is being developed at IPEN and, in addition to the improvement carried out in the purification and growth methodology, efforts have been concentrated to investigate the polarization effects and long-term stability of the TlBr crystal detectors. Besides, electronic systems and pulse-processing techniques are being investigated to improve the TlBr crystal detector performance. A good stability over 40 days measurements, carried out at room temperature without removing the bias voltage, was found. The characterized TlBr crystal as a detector has shown good response to gamma radiation and has great potential to be used as a semiconductor detector in radioguided surgery probes.

Development of an industrial computed tomography system for multiphase system

Today, the interest of a wide range of industries, such as chemical and oil sectors, in the use of computed tomography began to grow, for improving design, operation and troubleshooting of industrial processes. To follow this trend, the IPEN laboratory began a study for the development of the computed tomography methodology for multiphase systems. Two computed tomography (CT) systems were developed for industrial applications. The first one was a first generation CT, which use a 5.08 cm NaI(Tl) detector. The second was developed with a 40 cm bar scintillator detector, using two phototubes in its extremities in order to be used as a sensitive position detector. The data acquisition board, the mechanical control interface and the software were specially developed in our laboratory for this project. The tomography systems were tested using a phantom placed between the detector and a collimated ^{60}Co source. The phantom used was a standard polypropylene phantom designed and prepared by the University of Bergen, Norway, granted by the International Atomic Energy Agency. A good resolution was observed for all images reconstructed from the developed CT systems. The attenuation coefficient found for the different density materials used to fill the phantom holes are comparable to the theoretical values described in the literature for these materials.

Scintillator crystals applied in X-ray equipment for inspection in real time

For the industrial inspection and quality control, the artificial vision has appeared as a new and important technique that can provide accurate inspections in pieces, increasing the product quality significantly. The artificial vision has also been used as calibration tool during the production, to correct defective pieces generating processes. In this work, the feasibility of using the CsI (Tl) scintillator developed at IPEN, as a radiation sensor in the X-ray equipment has been studied, aiming its application in artificial vision of dynamic inspection systems for X-ray, with the cooperation of a Brazilian private company. This project has the The State of São Paulo Research Foundation support. This equipment will be used to detect undesirable elements (metals, stones, bones, plastics) in foodstuff and thus meeting the procedures of (a) Alimentary Safety and (b) Public Safety.

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 Centro de Tecnologia das Radiações (CTR) at IPEN/CNEN-SP. Until now, the diode's response as a function of the gamma-ray doses is linear in the interval of 5 Gy to 100 Gy.

MCZ Diode Response as a high-dose gamma radiation dosimeter

A high-resistivity Magnetic Czochralski (MCZ) silicon diode processed at the Helsinki Institute of Physics has been successfully used as a high-dose gamma dosimeter in radiation processing, within total doses from 100 Gy up to 3 kGy at a dose rate of 3 kGy/h. In this interval, the dosimetric response of the diode is linear.



Figure 8. Magnetic Czochralski (MCZ) diode

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 high uniform electric fields. The heart of the chamber is an RPC like cell with a bulk aluminium 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.



Figure 9. RPC like cell with a bulk aluminium anode and a glass cathode

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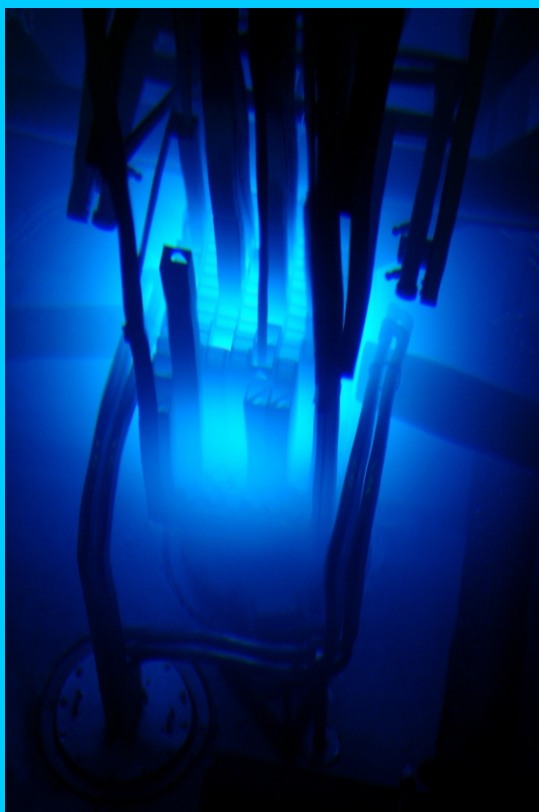
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Nuclear Science and Technology



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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 γ -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 computation 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. A major operation was carried out in 2007, with the cooperation of the United States Department of Energy (USDOE), which enabled the transportation of a second batch of 33 spent fuel elements of US origin, stored in the wet storage facilities of the reactor pool since 2000, back to the United States. In the year 1999 a similar program transferred back to US 127 spent fuel elements.

The Radiation Metrology Center was established in October 2002. 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.

The Radioprotection Service cares for the radiological safety of IPEN workers and general population, through radiation control programs and using national and international standards. Research related to the main activities is also performed.

The Radioactive Waste Management Laboratory receives, characterizes, treats and stores radioactive wastes generated at IPEN and at external communities, using national and international standards. Several research projects related to the main activities are also undergoing.

Study of the crystalline and magnetic structures of materials by neutron 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. The neutron diffraction patterns are measured in the IPEN-CNEN/SP PSD neutron powder diffractometer. The neutron powder diffractometer was constructed at the IPEN machine shop and installed at the IEA-R1 research reactor of the Institute. It is an extensive upgrading of the old IPEN-CNEN/SP multipurpose neutron diffractometer "Aurora". The old diffractometer was a single-detector instrument with a boron trifluoride (BF_3) detector and a flat copper mosaic single crystal monochromator. The main modifications introduced in the old instrument was the installation of a position sensitive detector (PSD) and a double-bent monochromator. The PSD is formed by eleven ^3He linear detector elements clamped together at each end to form a rigid plane. Placed at a distance of 1,600 mm from sample, the PSD spans an angular range of 20 of a diffraction pattern, with a quite good resolution. An extended powder diffraction pattern can be obtained by moving the detector and collecting the data in 20 segments in a 2 interval ranging from 5 to 130. In order to increase the neutron beam flux at the sample position, a focusing Si perfect single crystal monochromator was installed in the instrument. The monochromator is made of 9 vertically stacked Si single crystal blades, mechanically bent in the horizontal plane and quasi-bent by segmentation in the vertical plane. With a take-off angle of 84, the monochromator can be positioned to produce 4 different wavelengths, namely 1.111, 1.399, 1.667 and 2.191 Å (nominal values). A rotating-oscillating collimator (ROC), placed at the entrance to the detector shield, eliminates parasitic scattering from furnace or cryorefrigerator heat shields in the vicinity of the sample, while only reducing the scattered intensity by ca. 10%. The collimator also makes the PSD less sensitive to the ambient background leaking in through the shielding entrance. A beam shutter installed in the instrument protects the operator during sample manipulation or installation of any device at the sample position. Recently, a sapphire filter has been installed in order to remove fast neutrons from the neutron beam that impinges the sample. This lowers the background level in the diffraction patterns obtained with the instrument. In comparison to the former instrument, the new diffractometer has a better resolution and is ca. 600 times faster in data acquisition. The IPEN-CNEN/SP PSD neutron powder diffractometer has been designed mainly

for crystalline and magnetic structures determination and for application of the Rietveld method in quantitative phase analysis, although 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 , $^{111\text{m}}\text{Cd}$ besides the usual ones like ^{111}In and ^{181}Hf . A 4-BaF₂ 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 ^6Li ion beam from the Pelletron accelerator in IFUSP to implant ^{111}In probe into the sample through $^{106}\text{Pd}(6\text{Li},3n)^{111}\text{In}$ nuclear reaction was developed and it is also available. The compounds which are being investigate are: 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 $^{111}\text{In} \rightarrow ^{111}\text{Cd}$ and $^{181}\text{Hf} \rightarrow ^{181}\text{Ta}$ nuclear probes which were introduced into the samples through a chemical process and $^{140}\text{La} \rightarrow ^{140}\text{Ce}$ produced by irradiating the sample with

Experimental Nuclear Physics and Condensed Matter

quadrupole interaction parameters as well as the magnetic hyperfine field was obtained for each compound. Diluted Magnetic semiconductors: new families of semiconductors, which are doped with magnetic materials, are under intensive investigation as they can be used for spintronics. ZnO 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. 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. A very precise ab-initio 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 WIEN97 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 have been initiated.

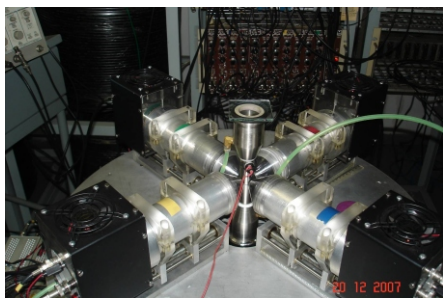


Figure 1. Angular correlation Spectrometer with 4 conical BaF_2 detectors and a sample heating furnace

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 $4\pi\beta\text{-}\gamma$ 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 $4\pi(\text{PS})\beta\text{-}\gamma$ which was recently developed at LMN. The disintegration rate is obtained by the application of the efficiency extrapolation technique. During the period of 2005-2007, the following radionuclides have been standardized by this technique: ^{51}Cr , ^{54}Mn , ^{55}Fe , ^{90}Sr , ^{90}Y , ^{182}Tl and ^{201}Tl . The Nuclear Metrology Laboratory at IPEN has also been involved in the development of radioactive water-equivalent solid sources prepared from an aqueous solution of acrylamide, polymerised by high dose ^{60}Co irradiation. The sources have been prepared with ^{57}Co , ^{137}Cs and ^{133}Ba radioactive solutions and have density similar to water as well as good uniformity. Therefore, they are suitable for performing constancy and accuracy tests, mainly for Medical Radionuclide Calibrators. 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 $4\pi\beta\text{-}\gamma$ 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. This methodology has been successfully applied to achieve primary standardizations of ^{35}S , ^{133}Ba , ^{134}Cs and ^{182}Tl . This approach has got an enthusiastic approval during the ICRM 2005 and 2007 international conferences. 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 cross sections and resonance integrals for ^{41}K (n, gamma) ^{42}K and ^{165}Ho (n, gamma) ^{166}Ho nuclear reactions have been determined experimentally.

Radiation spectroscopy and spectrometry / radioactive decay

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

Nuclear Data

Using single gamma spectroscopy coupled to gamma-gamma coincidence and angular correlation analyses, the group at LEER have been measuring relevant nuclear data on nuclei produced via neutron irradiation in the IEA-R1 reactor, as gamma transition energies, intensities and electromagnetic nature, determining transition placements in the decay scheme, deducing beta-feeding for the excited levels, and measuring thermal neutron cross activation sections;

Nuclear Instrumentation and Methodology

Due to the exploratory nature of the work undertaken by the LEER, the group has also 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 and data analysis softwares, as well as the development of a neutron irradiation facility that doesn't rely on a nuclear reactor;

Semi-Parametric Neutron Activation Analyses

The group at LEER has also been developing a line on neutron activation analyses that replaces the use of certified standards with neutron flux monitors, using the Cd-ratio technique with Au foils.

Highlights 2005-2007:

- Analysis of the decay of ^{155}Sm , using both singles spectroscopy and gamma-gamma coincidence to find new gamma transitions and propose new excited levels, as well as performing theoretical calculations using nuclear models;
- Analysis of the decay of ^{193}Os via gamma-gamma coincidence and angular correlation, finding several new gamma transitions, proposing new excited states and strongly improving the literature information on transition intensities, nuclear models were also used to assess the

Physical nature of the low-lying excited states;

- Development of an Am-Be neutron irradiator, using Monte Carlo techniques to assess its performance and to model a new one with enhanced flux, either using more Am-Be sources or a d-d neutron generator;
- Development of methods to extract precise gamma transition intensities from both gamma-gamma coincidence and angular correlation data;
- Determination of reference values for humans on whole blood for several elements using NAA;
- Elemental analyses on genetically modified food using NAA;
- Determination of several elements' concentrations on biological samples using NAA.

Neutron radiography technique

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 radioactive samples. The radiography is obtained by irradiating the object in a 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 is obtained in real-time. For both cases the radiographs are 2-D projections of the internal structure of the object. The neutron radiography - NR 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 m range were also developed. Nowadays the IPEN possesses also two digital systems to process radiographic images with which has provided services and developed high level researches. In this period (1988-2007) four Msc

and one PhD thesis have been advised and in the present three new PhD thesis by using this same neutron radiography facility are under advising. The figures 2 and 3 show examples of some typical obtained radiographs at the present facility. The NR working group intends to install a neutron tomography system in the NR facility of IPEN-CNEN/SP. Since the neutron radiography is able to provide images of objects which could not be investigated by the standard radiography techniques with X-ray or gamma radiation, the proposed tomograph will be very useful because it is the best alternative to investigate these same objects in 3-D. As has occurred in other countries, the availability of a neutron tomograph will spread the use of the neutron radiography technique in Brazil since it will reach those industrial, technological and research fields not yet reached by the conventional radiography techniques.

Highlights 2005-2007:

- Development of the neutron induced radiography (NIR) techniques to inspect thin samples.
- Development of a quasi real-time device to inspect radioactive samples by the neutron radiography.
- Development and implementation of a digital system to inspect radiography images in track detectors.
- Development and implementation of a digital system to characterize track detectors.

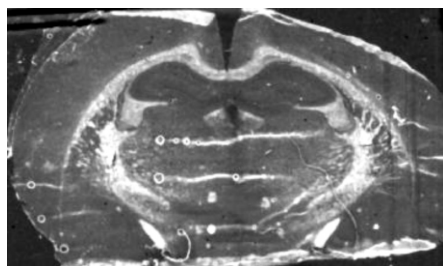


Figure 2. Biological tissue (brain)

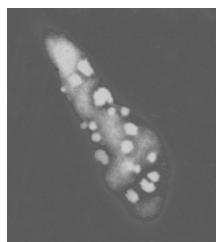


Figure 3. Bacteria colony

Scientific Computing applied to the Nuclear Area

Computing is a tool as important as experimentation and theory in solving the scientific challenges in the nuclear science of the twenty-first century. Central to the mission of our group is enabling the use and exploration of computational science to investigate fundamental problems in nuclear and related areas that require massive calculations and have broad scientific and economic impacts. Examples of these problems include dosimetry and micro-dosimetry modeling and simulation, radiotherapy and nuclear medicine modeling, simulation and planning, nuclear experiments modeling and simulations, and many more. The group's mission also encompass the development of new scientific software for analysis, visualization and exploration of data, employing advanced technologies from computer science and related areas. All this is done with a strong commitment to leverage student's abilities in scientific computing applied to the nuclear area, contributing to form competent scientists for the Brazilian Nuclear Program. Scientific Computing is considered nowadays a third mode of science, besides experimentation and theory. It now plays an equal role to theory and physical experiments in discovery-driven scientific research. Numerical simulation technology offer the possibility to model and simulate, maybe otherwise impossible, experiments that require expensive and inaccessible systems. It's even possible to model a complete nuclear physics facility, including radiation sources, filters, detectors and other parts, execute the simulation and validate the data against published experimental results. To accomplish such studies, however, solid computing skills are required. It's essential to deal with several programming languages, distributed processing and large scale software development, installation and management. Also the integration of several different software systems are essential to be able to successfully exploit computing systems for scientific use. Our group has been working with a Monte Carlo simulation software, mainly Geant4, applied to dosimetry, medical instrumentation and detector studies. Personal dosimeters as well as dose equivalent coefficients have been studied. X ray beams used in radiotherapy and diagnostic have been simulated; we also collaborate with international scientists who are also using Geant4 for dosimetry studies. We also developed a scientific software applied to Instrumental Neutron Activation Analysis; the software is now in the final testing phase before going to production.

This software will allow the incorporation of several gamma spectrum analysis techniques. Also planned is the networked version, which will bring to the laboratory, besides the client/server technology, the database persistence and reliability, allowing, in the future, mining techniques to uncover hidden features in data. The programming language of choice for this software was Python. Being widely used by the scientific community around the world, Python features, such as rapid development, will allow new techniques and algorithms to be quickly tested in the software.

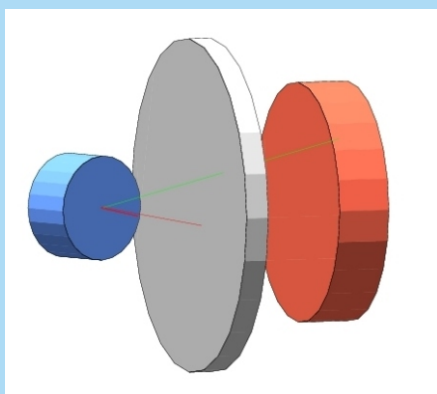


Figure 4. Simulation of a X ray tube: tungsten anode (blue cylinder) shot by an electron beam (red line), producing X rays (green lines) that cross the beryllium window (grey cylinder) and reach the volume to be irradiated (red cylinder)

Neutron Activation Analysis

Nutritional studies in foodstuffs and diets

The forms of malnutrition are numerous and varied, that may lead to serious health problems such as under-nourishment or obesity. In the developing countries, the nutritional problems are due mainly to specific nutritional deficiencies. About 2 billion people suffer from "occult" hunger: deficit of micronutrients such as iron, vitamin A, iodine and zinc. Children and women are particularly vulnerable of food insecurity. Information about trace element distribution is of significant importance in assessing the adequacy of human diets with respect to the intake of essential elements and in evaluating the potential health risks arising from exposure to toxic elements in foodstuffs. Essential and toxic elements have been measured extensively in Brazilian food and diets of different groups by Neutron Activation Analysis. Studies carried out in the period 2005-2007:

Iodine daily dietary intake in a group of Brazilian workers

The main role of iodine in nutrition arises from the important part played by the thyroid hormones in the growth and development of humans and animals. The recommended dietary allowance (RDA) for adult men and women is 150 g/day and the tolerable upper intake level (UL) for adults is 1,100 g/day. Iodine deficiencies can be prevented or reduced by increasing of its dietary intake through fortification of food. There is an intensive international effort to fortify the cooking salt with iodine in several countries, including Brazil. In this study, iodine dietary intake was evaluated through determination of iodine in duplicate portion diet samples by epithermal neutron activation. The collection of samples was carried out in a group of twenty-six workers from a steel industry of São Paulo city. The average daily dietary intake found for worker's group was 840 g/day, ranging from 400 to 1540 g /day. Some of the daily iodine intakes were about 10 times higher than the RDA value.

Total Diet Study: the nutritional elements variability

Since Total Diet Study (TDS) was first conducted in the U.S, in 1961, developed countries have been carrying out their own studies periodically. This study is the first attempt to establish a baseline for TDS in Brazil. Food consumption data source was from a recent household food budget survey (2002-2003) by the Brazilian Institute for Geography and Statistics, which contained 5441 foods. This study provides the nutritional elements contents of Na, K, Ca,

Fe, Zn, Se and Cr in 37 most consumed foods (more than 2 g/day/person) in among people in the state of São Paulo, Brazil. Foods were grouped into 15 food groups, according to their chemical characteristics. Food samples from restaurants of the University of São Paulo were prepared table-ready and analyzed by Instrumental Neutron Activation.

Essential and toxic elements, natural and artificial radionuclides in edible mushrooms

Mushrooms are excellent nutritional sources since they provide proteins, fibers and mineral, such as K, P and Fe. They have also been the focus of environmental studies. In Brazil mushrooms are not consumed in large quantities by the general population since people know little about the nutritional and medicinal benefits that mushrooms offer. Hence, this study intends to contribute to a better understanding of the essential and toxic element content in edible mushrooms, which are currently commercialized in São Paulo state. Concentrations of As, Br, Cr, Cd, Fe, K, Na, Se and Zn and the activities of ^{40}K , ^{137}Cs , ^{238}U and ^{232}Th were determined in edible mushroom species: Shitake (*Lentinus edodes*), Shimeji (*Pleurotus* ssp), Paris Champignon (*Agaricus bisporus*), Hiratake (*Pleurotus* ssp) and Eringue (*Pleurotus Eryngii*).

Essential element determinations in diet and light foods

The consumption of dietetic products, diet and light food, has become a significant part of the Brazilian population's eating habits. It seems to be connected to people's concern with their quality and style of life. Despite an increased emphasis on dietetic foods in traditional Brazilian diet, the knowledge of their chemical compositions in the field of nutrition and toxicology is restricted. The aim of this study is to determine essential elements in diet, light and their similar regular foods by Neutron Activation Analysis and to compare the results of these different categories. For sampling, diet, light and regular food samples: artificial sweetener, cappuccino and chocolate milk were collected in the market of São Paulo city.

Essential elements in milk formulas

The World Health Organization (WHO) recommends that infants up to 6 months be exclusively breastfed. Breastfeeding should continue along with complementary feeding until 2 years of age. However, this is not always possible due to the fact that not all mothers are able to produce sufficient amounts of milk for their infants. According to Codex Alimentarius, the best substitute

choices for maternal milk are infant milk formulas, and when prepared under proper hygienic conditions can be used to feed infants. There are no published data on element content in infant milk formulas marketed in São Paulo city. The objective of this study was to determine element (Br, Ca, Na, K, Fe, Rb, Se and Zn) content by Neutron Activation Analysis in the infant milk formulas consumed in São Paulo city and to compare with the printed labels.

Determination of mineral and trace elements in foodstuffs and diets from the Amazon region

The Amazon region is characterized by the availability of fruits rich in pro-vitamin A and fishes with high protein content. However, there is little information about the nutritional potentiality of these foodstuffs mainly concerning the content of fibre, phytate and minerals in the fruits and vegetables. The purpose of the present study was the establishment of the chemical composition of these foodstuffs by using the instrumental neutron activation analysis. About 25-30 different species of regional fruits and vegetables (only the edible part of these materials) were analyzed. This work is being carried out with the cooperation of Dr. Lucia K. Yuyama from INPA (Instituto de Pesquisas da Amazônia). Also different pre-school diets from this region were analyzed during this period.

Assessment of the Content of Mercury, Methylmercury and Other Elements of interest in Fish, Hair and Diets of pre-school children of the Amazon region

The Amazon region suffers mercury (Hg) impacts as a direct result of both natural and anthropogenic processes. Jaú National Park (PNJ) is the only National Park in Brazil that protects an entire black water basin (Jaú River), flood land and tropical reserve. These conditions favor Hg methylation in the aquatic biota. Preliminary studies of pre-school diets from PNJ communities have shown that these diets have a worrisomely high Hg content. The present study assessed total Hg content, micronutrients (Ca, Fe, K, Na, Se and Zn) and macronutrients (proteins, lipids, ash, energy, carbohydrate) in pre-school diets in the PNJ and surrounding communities. Furthermore, total and MeHg levels were also determined in hair samples of these children as well as those living in several neighborhoods of the city of Manaus. Included in this determination were the fish most consumed by these populations. From these results it was possible to evaluate the nutritional content of the diets and the exposure of the children to Hg and MeHg.

Study on the Element Migration from Plastic Packaging to food using Radiometric Method in the last few years, problems related to food contamination by substances or elements that can be a risk to human health have been concerned, not only to government authorities, but also to

general population as well. Within this context, plastic packagings can constitute a source of food contamination since plastic manufacturing processes involve the use of catalysts and different types of additives that may contain toxic elements. In this project a radiometric method was developed for element migration determination from plastic food packaging to food simulating or to the food itself, according to National Health Surveillance Agency (ANVISA) regulations. Element migration was determined for plastic packaging used for soft drinks, drinking water, milk, dairy products, juices and fatty foods and the results indicated the migration of Cd, Co, Cr and Sb present in these plastics. In some packagings, migrations of only some these elements were observed and in these cases the detection limits were determined. Results obtained indicated the potentiality of using radiometric method in the elements migration evaluation from plastic packages to their contents. Among the main advantages of radiometric method for element migration evaluation are its simplicity, no need of blank analysis and the possibility to obtain element migration to the food itself.

Correlation studies of trace element concentrations in human tissues and the Brazilian population health

Determinations of trace elements in human tissues are of great interest in the health area. 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 2005-2007 we focused in the analyses of blood serum of healthy elderly population and brain samples of normal and demented individuals of São Paulo Metropolitan region. Reliable protocol was established for serum sampling, reference population selection, control of possible sample contamination and quality control of the analytical results since recent studies have been concerned with reliability of the serum analysis results and their usability in clinical chemistry and medical investigations. To avoid contamination of sample the handling of the biological samples was performed inside a laminar flow hood (Fig1.). The evaluation of trace element levels in sera of an elderly population is of particular interest especially in regard to healthy longevity. The ageing is a period of life characterized by trace element imbalance due to many factors such as physiological alterations with lower absorption of nutrients, lower food intake and reduction of physical activities. On the other hand, presently

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the fastest growing population group in the world is that over 60 years old, however data on serum trace element analysis for this group are very scarce. Serum Ca, Fe, Rb, Se and Zn concentrations were obtained by INAA at the IPEN-CNEN/SP and serum Cu, K, Mg, Na and P, as well as, biochemical and hematological parameters were determined at the Central Laboratory Division, HC-FMUSP. Serum element concentration ranges obtained for healthy elderly from the "Successful Ageing" Program of FMUSP indicated that they are within reference ranges and, 32% of elderly presented elevated total cholesterol levels but 93% showed desired HDL and 59% of desired LDL concentrations. Cerebral tissues of an over 50 year old population of both genders provided from the Brain Bank of the Brazilian Aging Study Group (BBBABSG) were analyzed in order to elucidate about neurodegenerative diseases resulting from the gradual and progressive loss of neural cells which leads to the dysfunction of the nervous system. Trace elements may contribute to the pathogenesis of cognitive disease since they may increase oxidative stress and also can influence the homeostasis of other elements and accelerate the aggregation of amyloid beta peptides. Concentrations of Br, Fe K, Na, Rb, Se and Zn were determined in different compartments of brain. Significantly higher concentrations of some elements were found in the hippocampus of demented individuals than those presented by the normal group in the corresponding brain parts. Partnerships: IBt and FMUSP. Financial Resources Agencies: FAPESP and CNPq.



Figure 5. Laminar flow hood for handling human biological samples

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 contamination of Sepetiba Bay (Rio de Janeiro) using a multi-element approach was estimated from sixty bottom sediment samples. The elements Pb, Cd, Cu, Ni and Zn were determined. The results showed very strong contamination of zinc (up to 1000 $\mu\text{g g}^{-1}$). Cd and Zn presented values which may cause hazards to aquatic organisms. The contamination of Rio Grande tributary, Billings reservoir, in the Metropolitan region of São Paulo, by determining metal concentration and other elements of interest using three analytical techniques (INAA, AAS and ICP OES) were assessed in bottom and core sediment samples. The chosen chemical parameters for this characterization were Al, As, Ba, Cd, Cu, Cr, Fe, Pb, Mn, Hg, Ni, Se and Zn. From these assessments the most adequate technique was selected for the routine analysis of sediment samples for each element concentration determination. 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. 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. 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 studying pollution caused by anthropic discharges. Most of the experiments are based on collection and analysis of native organisms (passive biomonitoring), but this method has the disadvantage of dealing with many natural variations. In this work, the marine bivalve *Perna perna*, very abundant in the coast of the State of São Paulo, Brazil, was transplanted from a mussel farm (active biomonitoring) 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 ($1.0\mu\text{g.g}^{-1}$ for As and $0.10\mu\text{g.g}^{-1}$ for Se), in all sites of study, including the control site. For the potentially toxic elements Cd, Hg, Pb and Zn, the

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- Reference materials are important tools in the quality assurance of analytical results as they may be used in method validation, calibration of instruments and in the realization of the traceability of analytical results to stated references. This study is a contribution to metrology in chemistry in Brazil and describes the preparation of a mussel reference material using the *Perna perna* species. This mussel species is abundant in most part of the Brazilian seashore and is currently being used in biomonitoring studies in our laboratory. It also has economical importance due to its growing cultivation in aquaculture farms for human consumption. The preparation 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 are being performed, with emphasis on the assessment of the stability of the



Figure 6. Mussel sampling site and reference material bottles

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material, its homogeneity status and the layout for certified values assignment via an interlaboratorial program. 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.

- Lichens have long been considered one of the most valuable air pollution biomonitors. As such, they have been widely used to assess trace element atmospheric contaminants. In order to contribute to the understanding of the air pollution profiles of São Paulo Metropolitan Region (SPMR), *Canoparmelia texana* lichenized fungi samples collected in the sites of unpolluted areas of Atlantic Forest, SP and in the sites near automatic monitoring stations belonging to the Environmental Protection Agency of the State of São Paulo (CETESB) were analyzed by INAA. Element concentrations of As, Ba, Br, Ca, Cl, Co, Cr, Cs, Fe, K, La, Mn, Mo, Na, Rb, Sb, Sc, Se, U and Zn were determined in these analyses. Distribution maps of element concentrations found in lichen analyses were drawn and high concentrations of Ba obtained in lichens from São Caetano do Sul and surrounding regions that make up an industrial area called ABC Paulista Region. These high concentrations of Ba can be attributed to several industries and petrochemical complex located in this region. Br distribution in the SPMR indicates that its emission source can either be from vehicular or industrial origins. For the case of Co, lichens collected in sites located in the Eastern part of the SPMR presented high level of this element. The high concentration of Co in the São Miguel Paulista site can be associated to the emission of a metallurgical industry that produces about 600 tons of this element per year. As expected, the distribution maps of element concentrations showed the highest values around the petrochemical complex, metallurgical industry and in urban areas affected by vehicular emissions. Exploratory analyses revealed that the accumulation of toxic elements in *C. texana* may be of used in determining the human risk of cardiopulmonary mortality due to prolonged exposure to ambient levels of air pollution. Partnerships: IBT and FMUSP. Financial support agencies: FAPESP, CNPq and IAEA.

INAA: a link between past and present in retrieving of peoples' identities

The chemical analysis on archaeological ceramics samples can provide information about production 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. During 2005 and 2007 years, INAA method at IPEN-CNEN/SP was used on hundreds of archaeological ceramics specimens in sites from several states such as São Paulo, Pará, Sergipe, Mato Grosso do Sul and Amazonas in collaboration with various archaeologists. The ceramic powder samples are obtained by cleaning the ceramics' outer surface and by 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 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 $8.92 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ 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 weeks' time. 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. These objectives are: 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. These studies are made by means of data set using several multivariate statistical methods, such as Mahalanobis distance, cluster analysis,

principal components analysis, discriminant analysis, and other analysis like kernel density, Procrustes analysis, neural network to cite just a few of the statistical methods used. Figure 7 is an archaeological ceramic with details of lizards, reptilians including snakes, caymans that are common on Marajó Island. Combinations of human faces and reptilian imagery on large funerary vessels illustrate the close link between human and animal in Marajoara symbolism.

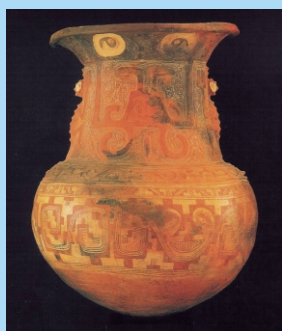


Figure 7. Funerary urn from Marajó Island (Musée Barbier-Mueller, Genève, Suisse)

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 several research projects in collaboration with different Universities (USP, UNICAMP, UFRGS) aiming trace elements determination in different kinds of rocks by INAA. The analytical procedure consists 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 10¹² 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 ⁶⁰Co. The gamma-ray spectra were

processed by using the VISPECT gamma-ray software which locates peak positions and calculates the energies and net areas. The LAN-IPEN has participated in the GeoPT - International Proficiency Test for Analytical and Geochemical Laboratories. The program GeoPT is designed to be part of the routine quality assurance scheme of analytical geochemistry laboratories. The organizers of the tests evaluated statistically all contributed data by using the Z-score criterion, which consider "satisfactory" the results falling between ± 2 . The elements Ba, Cs, Co, Hf, Rb, Fe, U, Th and the rare earths La, Ce, Nd, Sm, Eu, Tb, Yb and Lu were analyzed by INAA) and, as a general trend, Z-score values between ± 2 were obtained for most elements confirming the good quality of the analytical results.

Characterization of micronutrients and contaminants in fertilizers and agroindustrial by-products

Utilization of fertilizers, containing macro and micronutrients, is essential for obtaining productivity necessary to feed increasing world population. Brazilian fertilizer legislation considers the total concentration of micronutrients as being the guarantee of their presence in fertilizers. This fact allows the use industrial by-products as micronutrient source for fertilizers, which brings the risk of presence of high amounts of inorganic contaminants that may lead to soil contamination, affecting harvest and the quality of the agricultural products. Elements B, Cl, Co, Cu, Fe, Mn, Mo, Ni and Zn are considered as micronutrient for the plants and microorganisms. However, they are toxic at high concentration in soil solution. The elements As, Cd, Hg and Pb are considered toxic for the animals. Uptake of these elements by plants consumed directly or indirectly by humans can be one avenue of entry to food chain. Besides the presence inorganic constituents mentioned, the analytical method of INAA is a tool very adequate for the monitoring of no routine elements such as: Br, Eu, La, Sc, Sm, Th and U, which may have influence in the environmental impact over the time. This project is being conducted in partnership with CENA/USP and EMBRAPA. Steps carried out in 2005-2007:

- Multielemental characterization of thirty-one commercial fertilizers from different manufacturers: eighteen of the type F.T.E. (Fried traces elements) and thirteen Brazilian phosphate fertilizers.
- Multielemental characterization of five organic fertilizers: three sewage sludge, "biossolid", from different treatment plants and two composed of urban garbage.

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- Phytoavailability evaluation of micronutrients and contaminants in twelve commercial fertilizers of the type F.T.E. and in six brazilian phosphate fertilizers. To assess the microelemental phytoavailability, rice plants (*Oryza sativa* IAC 201 cultivar) were grown in pots containing 1 dm³ of soil (FIG. 8). The trial was performed at green house of Centro de Energia Nuclear na Agricultura-USP, Piracicaba, SP, Brazil. The Typic Quartzipsamment and Typic Oxisols were the soils used in this study.



Figure 8. Partial view of green house

Environmental radioprotection

An assessment of radon and thoron concentrations was carried out in the most visited caves of the natural state park in Brazil, 'Parque Estadual do Alto Ribeira' (PETAR), by using Makrofol E nuclear track detectors placed in twin chambers. Tour guides were evaluated for committed effective doses caused by radon and thoron inhalation, giving results between 0.2 and 4.0 mSv.y⁻¹ for radon and 0.005 and 0.017 mSv.y⁻¹ for thoron. Phosphogypsum, a fertilizer industry by-product, has been worldwide stockpiled, posing environmental concerns. Since this material contains natural radionuclides in significant concentrations, its use as a building material and in agriculture has radiological implications. In order to confirm the feasibility of the use of phosphogypsum, an experimental house was recently built, having one of its rooms entirely lined with this new material. A comprehensive radiological evaluation was performed, including measurement of the external gamma exposure and radon concentrations. The results show that the annual increment in the equivalent dose to a hypothetical inhabitant of the house will remain below ICRP 60 dose limit for public of 1 mSv.y⁻¹. In order to study the radiological impact of using phosphogypsum in agriculture, a conservative scenario was defined considering a long term exposure due to successive annual applications of phosphogypsum in agriculture for 10, 50 and 100 years. The doses evaluated due to the ingestion of food produced by using soils tilled with phosphogypsum were always below 0.5 mSv.y⁻¹, and of the same order of magnitude as those obtained by using Brazilian phosphate fertilizers. The Environmental Radiometric Laboratory at IPEN-CNEN/SP performs, on a regular basis, analysis of potassium-40, cesium-134 and cesium-137 concentrations in foodstuffs and food commodities imported and exported by Brazil. During the 2005-2007 period the radioactivity present in all analyzed products was within the international limits, allowing concluding that consumption of such goods offered no health risk derived from radiation.

Application of radionuclides in environmental studies

In Ubatuba coastal area, northernmost part of São Paulo Bight, time-series measurements of ²²²Rn activities showed a negative correlation between SGD rates and tidal stage. Large fluctuations of SGD were observed at sites situated only a few meters on the shore (from 0 cm d⁻¹ to 360 cm d⁻¹), as well as during few hours (from 0 cm d⁻¹ to 110 cm d⁻¹), strongly depending on tidal fluctuations. The average SGD flux estimated from continuous ²²²Rn measurements is 17 ± 10 cm d⁻¹. Integrated coastal SGD flux estimated for the Ubatuba coast using radium isotopes is about 7x10³ m³ d⁻¹ per km

of the coast. The fluvial fluxes of the radium isotopes and dissolved barium for Ubatuba embayments, was evaluated in 2007 and covered 12 small rivers that belong to the major surface drainage system of such coastal area. Activity concentrations of ²²⁶Ra in riverine waters discharging to Ubatuba embayments varied from 24 mBq 100L⁻¹ to 705 mBq 100L⁻¹ (in Perequê-Mirim River), while ²²⁸Ra concentrations ranged from 1119 mBq 100L⁻¹ to 3033 mBq 100L⁻¹. Natural radium isotopes have been used as tracers to study the movement of shelf water, which is believed to be a primary source of iron to the Southern Ocean (Fig.9) and ²³⁴Th as a tracer of particle flux in the upper ocean, since POC export via sinking particles is the primary mechanism of carbon sequestration in the Southern Ocean. In November 2007, the distribution of ²²³Ra and ²²⁴Ra in surface waters of Bransfield Straight and Drake Passage were determined.



Figure 9. Comandante Ferraz Brazilian Station at Antarctica (EACF) at King George Island

First results indicate a marked increase in ²²⁴Ra from Bransfield Straight close to Deception Island. These activities were in most cases lower than 0.1 dpm 100 L⁻¹ of seawater. In the framework of AMANDES project, the distributions of the four natural radium isotopes, U and Th have been analysed in a set of 40 seawater and particulate samples collected during two campaigns carried out in October 2007 and April 2008 on board of R/V ANTEA in the Amazon region. In October 2007 campaign, ²²⁴Ra activities up to 41 dpm 100 L⁻¹ were observed in Amazon river waters, while ²²³Ra activities ranged from 0.01 to 8 dpm 100 L⁻¹. The sedimentation rates and dating of a sediment core were determined by using ²¹⁰Pb method. The samples were collected at Lago Puruzinho, Amazônia Ocidental Brazil, in the same place where a previous Hg contamination was found. The age of sedimentary column was about 90 years and the sedimentation rate was estimated in 0.5 cm y⁻¹. The maximum concentration of Hg was found in 1982, the same period in which higher amounts of Hg were released in atmosphere.

Internal dosimetry

As a part of a continuous improvement of the monitoring programme for occupationally exposed workers at IPEN, a Web based system was developed to access the internal dosimetry database. The system was implemented using the Hypertext Preprocessor, PHP, and a PostgreSQL database. The database maintains information about worker identification, physical and chemical characteristics of the radionuclide, type of monitoring, measurement data and the dose.

External dosimetry

Aiming to improve the neutron dosimetry in Brazil a gamma-neutron mixed field dosimeter using the techniques of TLAD and SSNTD was developed. Commercially available materials were preferentially used. The dosimeter was projected to evaluate gamma, intermediate (Albedo) and fast neutron doses from $^{241}\text{Am-Be}$ sources.

High doses and accident dosimetry

In order to standardize a method for retrospective dose determination, the ESR response of tooth enamel irradiated with low energy X-rays and ^{60}Co gamma radiation was evaluated and compared with the ESR response of synthetic hidroxyapatite in the dose range between 0.2 - 10 Gy. Practical considerations of tooth selection, sample preparation and ESR spectrometer parameters settings were studied. The main dosimetric properties of Brazilian silicates of the jade family were studied for application in high dose dosimetry: tremolite, actinolite, rhodonite and diopside, using the thermoluminescent, thermally stimulated exo-emission and electronic paramagnetic resonance techniques. The results obtained showed the possibility of their use as irradiation detectors and for high dose dosimetry.

Metrology in diagnostic radiology

Computation systems were applied to simulate new ceramic compounds suitable to improve the radiodiagnostic room shielding. Different ceramic compounds were formulated theoretically using SiO_2 , Fe_2O_3 , Al_2O_3 , BaO , PbO e Ti_2O_3 compounds that were mixed in different concentrations and the shielding properties were simulated to choose the sample with the best shielding properties. Ceramic plates were produced and attenuation properties were evaluated and compared with the theoretical results.

A new X radiation system (160 kV) was characterized and the qualities from the IEC 61267 were established. As a part of its characterization, the spectrometry of this system was realized in order to verify the real kVp values. This system has been used to calibrate diagnostic radiology instruments. Since then, 490 instruments were tested. A special method to calibrate the CT ionization chambers was developed. A control and data acquisition program for the calibration of instruments for diagnostic radiology was established using the Labview software. The practical peak voltage quantity for diagnostic radiology clinical equipments was evaluated and a quality control program to be applied in dental diagnostic radiology systems was finalized. A methodology for correlations between doses and detectability in standard mammographic images was established. This methodology was applied in São Paulo State, with measurements performed in 50 health establishments using mammography units. Protective devices of five manufacturers were evaluated according to the international standards NBR/IEC 61331-1 and NBR/IEC 61331-3. Three different methodologies were applied for the determination of the attenuation equivalent. The discrepancy between the results obtained and the requirements of the standards show the need to adopt a compulsory certification process for protective devices, thus contributing for the increase of the radioprotection of users. An intercomparison program of radionuclide calibrators for nuclear medicine was established and performed for ^{67}Ga , ^{201}Tl and $^{99\text{m}}\text{Tc}$ sources at the Center for Radiopharmacy/IPEN.

Metrology in radiotherapy

The primary purpose of radiation therapy is to deliver sufficiently high dose to the tumor cells, maintaining at the same time the dose to surrounding normal tissues as low as possible. In this way, the Medical Physics Group at IPEN develops research projects in conjunction with some hospitals (A C Camargo, Clinicas, Albert Einstein, Syrian-Lebanese Hospitals and Pediatric Center Boldrini) in order to establish better procedures of clinical measurements for the new technologies in Radiotherapy, such as intensity modulated radiation therapy, radiosurgery and quality control on computerized treatment planning systems. As a result, new quality assurance programs will be implemented at these hospitals. The margins required to define a planning target volume (PTV) for adequate treatment of the mobile tumors such as prostate or those located in areas with less mobility as the ones in head and neck region, in the absence

of daily localization imaging based was studied. The impact caused by the PTV, in terms of dose, to the critical structures surrounding the PTV and its influence when inverse planning is used in the intensity-modulated radiation therapy (IMRT) was also evaluated. The results showed the importance of putting margins in the clinical target volume to assure an adequate treatment, and it was also showed that isocenter daily variation can cause an increase to the dose greater than the tolerance level to the critical organs. A DL-Alanine, Fe-II based gel dosimeter to be applied in Co-60 gamma-radiation fields in the dose range of radiation therapy procedures was developed. The dosimetric gel showed a linear behavior of the spectrophotometric response in the dose range between 0.5 and 40 Gy. The present DL-Alanine, Fe-II based dosimetric gel, presents an excellent potential for the determination of the dose fields in radiation therapy and its future application as a standard in the determination of these fields, in 3D, using the Magnetic Resonance Imaging technique (Fig. 10).

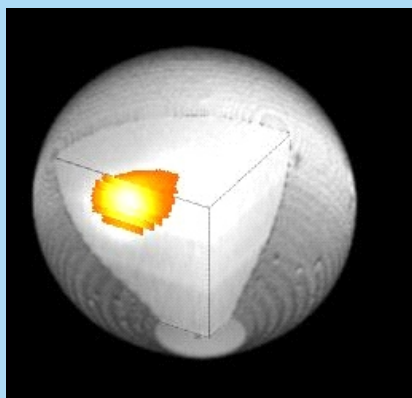


Figure 10. Head phantom image obtained with the magnetic resonance technique and irradiated using gamma knife system

Microdosimetry

Microdosimetric measurements were performed using a Wall-less Tissue Equivalent Proportional Counter - TEPC with spherical cavity. The fast neutron beam from a nuclear reactor was filtered using the heavily filtered fast neutron irradiation system (FNIS) to obtain a decrease of neutron radiation contamination by gamma rays. To obtain the neutron microdosimetric spectra, as the frequency distribution of lineal energy, the dose distribution of lineal energy with good precision, and another quantities as frequency-mean of lineal energy, dose- mean of lineal energy, absorbed dose, equivalent dose and average quality factor of fast neutron were evaluated. The obtained results were satisfactory, with the neutron microdosimetric spectra showing a gamma ray contamination under 5%, especially for dose distribution of lineal energy.

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; ²⁴¹Am source leakage tests; Determination of uranium, thorium and radium isotopes; Determination of ²¹⁰Pb, ²¹⁰Po and ²²²Rn; Determination of potassium-40, cesium-134 and cesium-137 concentrations in foodstuffs and food commodities imported and exported by Brazil.

Internal dosimetry

The whole-body and thyroid measurements are routinely performed on IPEN workers, visitors, trainees, and contract workers. During 2005-2007, the Laboratory carried out occupational monitoring of workers involved in radioactive waste management, radioisotope production, research, students and visitors. In this period 1.453 of thyroid and 1.496 whole body measurements were carried out. Routinely the analyses of biological samples from people occupationally exposed to radionuclides with risk of internal contamination are performed. The most frequently requested radionuclides, and the total number of analyses during the period 2005-2007 were: U-nat., 635; U (isotopes), 103; ²³²Th, 83; ³H, 87; γ emitters, 27; Actinides, 46. In addition to bioassay measurements, the Laboratory participated in national (Brazilian Intercomparison Programs) and in international intercomparison (Promotion du Controle de Qualite 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. During the period 2005-2007, a total of 2972 evaluations were performed for dose assessment, as part of the internal monitoring program. No worker received dose higher than the annual limit on intake.

Dosimetric pellets of CaSO₄:Dy

The Dosimetric Materials Laboratory has produced the CaSO₄:Dy crystals and dosimetric pellets during many years. The pellets are destined mainly to the personal, area and environmental monitoring. Furthermore, they are applied to high dose, accidental and medical dosimetry. During 2005 to 2007 about 15000 pellets were produced to fulfill the solicitations from many institutions.

External dosimetry

Around 1500 users are individually monitored monthly for external exposure, using trunk and extremity thermoluminescence dosimeters. There are 100 locations of environmental thermoluminescence dosimetry and 278 locations of workplace monitoring. The routine individual monitoring service for external exposure satisfies completely the regulatory norms of CNEN (Brazilian Nuclear Energy Commission). During 2005 to 2007 12,506 TLD evaluations for different applications were performed.

Calibration of Instruments

Many types of instruments were calibrated: several kinds of ionization chambers, pen dosimeters, survey meters (including superficial contamination detectors), alarm dosimeters, activimeters, clinical dosimeters, and others. The distribution, in terms of calibration levels was: 3% for radiotherapy, 8% for diagnostic radiology and 89% for radiation protection level. In this period, 6599 instruments were calibrated: 14% from IPEN, 20% from hospitals and clinics and 66% from industries. Besides this service, 475 samples including thermoluminescent dosimeters, alanine and other samples were irradiated, with beta, gamma and X radiations for dose-response curves.

Radioprotection Service

The main task of the Radioprotection Service of IPEN is to provide IPEN workers and general population with adequate protection against ionizing radiation. The Radioprotection Service implements adequate procedures and monitoring techniques according to the national and international regulations and standards.

The Radioprotection Service delivers professional radiation protection advice to departments which are involved in the use of radioactive material or ionizing radiation.

The group helps radiation employers to comply on their statutory obligations by providing Radiation Protection Advisers who may be integrated into existing project teams to ensure that all work, whether it be small movements of radioactive materials or large complex engineering operations, is carried out within a robust system of radiological protection.

The services include advice on compliance on statutory requirements for work with ionizing radiations, and they include:

- Preparation of local rules;
- Designation of radiological areas;
- Control and accounting of radiological material;
- Restriction of exposure;
- Dosimetry (internal and external);
- Occupational and environmental radiation and contamination monitoring;
- Dose assessment;
- Contingency planning and radiological risk assessment;
- Statutory training in all areas of radiological protection.

When required, the Radioprotection Service can provide the following services: assistance in the preparation of radiological protection aspects of safety documents; review of radiological protection aspects of safety documents; advice and assistance on radiological aspects of categorization of plant and plant 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; emergency and incident support and investigation (including advice to plant operations staff and site emergency services outside normal working hours); and response to national emergency response.

The Radiation Protection Advisers can provide a comprehensive support to all operations and facilities; as commissioning, and modifications to decontamination and decommissioning. The Radioprotection Service is available to the customers 24 hours a day, 365 days a year.

During 2005-2007, the IPEN/ Radioprotection Service cooperation in the field of emergency situations in Brazil has been carried out according to the expected work program.

Concerning the program for the improvement of infrastructures at IPEN, the Radioprotection Service is managing the activities of radiological survey of access areas under the direction and instructions of Radioprotection Service staff.

Finally, the Radioprotection Service is updating in a continuous way its procedures in order to fulfill the new legal requirements derived from the regulations.

Nuclear and radiological emergency response

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, state and local agencies to monitor, contain, and clean up the release of radioactive material while protecting people and the environment from harmful exposure to radiation. All the thirty-five notifications of reportable incidents occurred during the period 2005-2007 were cleared up.

Other important points to consider were the investments in infrastructure, as new facilities and radiation monitors for the emergency response, and the review of the Radiological Emergency Plan of IPEN.



Figure 1. Radiological emergency response

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 service has been established to satisfy: the training requirements for IPEN workers for various levels; to emergency response personnel, such as 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, than those who have a low potential exposure as a result of occasional activities in areas where exposure may occur. The training of the principles of radiation protection are based on the recommendations of the CNEN (National Commission in Nuclear Energy) 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.

Training in radiation protection is provided by classroom based training, and on the job training, and the courses provide the theoretical background necessary for the radiological protection requirements for ionizing radiations.

In the last three years, the courses were offered periodically. Upon completion of the course, an examination will be given to authenticate completion of the program requirements. A Certificate of Achievement was presented to those who have successfully completed the course, and a permanent record of training completion will be on file in the Radiation Protection 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, and the Federal regulations and IPEN procedures. After this basic course all workers are trained in this specific practice in each work area.

Occupational epidemiology

Epidemiology is the study of the distribution and determinants of disease in human populations. Occupational epidemiology involves investigating the frequency of occurrence and causal factors for health effects that have non occupational as well as potential occupational etiologies. The main evidence for the presence or absence of various health outcomes is provided by epidemiological investigations. It is necessary, however, to review carefully and understand the methodological issues and limitations of them in order to properly interpret the results. As background of the evaluation and as starting point, the United Nations Scientific Committee on the Effects of Atomic Radiation (UNSCEAR), presented these studies.

The main objectives of the research group, started in 2006, 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 (ex., study designs), in particular the radiation epidemiology;
- To determine the possible health consequences of workplace exposures (exposure standard setting) and to recommend remedial efforts when indicated.

Environmental risk assessment

Evaluation of the contamination risk caused by lightning rods disposed at uncontrolled garbage dumps

The Radioactive Management Laboratory (LRR) is conducting experiments to evaluate the contamination risk caused by lightning rods disposed at uncontrolled garbage dumps, using lysimeters as shown in figure 1, filled with waste and radioactive sources in order to evaluate the risk of contamination caused by radioactive lightning rods. The generated leachate is periodically monitored and analyzed to determine its characteristics as pH, Eh, solid content. Microbial growth is also being evaluated using a direct method to count the number of cells in a culture. The equivalent dose to members of the public was calculated considering the ingestion of drinking water, the most probable mode of exposure. The final result was about 145 times below the effective dose limit of $1 \text{ mSv} \cdot \text{year}^{-1}$ for members of the public, established by the International Commission on Radiological Protection (ICRP), demonstrating that the risk caused by lightning rods disposed at uncontrolled garbage dump is low. Microbial growth may be affected by the radioactive element (Am-241), however more assays have been conducted to elucidate this behavior.

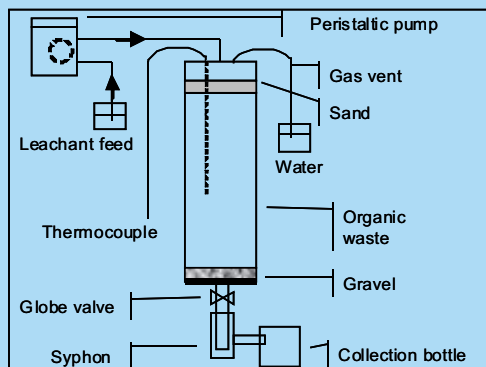


Figure 1. Sketch of the experimental design

A model for determination of screening level for radioactive elements in soil

At the present, decision about clean-up of Brazilian sites contaminated with radioactive isotopes is addressed on a case-by-case basis, since there is no general guidance or recommendation to support actions in early phases of the problem identification. For chemicals, CETESB - the governmental organization responsible for preventing and controlling environmental pollution in São Paulo State - established background values, prevention and intervention, as the first step to implement a remediation actions based on human health risk assessment.

The aim of this study was to develop a methodology for the establishment of target values for radioactive soil contamination, as far as possible consistent and compatible with the approach adopted by CETESB for sites contaminated with chemicals. The following steps have been addressed in this study: conceptual scenario and model development; codification of the equations in an electronic spreadsheet; selection of proper range and statistical distribution of the input values; derivation of the intervention levels for selected radionuclides using Monte Carlo methods. The mathematical model developed was mainly based on the equations used by the U.S. Environmental Protection Agency (EPA) and by the National Council on Radiation Protection and Measurements (NCRP) for soil screening purposes.

Intervention and prevention values are presented for an adult and for a 10 years old child, considering three exposure scenarios: agricultural, residential and industrial; the following radionuclides were considered: ^3H , ^{14}C , ^{32}P , ^{35}S , ^{45}Ca , ^{51}Cr , ^{90}Sr , ^{125}I , ^{131}I , ^{134}Cs , ^{137}Cs , ^{210}Pb , ^{226}Ra , ^{228}Ra , ^{232}Th , ^{238}U , ^{239}Pu and ^{241}Am . Quality reference values were determined for ^{40}K , ^{137}Cs , ^{210}Pb , ^{226}Ra , ^{228}Ra , ^{228}Th , Th-nat e U-nat. Results obtained in this study showed a good agreement with those reported by NCRP, considering that the equations and the input data used in both models are not the same ones.

Radioactive waste characterization, treatment and disposal

Characterization of ion exchange resins and activated charcoal

The Radioactive waste characterization program includes radioanalysis of wastes generated at IPEN and, in some cases, a proposition for their treatment. Our program follows the procedures outlined in CNEN-NN 6.09, about intermediate and low level waste acceptance criteria and some procedures are currently being implemented. The main subjects under development are characterization of ion exchange resins and activated charcoal generated at the research reactor water treatment system. The radiochemical characterization of the ion-exchange resins and activated charcoal wastes revealed the presence of significant amounts of ^{60}Co and ^{137}Cs , out of a list of 20 fission and activation products. The activities of the remaining radionuclides were below detection limits. Forty nine samples were taken from the 21 waste drums and analyzed by gamma spectrometry. As many as five samples were taken from some drums because of the stratification of the waste.

Radioactive Waste Management

Optimization of the radioactive waste storage

The Radioactive Waste Management Laboratory started a project to apply the concepts of clearance levels and exemption limits to optimize the radioactive waste storage capacity, taking into account that a fraction of these 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. This study was carried out by determining the doses and costs related to two main options: either to maintain the present situation or to open the packages and segregate the wastes that may be subject to clearance, using the national and three international clearance levels. These doses and costs have been evaluated by using the technique to aid decision making, known as cost-benefit analysis. The results pointed out that to maintain the present situation is not the better option, even though some parameters of each scenario may vary.

Artificial neural network applied to isotopic characterization of radioactive waste drum

In this study, the Monte Carlo method and the artificial neural network approach have been applied for waste isotopic characterization in a 200 L drum. A system with 10 detectors and 5 cross sectioned drum layers was designed, with the detectors positioned parallel to the vertical axis of the drum. Situations where the drum layers are filled with a Co-60 source are created and the responses in each of the detectors are simulated using the MCNP-4C computer code. The Co-60 source can assume 10 relative intensities between 0 and 74 and the detector responses are combined to generate a data file, containing all the possible situations in which the sources can occupy the layers of the drum. The data file is separated in input and output files and then used for training a neural network, where the inputs are the detector responses and the outputs are the position and relative intensity of the sources in the radioactive waste drum. The results of this study show that the neural network, in the training condition, is able to evaluate satisfactorily the activity of the radioactive waste contained in the drum. Nowadays, other radionuclides are under investigation for this methodology.

Treatment and disposal of disused sealed sources

The R&D work undertaken at the LRR in connection with the management of lightning rods, sealed sources and smoke detectors is divided in designing the facilities to handle

these sources; developing decontamination process; and developing the concept of a deep, fully-dedicated repository to dispose the sealed sources.

During the reported period, ancillary equipment were designed and fabricated for the hot cell designed to handle the sealed sources (Figure 2), under construction at the LRR. These include a mobile shielded transport overpack for the disposal containers; a hydraulic press stopper fastener to tightly close the disposal container; a workbench capable of holding the source's original shielding during the dismantling work.

For the lightning rods, an investigation on the decontamination process was started aiming at recycling of the metal scrap originated from their disassembling. Figure 2 shows the apparatus for monitoring the scrap.

The contribution of the LRR in final disposal of sealed sources is the development of a concept of repository that is technically and economically feasible for developing countries facing the problem of securing large inventories of disused sealed sources. An investigation work was started in this period aiming at the estimation of the durability of cementitious material under the deep borehole repository environment. This research is partially founded by AEA under a research contract that is part of the IAEA's Coordinated Research Project "Behavior of cementitious Materials in Multipurpose Packaging for Transportation, Long Term Storage and Disposal".



Figure 2. Apparatus for monitoring lightning rod plates

Use of *Saccharomyces cerevisiae* in radioactive waste treatment

Waste management plays an important role in reducing the volume of radioactive waste streams, minimizing the cost of the final disposal and the impact on the environment. In this context, new research should focus on the development of simpler and cheaper techniques which may improve the waste processing. The use of biomass in processes concerned with the removal of heavy metals

and radionuclides offers significant potential in the treatment of waste-liquid streams. The aim of this work is to study the potential of the *S. cerevisiae*, immobilized in bentonite, in removing Americium-241 from radioactive liquid streams occurring at the Radioactive Waste Laboratory of Nuclear and Energy Research Institute, IPEN - CNEN/SP. Parameters such as pH, biomass concentration and equilibrium time were evaluated. Preliminary results indicated that this technique may be an effective technique for the treatment of radioactive waste liquid contaminated with Am-241.

Degradation of radioactive organic waste using consortium and bacteria

The Radioactive Waste Management Laboratory is responsible for the treatment and storage of many radioactive waste streams, generated by the own Institute and other external radioisotopes users. The Laboratory receives liquid wastes, and sometimes they are constituted by a mixture of aqueous and organic phases. As they should not be directly immobilized in cement and also the small volume stored which does not justify the investment in an evaporator, the Laboratory is searching for a cheaper alternative and simpler methods of treatment. The aim of this work is to develop a technique to degrade organic compounds by using consortium and bacteria. Nowadays, liquid wastes were partially analyzed, and preliminary results revealed that the main organic compound is the tributyl phosphate; and for volatile organic compound it is the ethanol. The next step will be to test the degradation potential of the microorganisms in contact with the wastes.

Products and services

The Radioactive Waste Management Laboratory is responsible for reception, treatment and temporary storage of the radioactive wastes generated at IPEN, as well as those generated at many other radioactive facilities all over the country. The main features of the Laboratory include basically units for: waste reception and segregation; decontamination of small pieces; liquid waste immobilization and conditioning; in-drum compaction of compressible solids; spent sealed sources and lightning rods disassembling; primary and final waste characterization; storage of untreated and treated wastes.

Safeguards Service of the IPEN

The Safeguards Service - SS at IPEN acts in collaboration with several operators from different Materials Balances Areas (MBAs/IPEN).

It makes a annual planning schedule and pre-inventory procedures to accomplish the Physical Inventory Taking (PIT), check the Design Information Questionnaire (DIQ) and carry out 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) that complies with Quadripartite Safeguards Agreement (INFCIRC 435) in 2005, 2006 and 2007. This agreement was signed between Argentina, Brazil, ABACC and IAEA. This agreement adopted the Common System of Accounting for and Control of Nuclear Materials SCCC for Brazil/Argentina.

In 2007 the Safeguard Service carried out and followed the transfer fuel elements from IPEN Nuclear Research Reactor Facility to the United States, with CNEN and ABACC inspectors.

During the development of the Brazilian Nuclear Fuel Cycle in the 80's, IPEN acquired from INB (Indústrias Nucleares Brasileiras) Uranium as U_3O_8 , to be used as a raw material in the Nuclear Fuel Cycle Facilities.

The refund of this material was conducted and managed by the Technical Advisory (SAT) and the Safeguards Service (SS) of IPEN. They managed and executed the strategic planning, the coordination and supervision of the tasks.

This material was re-packed and distributed in several chemical compounds. They were standardized to attend the international standards.

During the tasks several measurements were performed such as air monitoring, dose rate etc and none of them presented values above the standards limits adopted by CNEN. A certain amount of it was sent to Canada to be processed into UF_6 and the remainder is expecting authorization from CNEN/RJ to be sent to Caetite Facilities in Bahia, Brazil.



Figure 1. Re-package of nuclear material

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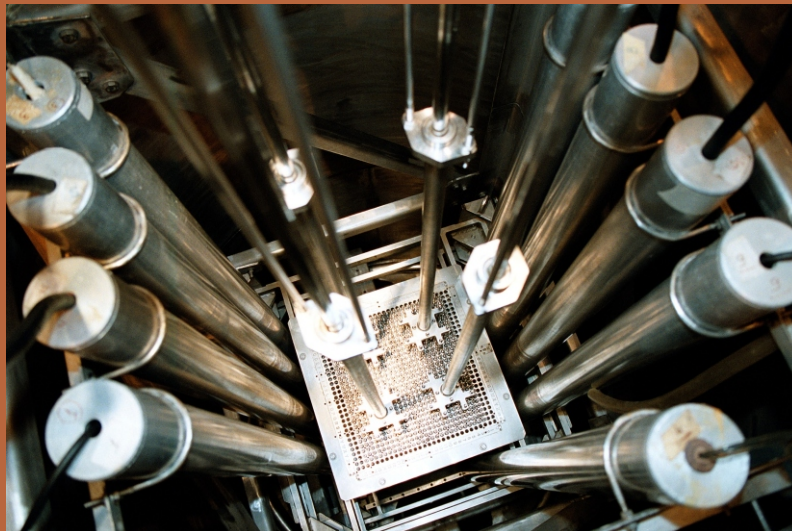
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Honor Mention and Awards

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“Citotoxicidade de ligas metálicas utilizadas como biomateriais” - LATINCORR 2006.

Nuclear Reactors and Fuel Cycle



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Introduction

The years between 2005 and 2007 will be known as historical ones due to the countless discussions and publications on the new start of the use of nuclear energy worldwide. In Brazil, itself, the nuclear community was granted with governmental decisions that indicate the retaking of the necessary investments and the adoption of a long term nuclear program.

As to the nuclear reactors, the construction of Angra 3 and at least four more nuclear power plants until 2030 are predicted. There is also a high chance that a new research reactor will be built. Concerning the fuel cycle the goal is to become self-sufficient in the production of nuclear fuel. The nuclear reactors and fuel cycle areas at IPEN-CNEN-SP are fully prepared to attend the challenges that will come with the retaking of the Brazilian nuclear program.

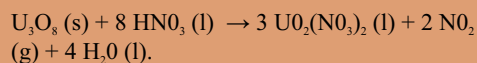
The Nuclear Engineering Center (CEN) has shown during all these years high competence in the field of nuclear reactors providing specialized engineering services, research and development activities and educational competence for education activities. The specialized engineering services are mainly provided for the nuclear power plants Angra 1 and Angra 2. The research activities are mainly focused on the power and research reactors in operation nowadays. However, research is also performed on advanced and innovative nuclear reactors in order to prepare a competent staff for the challenges of the Brazilian nuclear program to be launched.

The Research Reactor Center (CRPq) installed in 2007 a new heat exchanger that will enable the IEA-R1 research reactor to operate at higher power, increasing the production capacity of radioisotopes, sample irradiation, tests and new experiments that could be needed for the operations and will represent an important tool for the measurement of parameters in the field of reactor physics. Both reactors will be playing a decisive role at the formation and training of the human resources needed in the nuclear area.

At the Nuclear fuel Center (CCN), the design and construction of a new factory of MTR fuel elements are being developed. This new plant will allow the production increase of research reactor fuels and benefit any governmental decision relative to building a new research reactor. The development of CCN concerning the production procedures of $\text{UO}_2\text{-Gd}_2\text{O}_3$ nuclear fuel shows that the Center competency could also be expanded to respond to new challenges from the Brazilian Nuclear program.

Uranium recovery from slags of metallic uranium

The Nuclear Fuel Center of IPEN-CNEN/SP has already concluded its development program to fabricate uranium silicide fuel type (U_3Si_2 -Al) for nuclear research reactors as IEA-R1. The process of fuel fabrication starts at LEU UF_6 . The process to produce U_3Si_2 involves metallic uranium as an intermediate product, through magnesiothermic reduction which produces slags containing uranium. The recovery process consists on slag lixivium of calcined by-products from metallic uranium reduction. The results from researching this process confirmed that this method could be integrated in treatment and recovery routines of uranium. The chemical routes avoid to deal with metallic uranium since this material is instable, pyroforic and extremely reactive. On the other hand, U_3O_8 is a stable oxide with low chemical reactivity, and it justifies the slags calcination of metallic uranium reduction by-products. This calcination occurs in oxidizing atmosphere and transforms the metallic uranium into U_3O_8 . Some experiments have been carried out using different nitric molar concentrations, acid excess contents and temperature control of the lixivium process. The nitric lixivium main chemical reaction for calcined metallic uranium slags is represented by the equation:



It was adopted the following process parameters:

- temperature and time: calcination of slag of metallic uranium at 600°C during 3h;
- granulometric control: sieving and segmentation of calcined slag between at 100-200 mesh;
- concentration: lixivium adjustment of HNO_3 at 1 molar; HNO_3 excess (120%);
- lixivium temperature: 40 - 50°C;
- agitation: 300 rpm, turbine stem type (45 degrees inclination).

As results, we have: lixivium occurrence minimum time: 9 hours; fluoride concentration in lixivium: 0,002g/L. Lixivium made at low temperatures and low nitric concentrations reduce both the solubility of magnesium and calcium fluoride and the corrosion effect caused by fluoride ions, ensuring a stable and secure lixivium from operational point of view. The nitric dissolution of metallic uranium sludges produces uranyl nitrate solution, which is the utilized compound to feed the uranium purification system by solvent extraction method, with diluted n-tributylphosphate. The purified uranium product is precipitated as ammonium diuranate (ADU) at 60°C, by injecting gaseous ammonium diluted with air. Aiming at returning the recovered product to the fuel fabrication cycle

with nuclear quality level, the purified ADU has to be converted to uranium tetrafluoride (UF_4) by U_3O_8 route. The yield of 94%, which is obtained by this process, proves the viability of this slag recovery of uranium magnesiothermic reduction. Figure 1 displays the sequence of recovery operation presently used in CCN/Chemical Laboratories for this recovery.

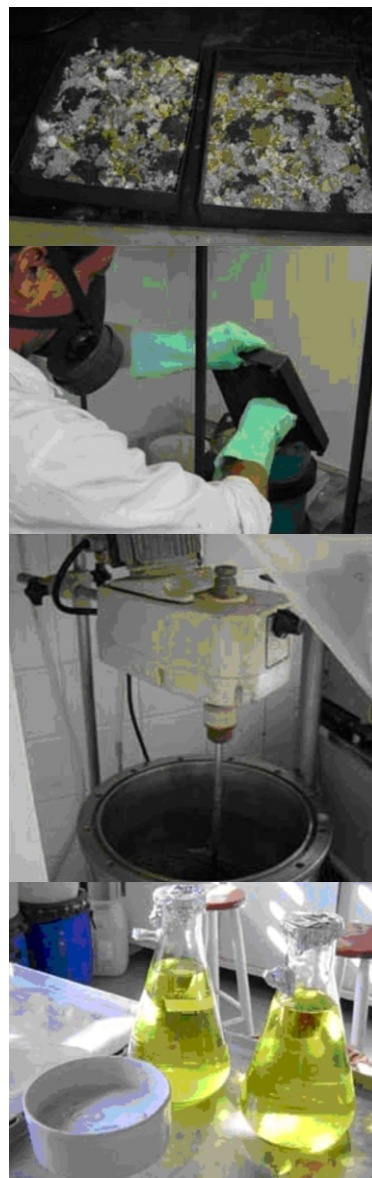


Figure 1. Development of uranium recovery

Uranium-Molybdenum technology

Since the start of the RERTR (Reduced Enrichment of Research and Test Reactors) program, IPEN-CNEN/SP/CNEN-São Paulo is working to fulfill its requirements. The conversion of high to low enriched fuels requested the development of the U_3Si_2 plate fabrication technology, but it is known that silicides, even being the current dispersion fuel of IPEN-CNEN/SP IEA-R1 reactor, are beyond the expected density goal of 9 gU/cm³, necessary to convert HEU high production reactors in the world to LEU. U_3Si_2 is inadequate for HEU reactors for its technological and compatibility concerned to rolling and under irradiation behavior. To avoid these problems, alloys of UMo, in the range of additions from 5 to 10% weight Mo, are the most studied and employed in the fabrication of the plates. Its gamma allotropic form is the most suitable for the use as fuel for research reactors. With the technical cooperation project Brazil / AIEA BRA 4053, "Development of Alternative High Density Fuel Based on LEU UMo Alloy" and the Ph.D. thesis "Development of a High Density Fuel Based on the UMo Alloy with High Compatibility in High Temperatures", IPEN-CNEN/SP is in the way to produce its first UMo fuel plates for seeking of its own fabrication technology also in this kind of nuclear fuel material. In addition, there are at our fuel center two projects with large budgets approved to upgrade our facility for fuel fabrication, mainly to produce U_3Si_2 but also to produce and research on UMo fabrication line. Two new MSc projects are underway to help the comprehension on UMo-Al chemical interaction and compatibility studies. The first step studied in the development of the technology of UMo alloys and plates fabrication was their synthesis and characterization. Here, factors like the condition of melting (arc or induction), hardness, grain size, gamma and alpha content, were determined by the usual techniques of characterization. The right thermal treatment conditions were also studied, enabling us to state and to reach the correct grain dimensions. A second and important step was the compatibility studies, where the interaction between oxygen, aluminum and UMo alloys were analyzed by means of the thermogravimetric and differential thermal analysis techniques. Both studies enabled us to state conditions of stability in high temperatures, as a function of the Mo addition. The third step was the production of UMo powders, and the route considered here was hydration-dehydration (HDH), instead of the widely utilized atomization and mechanization, for

experimental reasons. The behavior of the alloys was studied under several conditions of thermal treatments, mainly times, temperatures and hydrogen pressure, by means of the thermogravimetric technique. To produce higher amounts of powder, in the order of 30 to 60g of UMo, the hydration-dehydration facility in the Magnetic Material Laboratory of IPEN-CNEN/SP was utilized, with good results mainly in the percentages between 5 to 7% Mo. The fourth step will be developed soon, since we have approved a project which will enable us to study and prepare several dispersion fuels, including UMo, to the determination of its behavior under rolling. Also, productions of large amounts of UMo powder of about 500g, are comprised by this project, which will be necessary to our production facility. In the next figures, there are some of the results of the first three steps. Figure 2 shows a typical curve of UMo behavior under hydrogen atmosphere, its powder correlated micrograph.

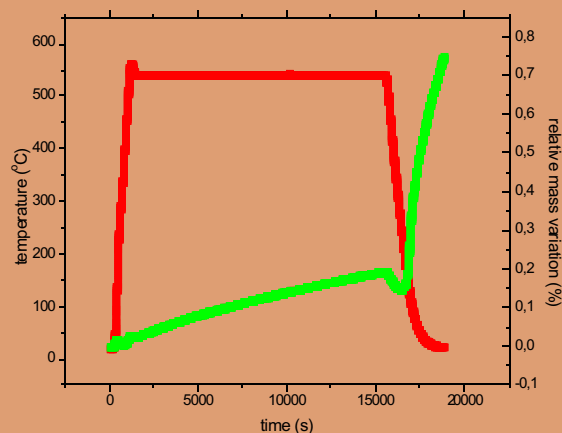


Figure 2. Typical mass variation curve showing the H₂ acquisition

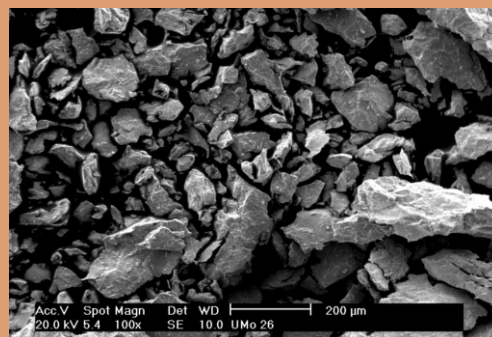


Figure 2.1. Produced powder structure

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 (Comissão Nacional de Energia Nuclear - National Commission of Nuclear Energy, which is a federal institution of Science and Technology Ministry in Brazil) 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. There has been a significant increase in the demand of radioisotopes over the past years. Between 2002 and 2004 the increase was about 30%. The distributed production supplied Brazilian hospitals and clinics. In 2006, it was attended more than 3 million patients, with an increase of about 10% relative to the previous year. To face this scenario, IPEN-CNEN/SP has been increasing continuously its production of radiopharmaceutical medicines to come along with the expanding demand imposed by Brazilian welfare. One of the most important project aims at own production of Mo-99 in order to provide cheaper Tc-99 generators than the ones produced from imported raw materials. So, this medicine will be accessible to a bigger amount of patients. The IPEN-CNEN/SP's research reactor IEA-R1 is the facility responsible to produce the radioisotopes for use in medicine. IPEN-CNEN/SP reactor's crew, for many years, has been working on upgrading mainly to increase its power from 2 to 5 MW and its operational time from 64 to 120 hours by week. The reactor control board and ventilation system were upgraded and additional safety items were incorporated. Besides, CNEN is planning to build a new facility in order to expand and spread the use of radiopharmaceutical medicines all over the country. Due to uncertainty in the international nuclear fuel market and in order to assure a continuous provision of fuel for its reactor, IPEN-CNEN/SP decided to fabricate its own fuel. The history of the fuel element fabrication technology in IPEN-CNEN/SP is very old. The work started in 1960 aiming at fabrication of the fuel for the ARGONAUTA research reactor. Between 1964 and 1965 the fuel elements were manufactured with 20% enriched U_3O_8 powder provided by IAEA in the program Atoms for Peace. In spite of the low demand of the ARGONAUTA fuel (very low power), a seed was planted and germinated 20 years later, in the decade of 80, and matured definitively in the decade of 90, when IPEN-CNEN/SP dominated the fabrication technology and began the production of the IEA-R1 fuel. The relative high power (2 MW) demanded a significant technological progress in the fabrication techniques. Starting from 1980, IPEN-CNEN/SP intensified its efforts to develop the

fabrication technology of dispersion fuel element, aiming at improving the manufacturing technology for more advanced fuels. At that time, IPEN-CNEN/SP could not buy fuel elements from the international market to supply its research reactor, since there was a political growing difficulty to get fuel elements in international market. This upheaval acted as an initial impelling force for IPEN-CNEN/SP to push ahead its program for fuel element fabrication. The technology previously developed in the 60's was updated since 1985, based on the recent technological advances in this area. Between 1985 and 1988, IPEN-CNEN/SP worked in assembling a small fuel fabrication facility as a laboratory level, with capacity to produce 6 fuel elements by year. This was enough to supply the IEA-R1 reactor operating at 2 MW and 40 hours a week. On August, 31st, 1988, as part of the commemorations of their 32o anniversary, IPEN-CNEN/SP provided the IEA-R1 reactor with its first fuel element fabricated in Brazil, only fifteen days before the full exhaustion of the reactor fuel feeding. The fissile material at this period was the same U_3O_8 powder previously used for the production of the ARGONAUTA fuel. There was a reserve of about 30kg of this material. From 1988, after the production of the first fuel element, IPEN-CNEN/SP began a continuous production of fuel element, which continues until nowadays. After the production of 26 fuel elements, the enriched U_3O_8 powder finished in 1996. So, in 1994 IPEN-CNEN/SP started developing the processes for UF_6 conversion to U_3O_8 and for recovering the uranium scraps generated in fuel plate fabrication. In 1996, IPEN-CNEN/SP did the conversion of about 20kg of imported enriched UF_6 . IPEN-CNEN/SP was then prepared for the routine production of fuel elements starting from UF_6 as raw material. In 1997, IPEN-CNEN/SP raised the fuel production capacity from 6 annual fuel elements to 10, which was the maximum considering the available facilities. In order to increase the radioisotope production of IPEN-CNEN/SP, the IEA-R1 reactor power capacity was increased from 2 MW to 5 MW. In 1997, the development of a new higher uranium density fuel started in order to attend the reactivity needs for reactor continuous operation. It has a compact core for better irradiation flux and also to dispose lower number of irradiated fuel elements to be stored at the spent fuel pool. The new fuel was based on the U_3Si_2 -Al dispersion with uranium loading of 3.0 gU/cm^3 . In 1998, the fuel plate fabrication technology of the new silicide fuel was implanted. At that time, U_3Si_2 powder was imported from France. Between 1999 and 2000 sixteen silicide fuel elements were manufactured. Starting on 1998, the efforts to develop the U_3Si_2 powder production technology began, aiming at the nationalization of all the production process: LEU enriched UF_6 reduction to produce UF_4 ; its

reduction to metallic uranium; from uranium producing the intermetallic U_3Si_2 , with powder fabrication and, finally, arriving at the fuel plate fabrication and fuel element assembly. At this time, IPEN-CNEN/SP participated in an international cooperation through IAEA under the Technical Cooperation Project TC BRA/4/047.

In 1999 IPEN-CNEN/SP got the technology of UF_4 production using $SnCl_2$ as reducing agent. In the area of metallic uranium, IPEN-CNEN/SP had a valuable previous experience in producing up to 150kg of natural uranium ingots in 90's decade. Based on this experience, IPEN-CNEN/SP initiated efforts to scale down the size of the metallic uranium ingot, trying to produce pieces less than 3kg using 20% enriched material (LEU) as raw material for the U_3Si_2 production. In 2002, the process for producing metallic uranium was dominated, which allowed the development of the U_3Si_2 intermetallic. In 2004, IPEN-CNEN/SP produced the first lot of natural U_3Si_2 powder, manufactured with national technology, dominating then the "uranium silicide cycle". In 2006, IPEN-CNEN/SP consolidated the fabrication technology of the silicide fuel by manufacturing the first fuel element (figure 3), with fully national technology. This fuel element was put in the IEA-R1 reactor core on June 26, 2007. The nationalization of whole the production cycle of dispersion fuels for research reactors have been accomplished. After the mining and enrichment steps, IPEN-CNEN/SP became capable to execute all the other fabrication steps of dispersion fuel. The first U_3Si_2 fuel element manufactured with materials and technology totally national began to operate in the IEA-R1 nuclear reactor of IPEN-CNEN/SP. So, Brazil became totally independent in terms of materials and technology to supply fuel elements for their research reactors for producing radioisotopes. This was an important technological conquest, because it puts the country inside the international market, as part of a restricted group of commercial suppliers of this kind of fuel. Besides, the Al-dispersed fuel technology was developed also as a base for developing the fabrication technology of advanced dispersion based fuel for compact necessary for high performance nuclear power reactors for nuclear propulsion.



Figure 3. First U_3Si_2 fuel element fabricated at IPEN

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 elements 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 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 elements a year, representing the maximum production capacity in laboratorial scale for the available facilities at that time. That production capacity, reached its limit, which was enough to keep IEA-R1 reactor operating at 3-4 MW 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 for radioisotope production was cogitated to be constructed in the Northeast region of Brazil. This decision would be very significant in near future, since IEA-R1 reactor is already quite old (more than 50 years) and this reactor is practically the only one producer of radioisotope in the country. The new reactor (probably 20 MW) would consume about 30 yearly U_3Si_2 -Al fuel elements. Therefore, a planned demand of about 50 fuel elements a year was quite realistic at that time. Based on this forecast demand for fuel elements, IPEN-CNEN/SP began a project in 2001 in order to adapt the production facilities seeking on improving

the producing capacity. The first phase of that project is now concluded (figure 4). The fabrication steps regarding to the dispersion preparation, 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 under way. Once ready the whole project, all the production will be made through an integrated line in facilities with industrial character, which will operate according to international nuclear quality and safety standards, established by IAEA. Nowadays, this project is being completed. The new facility is planned to have nominal capacity for producing 30 fuel elements yearly. That will attend entirely the fuel element demand in a medium period. The producing capacity of the new facility could incremented to reach 80 fuel elements in a year, supplying so a new research reactor which has been planned to be constructed. The conclusion of this project is foreseen for 2008-2009.



Figure 4. Partial view - concluded part of the new CCN plant for fuel elements fabrication

Technological aspects concerning the production procedures of $\text{UO}_2\text{-Gd}_2\text{O}_3$ nuclear fuel

The activities regarding the $\text{UO}_2\text{-Gd}_2\text{O}_3$ fuel were continued. The mechanism that explains the sintering behavior of $\text{UO}_2\text{-Gd}_2\text{O}_3$ fuel prepared by the dry mechanical blending method was studied on early years and it was found that the bad sintering behavior is based on the occurrence of the Kirkendall effect. A significant difference in the interdiffusion coefficients of the gadolinium into UO_2 and of the uranium into Gd_2O_3 causes a misbalance in material transport during formation of the solid solution. As a consequence, the densification during sintering occurs simultaneously with the formation of pores in locations where Gd_2O_3 agglomerates were originally present. The diameters of these pores are proportional to the initial diameter of these agglomerates. The pores formed are stable, since they are formed at high temperature in an essentially closed pore structure. Given this situation, the elimination of these pores after their

formation is not possible in the subsequent sintering process. The pores remain in the sintered pellet and are the cause of the low densities observed. The work was continued in order to study possible adaptations in fabrication procedures for overlapping the sintering problem. The actions that could minimize or eliminate the problem would be based on changing certain fabrication procedures, as follows:

- Activity optimization of the UO_2 powder used in the preparation of $\text{UO}_2\text{-Gd}_2\text{O}_3$ mixed powder, which can be achieved by adjusting the conditions for AUC reduction;
- Sintering cycle optimization by adjusting the heating rate, temperature and duration of the isothermal sintering step;
- Adjustment in the homogenization procedure of the UO_2 and Gd_2O_3 powders aimed at obtaining a mixture presenting a high level of homogeneity.

Activity optimization of the UO_2 powder

The formation of pores due to solid solution formation began to occur at temperatures above 1000°C , during the second stage of sintering, and ended around 1350°C . One possibility for acting on this process would be to delay the sintering process. In other words, to act on the sintering kinetics in order to determine that it occurs at higher temperatures, after the formation of the solid solution and, consequently, after pore formation. The delay in the sintering process could be compensated in the isothermal stage of the sintering cycle. One way to achieve this objective is to reduce the activity of the UO_2 powder used to prepare the mixed powder. This possibility was tested using UO_2 powder derived from AUC with different areas of specific surface when preparing the $\text{UO}_2\text{-Gd}_2\text{O}_3$ mixed powder. Controlling the activity of UO_2 powder was possible through controlling the parameters for AUC reduction. There is an optimal specific surface which provides the UO_2 powder a reserve of activity in order to guarantee that sufficient sinterability exists for densification at high temperatures, after solid solution formation. This reserve of activity for sintering at high temperatures would promote the efficient elimination of the pores formed due to the Kirkendall effect and optimize densification. The best result was obtained using UO_2 powder with a specific surface of $4.5 \text{ m}^2/\text{g}$.

Sintering cycle optimization

Other possibility of acting on the sintering kinetics by delaying the sintering process in order to determine that it occurs after pore formation by the Kirkendall effect, would be to increase the heating rate of the sintering cycle. If the kinetics for solid solution formation, or for pore formation due to the Kirkendall effect, occurs faster than sintering kinetics, the effect of increasing the heating rate would be beneficial in terms of

residual porosity, since a larger fraction of pores formed could be eliminated in the sintering process subsequent to pore formation. A delay in the sintering process could be compensated in the isothermal stage of the sintering cycle. Figure 5 illustrates the sintering process under different heating rates. The positive effect of increasing the heating rate can be verified in the final sintered density. Elevation of the heating rate from 5°C/min to 30°C/min led to an increase in the final sintered density of almost 1.5 vol%. When the heating rate was very low (1°C/min), pore formation during the formation of the solid solution was very clearly observed and densification in the isothermal period was very low. In contrast, when the heating rate was superior to 10°C/min, the decrease in the shrinkage rate due to pore formation was smaller and densification at the onset of isothermal treatment was pronounced, resulting in a beneficial effect in eliminating porosity, which led to higher densities in sintered fuel pellets.

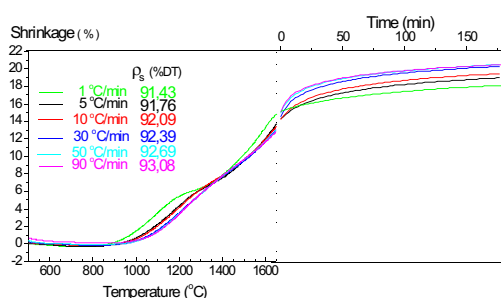


Figure 4. Effect of the heating rate on the sintering process of the $\text{UO}_2\text{-Gd}_2\text{O}_3$ fuel

Adjustment in the homogenization procedure

Previous experimental results demonstrated that homogeneity in the Gd_2O_3 distribution within the mixed powder exerted a decisive influence on the sintering process of $\text{UO}_2\text{-Gd}_2\text{O}_3$ fuel. In other words, the better the homogeneity in the mixed $\text{UO}_2\text{-Gd}_2\text{O}_3$ powder, the higher the final sintered density. This behavior is explained based on the Kirkendall effect mechanism. The development of an alternative method for UO_2 and Gd_2O_3 powder homogenization, which permits good homogeneity at a microscopic level, is another possible action that could improve the sintering behavior of $\text{UO}_2\text{-Gd}_2\text{O}_3$ fuel. The homogenization method should preserve the original morphology of the particles of the AUC powder, which gives good flowability to the

UO_2 produced and the desirable direct pressing procedure. The use of comilling, in spite of resulting in good sintered densities (microscopic level of homogeneity), is not technologically interesting, because this procedure destroys the original morphology of the UO_2 powder, which implies the incorporation of a granulation procedure. The coprecipitation process via AUC permitted the acquisition of good homogeneity, at a microscopic level, in the distribution of Gd_2O_3 in the $\text{UO}_2\text{-Gd}_2\text{O}_3$ mixed powder, which resulted in an adequate densification level during sintering. In fact, coprecipitation does not occur in the AUC case. Probably, simultaneous precipitation occurs and homogenization is achieved in the liquid phase. Although this method for Gd_2O_3 incorporation during the precipitation stage resulted in a homogeneity level that was adequate for obtaining sufficiently good sintered densities, this method has the disadvantage of contaminating the precipitation reactor, which would demand exclusive equipment. To avoid duplication of the precipitation installation, an alternative would be the incorporation of the Gd_2O_3 powder into the AUC suspension prior to filtration. Thus, homogenization would be achieved in a liquid media, which is much more efficient at allowing the desegregation of Gd_2O_3 powder and would permit its dispersion in individual particles among the AUC crystals. In this case, after AUC precipitation in the traditional reactor, the suspension would be pumped towards a homogenizing tank and then towards a second filter, different from that used in the process for producing the standard UO_2 fuel. In this case, the duplication of the filtration system and the installation of an additional tank for homogenization would only be required. The development of alternative techniques for homogenizing the powders, together with the use of Gd_2O_3 powder with special characteristics (low tendency to agglomerate), would probably allow the obtaining of $\text{UO}_2\text{-Gd}_2\text{O}_3$ fuel pellets with the minimum specified density. However, a specific work in that area should still be conducted.

Reactor physics benchmarks at the IPEN/MB-01 reactor

Since 2004, the reactor physics group of the Nuclear Engineering Center of IPEN-CNEN/SP 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:

- 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;
- Evaluate the data and quantify overall uncertainties through various types of sensitivity analysis;
- Compile the data into a standardized format;
- Perform calculations of each experiment with standard criticality safety codes;
- 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 located in the area of the Nuclear Engineering Center of

IPEN-CNEN/SP. 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 70 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 ANGRA-II to save storage space. Another very interesting experiment was a central void simulation with an 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 2007 was the measurement of several effective delayed neutron parameters without the need of any correction factor or data from other experiments. The experiment as approved to be included in the handbook of reactor physics benchmarks to be issued in March 2008. Also, in the same subject, IPEN-CNEN/SP and the Nuclear Center of Cadarache are working jointly in an international collaboration inside of the BRAZIL-FRANCE cooperation program. IPEN-CNEN/SP received a series of miniature fission chambers from Cadarache which will allow the measurement of several spectral indices in the IPEN/MB-01 Reactor. A mission from France visited IPEN-CNEN/SP in September 2007 to support the measurements at IPEN-CNEN/SP and to train the reactor physics staff on the utilization of the equipments.

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

The development of a reactivity meter at IPEN-CNEN/SP 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-CNEN/SP 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

A pos-doctoral student worked during 2006 and 2007 and developed a novel theoretical model to describe collisions among charged particles in an electric field. This model presents a novel form to treat coulomb interactions between charged particles. In this model the coulomb interaction is divided into two parts: interactions caused by the mean potential field generated by the electric field (electric field caused by the electrodes) and a short range interaction between two ions that takes into account the local effective potential. The short range interaction is used to correct the mean potential due to local changes when two charged particles are closed. The short range interaction is defined by a collision operator which is introduced in the Boltzmann equation. This modified Boltzmann equation allows a more realistic solution for the particle distribution in an ion field and is simple to be solved numerically. This model is now being adapted to study the space and energy distribution of the ions inside an IECF device. A master student is working since the beginning of 2007 to design an IECF device based on spherical electric grids. To provide subsidies for the design a parametric study is under way to verify the influence of the electric potential and the distances between the grids on the nuclear fusion rates. In this study a simple model that does not considers ion-ion collision is being used to calculate the ion space and energy distribution.

Numerical simulation and experimental validation for application in medical physics

The goal of this multidisciplinary research group is the development of new numerical dosimetry methodologies using Monte Carlo techniques for the application in medical physics area comprehending: radiotherapy, radioprotection and nuclear medicine. During the last three years the group has accomplished some important tasks to help medical physicists in clinical and instrumentation issues. In the period of 2005-2007 it was developed a project to completely characterize the electron beam of various energies produced in a specific linear accelerator used for patient treatment in radiotherapy. The construction of the energy spectra for each clinical energy beam enabled the simulation of a series of experiments in phantoms used for the system calibration. Monte Carlo simulation using voxel phantoms also allowed the estimation of dose distribution in patients under treatment. The results provided insights for the medical physicists about the effectiveness of the treatment proposed. Figure 5 shows an

example of an output of the methodology developed.

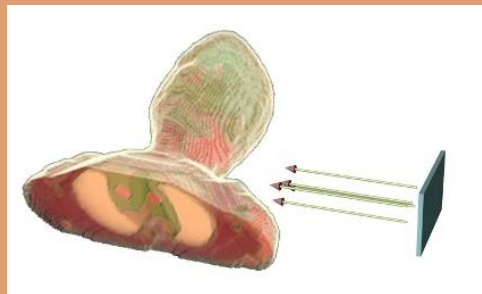


Figure 5. Head and neck irradiation with a typical clinical electron beam

Later, many efforts have been done to adapt the present methodology to be able to provide several dosimetric data for the implantation of new clinical modality: the total skin irradiation. The ultimate goal is to capacitate the radiotherapy service to offer an alternative treatment for patients with skin cancer.

Innovative and hybrid reactors

The renaissance 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 waste burner reactors. The activities under way are mainly related in development and qualification of calculation methodologies and fuel cycle simulations. We are participating 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 2005-2007 in innovative hybrids reactors are:

- Participation in the 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 radionuclides, 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.

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

With the financial support of the IAEA the Nuclear Engineering Center has designed an instrumented MTR fuel element, with the objective to measure the temperatures in lateral and central plates of the fuel element and compare them with the temperatures calculated with thermo hydraulic computer codes available in this Center. In this design was considered the installation of 8 thermocouples, 4 in lateral channel and 4 in the central channel. In the central channel there are 3 thermocouples to measure the clad temperature and one thermocouple to measure the fluid temperature near to the outlet channel. In the lateral channel there are also 3 thermocouples to measure the clad temperature and one thermocouple to measure the fluid temperature near to the outlet channel. A

dummy fuel element was constructed by the Center of Nuclear Fuel (CCN) to solve some construction problems, and the next step will be the construction of the instrumented fuel element to be used in the research reactor IEA-R1 of IPEN-CNEN/SP.

Natural circulation in nuclear power reactors (theoretical and experimental)

One of the most serious problems for a nuclear reactor in accidental conditions is the residual heat removal from the decay of the fission products. The design of a nuclear reactor providing the establishment of natural circulation in the primary system may be an alternative to refrigerate the reactor core. In order to understand the complex phenomena involved in two phase natural circulation and to generate data for validating computer codes, in 1994 an experimental rectangular glass loop was designed by IPEN-CNEN/SP and the Chemical Engineering Department of USP, with financial resources of Chemical Engineering Department. During the period 1994 to 1999, the operation of this circuit has generated several studies, articles and a master dissertation. In 2003 researchers of IPEN took the initiative to put into operation this natural circulation circuit and in the recent years, 5 students of basic scientific research, guided by researchers of the Centre of Nuclear Engineering, have worked to put this circuit in operation again with the replacement of broken thermocouples; leaks fix, replace of the heater; installation of flow meters for the secondary circuit; design, development and implementation of a new data acquisition system based on new tools. All these modifications and repairs were carried out with resources of IPEN-CNEN/SP.

Safety analysis of Angra-2 nuclear power plant

The Safety Analysis group is involved with the activities related to the licensing process of 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 Angra 1 and 2. The main 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 are also in development.

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 IEA-R1 nuclear reactor is designed for 5 MW heat transfer load. The following additional factors were considered in the design:

- 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;
- 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;
- 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 IEASA 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 IEASA 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 and IPEN/MB-01 Brazilian research reactors

In January 2000, the IAEA initiated the Coordinated Research Project (CRP) "To Update and Expand the IAEA Reliability Database for Research Reactor PSAs". The main objective of this CRP was to update and expand the existing IAEA reliability database, published as the IAEA-TECDOC-930, compiling, processing and including adequately all the data collected by the participant institutions in their research

reactors. The second objective of this CRP was to promote the development and use of Probabilistic Safety Assessment (PSA). As these objectives presented interests in common with the ones of IPEN/CNEN-SP, this Institute signed a contract with the IAEA in March 2001, aiming to carry out a research project with the following purposes:

- Development of a component reliability database for IEA-R1 and IPEN/MB-01 Brazilian research reactors;
- Development of a computerized system to store and to process reactor operational, maintenance and failure data;
- Collection of component failure data to compose the IAEA Reliability Database;
- Documentation of findings on the research areas of Accident Initiating Events, Human Errors and Common Cause Failures;
- Contribution to the new IAEA technical document that will supersede IAEA-TECDOC-930.

The database software was developed in order to store and to process operational, maintenance and failure data of IEA-R1 and IPEN/MB-01 components. It also includes the storage of components descriptions and provides the user with several types of reports. Microsoft Access has been used to develop the database software. PSA analysts and the personnel in charge of operation and maintenance of the facilities led data collection activities at IEA-R1 and IPEN/MB-01 reactors. The data collected and reported in this study cover major components of selected systems of the two Brazilian research reactors. Concerning IEA-R1 reactor, failures of 49 component types were compiled within an observed period from January 1999 to December 2003. Concerning IPEN/MB-01 reactor, failures of 59 component types were investigated within an observed period from October 1997 to March 2004. The component failure rates and associated 90% confidence limits derived from data collected at the reactors were tabulated and sent, via Internet, to the IAEA Project Officer. These results were then incorporated in the IAEA Reliability Database together with data collected by other participant Institutes in their research reactors. The descriptions of the component types (component boundaries, equipment ratings, size, application, etc.) of both IEA-R1 and IPEN/MB-01 reactors were also provided. As a completion of the objectives of the research project, IPEN/CNEN-SP elaborated the topic "Statistical Data Analysis" to be included in an Appendix of the new IAEA TECDOC. Finally, the research project carried out at IPEN/CNEN-SP has shown that it is essential to promote an integration of data collection activities with the existing administrative routines of the facilities in order to minimize the efforts and to provide good quality data.

Safety culture enhancement at the nuclear research reactors of IPEN-CNEN/SP

For the IEA-R1 research reactor, the following activities were carried out:

- Survey of the state of the art in safety culture. This task was accomplished through seminars given by invited speakers and panel sessions where the participants made presentations based on literature studies in this area;
- Safety culture self assessment at IEA-R1 research reactor. The aim of this self assessment was to evaluate the safety culture at the reactor objectively. For this purpose, it was used the self assessment questionnaire proposed in the document Safety Series No. 75-INSAG-4 published by the IAEA. Important improvement opportunities in the safety of the reactor were identified in these appraised aspects. It is worth mentioning two examples: 1. the safety policy of the organization must be rewritten in a clearer and complete format; 2. training program of the reactor performance must be focused on safety issues. Presently, these aspects are being addressed by the reactor management.

For the IPEN-MB/01 research reactor, an assessment of its safety culture was performed, based on Three Level model of safety proposed by Edgar Schein, with the participation of the whole staff of the reactor. This assessment was developed during a workshop in which the main concepts of the book "Organizational Culture and Leadership" and the guidelines IAEA-TECDOC-1329, "Safety culture in nuclear installations", were also presented.

Qualification program of research reactor U_3O_8 -Al and U_3Si_2 -Al dispersion fuels manufactured at IPEN-CNEN/SP

The strategy adopted by IPEN for qualification the own fuel elements is to conduct the fuel tests and fuel performance evaluations in pile, during IEA-R1 reactor operation. The program for fuel element evaluation include monitoring the reactor power; time of operation; neutron flux calculation at the position of each fuel assembly; burn up calculation; inlet and outlet water temperature in core; water pH; water conductivity; chloride content in water; radiochemistry analysis of reactor water. Periodic underwater visual inspections and eventual sipping tests for fuel assembly have been carried out. A special equipment to perform underwater thickness measuring was projected by the Fuel Engineering Group of IPEN-CNEN/SP to evaluate the fuel miniplat swelling during the irradiation time. A Brazilian supplier constructed the mechanical parts and the electronic measurement instruments

were acquired by IAEA. Since the 80's IPEN has been producing and qualifying its own LEU MTR fuels (19.75% ^{235}U). Fuel element assemblies have been constructed with U_3O_8 -Al dispersion fuel plates with densities of 1.9 gU/cm^3 (from 1988 to 1996) and 2.3 gU/cm^3 (from 1996 to 1999). Since 1999, IPEN has been fabricating 3.0 gU/cm^3 U_3Si_2 -Al dispersion fuels. Regarding the qualification in-use of MTR fuel assemblies, until 2007, the 3.0 gU/cm^3 U_3Si_2 -Al dispersion fuels achieved an average burnup of 42.49%. The in-pile irradiation of 2.3 gU/cm^3 U_3O_8 -Al dispersion fuel achieved an average burnup of 40.50%.

Studies on irradiation performance of U-Mo dispersion fuels

A multidisciplinary team of the Nuclear Engineering Center of IPEN-CNEN/SP has been studying on irradiation performance of the U-Mo alloy dispersion fuels aiming for generating technical specifications for fabrication of U-10Mo-Al dispersion fuel to be irradiated in the IEA-R1 Research Reactor. The irradiation performance aspects were associated to the neutronic and thermal-hydraulics aspects in order to propose a new core configuration to the IEA-R1 Reactor using U-Mo dispersion fuels. Core configurations evaluations with the computational programs CITATION and MTRCR-IEAR1 showed that U-10Mo-Al dispersion fuel with uranium densities ranging from 3 to 5 gU/cm^3 would be adequate to be used in the IEA-R1 Reactor and would present a stable in-reactor performance even at high burnup. It was proposed the meat uranium density of 5 gU/cm^3 for the miniplates of U-Mo dispersion fuel to be fabricated at IPEN-CNEN/SP and tested in the IEA-R1 Reactor. With this uranium density in the fuel meat, the number of fuel elements used in the IEA-R1 reactor would be reduced, bringing economic advantages and reducing the number of spent fuel elements to be stored in the reactor pool.

Options for the interim storage of the IEA-R1 research reactor spent fuels

A multidisciplinary team of the Nuclear Engineering Center and from CDTN-CNEN/SP has carried out an analysis of different options for the interim storage of research reactor spent fuels. The analysis results showed a dual-purpose metallic cask as one of the most feasible options for the interim storage of the spent fuels of the IEA-R1 Research Reactor. In case this reactor keeps its continuous operation at 5 MW in

the next years, it will be necessary to transfer the spent fuels, today located in the reactor storage pool, to a spent fuel interim storage, until a definition of the Brazilian national policy about the final disposal of these spent fuels be established. Those conclusions were shared with other Latin-American institutions in the IAEA Latin American Regional Project RLA/4/018 (IAEA-TECDOC-1508, 2006). The conceptual design of a dual transport cask for the interim storage of IEA-R1 spent fuels was finalized and a scale prototype was manufactured in Argentina for qualification tests to be held at CDTN-CNEN/SP in 2008. The dual-purpose cask Safety Analysis Report (SAR) is being prepared for submission to the licensing authority in Brazil. IPEN is now developing the general requirements and the design aspects of the conceptual project of a spent fuel storage facility that will accommodate the dual-purpose casks.

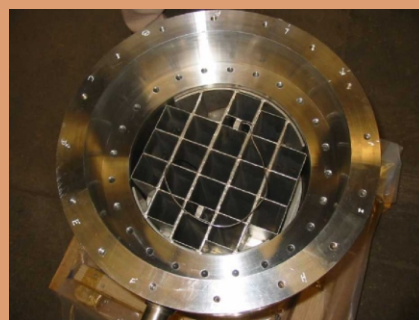


Figure 6. Dual purpose metallic cask - main body and basket

Modeling for release of fission products from a breached fuel plate under wet storage

MTR fuel elements burned-up inside the core of nuclear research reactors are stored worldwide mainly under the water of storage pools. When cladding breach is present in one or more fuel plates of such elements, radioactive fission products are released into the storage pool water. This work proposes a model to describe the release mechanism considering the diffusion of nuclides of a radioactive fission product either through a postulated small cylindrical breach or directly from a large circular hole in the cladding. In each case, an analytical expression is obtained for the activity released into the water as a function of the total storage time of a breached fuel plate. Regarding sipping tests already performed at the IEA-R1 research reactor on breached MTR fuel elements, the proposed model correlates successfully the specific activity of ^{137}Cs , measured as a function of time, with the evaluated size of the cladding breach.

Characterization of spent fuel elements stored at IEA-R1 research reactor based on visual inspection and sipping tests

Aluminum spent nuclear fuels are susceptible to corrosion attack, or mechanical damage from improper handling, while in pool reactor storage. Storage practices have been modified to reduce the potential for damage, based on recommendations presented at 2nd WS on Spent Fuel Characterization, promoted by IAEA. The Fuel Engineering Group of the Nuclear Engineering Center carry out an inspection program proposed to the IEA-R1 stored spent fuel elements, in order to provide information on the physical condition during the interim storage time under wet condition at the reactor pool. The inspection program is based on non-destructive tests results (visual inspection and sipping tests) already periodically performed to exam the IEA-R1 stored spent fuel and fuel elements from the core reactor. To record the information and examination results it was elaborated a catalogue containing the proposed inspection program for the IEA-R1 stored spent fuel, the description of the visual inspection and sipping tests systems, a compilation of information and images result from the tests performed for all stored standard spent fuel element. That document constitutes an important step of program's effective implementation, and it has been used to address regulatory and operational needs to guarantee a safe storage throughout the pool storage period.

Evaluation of fuel failure in nuclear power plants

Efforts are being made by Fuel Engineering Group of the Nuclear engineering Center to understand and evaluate the mechanisms involved in the fuel degradation subsequent to postulated primary failure in Brazilian nuclear power plants. Controlling the degradation process is extremely important to avoid large radioactivity levels in the plant and unscheduled shutdowns for removing the leaking assembly. On the majority of the cases, the inquiries on primary and secondary failures in PWR fuel rods are based on results of analysis were made use of the non-destructive examination results (coolant activities monitoring, sipping tests, visual examination). The complementary analysis methodology proposed includes a modeling approach to characterization of the physical effects of the individual chemistry mechanisms that constitute the incubation phase of degradation phenomenon after primary failure that are integrated in the reactor operational history under stationary operational regime, and normal power transients. The computational program DEGRAD-1 was developed based on this modeling approach. The practical outcome of the program is to predict cladding regions susceptible to massive hydriding. The applications

presented demonstrate the validity of proposed method and models by actual cases simulation, which (primary and secondary) defects positions were known and formation time was estimated. By using the modeling approach, a relationship between the hydrogen concentration in the gap and the inner cladding oxide thickness has been identified which, when satisfied, will induce massive hydriding. The novelty in this work is the integrated methodology, which supplements the traditional analysis methods (using data from non-destructive techniques) with mathematical models for the hydrogen evolution, oxidation and hydriding that include refined approaches and criteria for PWR fuel, and using the FRAPCON-3 fuel performance code as the basic tool.

Evaluation of Debris fretting failures on PWR fuel by post-irradiation examination and modeling in the DEGRAD-1 Code

One of the major recognized causes of fuel rod failures is fretting of the clad due to the entrapment of debris in a fuel rod spacer that inadvertently dropped into the primary system during maintenance operations. Debris, such as metal cuttings, electrical connectors, and metal fittings, pieces of wire, and small nuts, bolts can become trapped between fuel rods in a spacer where hydraulically induced vibrations can cause fretting failure of the fuel rod. Efforts are made by Fuel Engineering Group to evaluate debris-fretting failures on PWR fuel. The inquiries on fuel rods failures are based on results of analysis using post-irradiation non-destructive examination. The complementary analysis includes a modeling approach by DEGRAD-1 code to characterize the degradation phenomenon after primary failure integrated in the reactor operational history.

Evaluation of irradiation damage of structural materials

It is a know fact that radiation damage of PWR structures is a time limiting factor on its operation, the study of detrimental radiation effects on materials structures is one of the objects of research at IPEN. The R&D activities are basically performed in two fields:

- Establishment of an experimental infrastructure, by designing and fabricating the materials irradiation device CIMAT (Materials Irradiation Capsule), with controlled atmosphere and temperature (from 25 to 700OC) allowing for in-pile irradiation and on-line acquisition of parameters of interest. Priority is given to post irradiation evaluation to be performed by means of mechanical methods: creep, hardness, micro hardness, strain-stress, etc., at fluence of 1019 nvt, which represents a critical dose for the reactor

pressure vessel (RPV) steels. Operational tests of CIMAT have been completed and a modified device is under construction. Another device CIMAT 2 is under completion;

- Pre-irradiation characterization has been performed on samples of RPV steel AS 508 cl.3, of two origins: one from ELETROMETAL (Brazil AE) and the other from VITKOVICE (Czech AV), in as delivered condition. The samples were studied by means of metallographic, TEM, SEM, hardness, internal friction and X-ray diffraction. The samples show martensitic (AE) and bainitic (AV) structures, due mainly to different heat treatments. Rockwell (60 fkg) and Vickers (1 fkg/mm²) hardness data provide a maximum toughness of 600-630 MPa, which is in good agreement with the suppliers information. This characterization will be repeated after an annealing at 300OC, corresponding to the time interval of irradiation of the samples at 300OC (approx. 300 h), to equalize the thermal state of the material.

Effect of alloying and heat treatment on structure and mechanical properties of cobalt-free corrosion-resistant maraging steel

Effect of alloying and heat treatment on structure and mechanical properties was investigated in economic alloyed cobalt-free maraging steel with increased corrosion-resistant. These R&D activities counted on a technical cooperation between the Fuel Engineering Group and the St-Petersburg State Marine Technical University researchers. The developed and tested experimental steel present characteristics of a martensite class steel with increased strength provoked for the presence of carbon and intermetallics. The optimal regimen of thermal treatment regarding to the maximum strength is attained when the samples are submitted to quenching at 1050°C for 1 hour and aging to 520°C for 5 hours. Triple-austenitizing provides sufficient homogeneous grain structure. High-temperature microscopy verifies this observation. At 560°C for times higher than 3 hours, the presence of reverted austenite in the microstructure, confirmed by X-ray Diffraction measurements, causes a continuous decrease in the strength (hardness). Scanning Electron Microscopy of the fracture surface gives additional evidence about reduction deformation capacity of the steel. The present results constitute the initial phase of the study. In the next one, the steel will be radiated with neutrons fast in IEA-R1

Reactor, in high temperature and controlled atmosphere, simulating the conditions of work of power reactors, and using the device CIMAT.

Structural analysis & integrity assessment and life management of nuclear components

The research in this area was focused mainly in the Steam Generator Tubing Integrity Assessment, some aspects of the project by analysis covered in the nuclear standards and Life Management and Life Extension of PWR NPP. The continuous safe operation of a SG requires a tube integrity analysis at every outage with, in general, 100% of the tubes being inspected NDE techniques like the Eddy Current one. All uncertainties, like those in the material strength, defect dimensions, etc., should be considered to assess the structural limits of the SG tubes in terms of critical defect dimensions. Usually this is done using a Monte Carlo statistical approach. To assure that the operation did not challenge the adopted criteria based on regulatory limits, some defect can be tested in situ and if it passes the test the criteria remains valid for future assessments. Otherwise the criteria should be revised. Within the scope of this research area, several studies have been performed. Among them can be mentioned: the Structural Analysis of the main components of a Nuclear Power Plant, as well as Research Reactors, like the inlet nozzle of the new IEA-R1 Heat Exchanger; the evaluation of the stress concentration factor in Y fittings, the evaluation of stress intensity factors KI and KII in an postulated inclined crack within a weld for a particular geometric pressure vessel configuration; the continuous development of in-house programs to establish the critical defect dimensions in Angra-1 SG tubing.

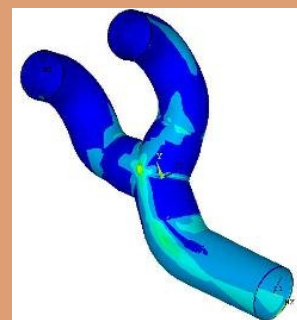


Figure 7. Stress analysis of a Y fitting according to ASME Section III, Division 2

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

The requirements for the qualification of transportation and/or storage packages for nuclear research reactors spent fuel elements are quite severe. Among the postulated accident conditions to be analyzed there is the 9 m free drop on a rigid surface. To avoid high decelerations in the package content, which could jeopardize the required nuclear safety standards, it is allowed to use impact limiters made with energy absorbing materials which will absorb the kinetic energy by means of a great amount of deformation. Their destruction is allowed during this process. The package itself has several components connected to each other in different ways (welded parts, flanged connections, surface contacts, etc.). This research involves other groups in Brazil and abroad (mainly, Argentina) and it is sponsored by AIEA. In its first phase a scale model was designed, it is being constructed and will be tested. Some material tests and properties characterization were already performed as well as some numerical analyses. All analyzes, were and will be performed, using the finite element method and explicit algorithms to simulate the 9 m free drop test condition, to show the worst drop position (the one that causes the maximum damage in the package). Some of the considered aspects are: the materials properties nonlinear modeling, the different stiffness of the package materials, 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.

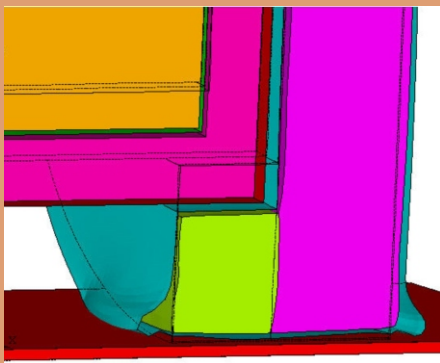


Figure 8. Final deformed for the corner impact (at 45°)

Monitoring and diagnosis of reactor systems and components

Since 2000, the Nuclear Engineering Center is developing techniques for the monitoring and diagnosis of nuclear systems and components using advanced methods for data processing and

analysis. This work has been supported by two IAEA technical cooperation contracts being one of them a Latin American regional cooperation project and three CNPq research projects being one of them a bilateral agreement in partnership with the National Science Foundation/University of Tennessee. The total budget up to date amounts to about a million reais. The general idea of this project is to improve reliability, safety and availability of critical components and systems of a nuclear power plant thus also allowing for the plant life extension. Well establish techniques were used as well as advanced methods for data processing such as artificial intelligence, time-frequency domain transforms and higher order statistics were studied. The main activities are summarized below:

Plant condition monitoring and fault detection

During the last years, model based fault diagnostics methods have received increasing attention in both research and application. This approach is based on the concept of analytical redundancy as opposed to physical redundancy (by hardware) that uses measurements from redundant sensors for fault diagnosis purpose. In this work, the system analytical model is obtained by using the Group Method of Data Handling (GMDH) which uses data from the reactor instrumentation to generate a normal condition model for different operational conditions, a fault free database. The GMDH is a form of artificial intelligence and is based on a sequential learning networks which are networks of mathematical functions that characterize complex, nonlinear relationships in a compact and rapidly executable form. The method was tested successfully in the IEA-R1 research reactor instrumentation and control system.

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.

Leak detection and localization in pressurized tubes and vessels

Leaks in pressurized tubes and vessels generate acoustic waves that propagate through the walls of these tubes, which can be captured by

accelerometers or by acoustic emission sensors. In this work an analytic model was implemented, through the solution of the equations of motion of a cylindrical shell, with the objective to understand the behavior of the tube surface excited by a point source. The theoretical results were validated through measures accomplished in an experimental setup composed of a steel tube ending in sand boxes, simulating the condition of an infinite tube. To determine the location of the point source on the surface, the process of inverse solution was adopted.

Monitoring and automated diagnosis of rotating machinery

Rotor unbalance, shaft misalignment, and bearings faults are usually detected using conventional frequency domain Fourier transform of the accelerometers signals. In this work we tested the use of Wavelet transforms to obtain new characteristics for the defects with improved diagnosis capabilities. The ability to differentiate localized defect from distributed corrosion type defect is one of the advantages. Also, in order to automate the diagnosis decision a para consistent fuzzy logic engine were developed to process the several characteristics and to indicate a defect automatically. The methodology was tested in a rotating machine defect simulator and the results are quite satisfactory when compared with actual data from IEAR1 research reactor.

Development of an artificial neural network for monitoring and diagnosis of sensor fault and detection in the IEA-R1 research reactor at IPEN-CNEN/SP

The increasing demand on quality in production processes has encouraged the development of several studies on Monitoring and Diagnosis Systems in industrial plant, where the interruption of the production due to some unexpected change can bring risk to the operator's security besides provoking economic losses, increasing the costs to repair some damaged equipment. Because of these two points, the economic losses and the operator's security, it becomes necessary to implement Monitoring and Diagnosis Systems. In this work, a Monitoring and Diagnosis Systems was developed based on the Artificial Neural Networks methodology. This methodology was applied to the IEA-R1 research reactor at IPEN. The development of this system was divided in three stages: the first was dedicated to monitoring, the second to the detection and the third to diagnosis of

failures. In the first stage, several Artificial Neural Networks were trained to monitor the temperature variables, nuclear power and dose rate. Two databases were used: one with data generated by a theoretical model and another one with data to a typical week of operation of the IEA-R1 reactor. In the second stage, the neural networks used to monitor the variables were tested with a fault database. The faults were inserted artificially in the sensors signals. As the value of the maximum calibration error for special thermocouples is $\pm 0,5^{\circ}\text{C}$, it had been inserted faults of $\pm 1^{\circ}\text{C}$ in the sensors for the reading of the variables T3 and T4. In the third stage a Fuzzy System was developed to carry out the faults diagnosis, and it was considered three conditions: a normal condition, a fault of -1°C , and a fault of $+1^{\circ}\text{C}$. This system will indicate which thermocouple is faulty.

Time response measurements of temperature and pressure sensors of Angra I nuclear power plant

The Instrumentation, Monitoring and Diagnosis group of IPEN-CNEN/SP currently performs time response measurements of temperature and pressure sensors of Angra-I nuclear power plant protection system. Response time test is a regulatory requirement and it need to be performed in each outage for refueling or in case of one transmitter need to be replaced. Sensor time response measurement represents an important requirement to be observed in the maintenance of a nuclear reactor protection system. This measurement has to be performed periodically to ensure that the protection reactor limits are respected.

Loop Current Step Response - LCSR

The methodology for temperature time response measurements is the Loop Current Step Response LCSR, used to determine time constant for Resistance Temperature Detector-RTD sensors. The test consists in applying a small current to the RTD leads that heats the sensor filament and the temperature transient due to a step change is analyzed to determine the response time that would have followed a fluid temperature change. The LCSR data gives the sensor response of an internal heating perturbation, but the response of interest is the one that results from a fluid temperature perturbation. An analytical transformation was developed to predict the response to a fluid temperature perturbation by using information from the LCSR data record.

Noise Analysis

The methodology used for pressure sensor time response measurements is the Noise Analysis methodology. Random noise techniques for measurements on nuclear reactor systems have been developed as a tool for system surveillance or to analyse dynamic behaviour with a minimum of interference during normal operation. Random variations in the neutron flux density, temperature, steam flow or pressure may be used to derive useful information about the system dynamics or to monitor sensor characteristics. The frequency domain method consists of evaluating the Power Spectral Density (PSD) function. The information needed for time constant estimation can be obtained by fitting an all-pole transfer function to this power spectral density. If the system has only one dominant pole, then the time constant of interest can be obtained from the break frequency of the Power Spectral Density curve. Sensors' signals are filtered and amplified before acquired using a data acquisition system connected to a PC (Figure 9).



Figure 9. Isolating amplifiers used to data pre-processing in the noise analysis methodology

Development of a wide-range reactor power measuring channel

The main objective of this research is the development of a digital wide-range reactor power measuring channel (hardware and software) to be used in control and protection systems for nuclear research reactors. The system is composed of two neutron detectors (pulse and current modes) arranged to cover 10 decades of reactor neutron flux and associated electronics. The conditioned signals are processed by distributed microcontrollers. Hardware and software tests are currently running and the estimated date for experimental installation in IPEN/MB-01 research reactor is set for end of 2008.

Using cumulants and spectra to model nuclear radiation detectors

A general mathematical methodology is presented to model a nuclear radiation detector. This is accomplished by using a proposed generalization

of Campbell's theorem, which employs nth-order cumulants and spectra analysis and a vector of random parameters to describe the current pulses. This allows a more elaborate, higher order statistical characterization of the radiation detection process, as compared to the usual second order treatment. To demonstrate the method, it is applied to a neutron ionization chamber (fission or ^{10}B). A deterministic model for the chamber current pulses is developed and their probability density functions are used to define the characteristics of the vector of random parameters. Computer simulations of the proposed theoretical methodology were developed and potential applications suggested.

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 CEN-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 CEN-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 utilization of Reactor IPEN-MB/01

The first criticalization of the IPEN/MB-01 reactor (Figure 6) was obtained on November 9th, 1988. Since this date more than 2150 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 configurations (Benchmarks to OECD - NEA). 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. Beyond experimental purposes the IPEN/MB-01 reactor is used also to reactor operators training and educational purposes (Graduated and Post-graduated).

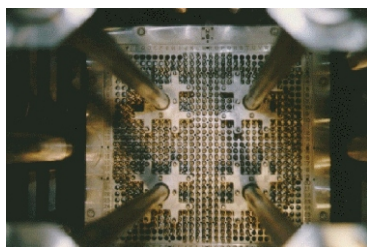


Figure 10. IPEN/MB-01 Reactor core

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 using economics resources of the Centro de Engenharia Nuclear (CEN). Normally are reserved until 15 weeks by year to maintenance of the IPEN/MB-01 Reactor. Basically the reactor has two kinds of activities: Operational and Experimental. 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 partners (CTMSP-Brazilian Navy).

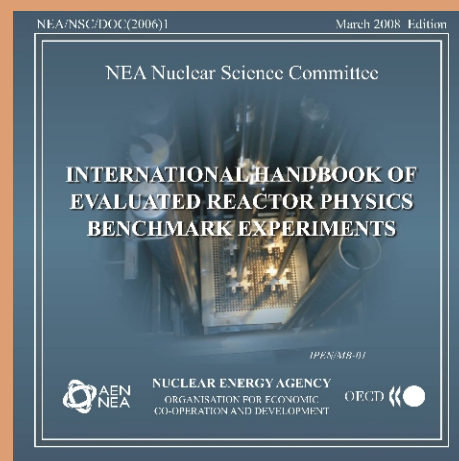


Figure 11. International Handbook of Evaluated Reactor Physics Benchmark Experiments Containing IPEN/MB-01 Reactor Experiments

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 other IPEN centers 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 with other institutions that

already have tradition in this area. During the last three 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. In fact, the origin of the phenomenon of the global warming (intensive energy use), and the global social deficit, is the incentive for the demand of products and services that are not necessary to a worthy life of the humanity (consumerism). It is observed that the benefits for these products and services go to a lowermost part of the world-wide population, widening each time more the abyss that separates the supplied populations of the great majority of the poor persons and that, in not rare times, do not make use of the minimum for the proper survival. Another point is the study of the emergy methodology and its scientific fundamentals that takes into account the embodied energy during a transformation process, making possible to measure the nature contribution, until now considered free, in the same calculation basis, such method comes to increase the resources utilized to evaluate the natural systems sustainability. 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 considering activities directly related to cores of nuclear reactors, waste management, thermal and hydraulics and safety analysis of plants and systems. The technical expertise of the professionals in the above specialties is directed to solve problems related to conceptual design,

operation, nuclear fuel design and performance evaluation, life extension, reliability and safety analysis. The following areas were covered: thermal and hydraulics, nuclear fuels, safety assessment, reactor physics, training of operators, and plant engineering. Complementing the services described in the nuclear engineering field, other specialized technological services were also performed in design and analysis of systems and equipments of nuclear reactors, fuel cycle installations and cogeneration power plants. The services were directed to solve technical problems related to conceptual design, operation, aging of components, in service inspection, and so on. The background acquired is also applied to conventional thermal and general process plants. The following areas were covered: instrumentation and control, electricity, structural mechanics, conventional power plants, calibration and metrology, and fuel cell. All the services and specialized services described above were conducted by a team of 40 (forty) professionals that are working at the Nuclear Engineering Center. A total of approximately 30.000-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), the Brazilian nuclear utility (named Eletronuclear) and the Brazilian navy technological centre (name CTMSP). All the engineering services provided by the Nuclear Engineering Center are certified with the ISO 9001:2000.

Nuclear Research Reactors, Operation and Utilization

In the triennial 2005/2007 the IEA-R1 Research Reactor has been operated most of the time at a reactor power of 3.5 MW and the reactor operation schedule of 62 hours per week. The Operation and Maintenance Area of the IEA-R1 Research Reactor is composed by three groups as follows:

- operation group
- maintenance group
- technical support group

Operational group has a crew of 23 operators responsible for the reactor operation, placement and disposal of irradiation samples in the reactor core, tests, experiments and control of operational documents. Maintenance group has 9 technicians for the preventive and corrective reactor systems maintenance, study of ageing and maintenance registrations. Technical support group, formed by two persons, is dedicated to neutronics and termohidraulics calculations. In this triennial, to given an example of previous years, several changings in order to achieve reactor modernization has been done. These works are listed below:

New heat exchanger acquisition and installation

The heat exchanger new project was developed by Ipen nuclear engineering group. The construction and installation was carried by Brazilian company IESA. The old equipment removal and assembly of the new one were followed by the reactor operation and mantance group, leased by the Ipen's Radiological Protection.



Figure 12. New heat exchanger

New irradiation pneumatic system construction

The old system has presented infiltration and contamination by the swimming pool water. The new system was projected and constructed by the Ipen technicians.

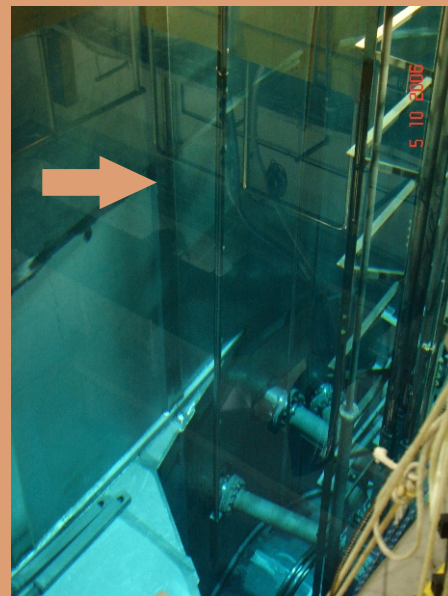


Figure 13. New irradiation pneumatic system

The start of the construction of pneumatic system between the reactor and radiopharmacy center

This system will allow to send samples from the reactor swimming pool hall to the radiopharmacy center cells of processing, avoiding the use of shields. With this, time will be saved and the level of radioisotope activity will be increased.

Restoration of the building reactor wall

After 50 years, the wall concrete has presented cracks, hardware and internal humidity. The wall restauration allowed the correction of the problems in the concrete, waterproofing and painting.



Figure 14. IEA-R1 Research Reactor

Transport of 33 reactor spent fuel elements to United States

This task has continued the transportation of spent fuel elements to Savannah River Site Laboratory in South Carolina. In 1999, it has been transported 127 fuel elements to United States. In November 2007 has been transported 33 elements. In this operation it has been used one LWT cask, developed by an American company NAC.



Figure 15. transportation of spent fuel elements

Acquired equipments by AIEA program

- Spectrometer of HPG;
- 2 fission chambers;
- Digital personal dosimeters;
- SPND detector (flux detection);
- Gateway radiation protection monitor;
- 2 ducts radiation detectors (cintilator);
- 2 area radiation detectors (gieger muller);
- Lead viewfinder;
- 2 ionization chambers (flux detection);
- Surface contamination monitor;
- Contamination detector;
- Vibration system (primary pump monitoring).

Monthly, the Reactor Operation and Maintenance Area issues a report containing the activities of the reactor. This report encloses the number of reactor operation schedule, energy dissipated in this period, reactor core configurations, chemical and physical characteristics of the water systems, 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. The main operational parameters of the triennial 2005/2007 were:

- Reactor power: 3,5 MW
- Time of reactor operation in hours: 7850.91
- Energy dissipation in MWh: 24823.33
- Time of low power operation in hours: 76.06
- Number of samples irradiated in the grid plate: 3309
- Number of samples irradiated in the pneumatic

system: 319

- Number of new core configurations: 14

In 2002 the Research Reactor Center (CRPq) started the process of implementation of a quality management systems (QMS) in compliance to NBR ISO 9001 version 2000. This process resulted in the accreditation of the Operation and Maintenance of the IEA-R1 Reactor and Irradiation Services by Fundação Carlos Alberto Vanzolini on December 13, 2002. Since then, the reactor has operated with this certification, which is renovated every year by external and internal audit.

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Environmental Science and Technology



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Introduction

The Environmental Science and Technology Program was structured based on the continuous growth of environmental activities on areas related to nuclear programs at IPEN. The program comprehends five main areas:

- Environmental analysis
- Chemical Technology
- Polymer technology nucleus
- Chemical and Isotope Characterization
- Analytical Chemistry for the Nuclear Fuel Cycle Samples

Recent activities are highlighted bellow:

- Development and use of modern analytical technologies for the characterization of pollutants and contaminated areas;
- Development of chemical fingerprint methodologies for the identification of materials for forensic purposes (cannabis origin, gunshot residues, cocaine origin and nuclear signatures);
- Studies of trace gases as part of LBA project (Large Scale Biosphere-Atmosphere Experiment) in the atmosphere of Amazonian region and greenhouse gases of urban areas;
- Cooperation with SABESP (Water and Sewage Company) and CETESB (State Environment Agency) in programs for the development of public policies, such as the critical revision to the guidelines of drinking water standards.
- Studies and development of recycling technologies reuse of materials, waste storage and decontamination;
- Studies and developments in biodegradable polymers, polyolefins and advanced methods for polymer and rubber destruction etc.;
- Studies on the production of Hydrogen from renewable sources for fuel cell purposes.
- Electrochemical techniques for the dissolution of metals and treatment of industrial and radioactive wastes
- Clean Technologies
- Synthesis and development of magnetic nanoparticles and participation on the RENAME (network on nanotechnology research) project.

These achievements were conducted with support from national and international funding agencies - FAPESP, CNPq, FINEP, CAPES, IAEA - as well as cooperation with partners and clients, such as SABESP, CETESB, 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 polycyclic aromatic 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 supporting by CNPq and FAPESP in partnership with Environmental Agency State (CETESB) and SABESP were developed:

Identification of the influence of sludge discharges from water treatment plants

The growing concern of environmental surveillance of the quality of hydric resources guides the development of research on management of residues generated in water treatment plants (WTP). Approximately 8.000 WTPs in Brazil operate without a treatment program of the residues, disposing these effluents in the environment. This work evaluated WTP discharges into watercourses by collecting superficial waters, sediments and benthic samples at the town of Registro, São Paulo State, Brazil. Even though superficial waters and benthic samples showed no further contamination, sediment analysis pointed out that aluminum deposits detected near sludge discharges may represent a potential risk to the environment.

Assessment of agriculture impact on environmental preservation areas of the Ribeira de Iguape River, São Paulo

In order to ascertain the impact of agricultural activities on water quality of the Ribeira de

Iguape River Basin in the state of São Paulo, surface water areas for catchment and drinking water have been characterized. The sampling period covered March/2002 - February/2003 and January/2004 at 10 different catchment points. SPE-LC-UV/Vis was used to monitor various pesticide classes such as carbamates, triazines and nitro anilines. The results revealed that water quality is associated with seasonal variation. Of 152 samples analyzed, only 24% showed the presence of pesticides, particularly during the wet season. High variability in pH, turbidity and color were observed.

Environmental water quality in Pedrosó Reservoir (Billings's watershed)

The present work aimed to evaluate the environmental water quality in Pedrosó Reservoir (Billings watershed) situated in Santo André - São Paulo State. Sixty four water samples (41 drinking water samples and 23 surface water samples) Ag, Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Hg, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, V, Zn, NO₃⁻, SO₄²⁻, PO₄³⁻, F⁻ and Cl⁻ were evaluated. The sampling period covered June 2005 to December 2006. For the drinking water samples all elements analyzed were detected in concentrations below the limit established by the Brazilian Drinking Water Standard - Portaria N° 518/MS/04. For the surface water samples of all elements analyzed, only to Ag, Al, Fe and P were detected in concentrations, above the limit established by CONAMA 527/05.

Evaluation of water quality using physicochemical parameters and a modified water quality index

The Brazilian Federal Designation N° 5440/05 establishes definitions and procedures for water quality control of supplying systems and defines mechanisms and instruments for transmitting information about human consumption water quality to general consumer. The current guidelines for drinking-water standards (Portaria N° 518/04), in article 9° of section IV: charge the responsible of the operation of the water system to supply all the consumers' information about the quality of the distributed water, in the terms of the Code of Defense of the Consumer. A Water Quality Index (WQI) is a numeric expression used to evaluate the quality of a given water body and to be easily understood by managers. Therefore, the purpose of this paper is to identify, in some of the several water quality indices described in the literature, a dynamic index of water quality for supplying, which will be used as a tool to simplify the report of the water quality data.

Contribution of Taquaruçu Grande Basin to Luis Eduardo Magalhães Reservoir - TO

Many reservoirs have been found to receive significant pollutant loading from their upstream watersheds. Wide usage of agricultural pesticides and fertilizers, inappropriate landuse management

high intensity rainfall and steep-slope farming are major factors that contribute to the degradation of reservoir water quality. The sub-basin of Ribeirão Taquaruçu Grande located at Palmas city, has preserved sources, contains agricultural areas and by the end of their course receives influence the process of urbanization. This project aims to assess the physical, chemical and biological contribution of the Ribeirão Taquaruçu basin through the analysis of the limnological variables, metals and trace elements determination, and the identification of phytoplankton community.

Soil contamination

The metal partition coefficient (K_p) is ratio between the concentration of metal bond to the soil and metal concentration in soil solution. The K_p may vary by several orders of magnitude because of the different soil characteristics. For human health risk assessment it is important to determine the specific soil metal K_p . One study the metal K_p of soil samples were determined for the coal-fired Figueira (Figueira county, north of Parana State) power plant surrounding soil. Another study the K_p of benzo(a)pyrene was determined in tropical soil. Radionuclides of the natural series, such as ^{238}U , ^{228}Ra , ^{226}Ra , ^{210}Pb and ^{40}K , in raw material samples of coal used by the main power plants in Brazil (RS; SC; PR) were evaluated. The results showed that the uranium concentration in coal from (PR) was at least three times higher than concentration found in other coal samples in Brazil, RS and SC. The natural radionuclide contents in pulverized coal, furnace bottom ash and fly ash samples in Figueira coal-fired power plant were evaluated. High enrichment factor was observed for the last stage filter fly ash. The concentration of the uranium series found in the ashes is close to the limit adopted by the Brazilian guideline. Therefore, it is advisable to evaluate the environmental impact of the installation. Soil columns with batteries placed at the top were leaching with an acid-rainwater solution during one year. Zn and Mn showed a high tendency to be retained at the upper layer of the soil column.

Autochthonous species of invertebrates in aquatic toxicity test

Daphnia laevis, *Ceriodaphnia silvestrii* and *Chironomus xanthus* are among the autochthonous species whose biology has been studied and which have been used as test-organisms. However, routine actions are required to be introduced in assay labs, such as sensitivity evaluations and establishment of control-charts. Based on *Daphnia laevis*, *Ceriodaphnia silvestrii* and *Chironomus xanthus* continuous cultures in laboratories

involving biology studies for methods standardization, this research shows species sensitivity based on tests with reference substances, a fundamental requirement for them to be considered eligible for toxicity evaluations within the specific sensitivity range for a reference substance. This requirement meets what is established in quality systems for assay laboratories, according to ABNT ISO/IEC 17025 (ABNT, 2001) and Good Laboratory Practices (OECD, 1998). Standardized cultivation procedures have been developed for *Daphnia laevis*, *Ceriodaphnia silvestrii* and *Chironomus xanthus* species, according to ABNT ISO/IEC 17025 (ABNT, 2001) and Good Laboratory Practices (OECD, 1998), from biology studies of species based on existing literature and guidelines by ABNT (1993), CETESB (1991), ASTM (2000) and EPA (2002, 2004).

Occupational and environmental exposure assessment levels to emerging pollutants, metals, endocrine disruption and organic compounds

Pharmaceuticals, personal care products, and potential endocrine disrupters are part of a large and diverse group of organic compounds, labelled as emerging contaminants, which have received much attention in recent years. Many of these contaminants enter the environment by way of wastewater systems and their fate and the consequent environmental risks are only now being considered in detail through ecotoxicological studies. This is partly due to the improved sensitivity of analytical techniques but also because reuse of wastewater is becoming more common worldwide, driven by pressure on other sources of fresh water.

For endocrine disrupting chemicals in the environment, concerns arise primarily from the effects that may be induced in wildlife. A well studied example is estrogenic chemicals in the aquatic environment and their effects on fish. Directly measuring effects, in fieldwork studies, is an expensive and time-consuming approach that is fraught with many difficulties, ranging from study design right through to data analysis and interpretation. Several chemical substances suspected of endocrine disruption are currently being used in industrial and agricultural activities in Brazil. Virtually nothing is known about the related health effects, highlighting the need to implement a national program to evaluate such impacts. Scientific Cooperation programs supporting

by CNPq and FAPESP in partnership with SABESP were developed focus:

Determination of endocrine disrupters in public water supply on Paraíba do Sul River region, Brazil

This research line focuses on investigate the presence of some endocrine disrupters agents (EDs) and pharmaceuticals and personal care products (PPCPs) which may affect the quality of public water supply on Paraíba of the South River Basin. The study has been developed according to: (1) occurrence of EDs and PPCPS in surface and drinking waters; (2) optimization of the analytical procedures proposed to quantify a specific class of chemical contaminants; and (3) determination of EDs and PPCPs in surface and potable waters and effluents of a municipal sewage treatment plant (STP).

Geochemistry distribution of Polycyclic Aromatic Hydrocarbons (PAHs) in sediments of Park Pedroso Reservoir, Santo Andre, Brazil

An environmental diagnosis identifying the presence of PAHs in sediment and surface water has been carried out. The methodology involves the identification of HPA's and the combination of other physical and chemical parameters.

Selenium in ground water of the Northwest Area of the State of São Paulo

In some districts of the Northwest Area of the State of São Paulo the public provisioning is made mainly by reception of underground water of the Guarani aquifers, through tubular wells. The monitoring of Se was realized in four wells of the Northwest Area of the State of São Paulo, to follow the variation of the concentration of that element for a hydrologic cycle.

Identification of pesticides on sludge discharges from water treatment plants

Sludge from water treatment plant (WTP) is analyzed in this study. Many compounds can be concentrated in this material as it is an end-residue generated in this purification process. A WTP in the Vale do Ribeira, SP, region of banana crops, was chosen. Using liquid chromatography, several pesticides are determined after liquid-liquid extraction. Methodology validation was performed.

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

The development of adsorbent material to monitoring the water quality by the measurements of physical, chemical and biological aspects for water quality evaluation of the urban stream and the development of an adsorbent material with remain fixed for an immersion period in polluted urban waters, when submitted to a series of analyzing procedures, presents toxic compound

content equivalents to the waters existing ones with the information about the presence of toxic metals and organic composites with also the measurement of water eutrophic condition of rivers, streams, lakes and reservoirs with dense pollution as the urban streams Pirajuçara, Aricanduva, Tamanduateí and Sapateiro located in the Metropolitan area of Sao Paulo.

Application of zeolite materials for metals removal from aqueous solutions and soil

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 4.0×10^6 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 e alumina and also due to a low ratio $\text{SiO}_2/\text{Al}_2\text{O}_3$, 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. 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 treatment of the electroplating effluents, immobilization of heavy metals in soil, decontamination of actual acid mine drainage and removal of dyes from aqueous solution has been

evaluated. The results obtained in the project showed a great reduction in the pollutant concentration in treated waters and soil and demonstrated the high potential of the zeolites synthesized from Brazilian coal ashes as low-cost adsorbent material.

Green House studies in Amazon Basin and ozone precursors in Sao Paulo city 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 CO₂, CH₄, CO, H₂, N₂O and SF₆ have been measured above central Amazônia (Over Tapajós National Forest, a primary forest in Para State and over Cuieiras Biological Reserve, a primary forest in Amazonas State) and the Brazilian coast (Fortaleza over the ocean).

Samples are collected aboard light aircraft between the surface and either 4 km (Tapajós National Forest) or 5 km (Fortaleza) using the NOAA/CMDL semi-automatic portable flask package (PFP). The PFP consists of 17 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, which was severely hampered due to problems inherent in shipping samples between Brazil and the United States.

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 CO₂, CH₄, CO, N₂O and SF₆ in the flask and PFP samples. All measurements are calibrated to internationally accepted scales. The fluxes determination using atmospheric measurements from aircraft profiles over Floresta Nacional do Tapajós (SAN - 02°51'S; 54°57'W, over tower 67km) and Reserva Biológica de Cuieiras (MAN 02°36'S, 60°12'W, over tower K34). SAN profiles started December 2000 and MAN started December 2004, both running until 2007. Samples are collected aboard light aircraft between the surface and 4 km using the NOAA/CMDL semi-automatic portable

flask package (PFP), with 17 flasks samples. Using a column integration technique of CO₂ profile measured subtracted by CO₂ background, it was determinates the flux for each flight. The CO₂ background was determinates using a co-measured SF₆ 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. 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. Arembepe, Bahia Background station was start CO₂, CH₄, CO, H₂, N₂O and SF₆ measures since October 2006 in cooperation with INMET, NOAA and WMO. This is the first Brazilian station in global net work. This project opens the possibility to start others stations in others place in Brazil.

Ozone Precursors in São Paulo City

The pollution in the urban area of Sao Paulo city is mainly related with vehicular emissions and secondary with industrial emissions. The metropolitan area of Sao 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 NO_x, 50% of aerosols, and 55% of SO_x. Volatile organic compounds speciation and the contribution to the ozone formation have been investigated. The research has been carried out by means of an extensive sampling campaign between March 2005 to March 2006, using traditional methods of sampling (canisters) and analysis (CG/MS/FID) for VOCs. It was made 2 sampling campaigns to following trace gases measurements: CO, NO_x and O₃ in winter and summer period. Meteorological data was collected to understand the transport process of these compounds. OZIPR mathematical model was used to determinate the principals VOCS in ozone formation. Financing by NASA, NOAA and CNPq.

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

X-Ray Fluorescence Laboratory (LFX) (Fig. 1) has worked in geological, environmental and biological studies establishing new analytical methodologies for different samples such as soils, sediments, air filters, used lubricating oils, domestic dust, metal

monitoring plants, Brazilian medicinal plants, diets, organic fluids like tissue, serum and whole bloods. The R&D activities have been supported by the IAEA, CNPq and FAPESP organisms. The main projects, carried out and ongoing, are listed below:

Food

The inorganic elements (Ca, Cl, Cu, Fe, K, Mg, Mn, Na, P, Se and Zn) in typical industrial Brazilian workers were determined for comparison with Neutron Activation Analysis data and participation in a network using analytical techniques, such as NAA, XRF, AAS, ICP-OES and HPLC, for certification of national in-house bovine liver reference material.

Environmental diagnostic

The inorganic elements (Na, Mg, Al, Si, Ca, Ti, Fe, P, S, V, Cr, Mn, Ni, Cu, Zn, As, Rb, Sr, Zr, Ba and Pb) in soil, ashes, coals and grasses were determined to study thermoelectric powder plant pollution.

Brazilian medicinal plants

The quantitative inorganic profile for Brazilian medicinal plants (*Anadenanthera macrocarpa*, *Schinus molle*, *Hymenaea courbaril*, *Cariniana legalis*, *solidago microglossa* and *Stryphnodendron barbatiman*) were established by Na, Mg, P, S, Cl, K, Ca, Mn, Fe, Ni, Cu, Zn, Rb and Sr determination.

Indoor pollution

The inorganic elements (Na, Mg, Al, Si, P, K, Ca, Ti, Cr, Fe, Ni, Cu, Zn, Se and Pb) and organic compounds (phthalate esters) in domestic dust in the metropolitan area of São Paulo city has been determined for urban and indoor pollution study.

Residue recovery

Reuse and recovery for used lubricating oils using ionizing radiation (60-Co source) has been studied. The removal degree was determined for Mg, Al, Si, P, S, Cl, Ca, Cr, Mn, Fe, Ni, Zn and Pb elements using different absorbed doses. The organic compounds degradation, related with different absorbed doses, has also been studied.

Metrology

Uncertainty of measurements for P-10 plant steel and 302 and 304 series stainless steel samples analyzed by WDXRF and OES-spark techniques has been calculated.

Clinical studies

The analytical method for K, P, Cl, Na, Fe and Ca determination in serum and whole blood, has been developed for comparison with Neutron Activation Analysis data, aiming the Brazilian reference value determination.

Hazardous substances

Analytical method for Cr, Br, Cd, Hg and Pb

determination has been developed, to meet general requirements of the RoHS (Restriction of the use of Hazardous Substances in electrical and electronic equipments), WEEE (Waste Electrical and Electronic Equipment) and ELV (End of Life Vehicles) directive.

Quality assurance

In the period, the LFX implanted de ISO IEC 17025, version 2005. The several topics of the Quality Manual were modified and adapted according to new requirements. The analytical methods validation and robustness were determined for 302 and 304 series stainless steels, sediments and igneous rocks chemical assays, calculating the uncertainty of measurements and the accuracy, using statistical tools. The annual internal audit by SGI/IPEN was carried out. The Laboratory participated in national and international inter-laboratory programs (steel and alloy samples: promoted by IPT/Brazilian Metrology on Chemistry Program, IPT/PBMQ ; bauxite by SGS-GEOSOL, MG, Brazil ; synthetic aqueous solution by Instituto Nacional de Tecnologia Industrial, INTI, Argentina and biological and herbal samples by the International Atomic Energy Agency, IAEA, Austria. The Laboratory participated in the RILCA program (Red Iberoamericano de Laboratorios de Calidad del Agua) supported by CYTED (Programa Iberoamericano de Ciencia y Tecnología para el Desarrollo). (<http://www.cepis.oms.org/bvsala/E/taller-relac/RILCA.ppt>). In 2007, the Laboratory participated in "I Taller Iberoamericano sobre Calidad de la Información Analítica en los Laboratorios de Calidad del Agua", as a Brazilian representative, lecturing about the Brazilian Water Quality Laboratories. Inside the RILCA program, a course on laboratory accreditation, according to ISO/IEC 17025/2005, was given in Facultad de Ciencias Exactas y Naturales, Paraguay (5th - 9th November, 2007).



Figure 1. X-Ray Fluorescence Laboratory (LFX)

Analytical chemistry for environmental diagnosis and to assist the Nuclear Fuel Cycle samples

The Laboratories of Chemistry and Environmental Diagnosis Center - CQMA, were established methodologies for evaluation of physical-chemical, chemical and toxicological parameters to support several research projects in development for environmental diagnosis and to assist the Nuclear Fuel Cycle Program.

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 have been participated in several international interlaboratory programs sponsored by: CETAMA (Établissement Comission des Analyse Méthods, France), IAEA (International Atomic Energy Agency), INTI (Instituto Nacional de Tecnologia Industrial, Argentina), SENAC (Brasil), ABACC (The Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials), SABESP (Basic Sanitation Company of the State of São Paulo, Brasil) and Rede Metrológica Rio Grande do Sul (Brasil). The Quality Handbook according to ISO GUIDE 17025 has been elaborated. The XRF Laboratory has been participated of Quality Control in Analytic Laboratories of the AIEA-ARCAL LVXXI Program - Test Aptitude. 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.

- Evaluation of selenium in groundwater and sediments samples by Atomic Absorption Spectrometry (AAS, HGAAS, CVAAS) Selenium is associated to some human metabolic processes; however, they become toxic when present at high levels.

- Metal evaluation in top water and drinking water.

- Metal evaluation for industry wastewater.

Biomedicine

Hydroxide-apatite Ca/P relation in teeth for laser prevention studies has been determined by ICP-OES.

Nuclear fuel cycle

- Evaluation for quality control of metal and rare earth impurities of Nuclear Fuel Cycle Program (U_3Si_2 ; UF_4 ; U_3Si_2-Al ,) using extraction chromatography and ICP-OES. The routine analyses related to nuclear materials by RFX (Si_3U_2 , aluminum powder and alloys, AgInCd alloys, uranium and thorium compounds).

- Development of method for Si and U determination in U_3Si_2 system by X ray fluorescence and gravimetric/volumetric techniques. This method is applied to the assembled fuel elements.

IEA-R1 Reactor

Evaluation of IEA-R1 water quality.

Industrial Partnership

- An analytical methodology was established for the determination of Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu and Zn in adhesive materials, applied in the automotive manufacturing, for SABÓ IND. e COM. de AUTO-PEÇAS LTDA.

- A similar method was developed for Mg, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu and Zn determination in adhesive ribbons, used in the electrical components isolation, for VOITH SIEMENS HYDROPOWER GENERATION.

Environmental Monitoring Programme (EMP-Q) to assist the no radioactive chemicals at IPEN

The environmental monitoring program of no radioactive chemicals compounds, is carried out by measuring samples of ground water and waste water discharge in the influence area of IPEN. The aim of this monitoring is to provide Ipen of technical documentation necessary for accomplish environmental laws force and accomplish the demands of the TAC IBAMA referring to

their facilities, to:

- Assess the environmental impact from activities of IPEN;
- Demonstrate compliance of the procedures adopted in the release of liquid effluents with the authorized limits and the appropriate legal requirements;
- Maintain a continuous recording of the effects of plants on the levels of natural chemical compounds, in the influence region under the campus of IPEN;
- Detect any weaknesses and plan corrective measures;
- Providing information to the general public.



Figure 2. Effluents monitoring plant

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 CQMA has dedicated attention and research initiatives in accordance with this approach.

Biosorbents for the wastewater treatment

Adsorption techniques are recognised as effectiveness for the removal of heavy metal ions and dyes at trace quantities from wastewater. Activated carbon has undoubtedly been one of the most popular adsorbents employed in wastewater treatment throughout the world. But the process has not been used extensively due to high cost activated carbon. For that reason, non-conventional and low cost materials as sorbent to replace the costly activated carbon have been highlighted in recent years.

Biological resources, known as biomass, are available in large quantities and many theirs have been proven for its effectiveness for sorption. Different types of biomass have shown different levels of contaminant uptake so they have been considered biosorbents.

Biosorbents include by-products or waste materials from agriculture, food processing industries are abundant in nature and are classified as low-cost and non-conventional adsorbents. Sugarcane bagasse, coir pith, banana pith, coal fly ash and chitosan have

been investigated as alternative biosorbents to commercial adsorbents for the removal of U, Th, Pb, Zn, Ni, Cr ions and dyeing wastewater. Their biosorption has been studied by means of batch equilibrium, isotherm equilibrium kinetic and thermodynamic. The equilibrium data have been analyzed using the Langmuir and Freundlich isotherm models. In the greater number of cases, the Langmuir model was found to best describe the data. Generally, the biosorption is favoured by an increase of pH and is directly related with the concentration of biomass in the suspension until to reach the equilibrium.

Kinetic and thermodynamic investigations of sorption process and sorption data were fitted to different sorption isotherms using pseudo first-order and pseudo second-order models and Vant'Hoff equation. In all cases, the pseudo second-order model was found to explain the rapid kinetic of biosorption of metal ions and dyes and Gibbs free energy indicated the spontaneous nature of the adsorption processes. The major interesting of the studied biosorbent materials are high tolerance for organics and regeneration possible. Furthermore, they are abundant and inexpensive natural resources.

The dynamic sorption of Ni ions was verified using a column process with bagasse. Sorption breakthrough curve showed that bagasse is a good candidate for use in filters of the wastewater treatment containing trace amounts of Ni. The biomass allowed the reuse in three subsequent adsorption-desorption cycles. The research project of sugarcane bagasse and coir pith biosorbents was supported by Conselho Nacional de Desenvolvimento Científico e Tecnológico - CNPq in the period 2005-2007. The banana pith biosorbent work was awarded the Young Scientist Prize in 2007.

Among studied biosorbents, chitosan was largely comparable to that of the commercial adsorbent. Chitosan has demonstrated a versatility in its ability to bind both cations and anions. The experiments conducted in this research have shown the sugarcane bagasse, banana pith, coir pith and chitosan were efficient for removal of U, Th, Pb, Zn, Ni, Cr ions and reactive orange 16 dye. Therefore, they are potential biosorbents for the wastewater treatment. This is especially advantageous from environmental point of view because the biosorbents are wastes or subproducts available in large quantities that represent pollutants and hence their application to the treatment of wastewater is considered an economic sustainable activity.

Electrochemical techniques for the dissolution of metals and treatment of industrial and radioactive wastes

Electrochemistry is considered as an important partner in the treatment of wastes and other chemical processes due to its no pollutant characteristics. For this reason, electrochemical processes have been under study for industrial and radioactive waste treatment at CQMA. The applied areas can be resumed as follows:

- Treatment of radioactive (Cs) and industrial (Cr) wastes using electrochemical ion exchange (Figure 3);
- Separation of rare earths (Eu, Sm, etc.) (Figure 4);
- Recovery of pure Pb from RETOTER (Rare earths and thorium waste);
- Electrodissolution (or anodic dissolution) of aluminum presents in irradiated uranium silicide fuels and incorporation of the radioactive material in a vitreous matrix for final storage;
- Electrochemical synthesis of rare earth chromates for application as corrosion inhibitors



Figure 3. A 4-stage electrolytic cell with ion exchange electrodes used in the treatment of wastes containing Cs or Cr

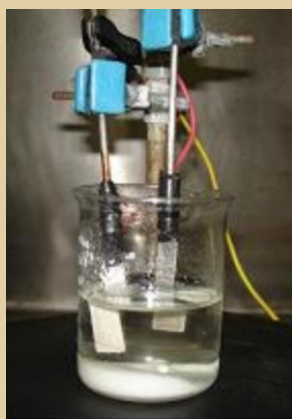


Figure 4. Electro-reduction/Precipitation of Sm from a RE concentrate containing Eu, Gd, Y, Nd and Tb

The mass spectrometry technique is a well known analytical tool to characterize many physical chemical samples. The scope of this research is to characterize chemically, contaminants and constituents on environmental samples using mass spectrometry techniques. The task is divided into three specific activities, one is the specific scientific research plan, second is students training to do research and the last one has the target to publish the research and apply the methodology developed into the environmental agency. The first task, concern about projects on mass spectrometry link to characterization of environmental contaminants. The second task, concern about 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 do routine analysis to the IPEN costumers. 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 of 2005 to 2007.

- Multiresidue method of benzimidazoles, carbamates and triazines pesticides in corn by liquid chromatography/Tandem mass spectrometry with electrospray ionization.
- Determination of chloramphenicol residues in industrialized milk and honey samples using LC-MS/MS.
- Residues determination study of glyphosate and aminomethylphosphonic acid (AMPA) in soybean and water samples using liquid chromatography coupled electrospray ionization tandem mass spectrometry (LC-ESI/MS/MS).
- Characterization of acrylate and methacrilate cross linked copolymer attained by photopolymerization for waveguide employment.
- Determination of hexachlorocyclohexane residues on blood plasma of exposed employees of the historical achieves of Joinville on 80's decade.
- Hydrogen characterization from methanol cracking for fuel cells / melting silicium oxides.
- Evaluation of the composition of the açai oil submitted to different doses of ionizing radiation.
- Characterization of the saturated and unsaturated fatty acids in Amazones oils: copaiba, buriti, maracuja, açai, and Para nuts by GC/MS.
- Direct and sensitive analysis of phenol and trichlorophenol by GC/MS.

- Diesel emissions significantly influence composition and mutagenicity of ambient particles: a case study in São Paulo, Brazil.

Synthesis and characterization of new rare earths compounds and their application as electrocatalysts in proton exchange membrane fuel cells (PEMFC)

The separation and purification of rare earths (RE) it is still today a matter of utmost concern. The technology and the procedures for the separation of macroscopic amounts of RE are always welcome. Brazil has a long tradition in rare earth technology, from monazite ores mining until the industrial separation and production of RE concentrates. These RE concentrates were studied and some high grade pure oxides were produced, like lanthanum, cerium, neodymium, samarium and yttrium, to be used as standards. The dissolution of the RE concentrates with ammonium carbonate was also studied and the resultants complexes treated with hydrogen peroxide for the precipitation of the corresponding peroxycarbonates. The behavior of the solubility of the RE and complex formation toward ammonium carbonate and the differential precipitation of the corresponding peroxides were investigated. The carbonate chemistry is of relevant importance to the rare earths from both analytical and industrial aspects. The RE were precipitated and dissolved by alkaline carbonate as well. The RE catalysts precursors thermally stable were synthesized by sol-gel method and characterized. An electrochemical method for the separation of europium and samarium from RE concentrates were developed. Praseodymium has been separated by ion exchange method and its purification is the final step. The preparation of nanoparticles of RE from high pure acetates are under study. This research is of great importance, since these materials will be applied as co-electrocatalysts in PEM fuel cells. The use of RE as co-electrocatalysts has been studied since 2005 in a project supported by CNPq.

Molten salt processes for waste treatment

The suitable final disposal of hazardous organic wastes such as PCBs (polychlorinated biphenyls), pesticides, herbicides, some organic radioactive wastes and hospital residues constitutes a serious problem. In some point of its lifecycle, these wastes should be destroyed, in reason of the risk that they represent for the human being,

animals and plants. The thermal decomposition has been used commercially in the waste disposal, mainly the incineration, whose most important characteristic is the combustion with flame. During the last three decades, incineration was usually a more practical and less expensive alternative. However, the conventional incineration, as way of destruction, presents some restrictions, due to the gaseous emissions eventually generated in the process. PCB-contaminated organic wastes are difficult to treat by incineration due to the formation of dioxins and furans, cancer-causing agents, at incineration temperatures, which are typically above 1200°C. An alternative to the incineration, for the treatment of a vast range of dangerous wastes, is the waste decomposition by molten salt oxidation. This process has several advantages over incineration. Molten salt oxidation is a thermal means of oxidizing (destroying) the organic constituents of mixed wastes while retaining inorganic and radioactive constituents in the salt. For this reason, 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 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. A special injection lance is used to introduce the organic waste and air into the bottom of the molten salt bed that is contained in the reactor vessel. 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 CO₂, and water. Due the flame absence, it is not considered an incineration process. Reactive species such as halogens (fluorine, chlorine, bromine, iodine), sulfur, phosphorous and arsenic, present in the organic waste, react with sodium of the molten salt to form the corresponding neutralized salts, such as, for instance, NaCl, NaF, NaBr, NaI, Na₂SO₄, Na₃PO₄, which are then trapped in the salt bath. Other non-oxidizable inorganic constituents, heavy metals, and radionuclides are captured in the salt, mainly as oxides, and most can be easily separated for disposal. The sodium carbonate can be reused after a dissolution-filtration recovery process and the salt can return to

the reactor vessel. In the figure 5, a schematic drawing of the molten salt process is presented.



Figure 5. Equipment scale

Microwave technology

The microwave technique has been studied all over the world to identify (qualitatively and quantitatively) and define the mechanism of microwave-material interaction, in rubbers devulcanization and in several types of petroleum hydrocracking and hydrotreating processes. In Brazil, the application of this process is a very recent field and has been studied as a new tool in materials processing, which uses high temperatures. 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's 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 does not require an appreciable amount of time to effect temperature changes such as the conventional methods and when the microwave device is turned on the effect of these electromagnetic waves is instantaneously

interrupted. Contracts between CENPES - Centro de Pesquisas da Petrobrás e IPEN:

- Project and Assembly of Microwave System Applied the Catalytic Reactors for Crude Oils Processing (Fev/2005-Fev/2007);
- Microwave Application in HDT and HCC Reactions: Batch studies and equipment construction for continuous operation (Jun/2007-Jun/2009);
- Project of Recovery of the Previously Laboratory for Zirconia Preparation at IPEN Amend building to attend Projects of PETROBRAS Interest in the future refinements Area (Set/2007- Ago/2008).

Petroleum or crude oil is a naturally occurring mixture of hydrocarbons and smaller amounts of organic compounds containing heteroatoms such as sulfur, oxygen, nitrogen, and metals. The petroleum products obtained from crude oil processing vary considerably, depending on market demand, crude oil quality and refinery objectives. In current industrial practices, crude oils are submitted to distillation under atmospheric pressure and under vacuum. The distillation fractions (including the residual fractions) undergo further catalytic refining processes so high-value products can be produced. The hydrogen content of petroleum products is an important index of their economic value. In conventional hydrocracking and hydrotreating processes, the hydrogenation reactions of aromatic compounds play a crucial role. Heavy residual compounds are normally aromatic in nature. The complete or partial saturation of these compounds by hydrogen addition is an important step in their cracking into smaller, more valuable compounds. Conventional heavy oil hydrocracking processes require relatively high temperature and very high pressure. In hydrotreating and hydroreforming processes, some catalysts become active only at the high temperature range. In order for reactions to take place at a favorable lower temperature range, metal catalysts are usually used to achieve good hydrogenation efficiency. Attempts have been made to find new classes of catalysts that would significantly lower the process parameters, while increasing the hydrogenation efficiency in terms of deep reduction of aromatic content. In another aspect, in Brazil microwave provides a new method of organic compound hydroprocessing. The method comprises the irradiation of hydrocarbons with a catalyst, by using high temperature and high pressure, having a reaction surface to produce a catalyst-organic compound mixture.

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Honor Mention and Awards

Prêmio Inventor 2007 Petrobras. Dispositivo de sistemas para processamento de cargas a altas temperaturas e pressão em presença de microondas. 30/11/2007 - Petrobras - RJ - Dra. Sumair G. Araujo e equipe.

3º lugar Prêmio Fernando Cerviño Lopes. Reciclagem de resíduos gerados em usina termelétrica para aplicação na remoção de corante em água. Sindicato dos Químicos, Químicos Industriais e Engenheiros Químicos do Estado de São Paulo - Dra. Denise Fungaro.

1º lugar categoria Graduado. Uso da casca de banana para o tratamento de efluentes radiotóxicos. XXII Prêmio Jovem Cientista - Gestão sustentável da biodiversidade: desafio do milênio - Boniolo, M.R.; Yamaura, M.

Prêmio Fernando Cerviño Lopes. Reciclagem de resíduos gerados em usina termelétrica para aplicação na remoção de corante em água. Sindicato dos Profissionais de Química do Estado de São Paulo - 2007.

Prêmio Internacional da Água e da Ciência. Simpósio de Água de Cannes, França, UNESCO; Universidade das Nações Unidas e Universidade de Nice Sophia Antipolis. Dra. Denise Fungaro.

Prêmio Polícia Cidadã da Fundação Sou da Paz, Governo do Estado de São Paulo e Rede Globo de Televisão. O resíduo gráfico azul. Coordenado por Regina Branco e realizado em conjunto com pesquisadores do Centro de Química e Meio Ambiente do IPEN, Instituto de Química da USP e Unicamp. Dra. Vera Salvador.

3º lugar Prêmio Fernando Cervino Lopes - Novas técnicas de reciclagem. Sistema de tratamento de efluentes e resíduos sólidos oriundos da indústria hidroletalúrgica e baterias exauridas. Sindicato dos Profissionais da Química do Estado de São Paulo - junho/2005 - Dra. Denise Fungaro, Dr. Alcídio Abrão, Fátima Maria.

Prêmio Bramex Ambiental 2005 - categoria Inovação. Contribuição à preservação ambiental em região de complexo carboelétrico. Câmara da Indústria, Comércio e Turismo Brasil-México - Dra. Denise Fungaro.

Prêmio Polícia Cidadã do Instituto Sou da Paz. Desenvolvimento de pesquisa que resultou na possibilidade de verificação científica da origem da maconha - 2005 - Dr. Jorge Sarkis.

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 established 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 few years ago the development of fuel cells for stationary and distributed energy applications. Previous studies were carried out at the Materials Research Center due to IPEN expertise on nuclear materials development. Based both on the good initial results and on the Brazilian Fuel Cell Program proposition by the Ministry of Science and Technology (MCT), IPEN decided to organize an institutional program on the subject. The objectives and goals of the IPEN program are based on the MCT national program.

In July 2003 the Fuel Cell Program was formally established at IPEN in order to contribute to the national development in this area. The Program was structured in a cross-cutting way involving human and infrastructure resources from IPEN Technical Departments. Three main areas were developed: PEMFC (Proton Exchange Membrane Fuel Cell); SOFC (Solid Oxide Fuel Cell); and REFORM (H₂ production from ethanol reforming).

More than 50 professionals were engaged at IPEN Program, although some in part time, including PhDs, MSc, and post-graduated students. Important scientific and technological results have been obtained and the main achievements can be observed by the submitted patents, the published international papers, the post-graduated courses given, and the graduate student's thesis advisory.

In October 2003, we organized the first IPEN Fuel Cell Meeting with participation of more than 150 attendees from IPEN and from all over the country. It was considered a very important event in the national scenario for the fuel cell development. In 2005 and 2007 the second and third IPEN Fuel Cell Meeting were realized with the same success as the first. In 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 1 kW of power. In the period of 2005-2007 news laboratories of SOFC, Hydrogen and Fuel Cell Systems have been implemented.

Due to the positive results obtained by the IPEN Fuel Cell Program, in 2007 a new center called Fuel Cell and Hydrogen Center was created. The financial resources were based on scientific funds from federal and state government (FINEP-MCT- ProH₂, FAPESP, CNPq and CAPES). Due to the good developments achieved, IPEN is considered as an important partner within the R&D networks established by the MCT-ProH₂ Program. Partnership with emerging enterprises from CIETEC (Incubator Center) led to advances and autonomous technological domain in PEMFC area.

Proton Exchange Membrane Fuel Cell (PEMFC)

The Proton Exchange Membrane Fuel Cell (PEMFC) Group aims the basic and technological developments of low power fuel cell for the direct oxidation of hydrogen, hydrogen/CO mixtures and alcohols (methanol, ethanol and ethylene glycol) concerning stationary applications, that means, distributed electric power generation. These includes: development of new methods of electrocatalyst production and new electrocatalyst formulations; development and production of new membranes (electrolytes) alternative to Nafion for special applications (Nafion-like) and High-Temperature operation (130°C, composites); Membrane-Electrode-Assembly (MEA) production and optimization; unit cells tests in laboratory and pilot scales; development of low power fuel cell stacks; and finally education in related fields. Highlights 2005-2007:

- Development of new methods of electrocatalysts (carbon-supported Pt-based nanoparticles) preparation: radiolysis process (gamma and electron beam irradiation) and hydrothermal carbonization.
- Development of new electrocatalyst formulation PtSnNi/C for ethanol electron-oxidation, among others.
- Development of modified carbon supports with polymeric chains containing sulfonic groups that improves the three boundary phase of the fuel cell electrodes.
- Development of Nafion-Titanate Nanotube and Nafion-TiO₂ Composite Membranes for PEMFC Operating at High Temperatures.
- Development of Membranes Nafion-like.
- Development of Membrane-Electrode-Assembly (MEA) by sieve printing method and preparation of MEA with 144 cm² of electrode area (FIG. 1).
- Modelling and Simulation of Fuel Cell components (bipolar plates) using CFD techniques.
- Development of a fuel cell stack of 1 kW.



Figure 1. Sieve-printing apparatus for Membrane-Electrode-Assembly (MEA) production

Solid Oxide Fuel Cell (SOFC)

Solid oxide fuel cells are considered as promising potential electrical energy sources owing to their efficiency and release of environment friendly gaseous end products. Essentially, SOFC 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. Thermal and chemical compatibility, good electrochemical and mechanical properties, and thermal stability are key issues regarding this technology. One of the most important tasks in SOFC research is the reduction of operating temperature from 800-1000°C down to 500-800°C, to reduce degradation of cell components, improve flexibility in cell design, and lower the material and manufacturing cost by the use of cheap and readily available materials. Among the various designs of fuel cells, the planar type is claimed to have the advantages of high power density per unit volume and low production costs. The research activities at IPEN are primarily concerned with the development of the SOFC materials, aiming to the use of simple and low-cost methods for high-performance SOFC components. The activities of the SOFC research group at IPEN have been focused on the synthesis, processing, and characterization of the SOFC components. Along with the traditional solid oxide reaction, several different chemical techniques have been used for the synthesis of ceramic powders. The methods chosen for processing cells are largely dependent on the materials configurations as well as on macroscopic properties desired and their microstructural appearance. In order to improve the fracture toughness of cubic stabilized zirconia electrolyte, without compromising its electrical properties, composites containing up to 40 wt % of alumina, in a 9 mol % yttria-stabilized zirconia (9Y-CSZ) powders were synthesized by coprecipitation route and ceramic samples were prepared by pressing and sintering. SEM micrograph of sintered ceramic is shown in (FIG. 2). Studies about conditioning of YSZ powders have been realized to investigate effect of temperature and time of calcinations step and high energy ball milling on the physical characteristics of YSZ powders. The perovskites oxides $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_3$ and $\text{Nd}_{x-1}\text{Sr}_x\text{MnO}_3$ ($x=0,10,0,30,0,50$); (FIG. 3), have been prepared by the polymeric precursor technique and traditional solid oxide reaction, respectively, as a cathode materials for SOFC operating at intermediate temperatures (500-800°C). The Strontium-substituted lanthanum manganite/yttria-

-stabilized zirconia (LSM/YSZ) composite samples were prepared for electrochemical performance of SOFC cathode to increase the three-phase boundary (TBP) sites.

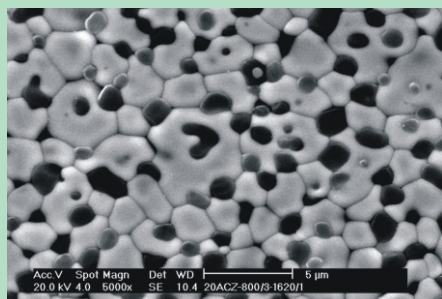


Figure 2. SEM micrograph of sintered zirconia alumina electrolyte

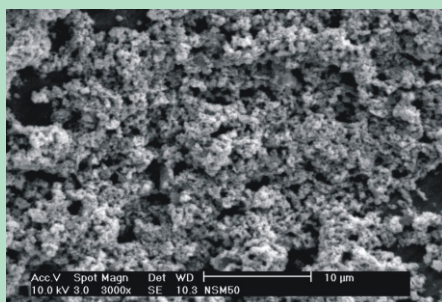


Figure 3. Scanning electron micrograph of the $\text{Nd}_{0.65}\text{Sr}_{0.35}\text{MnO}_3$ powders

Nickel oxide-yttria stabilized zirconia (8YSZ-NiO) anode materials were synthesized by combustion process involving metal nitrate-urea decomposition and by coprecipitation. The microstructural changes of the 8YSZ-NiO to occur in this composite when submitted to the reduction in tubular oven 800°C for periods between 5 minutes and 3 hours in controlled atmosphere with a mixture of 4% of hydrogen and 96% argon in atmospheric pressure. The chemical route has been also applied to the synthesis and characterization of zirconium titanate composites. Mechanical alloying processing has been investigated as a new SOFC anode preparation route. This method employs metallic Ni and YSZ co-milled powders leading to refined structures of dispersed constituent, which can improve the anode properties. Compacted powder sintering temperatures are reduced. The method can be designed with fewer processing steps. Thin films (green state of 500 microns) by tape casting process were attained as result of the rheological behavior studies of lanthanum chromite powders synthesized in our laboratories (FIG. 4). Main application of this material is for interconnect devices. A laboratory setup was designed and put into operation for the development of SOFC. The whole project consisted of the preparation of the component materials: anode, cathode and electrolyte, and the buildup of a hydrogen leaking-free sample chamber with platinum leads

and current collectors for measuring the electrochemical properties of single SOFC. Several anode-supported single SOFCs of the type $(\text{ZrO}_2\text{:Y}_2\text{O}_3 + \text{NiO})$ thick anode/ $(\text{ZrO}_2\text{:Y}_2\text{O}_3)$ thin electrolyte/ $(\text{La}_{0.65}\text{Sr}_{0.35}\text{MnO}_3 + \text{ZrO}_2\text{:Y}_2\text{O}_3)$ thin cathode have been prepared and tested at 700 and 800°C after in situ H_2 anode reduction. The main results show that the slurry-coating method resulted in single-cells with good reproducibility and reasonable performance, suggesting that this method can be considered for fabrication of SOFCs. The wet powder spraying technique was also used for fabrication tests in LSM/YSZ interfaces.

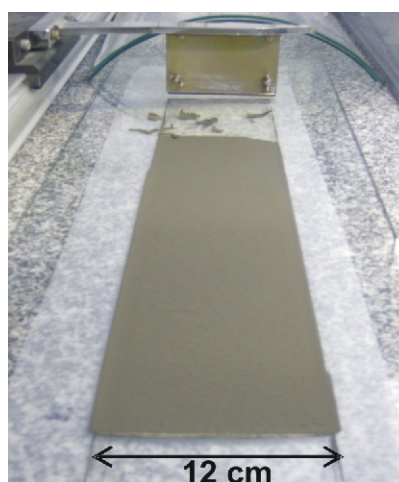


Figure 4. Tape of Lanthanum Chromite attained by tape casting process for SOFC interconnector application

Hydrogen production

The biohydrogen can be withdrawn from various types of biomass as the organic municipal waste, sewage, animal excrement, tailings from forestry and agriculture. The great motivation for the development of hydrogen obtaining procedures, for use in fuel cells is to accelerate the learning of this technology to have a strong environmental character. In Brazil, particularly, there is a necessity for a national technology development based on bioethanol, in our particular market, which has different characteristics from other countries. Bioethanol is a liquid fuel, produced from renewable sources, considered less toxic than methanol and is handled, stored and transported easily. Brazil has the infrastructure for production and distribution of bioethanol throughout the national territory. The Hydrogen Group has been developed catalysts for ethanol steam reforming based on zirconia microspheres containing transition metals prepared by the sol-gel method. The catalysts exhibited good performances for hydrogen production. Researches on membrane reformers to obtain pure hydrogen from the mixture generated in the reforming process have been investigated as

well the development of reactors for preferential oxidation and shift reaction. In 2006 was started up the research on the use of metal or ceramic membranes covered with palladium (FIG. 5), which will be integrated with a hydrogen reformer. In this process the reforming reaction and purification occur in a single stage (membrane reactor). The metallic membranes, palladium or their alloys especially, presenting itself as the most promising for the selective separation of hydrogen from reforming gases. The Hydrogen Group has conducted searches for obtaining these membranes with different substrates and recoveries with palladium.

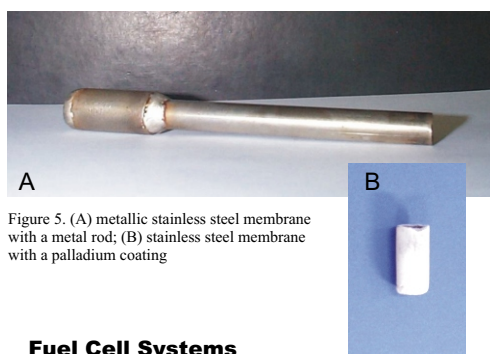


Figure 5. (A) metallic stainless steel membrane with a metal rod; (B) stainless steel membrane with a palladium coating

Fuel Cell Systems

The main objective of the group is to perform research, development, tests, certification of fuel cells and energy systems integration based on Fuel Cell technology. The Fuel Cell Systems Laboratory has been implemented and the following equipments and resources are installed:

- 50 kW_e PEMFC Module with 3 stacks. (FIG. 6)
- PEMFC Life Cycle Test Stand. (FIG. 7)
- Electrolyzer for hydrogen and oxygen generation with H₂ production capacity of 10.000 lt/h and O₂ production capacity of 5.000 lt/h both at 4 bar operation pressure.
- Gas piping line for hydrogen, oxygen and nitrogen. This piping line feeds the 50 kW_e PEM Fuel Cell Module and the PEM Fuel Cell Life Cycle Test Stand.



Figure 6. 50 kW_e PEMFC module



Figure 7. PEMFC life cycle test stand

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Honor Mention and Awards

Honor mention to the work "Direct ethanol fuel cell" presented at XIV Congresso e Exposição Internacional de Tecnologia da Mobilidade SAE BRASIL, São Paulo, 2005

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Introduction

The focus of the Materials and Nanotechnology Program is materials technology development related to processing, analysis, testing and characterization, in general. These are achieved through execution of R&D projects in materials science and engineering as well as collaborative projects with private / public sector companies, universities and other research institutions.

Besides technology development, one other objective of this Program is modernization of existing facilities to be more competitive and to explore new opportunities. This Program also fosters training and human resource development in relevant areas in association with universities and specific industrial sectors.

The Materials and Nanotechnology Program is divided into sub-programs in the following areas: Ceramic Materials, Composite Materials, Metallic Materials, Physical / Chemical Characterization and Nanomaterials. The sub-programs are further divided into broad topics in research, development and innovation. Within each topic, several R&D projects are carried out.

Highlights of this program include:

- Calcium phosphate based macro-porous ceramics;
- Glass matrices to contain industrial and radioactive wastes;
- Glass microspheres for radiotherapy;
- Porous biomedical alloys;
- Processes to recycle industrial wastes into chemically resistant silicate glass based ceramics;
- Solid oxide fuel cell materials, components and unit cells to produce energy from different fuel sources;
- Proton conductors like barium cerate;
- Titanium and strontium based ceramics as oxygen sensors;
- High temperature erosion-oxidation resistant nanostructured coatings;
- Chromium-free corrosion resistant coatings for aluminum alloys;
- Thin film nanocrystalline structures, nanowires and nanotubes;
- Intermetallic compounds by combustion synthesis;
- Sintered components for use as cutting tools and filters;
- New classes of permanent magnets;
- Environment friendly nickel-metal hydride batteries;
- Sintered parts for the automotive industry;
- Processes to recycle zirconium alloy machining chips;
- X-ray diffraction reference materials;
- Residual stress measurement techniques;
- Copper based electrical components;
- Cyclic and monotonic testing of materials.

High temperature degradation resistant materials and coatings

The high temperature erosion-oxidation (E-O) behavior of several alloys and coatings have been studied in a E-O test rig. Erosion-oxidation maps of many coatings and alloys have been plotted to aid in material selection and include; (a) steels like AISI 1020, 410, 304 and 310; (b) HVOF coatings of Ni20Cr, Cr₃C₂ and WC-Co. Nanocrystalline coatings of a variety of oxides of rare earths (Dy, Er, Ce, Sm, Y, La, Pr and Nd) have been prepared to improve high temperature oxidation resistance of chromium dioxide and alumina forming alloys. Other nanostructured coatings have been prepared and studied. These include titanium dioxide nanotubes and HVOF deposited Cr₃C₂-Ni20Cr and WC-Co.

Corrosion resistant coatings

The automobile and aeronautics industries currently use conversion coatings for corrosion protection of steels and aluminum alloys. There are increasing restrictions to the use of these coatings due to toxicity. Hence, other alternate surface treatments are being tested and these include the use of self-organized monolayers - SAM. The surface treatment of aluminium alloys using this chrome-free SAM process is presently being studied.

Corrosion behavior of biomaterials

The corrosion behavior of metallic materials for use as implants was studied. The corrosion resistance of different alloys and coatings are being investigated and these include: Ti alloys, AISI 316 L stainless steels (with and without coatings), special austenitic stainless steels with high N content, PVD coatings and bioceramic coatings obtained by ion beam deposition.

Surface treatments for protecting carbon steels

The effect of modifications to the phosphating process to obtain coatings with improved corrosion resistance is being investigated. Also, currently under study are corrosion inhibitors for carbon steels used as reinforcement in concrete structures.

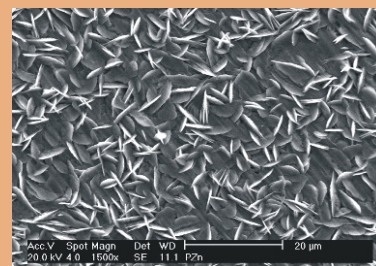


Figure 1. Scanning electron micrograph showing the phosphate layer obtained by surface treatment to protect carbon steels

Materials and technologies for a self-sustained environment

The main activities are related to the use materials science and technology for industrial process development with the aim of solving final waste problems. These include production control, inertizing, recycling or incorporation of industrial wastes in materials with increased economic value. The focus of the study included several industrial wastes like slags from metallurgical industries, residues from aluminum extraction plants, construction and demolition wastes. One other study focused on the recovery of platinum from fuel cells. Green stoneware tiles have been produced by incorporating galvanic wastes in industrial mixes containing kaolin clay, feldspar, quartz and recycled domestic glass. The process that was developed allows the incorporation of up to 20 mass% of metal waste for effective immobilization in a porcelain matrix and it also contributed towards coloring the matrix. The material obtained revealed a high chemical resistance. The microstructure observed in the FESEM micrographs was obtained after chemical etching and the crystalline phases contract from the glass matrix. In another study, the effect of the presence of white dross residue (WDR), produced during aluminum recovery from slag in the formation of glass-ceramic frits used in ceramic glazes was determined. Fig. 2.



Figure 2. Fritted glass produced with 30 mass% WDR

Applied crystallography

Standard materials were developed for use as references in the calibration and determination of instrumental parameters of equipments used in diffraction (conventional X-rays, synchrotron and neutrons). The following standard materials for diffraction were produced: silicon (Si), yttria (Y_2O_3), lanthanum hexaboride (LaB_6), α -alumina (Al_2O_3), ceria (CeO_2), hydroxyapatite, silicon nitride and others. These are currently being evaluated for use for SAXS, XAFS and for in situ measurements under high hydrostatic pressures. The first set of samples were tested in other institutions and at Brazilian universities and these have shown excellent results.

Biomaterials

Research and development is being carried out on several general topics and these include bio-inert, bio-active or re-absorbable ceramics and new biomedical alloys. Nanometric hydroxyapatite powders, tricalcium beta-phosphate were developed by the neutralization method with variation of several parameters and using ultrasound. This led to obtaining nanometric materials and the incorporation of cations and anions in the crystalline structure of the materials. The repair of bone defects was evaluated with these materials by conducting in-vivo tests in *Rattus norvegicus*. Histological evaluation indicated that bone repair was successful. Calcium phosphate ceramics were obtained either in the dense or macroporous form. The latter was obtained by the direct consolidation method using albumin. (Fig. 3) In-vitro tests of these materials were conducted. Adhesion and cellular proliferation as well as initial biocompatibility were observed. The anchoring behavior and proliferation of fibroblasts was investigated on the surface of the ceramic specimens prepared with different surface treatments. The dissolution behavior of calcium phosphate based ceramics with different microstructures in simulated body fluid - SBF medium and a cell culture medium (DMEM) has been studied. The reactivity in SBF medium was investigated and the biocompatibility in culture medium with osteoblasts obtained from the bones of newly born mice. The in-vitro results indicated that the materials were biocompatible and did not reveal cellular death, or drastic morphological alterations that could compromise the metabolism of the cells.

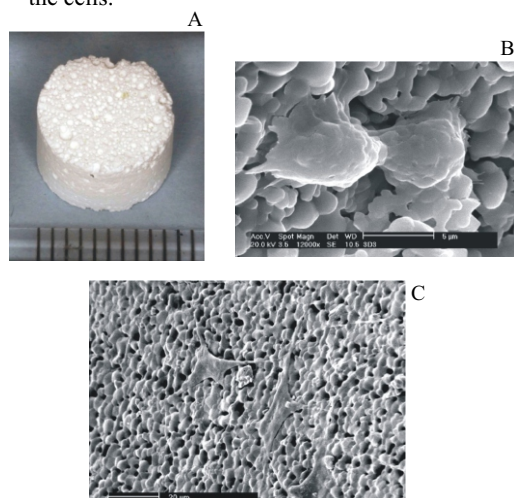


Figure 3. Scanning electron micrographs showing: (A) a macro-porous calcium phosphate based ceramic obtained using the albumin consolidation method; (B) osteoblasts originated in in-vitro culture test; (C) fibroblasts adhering to the ceramic surface in in-vitro test

Biomedical alloys

Ti13Nb13Zr alloy implants were obtained by the hydride route via powder metallurgy. The implants were then sterilized and surgically placed in the central region of rabbit's tibiae. Two double fluorescent markers were included and after an 8-week healing period, the implants were retrieved, non-decalcified sectioned and studied. The results indicated biocompatibility and osteoconductive properties of Ti13Nb13Zr processed by the hydride powder route.

Glasses

Glasses are being developed to immobilize industrial and nuclear wastes, to produce microspheres for radiotherapy treatments, and for other optical, electrical, and mechanical applications. A mathematical model using the Monte Carlo Method is being developed to determine the dose distribution in a human liver when neutron activated glass microspheres are directly injected in the organ through an artery. The purpose is to develop a fast, precise, and safe way to eliminate tumors in the liver.

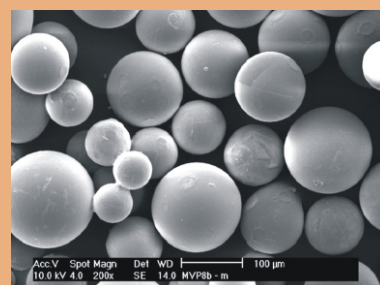


Figure 4. Glass microspheres containing phosphorous

Synthesis, processing, production and testing of SOFC unit cells

The properties of ceramic materials are strongly influenced by the chemical and physical characteristics of raw powders, such as particle size, surface area, chemical homogeneity and extent of agglomeration. Preparation of zirconia, rare earths, titania, alumina and silica based ceramics powders was done using various routes like powder mixture, co-precipitation, modified Pechini method, hydrothermal treatment and combustion synthesis. A scanning electron micrograph (SEM) of ZrO_2/NiO powder synthesized by co-precipitation is shown in Fig. 5a. Processing of high performance ceramic materials from the powders described above are done by milling,

classification and forming of ceramic bodies using techniques such as uniaxial and isostatic pressing, slip casting, tape casting, electrophoretic deposition and wet powder spraying. A SEM of the fracture surface of the electrophoretically deposited yttria stabilized zirconia (YSZ) layer on YSZ/Ni substrate is shown in Fig. 5b. and Fig. 5c a SEM of the cross section of strontium doped lanthanum manganite (LSM) deposited by wet powder spraying on YSZ electrolyte.

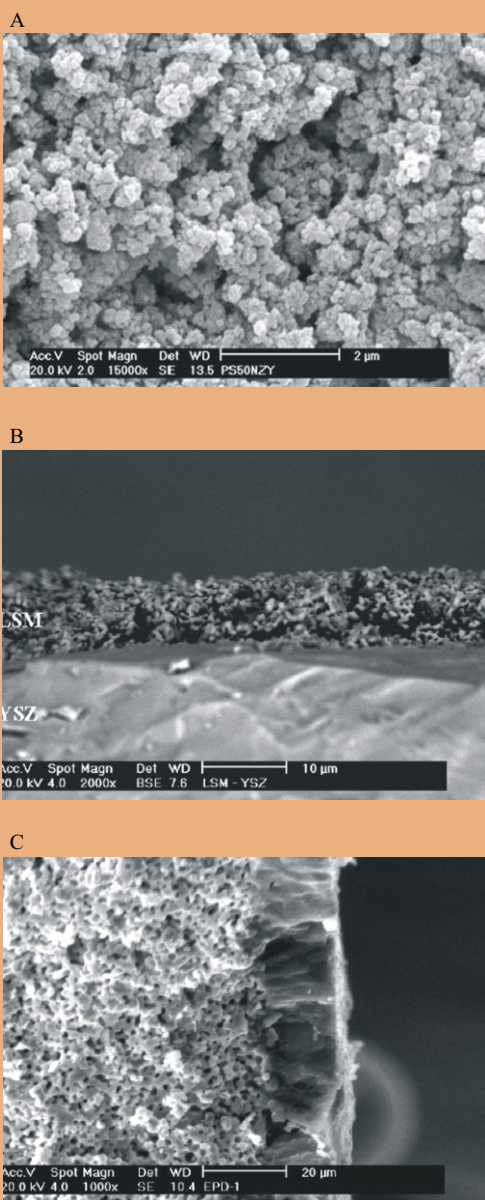


Figure 5. (A) ZrO_2/NiO ceramic powder synthesized by co-precipitation; (B) Electrophoretically deposited YSZ layer on ZrO_2/Ni substrate; (C): LSM layer wet powder sprayed on ZrO_2/Ni substrate

Electroceramics

Basic research work is currently focused on zirconia/ceria based ionic conductors and cerate / zirconate based proton conductors, particularly for the development of solid oxide fuel cells and oxygen sensors. The synthesis of ceramic nanopowders is carried out by several techniques: co-precipitation, using polymeric precursor, combustion synthesis and with 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 properties. Electrical measurements are done using impedance spectroscopy at a wide range of temperatures (LNT-1500 K) and oxygen partial pressures (10 ppm-1 atm). Microstructure evaluation is carried out using x-ray diffraction analysis, scanning and transmission electron microscopy and Raman spectroscopy. Current technology development work consists of: (a) operation of solid oxide fuel unit-cells with proton and oxide ion conductors under hydrogen, methane and ethanol; (b) testing oxygen lambda sensors. In the period 2005-2007, the main results were:

- Development and testing of anode-supported solid oxide fuel cells with slurry-coated electrolyte and cathode under ethanol and hydrogen;
- Impedance spectroscopic evaluation of sintering of zirconia-yttria ceramics;
- 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.

Ceramics

Advanced materials have tailored microstructures for specific applications in high technology industries. Among the many applications of advanced ceramics, cutting tools are prominent and these require specific properties such as high chemical stability, thermal shock resistance, high hardness and wear as well as tear resistance. Among the ceramic materials that have been developed, the more important ones are those based on silicon nitride, silicon carbide, zirconia and alumina. These ceramic materials are processed by different routes to achieve high relative densities and optimized microstructures to increase fracture toughness, a property considered important for the ceramic materials. When

Alumina based composites, with niobium carbide additions had their performance evaluated during machining of vermicular cast iron, the ceramic tools suffered abrasion and adherence, but presented acceptable performance under operating conditions. Acid attack of Si_3N_4 ceramics was studied to evaluate their potential use in aggressive atmospheres. Inorganic polymers play an important role in the processing of ceramic materials, either as an additive for forming parts or as a ceramic precursor. Polymer precursors enable, with appropriate treatments, different types of ceramics to be obtained and from which the removal of the precursor is not essential. The effect of adding polysiloxane as a precursor on the microstructure of alumina has been studied. The preparation of ceramic composites using small amounts of polymeric precursors indicated this method to be a simple and viable route to produce pieces containing refractory phases and with complex geometries. Fig. 6.

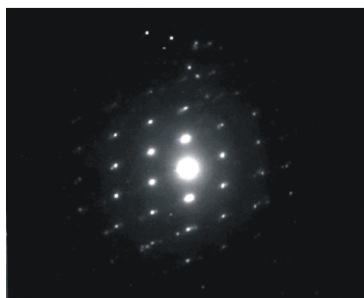


Figure 6. Electron diffraction pattern of the phase $\text{Si}_3\text{Al}_2\text{O}_5\text{N}_4$

Micro and nanostructured coatings

A metal-organic chemical vapor deposition (MOCVD) system was installed to develop micro and nanostructured coatings. The MOCVD technique is an extension of the CVD technique and enables the deposition of several metals and ceramics. Currently this system is being used to: (a) develop nanocrystalline deposits of TiO_2/TiNO and of Y_2O_3 on mono and polycrystalline substrates; (b) prepare nanostructured fine films and three-dimensional nanocrystalline structures such as nanofibres and nanotubes.

Synthesis using mechanoactivated combustion

The purpose of this project is to verify the influence of high energy milling on combustion synthesis of intermetallic compositions such as niobium tri-aluminide, NbAl_3 . Milling is used to prepare mixtures of the powders of the constituent elements of the intermetallic compound, mainly to

promote an intimate mixture and to reduce the particle size of the reactants (mechanical activation). The combustion is accomplished in compacted tablets of the mixture previously processed by grinding. Funding obtained enabled improvements in existing infrastructure, mainly to expedite characterization of the processed materials and for the construction and operation of a system to study hydrogenation/dehydrogenation of materials.

Polymer matrix composites

One of the projects in this area is related to the construction of a hybrid composite material tube with carbon and glass fibers, for use as a prosthetic extension of lower limbs and to be eventually manufactured for the Association for the Assistance of the Impaired Children - AACD. The aim was to evaluate and optimize cost and to reduce weight. Finite element modeling was used to optimize the component in terms of bio-mechanical functions required of a prosthesis. This modeling took into consideration the amount of material, number of layers as well as other requirements to minimize cost. A survey resulted in finding a cheaper route to manufacture the composite using the vacuum assisted resin transfer molding (VARTM) process and pre-forms of glass with carbon. A patent application has been submitted. In order to compare the quality of the composite tube, another tube was produced using the conventional filament-winding process. Currently the VARTM process relies on imported feedstock. Even though some of the properties of the VARTM composite tube require further improvement, overall it presented advantages such as weight reduction and increased impact absorption. (Fig. 7)

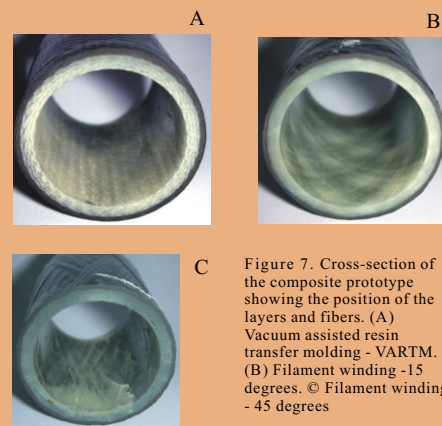


Figure 7. Cross-section of the composite prototype showing the position of the layers and fibers. (A) Vacuum assisted resin transfer molding - VARTM. (B) Filament winding -15 degrees. (C) Filament winding - 45 degrees

Powder metallurgy

Research and development of powder processing techniques. This involves support services for research and technology development in collaboration with industries, universities and other research institutions. Sintered components have been developed for applications as cutting tools, AISI M2 and T15 high-speed steels and filters. Samples of compacted stainless steel plates (80 x 140 mm and 2.0 mm thick) were prepared in collaboration with the company BRATS Special Materials.

Magnetic materials

R&D activities related to processing of metallic materials with rare earths using hydrogen technology and the powder metallurgy route. The microstructure and properties of magnetic alloys based on Sm-Co, Pr-Fe-B and Nd-Fe-B were studied. Nickel-metal-hydride based batteries (less aggressive to the environment than the lithium-cadmium batteries) are being developed with the alloys: La-Ni, La-Mg, La-Pr-Ni-Mg. Other activities include: processing of biomaterials with Y- or Nb-Zr alloys; development of uranium- molybdenum alloy powders for use in the nuclear area.

Recycling of Zircaloy

The production of fuel elements for the Brazilian Angra -I and -II PWR reactors involves machining of zircaloy tubes. A considerable amount of machining scrap are produced and since Brazil imports zircaloy a project to recycle zircaloy machining scrap was started. This project envisages development of the process to reuse Zircaloy scrap and consists of: adaptation of the vacuum arc remelt (VAR) furnace, melting, remelting or refining of consumable zircaloy electrodes using a non-transferable electric arc.



Figure 8. Macrograph showing a compact of stainless steel scrap and its consolidation in the VAR furnace

High-pressure cells

The purpose of this project is the specification and construction of a pressure cell for use in the Brazilian Synchrotron Light Laboratory - LNLS and in the IPEN neutron diffractometer. The objective was to initiate studies using the neutron diffractometer that is presently in the testing stage and for use eventually during the second semester of 2008. The design and construction of the pressure cell were based on the Paris-Edinburgh cell design. However, the new cell is lighter and more compact. As a consequence, the cell operates at lower pressures of up to 2 GPa. The design incorporates new materials that were developed at IPEN. A patent application for the design of the cell has been made. That cell will operate in the neutron diffraction facility installed in the IPEN research reactor, IEA-R1. The purpose is to provide the users the possibility of conducting neutron diffraction measurements on samples submitted to external hydrostatic pressures of up to 2 GPa.

The nanotechnology has been pointed as a high innovative technology allowing a deep and wide change in the materials production application. The molecular planning atomic in an atomic scale is a new technology concept starting that allows a new functionality of the matter. This leads to the control of the physical, optical, electronics, 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 it will increase in a short time. That new technology concept has been at development at CQMA, by the study of nanostructured functional materials. The following activities are in development at IPEN:

Lanthanides supermolecules as fluoroimmunoassays agent for disease detection. Prostate Specific Antigen (PSA)

Prostate cancer is the most common malignant tumor in men, corresponding to approximately 27% of all male cancer. In this work it is studied the use of lanthanide - diketone supermolecules with macrocyclics as markers for prostate specific antigen (PSA) detection in serum sample. Most specifically it was studied the interaction, supermolecules/antigen/antibody by electronic spectroscopy (fluorescence/absorption) using compounds based on lanthanides as biological markers. It was made a fluoroimmunologic assay using these compounds that showed an efficient luminescence and physical chemical properties for acting as luminescent probe. In this method, the luminescent probe is chemically conjugated to an antibody, which link in a specific way with the biomolecule or organism. The luminescent probe is sensible to the chemical environment and its luminescence can indicate the presence of the biomolecule or organism. The supermolecules that showed high stability were tested as fluoroimmunoassay markers for tests of PSA in serum. It was established a routine methodology for PSA detection aiming to implantation of this kit in the Brazilian market. This work is supported by CNPQ-RENAMI and FAPESP agencies.

Magnetic nanoparticles and their application in biotechnology. Synthesis and characterization of MnFe_2O_4

Magnetic nanoparticles are of great technological importance because of their use in magnetic fluid, information storage system, medical diagnostics, etc. Various preparation techniques have been used for the synthesis of fine particles of ferrites, which exhibit novel properties when compared to their properties in bulk. Nonconventional methods such as co-precipitation, thermal decomposition, solgel and hydrothermal methods have been widely used. Ultra fine ferrite particles can be prepared by the simple chemical co-precipitation method. In this field spinel ferrites have received considerable attention because their magnetic properties can be varied systematically by changing the identity of the divalent Me^{2+} cations ($\text{Me} = \text{Co}, \text{Mn}, \text{Zn}, \text{etc.}$) without changing the spinel crystal structure. It was synthesized and characterized magnetic nanoparticles of manganese ferrites and this nanoparticles doped with gadolinium and covered with a biopolymer, chitosan for biological application. Some variable, as: pH of precipitation (11, 12 and 13), type of alkaline solution (NH_4OH , KOH , LiOH and NaOH), molar concentration of the solution, annealing time (0, 1 and 2 hours), the addition of surfactant in the solution of metals, gadolinium percent doped (1, 3, 5, 7 and 10% molar) were studied.



Figure 9. Magnetic nanomaterials

Metals recognition by smart supported liquid membranes (SLM)

Liquid membranes have been an emerging technique used as an alternative for the separation and pre-concentration of several metallic species. It have been applied as one alternative technique in the separation of metallic ions originating from several areas of industry like the hydrometallurgical, gases separation, biotechnology, treatment of nuclear and non-nuclear waste. The recovery of those metals through that technique has good potential to reduce, in very low levels, pollutants in processes. The liquid membrane is a solution, that is an organic solvent, water immiscible and with low dielectric constant, that is used as a diluent for an extractor agent, also called the sequestering agent, loader or metal transport, that is absorbed in the microporous of a polymeric film and actuates as solid support of the liquid membrane. The extractor agent gets the metallic ion from the feeding solution, liberating it on the other side of the membrane, through stripping solution. Then, the extraction and stripping of the metallic species are carried out in a single step in the process, and has this advantage in relation to the liquid-liquid extraction that requests a large number of stages to obtain products with high purity. It was investigate the influence of several parameters in the process of extraction of lanthanide metals and uranium originating from a simulated radioactive waste using as polymeric support membranes, PTFE and as loader calixarenes. The calixarenes are part of a very versatile class of macrocyclic compounds that can be functionalised, being obtained like this a great variety of multifunctional receptors.

Materials based on uranyl ion and their potential for solar energy conversion cells

The search for renewable sources of energy has led to an increasing interest in photochemical cells because of their possible role as transducers of solar to electrical energy. On the other hand uranyl compounds (UO_2^{2+}) present a great potential as luminescent materials, for instance, applied in technology laser, luminescent probes, cells for conversion of energy, etc. In this work it was studied two compounds of UO_2^{2+} based on methanesulphonate and calixarenes for to be used as efficient Light Conversion Molecular Devices (LCMD) and/or in solar cells for energy conversion. The uranyl ion possess some properties which make it a potential component for a solar energy conversion, that is, absorbs light in the shorter wavelength range of the solar spectrum, producing a relatively long lived excited state. The excited state has fluorescence peaking at ~ 520 nm, a property which makes it relatively convenient to investigate its reaction.

The redox potential of the excited uranyl ion makes a powerful oxidizing agent. This may be of potential use in the photogeneration of oxygen, which is of great importance for the photocleavage of water. Up to now we have investigated the electronic properties of some uranyl compounds (UO_2^{2+}).

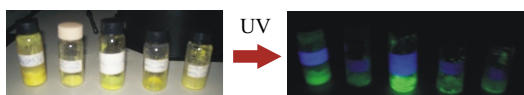


Figure 10. Uranyl Complexes as dye sensitized solar cells (DSSCs)

Preparation and spectroscopic study of nanoparticles of rare earth for biolabeling

The use of nanoparticles as labels in place of conventional molecular fluorophores has improved the biomolecular detection in terms of sensitivity and selectivity. A microwave method has demonstrated good results for preparing inorganic phosphor nanoparticle labels. The capping process adds a silane layers to the surface of the $\text{Y}_2\text{O}_3:\text{RE}^{3+}$ (RE=Rare Earth) particles and provides amine groups for biological conjugation. $\text{Y}_2\text{O}_3:\text{RE}^{3+}$ materials act as an inorganic phosphors with a spectrally narrow emission and a long emission lifetime. $\text{Y}_2\text{O}_3:\text{RE}^{3+}$ phosphors are prepared by Pechini and combustion methods and functionalized with 3-aminopropyltrimethoxysilane (APTMS) using microwave technique. Determination of amino groups in $\text{Y}_2\text{O}_3:\text{RE}^{3+}$ system is performed using a method involving ninhydrin. Luminescent properties of functionalized and unfunctionalized compounds are very similar.

Luminescence of natural polymeric magnetic films based on chitosan and lanthanide β -diketonate compounds

Chitosan is the deacetylated form of chitin, which is a linear polymer of acetyl amino d-glucose. Recently, chitosan that is used as an adsorbent has drawn attentions due to its high contents of amino and hydroxy functional groups showing high potentials of the adsorption of dyes, metal ions and proteins. Other useful features of chitosan include its abundance, non-toxicity, hydrophilicity, biocompatibility, biodegradability and anti-bacterial property. In this work we investigate the properties of a film based on chitosan doped with β -diketonate of europium. These films are candidates to be used as biosensor or in displays. The chitosan films were synthesized using a solution of chitosan in acetic acid and an alcoholic solution of $\text{Eu}(\text{TTA})_2\cdot 2\text{H}_2\text{O}$ in weight proportion (0.1%). For the magnetic film we use a manganese ferrite dispersed into the chitosan solution. The compounds were characterized by: thermogravimetry (TG),

differential scanning calorimetry (DSC) and infrared spectroscopy. The emission spectra of the Eu^{3+} -TTA complex doped in the chitosan films recorded at 298 and 77K exhibited the characteristic bands arising from the $^5\text{D}_0\text{-}^7\text{F}_j$ transitions ($J = 0, 2, 4$). The experimental intensity parameter, Ω_2 ; (34, 24) for non magnetic and magnetic films respectively, indicated that the Eu^{3+} ion in the precursor complex is in a more polarizable chemical environment than in the magnetic and non magnetic doped films. The emitter $^5\text{D}_0$ level lifetimes for doped samples show the luminescence decay curve profiles of second order exponentials. It was observed suppression of luminescence for the material with ferrite nanoparticles. The emission quantum efficiency (η) equal to 35.3 and 13.11 for the non magnetic and magnetic films is also discussed.

Synthesis and characterization of inorganic ion exchangers based on tin-titanium mixed oxide to be used in recovery of cadmium and nickel

This work presents the synthesis, characterization and adsorption studies of inorganic ion exchangers based on tin-titanium mixed oxide for recovery of cadmium and nickel metals from aqueous effluents, discarded in the environment mainly through NiCd battery. The exchangers were synthesized by sol-gel modified method using a mixture of tin(IV) chloride and titanium (III) chloride and ammonium hydroxide, precursor reagents. The materials obtained: $\text{SnO}_2/\text{TiO}_2$ e $\text{SnO}_2/\text{TiO}_2:\text{Eu}^{3+}$ were characterized by infrared spectroscopy, thermal analysis, scattering electronic microscopy (SEM), X-ray powder diffraction (XRD) and electronic spectroscopy (excitation and emission) for the europium doped exchanger. The same materials also were synthesized too in polymeric matrix and can be used in column, because the synthesized materials showed crystals size in nanometric scale. It was determined by the distribution ratios for metals taking as parameters the influence of pH, the concentration of metals (by adsorption isotherms) and the contact time. The inorganic ion exchanger presented high exchange capacity with adsorption percent above 90% for the studied conditions, heterogeneous exchange surfaces and physic adsorption. It was calculated Gibbs energy for the process of exchange and the results agree with a spontaneous process.

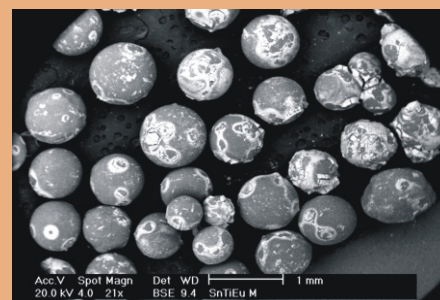


Figure 11. Inorganic ion exchange based on Ti/Sn oxide

Chitosan hydrogels membranes obtained by UV irradiation

Chitin, the second-most abundant natural polymer after cellulose, is the main structural component of marine crustaceans such as crab, krill, lobster and shrimp. Chitosan, a linear polymer of D-glucosamine, and the naturally unique alkaline polysaccharide, is easily obtained by alkaline N-deacetylation of chitin. In this work it was synthesized hydrogels based on chitosan biopolymer crosslinked by two-size polyethyleneglycol of different molecular mass PEG 300 and PEG 400 to be used in biotechnology applications. Those hydrogels were cured by two sources of energy γ -radiation (cobalt δ source with 25, 20, 15, 10 KGy and 60 Watts UV-radiation lamp). The hydrogels irradiated by cobalt source presented decomposition and were burned. 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 OH = and NH centered at 3446 cm^{-1} , Amide I band corresponding to 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 CN stretching vibration and the band due to C-O at 1089 cm^{-1} . 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 swelling kinetics and time dependent-swelling behaviors of chitosan/PEG300 hydrogels was obtained in deionised water (pH 7) at 25 degrees. To observe swelling response of the chitosan/PEG 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.



Figure 12. Decomposed membranes, UV spectra of UV irradiated membranes and their swelling behavior

Synthesis of superparamagnetic particles covered with chitosan for toxic and radioactive metals

Nanoparticles and the superparamagnetic particles possess a lot of applications in diverse areas of the human activities being, mainly in biotechnology and remediation of the environment. The superparamagnetic particles recovered by chitosan demonstrated to be good adsorbents of toxic metals and to other dangerous pollutants found in industrial effluents; therefore it was studied synthesis of such particles for coprecipitation in situ method and covering of the magnetic particles with chitosan methods by crosslinking in suspension, spray dryer and inversion of phases techniques. The particles had been characterized by transmission electron microscopy (TEM); scan electron microscopy (SEM), magnetization measurements, X-ray diffraction, infrared spectroscopy, conductimetric titration, thermogravimetric analysis and differential scan calorimetry. The performance of recovered superparamagnetic particles with chitosan in adsorption of Cr (VI) and Th (IV) was studied from experiments carried out through bath system, using the kinetic models of external diffusion, diffusion intraparticles (Weber and Morris), pseudofirst order, pseudosecond order, models of Langmuir, and Freundlich and the hybrid Langmuir-Freundlich isotherms, and the thermodynamic parameters of adsorption.

Synthesis and characterization of magnetic cobalt ferrite nanoparticles covered with 3-aminepropyltriethoxysilane for use as hybrid material in nanotechnology

Nowadays with the appear of nanoscience and nanotechnology, magnetic nanoparticles have been finding a variety of applications in the fields of biomedicine, diagnosis, molecular biology, biochemistry, catalysis, etc. The magnetic functionalized nanoparticles are constituted of a magnetic nucleus, involved by a polymeric layer with active sites, which ones could anchor metals or selective organic compounds. These nanoparticles are considered organic-inorganic hybrid materials and have great interest as materials for commercial applications due to the specific properties. Among the important applications it can be mentioned:

magnetohyperthermia treatment, drugs delivery in specific local of the body, molecular recognition, biosensors, enhancement of nuclear magnetic resonance images quality, etc. This work was developed in two parts: 1) the synthesis of the nucleus composed by superparamagnetic nanoparticles of cobalt ferrite and, 2) the recovering of nucleus by a polymeric bifunctional 3-aminepropyltriethoxysilane. The products obtained were characterized using the following techniques X-ray powder diffraction (DRX), transmission electronic microscopy (MET), scanning electronic microscopy (MEV), spectroscopy of scatterbrained energy spectroscopy (DES), atomic emission spectroscopy (ICP-AES), thermogravimetric analysis (TGA/DTGA), Fourier transform infrared spectroscopy (FTIR), differential scanning calorimetry (DSC) and magnetization curves (VSM).

Titanium dioxide nanostructured films: characterization and nanotechnological applications

Nanoscience and nanotechnology are scientific-technological fields determined in understanding as the structural control of the matter at the molecular level can be used for the preparation of new materials with unique and exclusive properties. A very promising material for nanotechnological applications is titanium dioxide nanostructured films prepared by the sol-gel process and dip coating. The films are morphologically characterized for atomic force microscopy; crystalline phase and crystallite size for X rays diffraction spectrometry; optical properties for UV-VIS molecular spectroscopy. Our studies seek mainly to the improvement of the photocatalytic activity of titanium dioxide nanostructured films for development of solar cells with high efficiency conversion of energy solar in electric power.

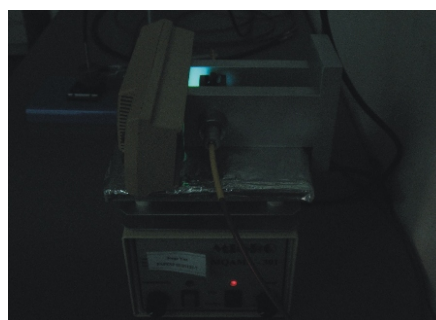


Figure 13. Photocatalytic activity evaluation of TiO₂ nanostructured films

Research project: zeolite-based nanostructured materials

Zeolite with highly regular, ordered cages and channels of nanometer scale is one of inorganic porous materials. The specificity, adsorption and catalytic properties of zeolites, besides the possibility of its pores to host different ions, atoms, molecules and clusters have opened up numerous opportunities of zeolites as advanced nanomaterials. The Brazilian coal ashes consist, basically, of aluminosilicate with high silicon and aluminium oxide contents. Since coal ashes are composed of a large amount of silica and alumina and also due to a low ratio $\text{SiO}_2/\text{Al}_2\text{O}_3$, 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 14 shows the SEM (Scanning Electron Micrographs) images of zeolitic material synthesized from fly ash.

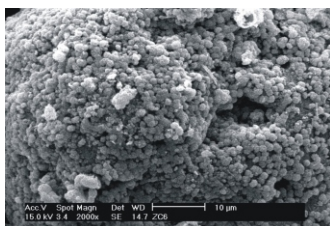


Figure 14. SEM photographs of zeolitic material synthesized from fly ash

The utilization of synthetic zeolites as adsorbent for the treatment of the electroplating effluents, immobilization of heavy metals in soil, decontamination of actual acid mine drainage and removal of dyes from aqueous solution has been evaluated. The results obtained in the project showed a great reduction in the pollutant concentration in treated waters and soil and demonstrated the high potential of the zeolites synthesized from Brazilian coal ashes as low-cost adsorbent nanomaterial for treatment of wastewater and soil.

Pt-Rare Earths/C electrocatalysts for PEM (Proton Exchange Membrane) fuel cells

Pt-Rare Earth/C electrocatalysts (Rare Earth = La, Ce, Pr, Nd, Sm, Tb, Dy, Ho, Er, Tm and Lu) were prepared in a single step by an alcohol reduction process (developed at

IPEN) using ethylene glycol as reduction agent and solvent and Vulcan XC 72 as support. The obtained materials (in the range of 6 to 15 nm) (Figure 15) were tested for ethanol oxidation and oxygen reduction in acid medium using cyclic voltammetry and chronoamperometry. The X-ray diffractogram of Pt-Rare Earth/C electrocatalysts showed the typical fcc structure of platinum and the presence of rare earth (III) hydroxides. All electrocatalysts showed higher current values than Pt/C in the potential range of interest for direct ethanol fuel cell (0.2-0.6 V). Further work is necessary to modify the electrocatalysts preparation methodology in order to decrease the Pt particle size and investigate these electrocatalysts in gas diffusion electrodes for tests in single direct ethanol fuel cell. (Project supported by CNPq)

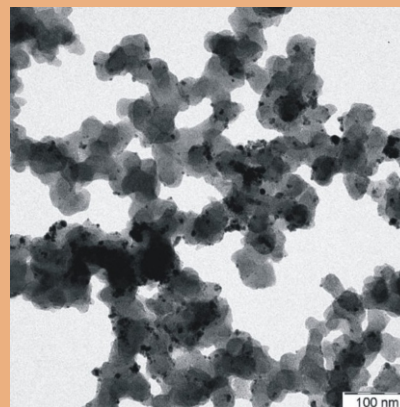


Figure 15. Transmission electronic micrograph (TEM) of Pt-La/C electrocatalyst prepared by the alcohol reduction method

Magnetic carrier technology as adsorbent

Synthetic magnetite has been prepared by precipitation process of the Fe^{2+} and Fe^{3+} ions with a basic solution. It is formed by nanoparticles and exhibit a strong magnetic response in presence of a magnetic field, without nevertheless becomes magnetic. The superparamagnetism, an intrinsic behavior of the magnetite nanoparticles, has been confirmed by magnetization measurements. The magnetite nanoparticles are good adsorbents of metallic ions and dyes and may be easily removed from wastewater using a magnet due to their superparamagnetic properties and as they do not hold a remanent magnetization, they can be disaggregated and reused after desorption of contaminants. The synthetic magnetite has been studied as adsorbent of U and Th ions from nitric solutions and has shown great perspective as an alternative adsorbent to conventional adsorbent. Another research focus has been

conducted for obtaining of magnetic carrier. The magnetite nanoparticles have been conjugated with adsorbent materials and organic complexants and their effect on enhancement adsorption capacity has been investigated. The technology of magnetic carriers for the wastewater treatment combines contaminant separation by sorption and magnetic recovery into a simple and compact process. Also called of magnetic adsorbent provides a simple way to remove contaminants from solutions under a wide range of chemical conditions. Dextran, citrate, chitosan, sugarcane bagasse and synthetic zeolite adsorbents were combined with the magnetite nanoparticles for obtaining of magnetic adsorbents. Characterization and adsorption studies of U, Th, Cr ions and dyes Reactive Orange 16, Indigo Carmine and Congo Red were carried out. The sorption were studied by means of batch equilibrium, isotherm equilibrium, kinetic and Gibbs free energy. The equilibrium data were analyzed by the Langmuir and Freundlich isotherm models. The magnetic adsorbents of the chitosan and dextran presented the best results of adsorption of the U, Th, Cr ions and Congo Red dye. 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. Recovery of 94% U from the magnetic chitosan by desorption process using carbonate ion was obtained. For all adsorption processes studied, the Gibbs free energy indicated the spontaneous nature and chemical adsorption. Magnetic zeolite was prepared by combination of magnetite nanoparticles and synthetic zeolite obtained from mineral coal fly ash. Under the studied conditions, about 80% of the Reactive Orange 16 dye and 42% of the Indigo Carmine dye were removed. The magnetic zeolite exhibited a potential application in treatment of textile wastewater as an efficient and low cost adsorbent for dye removal. Since 2006, the research project of magnetic zeolite has been supported by Conselho Nacional de Desenvolvimento Científico e Tecnológico CNPq (proc. n° 472746/2006-Universal). Also, the magnetic bagasse was investigated for oil spill cleanup applications on water. This material absorbed the oil and was easily attracted by a magnet to remove it from water. The studies shown that magnetic bagasse may be used effectively in the area of oil spill cleanup. The majority of the sorbed oil was removed from the oil-saturated biosorbent by a simple mechanical compression suggesting that the biosorbent can be used repeatedly in oil spill cleanup. Further investigations on a more detailed analysis of the preparation of magnetic adsorbent by different methods and their application to the removal of heavy metals and dyes are in progress.

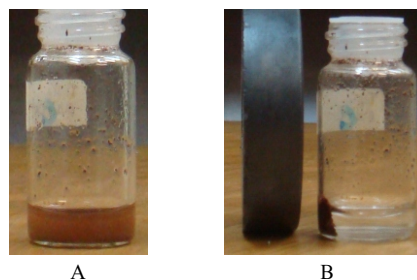


Figure 16. (A) Magnetic chitosan suspension in an aqueous solution; (B) Magnetic separation of the magnetic chitosan from the aqueous solution

Development of HMS-PP to achieve high level of long chain branches (LCB)

Study of the radiation effect on the LCB formation on polypropylene (PP) of different molar mass (M_w), related to different values of melt flow index (MFI) has used acetylene and gamma irradiation. The linear viscoelastic behavior of these samples was investigated by an oscillatory rheometer. Melt strength and drawability were determined by Rheotens equipment. The melt strength of a polymer (maximum force at which a molten thread can be drawn under standard conditions before it breaks) is more efficiently achieved by addition of long chain branches (LCB). Gel permeation chromatography - GPC analysis reveals LCB on modified PP samples. The increase of melt strength of each HMS depends, in part, of the low level cross-linking achieved. But, the pronounced increase of the melt strength and drawability values confirms the presence of long chain branching resulted from the main radiation modification in presence of the acetylene monomer.

Research and development of a process to obtain high melt strength polypropylene (HMSPP) in the presence of liquids monomers

From this study it has been possible to develop two different routes to obtain high melt strength polypropylene (HMSPP) using high-energy radiation like gamma rays from ^{60}Co and pure homo polypropylene resin. In all processes to achieve HMSPP it was added up different quantity of liquids monomers. The monomers studied were ethylenoglycol dimethacrylate (Egdma), triallylcyanurate (Tac), triallyl-isocyanurate (Taic) and trimethylolpropane trimethacrylate (Tmptma) in range concentration of 0 - 5.0 mmol/100 g of pure resin. The radiation process was carried out at room temperature and in two atmosphere conditions: one inert and another oxidative. It was analyzed the radiation doses of 10 and 20 kGy for both conditions. For all routes the samples were characterized by gel fraction, rheological properties, melt strength, extensibility and mechanical and thermal properties. From this project and using these two kinds of process it was possible to produce HMSPP of different values of melt strength and consequently strain hardening.

Study of correlation between rheological parameters and processability of different modified polypropylenes

In this research it has been studied three kinds of high melt strength polypropylene (HMSPP) achieved by modification of polypropylene resin with gamma rays and polyfunctional monomer. Two HMSPP were obtained by polypropylene homo polymer with Tac. In the case of HMSPP achieved by random polypropylene copolymer (RP) it has been studied different kinds of RP with EVA blends. All HMSPP were characterized by rheological properties and submitted to thermoforming and expansion process.

Membranes to proton exchange

A membrane to perform as polymeric electrolyte has to have a combination of requirements in order to maintain good separation and electrochemical capabilities. These include high ionic conductivity, low swelling behavior, high chemical resistance and mechanical integrity. Many groups all over the world have studied commercial fluoropolymer films, which were irradiated, grafted with styrene and sulphonated. Projects of new membranes based on perfluoropolymers and polyolefin was developed using radiation-induced graft polymerization in presence of low molecular mass monomers. Grafting conditions are playing an important role when determining the degree of grafting and the structure built up inside the grafted polymer. In addition, the solvent used for monomer dilution is of special interest as it is one of the essential elements towards successful radiation-induced grafting processes. Solvents are usually used during grafting to bring about swelling of the base polymer, therefore increasing the monomer accessibility to the grafting sites. The PTFE, PP, PE among others are the polymer matrixes in which monomers are grafted in special solvent conditions. Simultaneous irradiation method used for membranes with sequential sulphonation of aromatic rings in order to introduce ionic groups that will be responsible for the proton transportation.

Biomaterials: drug delivery systems

Hydrogels from PVP were developed at IPEN to be used as wound dressings. Silicone and hydrophilic polymers like PVP, PVAI, PEG, CMC and others are being used to synthesize matrices for drug delivery systems (DDS). Controlled drug delivery occurs when a polymer, whether natural or

synthetic, is judiciously combined with a drug or other active agent in such a way that the active agent is released from the material in a predesigned manner. The purpose behind controlling the drug delivery is to achieve more effective therapies while eliminating the potential for both under- and overdosing. Other advantages of using controlled-delivery systems can include the maintenance of drug levels within a desired range, the need for fewer administrations, optimal use of the drug in question, and increased patient compliance. It has been developed hydrogel matrices as DDS for delivery of resveratrol, Assai berry oil, and the glycoprotein Antarticine for cosmetic purpose as well as silicone matrix with papain, prostaglandin (hormone therapy and induction of labour) and timolol for glaucoma treatment.

Natural polymers

Biodegradable foams from cassava starch were developed with partnership of CBPAK and this technology was transferred for this industry. Also biodegradable films were developed.

Organic-inorganic hybrid materials

Developments of membranes composed of hybrid materials are application of conceptual knowledge of nanoscience. Our group reported the synthesis and characterization of organic-inorganic hybrid materials based on Cassava starch (biopolymer) and Hectorite clay (a layer silicate). The materials were prepared by solution exfoliation process. In this method, layered clays are exfoliated into single platelets using a solvent in which the polymer is soluble. After the synthesis, the strong interfacial interaction between biopolymer and clay has been evidenced for homogeneous gels formed. The analysis of FTIR confirms the presence of the layered inorganic material and also two major polymeric components of the cassava: amylase and amylopectin. The thermogravimetric data show that the inorganic clay sheets delay the temperature of the biopolymer decomposition. When small amounts of clay are added, the PXRD data indicate the formation of exfoliated hybrid materials, in which the individual inorganic layers are separated in the polymer matrix. On the other hand, when great amounts of clay are added the intercalated hybrid materials are formed in a regular fashion, as a result of the polymer chains insertion into the layered inorganic structure.



Figure 17. Aerial view of the Materials Science and Technology Center (CCTM-acronym in Portuguese)

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Biotechnology



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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). Besides research, they have also activities on 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 ¹³¹I 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. Studies on the alterations caused by microgravity in excitable media in order to further characterize the physical and thermodynamical behavior of propagating waves, one of the major mechanisms of neural physiology, were also developed. These experiments were performed on a sounding rocket in collaboration with the Brazilian and German space agencies.

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.

Recently, Quality Assurance actions have been started for all the quality control system in order to certify the center for pre-clinical assays to be performed in this facility, assuring that clinical investigation using human volunteers will be done according to national and international guidelines.

It was given continuity to research and production activities related to the five human pituitary hormones, namely growth hormone (hGH), prolactin (hPRL), thyrotropin (hTSH), follitropin (hFSH) and luteotropin (hLH). The basic mission is the development of applied research, especially emphasizing the interaction between the Academic and the Industrial world that, although has been neglected in Brazil, led, in this case, to the introduction of some important facilities, as for example the CIETEC (a spin-off experience), at IPEN. In this 3-year period (2005-2007), in fact, it was experienced a joint-venture with an important Brazilian Pharmaceutical Company (Biolab-Sanus) that provided conspicuous achievements for the Institute. At the end of 2007 a new joint-venture was set up with a young and successful biotechnology company (FK Biotecnologia) that demonstrated great interest in hormone and antibody production for therapeutic and diagnostic applications. It can not be denied that this type of interaction is particularly challenging and sometimes difficult in this country. What can be done, still keeping this goal in mind, is to give at the same time great emphasis to scientific production and to collaboration with well-known national and international research groups. Thus, in this 3-year period, 15 scientific papers, practically all in international journals, whose impact factor was in general between 1 and 5, could be published.

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 in Gene Therapy has been carried out using the hGH gene. This approach represents indeed a way of administrating biopharmaceuticals via genetical modification of human cells that can be transplanted "ex vivo". The goal is the development of an animal model (immunodeficient dwarf mouse, lit/scid) in which, using transduced keratinocytes, could be possible to obtain useful and sustained circulatory levels of hGH and of mouse GH. Three important papers have been published in this period: an Invited Review on "Animal Models for Growth Hormone Gene Therapy" (Current Gene Therapy, 2005) and two works on the utilization of organotypic raft cultures either with hGH-secreting or with mGH-secreting keratinocytes (Molecular Biotechnology, 2006 and Journal of Gene Medicine, 2008).

A new approach is also being carried out with basis on naked hGH or mGH DNA administration followed by electroporation in either muscle or skin of the same animal. This technique, that avoids the use of heterologous cells, is already showing some positive results.



Figure 1. Muscle electroporation of lit/lit mice after administration of DNA plasmid containing the GH gene

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.

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. Human TSH is related with 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, its 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 utilized for their industrial production being CHO cells. The laboratory has synthesized and characterized hTSH, having also the know-how for synthesizing

hFSH and hLH. In this period, though, three main objectives were focused: (i) HPLC (size-exclusion and reversed-phase) set up for analytical and preparative purposes; (ii) MALDI-TOF mass spectrometry determination of these heterodimeric glycoproteins and of their alpha and beta subunits; (iii) characterization of their carbohydrate moiety, essentially composed of complex N-glycan structures, whose determination was carried out in collaboration with the University of Vienna, Austria. Four major publications were concerned with these aspects. An invited Review described the state-of-art concerning HPLC analysis of human pituitary hormones for pharmaceutical applications, obviously including also hGH and hPRL (Current Pharmaceutical Analysis, 2006). An original research article has set up a reversed-phase HPLC methodology, specific for hFSH preparations, including MALDI-TOF-MS conditions that, for the first time, could provide, in a single assay, the molecular masses of heterodimer, alpha- and beta-subunit (Journal of Chromatography A, 2006). A semi-preparative reversed-phase HPLC column has also been used for setting up a practical laboratory-scale purification of hTSH (Journal of Chromatography A, 2007). Finally, still concerning hTSH, we studied a reduced CO₂ environment that led to a higher productivity, starting for the first time to characterize the N-glycan structures of our recombinant preparations in comparison with the only commercial preparation available (Thyrogen from Genzyme, USA). 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. This difference must be studied and evaluated, especially considering the repeated parenteral use of these biopharmaceuticals. More work is in progress concerning this aspect, especially considering the comparison of the N-glycans 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). Also more work is in progress, related to hLH characterization and to the synthesis of a human-like form of hTSH.

Prolactin (hPRL)

Prolactin (hPRL) 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 their analogs/antagonists - an anti-proliferation activity, especially concerning 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 for the first time in our laboratory, in their authentic forms. One published paper (Protein Expression and Purification in 2006) carried out a complete

physico chemical and biological characterization and comparison of the two antagonists, synthesized by CHO (Chinese Hamster Ovary) cells. Moreover one student working at the University of California at Riverside, where S179D-hPRL had been discovered, carried out advanced studies showing that this antagonist is a potent anti-angiogenic hormone (Endocrine Related Cancer, 2006), uses particular signaling pathways capable of inducing apoptosis (Endocrinology, 2006) and has an opposing effect in comparison with hPRL on the expression of cell cycle regulatory proteins (Oncology Research, 2006). A complete study, comparing hGH and hPRL behavior when synthesized in bacterial periplasmic space and setting up specific fermentation conditions for hPRL-secreting *E. coli* (Patent N. PI 070 1082-6, 2007) was also published in the Journal of Biotechnology (2008). Finally, in collaboration with the Neuroendocrine Unit of the "Hospital das Clínicas" of the FMUSP, a study on human macroprolactin and its biological activity in different groups of patients has been carried out and published in the Journal of Clinical Endocrinology and Metabolism (2006).

Cellular response to ionizing radiation

Ionizing radiation is a very-known genotoxic agent, but on the other hand it is a therapeutic modality used in medicine. Therefore, a better comprehension of the cellular response to ionizing radiation is of great value from the radiobiological as well as from oncological viewpoints. The present project has been developed in three interlinked aspects. In the first one (cytogenetic aspect) a comparative study of the effects of different radiation types and radionuclides in human and rodents cells has been carried out. Parameters such as survival curve, proportion of affected cells, distribution of lesions, kinetics of cell proliferation, cell viability and apoptosis were studied. So, cytogenetic (chromosome aberrations and micronuclei) and biochemical (comet assay and viability test) techniques were developed and standardized in the laboratory. A study on the cytogenetic effects of neutrons produced in the Reactor R1 of IPEN-CNEN/SP (in collaboration with the "Centro de Engenharia Nuclear") in human blood lymphocytes is being developed. Following this, dose-response curves for different types of radiation (γ , β , n) will be constructed for biological dosimetry (dosimetric aspect) purposes. Cytogenetic techniques will be applied for the quantitative estimate of absorbed dose, according to the criteria adopted by IAEA (2001). Another study that has been done is about radiosensitivity of tumoral (T-47D and MCF-7) and non tumoral (MCF-10) human breast cell lines exposed to various doses of γ -radiation, analyzing radioinduced damage, DNA repair capacity and cell viability. The data obtained showed that the tumor cell lines responded to the genotoxic action

of ionizing radiation with more intensity than non tumoral cell line. The analysis of the influence of the radioprotector (melatonin and propolis) in the induction of cytogenetic damage caused by γ -radiation of ^{60}Co in CHO cells has been another goal of our study. Preliminary data showed a promising reduction of the frequency of micronucleus induced by γ -radiation. Another aspect of the study (therapeutic approach) has been the analysis of the cytogenetic effects on blood lymphocytes of radiopharmaceuticals used in nuclear medicine, as ^{131}I administered to patients with thyroid diseases such as differentiated thyroid carcinoma with or without administration of recombinant thyrotropin (Thyrogen or rhTSH produced at IPEN). The data obtained in an animal model (rats) showed that the treatment did not cause any major damage in peripheral blood lymphocytes. Thyrogen, an imported product, and rhTSH produced at IPEN behave similarly.

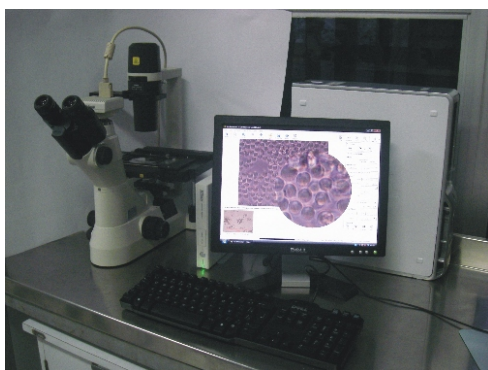


Figure 2. Inverted microscope showing blood lymphocytes

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.



Figure 3. Training the insertion of the experiment in the rocket payload

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. Synthesizes of polymeric biomaterials were also done, resulting in 3 (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 USP. The biofunctionality of the bovine pericardium with endothelial cells has been tested.

Recombinant proteins - Refolding from inclusion bodies using high hydrostatic pressure

Escherichia coli is the most efficient and cost-effective host for transgenic protein production for therapeutic or for scientific purposes which does not require post-translational modification. However, *E. coli* often is unable to fully refold the foreign protein during overexpression of recombinant proteins. Often bacteria accumulates these misfolded intermediates in their cytoplasmic as insoluble and misfolded aggregates known as inclusion bodies. Aggregated proteins do not possess biological activities and in many instances, injections of this kind of proteins induce immunologic reactions. A novel and robust method to disaggregate and refold murine globular protein was described. It was demonstrated that high pressure can successfully convert insoluble globular protein expressed in *E. coli* (like endostatin or QM) to a preparation with native tertiary structure and full biological activity. Endostatin can specifically inhibit endothelial cell proliferation and thus potentially inhibit angiogenesis and tumor growth. QM or ribosomal protein L10 is originally identified as a tumor suppressor protein. The QM gene encodes the ribosomal protein L10 that is necessary for joining the 60S and 40S subunits of ribosome. This study will analyze the structure and the function in the cell of this protein.

Biological effects of ionizing radiation

Ionizing radiation, in aqueous solution, produces several highly reactive species. The most important are hydroxyl radical (OH) and hydrated electron (e-aq.). These 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.

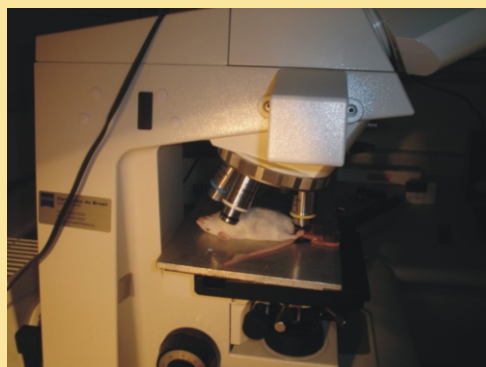


Figure 4. Facility used for Intravital microscopy

Ionizing radiation can also modify biological and structural properties of toxins as crotoxin, used here as a model. Biological alterations occurred in irradiated crotoxin were observed with intravital microscopy; native crotoxin causes a time-dependent vasoconstriction that suffers inversion with irradiated toxin (2kGy) leading to the vasodilatation. Structural analysis suggested alteration in tertiary structure, keeping the primary structure unbroken.

The Animal Laboratory Division (DALI) is a facility having 840 m², 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 5. 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.

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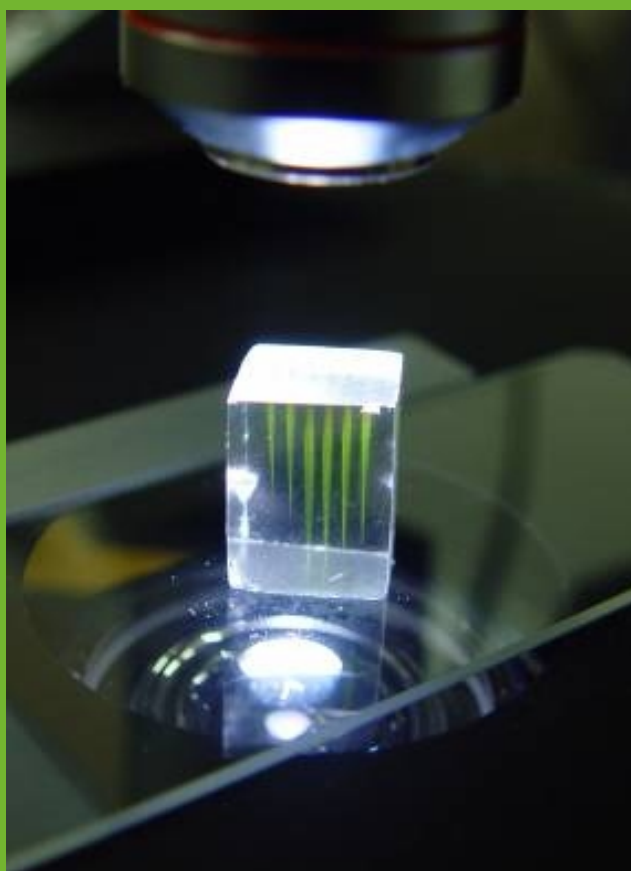
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Honor Mention and Awards

2006 Best Poster presented in IV Congress of Latin American in Biomaterials and Artificial Organs RODAS, A.C.D.; HIGA, O.Z.; MAIZATO, M.J.S.; LEIRNER, A.A.; PITOMBO, R.N.M. "Cytotoxicity and genotoxicity of bovine pericardium preserved in glycerol", Caxambú, 2006

2007 Top Cited Article 200-2006 Award, Journal of Chromatography - UEDA, E.K.M.; GOUT, P.W.; MORGANTI, L. - "Current and prospective applications of metal ion-protein binding", Holanda, 2007

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Introduction

The Lasers Technology Program of IPEN is developed at the Lasers and Applications Center. In recent years the Program was dedicated to the research and development of optical materials, as laser media and scintillation crystals; characterization, modeling and optical spectroscopy of solids, plasmas and biological materials; development of a compact diode pumped-solid state laser and the development of a high power (TW) laser system, one of the main projects. High power ultrashort pulses lasers based on CPA technologies and Ti:Sapphire gain media, allowed the generation of high peak powers in conventional optical tables, and the attainment of relativistic intensities (10^{18} W/cm²) at modest costs.

The Program is strongly committed to the study of Laser Applications on several areas: Nuclear, Medicine and Dentistry, Industry, Environment and Advanced research, aiming not only research but diffusion and innovation, in association with Brazilian Universities and commercial partners. Some highlights of the Program:

- Development of new fluoride materials for laser systems in cooperation with Institut für Kristallzüchtung, Germany.
- Characterization, modeling and optical spectroscopy of rare-earth doped crystals and glasses for the development of solid laser medium;
- Laser induced plasma spectroscopy technique for plasma properties investigation;
- A 600mW Yb:YLF diode pumping laser emitting at 2.3 μ m - best result of the literature;
- A hybrid Ti:Sapphire/Cr:LiSAF TW peak power laser system - 0.5 TW ultrashort already generated;
- New pulp vitality tests, done by Laser Doppler Flowmetry increased the test performance;
- Implementation of an Optical Coherence Tomography (OCT) resulted in collaboration with a cosmetic company allowing the development of diagnose tests and tissue characterization;
- Therapeutic processes of photosensitization and photobiomodulation were studied allowing the advance of new ones using innovative technology through low-power lasers;
- Physical characterization of biological tissues - optical properties and thermal responses - was performed for the development of new processes using high intensity lasers;
- Collaboration among IPEN, CTMSP and INB: laser processing of special materials: welding of Zircaloy-4 applied in assembling the fuel elements used in Angra dos Reis reactor;
- A ground-based elastic backscatter LIDAR system has been upgraded and two additional channels were added in order to achieve both Nitrogen and Water Vapor Raman signals in the atmosphere up to 4 km in altitude.

These achievements were supported by national and international funding agencies (FAPESP, CNPQ, FINEP, CAPES, DAAD), as well as, cooperation with partners such as Natura, Lasertools, Smar, and others. In the period about 2 millions dollars were invested in the program.

Crystal growth

The research on fluoride crystal growth performed in this period was focused in the investigation of the YLF:Yb:Tm, $\text{LiGd}_{1-x}\text{Lu}_x\text{F}_4$ ($0.5 \leq x \leq 0.75$), and BaY_2F_8 single crystals doped with Tb, Er, Pr and YLF single crystal fibers for optical applications. These projects were supported by CNPq (Edital/2004) for the investigation of Czochralski single crystals for laser application; CNPq/PADCT project in collaboration with Federal University of Sergipe (UFS), in the investigation of new scintillator materials; CNPq-Instituto do Milênio program in collaboration with Federal University of Pernambuco, in single crystal fibers growth investigation and CAPES/PROBRALDAAD project with the Institut für Kristallzüchtung (IKZ), Germany, which common research in the investigation of the phase relation of the binary subsystem YF_3 - GdF_3 ; YF_3 - LuF_3 and YF_3 - BaF_2 .

Fluoride single crystals growth

YLF crystals were grown by the Czochralski method; they were doped with 0.05 mol% Tm and/or 20 mol% Yb, and/or 1.3 mol% Nd. The objective was the identification of the most important processes that lead to an enhancement of thulium blue up conversion emission, under excitation around 792 nm. An enhancement in 475 nm-Tm³⁺ emission of almost 20 times was observed in the samples YLF:Yb:Tm:Nd compared to an YLF:Yb:Tm sample. The melting behavior of $\text{LiGd}_{1-x}\text{Lu}_x\text{F}_4$ ($0.5 \leq x \leq 0.75$) solid solutions was studied utilizing the melting zone technique, under a HF reactive atmosphere, to obtain high purity compounds. It was concluded that the $\text{LiGd}_{1-x}\text{Lu}_x\text{F}_4$ compounds melt congruently by x values around 0.75. Crystals were successfully grown from the melt, under an Ar+CF₄ atmosphere, with the stoichiometric compositions, $\text{LiGd}_{0.25}\text{Lu}_{0.75}\text{F}_4$ and $\text{LiGd}_{0.232}\text{Lu}_{0.75}\text{Nd}_{0.018}\text{F}_4$.

Phase diagram studies

The binary phase diagram YF_3 - GdF_3 was studied by differential scanning calorimetry (DSC). Yttrium fluoride and gadolinium fluoride show complete miscibility. The transformations at high temperature are of first order and occur at 1338.6 K (YF_3) or 1174.8 K (GdF_3). A second order shaped local maximum at 1333 K was observed for GdF_3 . The phase diagram GdF_3 - LuF_3 was determined by DSC and X-ray powder diffraction analysis. The mutual solubility of both components is unlimited in the orthorhombic room temperature phase. The maximum solubility of Lu in the high temperature phase of GdF_3 (tysonite type) is about 20% and the maximum solubility of Gd in LuF_3 (α - YF_3 type) is about 40%. Intermediate compositions of the low

temperature phase decompose upon heating in a peritectoid reaction to a mixture of both high temperature phases. The BaF_2 - YF_3 system was also investigated, with focus given to the BaY_2F_8 compound and its neighboring phases. Various difficulties hinder the thermal analysis investigation of this binary fluoride system however thermal results compared with data from the literature showed discrepancies. Thermal analysis curves show no variation in the polymorphic transition temperature of BaY_2F_8 in each side of the compound. Conclusive evidence of the presence of the solid solution range BaY_2F_8 mentioned in previous literature has not been found. All thermal peaks were observed to occur at temperatures much higher than described by literature phase diagram. The observed discrepancies are under further investigation.

Single crystal fibers growth

Regular and transparent BaLiF_3 and LiYF_4 :Nd and Er-doped single-crystal fibers were successfully grown. Several improvements were performed on the micro-pulling down system used for fiber pulling process. A new technique for welding of micro-crucibles was developed. The atmosphere control was determined as the most important factor to avoid impurity contamination from moisture and oxygen, preserving chemical composition and optical quality of the growth fibers. It is well known that optical properties of laser hosts are very important in determining laser performance.



Figure 1. BaLiF_3 single crystal fiber growth

Development of compact diode-pumped solid state laser sources

This activity comprises the investigation of laser systems with respect to their temporal and spectral characteristics with the purpose of improving their usage in future applications in industry, dentistry, medicine and pollution control. It also includes the production design of such systems, tests and experiments in order to make these lasers reliable and application ready. Our group has the capacity to generate know how in the area of optical resonators which allows us to achieve specific desirable characteristics of diverse nature such as temporal, thermal, spectral, beam quality and efficiency. Our diode pumped solid state lasers (DPSS) have output powers in the range of milliwatts to 50 watts and in most cases, close to Gaussians beam quality. We have build laser that operate frequency tunable in the NIR and IR.

Some highlights of this last period are:

- Single frequency Nd:LiGdF laser, continuously tunable during 150 GHz.
- TEM00 Tm:Ho:YLF emitting at 2 μm with 2.3 watts of output power.
- Compact, diode-pumped LiF:F2 laser emitting at 1.14 μm .
- 600mW Yb:Tm:YLF laser emitting at 2,3 μm (best result of the literature).
- Spectral conformation of laser diodes: we developed a proprietary technology to narrow the emission frequency (Figure 2) and generate dual frequency emission. This technique is useful for solid state laser pumping and telecommunications (wavelength division multiplexing).

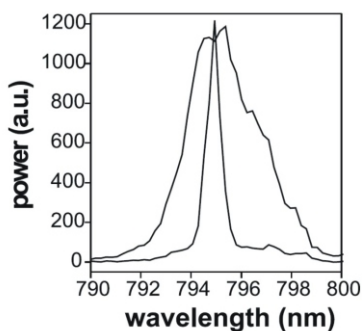


Figure 2. Frequency narrowing of a 40 watt diode

- Nd:YVO laser with 63% optical to optical efficiency and 20 watts of output power int

TEM00 mode (Figure 3).

- Nd:YLF laser operating at 1053 nm with 45% efficiency.

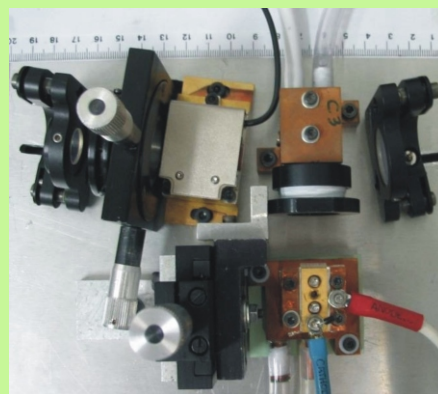


Figure 3. Very compact Nd:YVO laser with 20 watt of output power and 63% efficiency

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

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 (10 mJ), was used for lifetime and luminescence spectrum measurements of rare earth ions in glasses and fluoride 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 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 medium infrared and to improve light signal amplifiers based on thulium-doped materials that operate in the S-band of telecommunication (1470-1500 nm). A detailed investigation of the energy transfer process from first excited state of erbium (and holmium) caused by Nd^{3+} , Tb^{3+} and Eu^{3+} ions in LiYF_4 (YLF) crystal was carried out. By 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. Fluorozirconate (ZBLAN) and germanate glasses based on GeO_2 doped with 0.1 mol % of Tm^{3+} and codoped with (0.1 - 4 mol %) of Tb^{3+} , Eu^{3+} and Ho^{3+} were used to evaluate the 1.5 m

emission gain (or amplification gain) under 797 nm pumping. The ZBLAN glass doped with 0.5 mol% of thulium and codoped with 1 mol % of holmium showed a gain 10 times higher than one verified for Tm(0.1%):Tb(0.2%) in germanate glass. The rate equations solutions using the fourth order Runge-Kutta numeric method allowed us to get the population distribution of the three lowest levels of Er^{3+} (and Ho^{3+}) in YLF involved in the optical cycle of laser emission at $\sim 3 \mu\text{m}$. This investigation was applied to Er single doped, Er-Nd, Er-Tb and Er-Eu doped YLF crystals. It was observed that 20 mol % of erbium is the best concentration to maximizes the laser gain at $3 \mu\text{m}$ emission in Er-single doped YLF crystal. However, the best concentrations of Er:Nd:YLF is 4 mol % of Er^{3+} and 1.5 mol % of Nd^{3+} that has a frequency limit of operation increased to 315 Hz. The frequency limit of operation in Er (20 mol %):YLF was observed to be much lower, $\sim 15 \text{ Hz}$ for a pumping at 980 nm.

Characterization, modeling and optical spectroscopy of plasmas

Laser induced plasma spectroscopy technique was implemented in three setups: Nitrogen laser (UV, 10 ns), Ti:Sapphire laser (NIR, 45 fs) and Nd:YAG laser (NIR and green, 4 ns). Some experiments were carried out in order to characterize the plasmas generated by nanosecond and femtosecond lasers in several materials. The plasma can be completely characterized by its temperature and electronic density using the Boltzmann plot and Stark broadening methods. The temperature of a plasma generated in air by femtosecond pulses was determined as 130(74) 103K for a ms integrated gate time and 392(9) 103K for a ns gate time. The value for electronic density was $1.1(1) \cdot 10^{18} \text{ cm}^{-3}$ for both gates. Some stainless steel standards were irradiated by ns and fs regime, allowing us to observe that the femtosecond irradiated steel formed a plasma almost twice hotter but less dense than the nanosecond one (Figure 4). It was possible to determine the plasma evolution in soil samples irradiated by femtosecond laser, discriminating the white continuum from atomic and ionic emissions. The temperature of the plasma evolved from 114(12) 103 K in 30 ns to 12(1) 103 K in 130 ns and the electronic density diminished from $9(1) \cdot 10^{18} \text{ cm}^{-3}$ to $1.5(2) \cdot 10^{18} \text{ cm}^{-3}$. Recently, the Saha-Boltzmann method was implemented. The calibration free technique was applied to cultural heritage coins and the analytical determination of elemental composition were employed. For an “one thousand réis” coin from 1939 (Tobias Barreto) the plasma temperature was 10300(150)K, and the plasma density was $4.3(1) \cdot 10^{16} \text{ cm}^{-3}$ using Saha-Boltzmann plot and $2.5(2) \cdot 10^{16} \text{ cm}^{-3}$. The evaluated Cu, Al and Zn concentration agreed to the Edict Law for coin forgery.

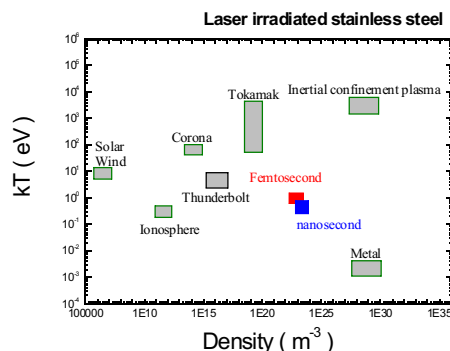


Figure 4. Comparison between the temperature (in eV) and densities (in m^{-3}) for some natural and human created plasmas and the values evaluated for stainless steel irradiated by nanosecond and femtosecond lasers

Teeth vitality testing via Laser Doppler Flowmetry

Pulp vitality tests based on assessment of pulp blood flow, measured by the laser Doppler Flowmetry (LDF), have been suggested in the literature. The relevance of the technique is justified by the limitations of the current methods for evaluation of pulpal vitality, based on sensitive stimulus, with fail after a trauma and in immature teeth. For non-invasive pulp blood flow measurements via LDF, an optical fiber delivers the laser radiation to the tooth enamel. A fraction of the transmitted radiation, by the enamel and dentin, reaches the pulp and via an inverse pathway is collected by another optical fiber. It is recognized that a fraction of the scattered radiation also reaches the periodontal and gingival tissues, and a fraction of the measured flow via LDF originates from these regions. As a consequence, generally the measured flow (F) from devitalized teeth is not zero. In addition, generally the measured flow from both vitalized and devitalized teeth varies largely among individuals, as well as among teeth of the same individual. Thus measured fluxes from both vitalized and devitalized teeth occupy a continue range of values, and generally the ranges are superimposed. In such conditions it is not possible to find a cut-off value of flow below which all devitalized teeth are correctly classified and above which all vitalized teeth are also correctly classified by the test. As a consequence, the discriminatory capability of the current vitality test based on LDF may be significantly decreased, depending on the amount of measured flow from other regions than the pulp. To improve the test accuracy, we developed two new discriminators, derived from the measured flow (F), aiming the minimization of intra and inter-subjects variability. We used the ROC curve analysis as an objective method to quantify and to compare the performance of three discriminators: I) the currently used, the measured flow F(UA); ii) the percentage of flow variation, F(%) and: iii) the difference of flow, F(Diff), being (ii) and (iii) between the interrogated tooth and its homologue vitalized. ROC analysis confirmed our expectation that the proposed discriminators allowed accuracy higher than 96% (here accuracy is the percentage of correct classification of any arbitrary pair of teeth one vital and one non-vital). The measured accuracy of current test was 22% lower. Accuracy of the current sensitive methods for vitality testing (Electric and Cold tests). The accuracies of two new, F(%) and F(Diff), non-sensitive methods (laser Doppler), are higher than sensitive methods,

and higher than the current laser Doppler flow-based discriminatory method.

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 in cutting and welding of some materials for important technological applications. One of the developed processes is the welding of Zircaloy-4, a zirconium alloy, which has begun in collaboration among IPEN, CTMSP and INB. This alloy is largely used in nuclear applications and, in this specific case, these welds have been accomplished in assembling of fuel elements used in Angra dos Reis reactor. Because this material is extremely reactive with oxygen, welding must be done in controlled atmosphere, where oxygen content must be kept very low (less than 20 ppm). Due to the complexity of this spot weld with non traditional geometry, process parameters must be carefully controlled and balanced, and the studies carried out with a Nd:YAG pulsed laser has brought significant improvements in the quality of these joins. Dissimilar stainless steel welding is another activity where research has brought significant results in applications of great importance in nuclear and aero spatial fields. Following remarkable advances in this kind of welding, joining austenitic, martensitic and nickel alloys, a study was realized to understand the process of nucleation occurring in the melting zone during cooling and solidification of the welding pool. The aim was to achieve more resistant and confident welds, avoiding common defects like cracks porosity and excessive melt. Titanium and its alloys, due to their good mechanical strengths when used in high temperature, also have had many applications in medical, nuclear and aero spatial areas. Studies were carried out to determine the gas shielding influence in the properties of laser welds of these materials and resulted in optimized joints with good cosmetic and mechanical quality. Laser cutting of these materials was also optimized aiming the production of improved cut edges free of nitrates, recast material and dross. Besides thermal processes accomplished by traditional lasers, another program has been

developed where thermal effects are absent in the region of interaction between laser focus and processed piece. This occurs when very short laser pulse length are employed, in the order of femtoseconds (10-14 a 10-13 s). To use such pulses a workstation was built and the first results were obtained allowing the achievement of ablation threshold for some metals and semiconductors. These data are essential to machining in micron or sub-micron scale and have been used to obtain such structures.

Main products and services - core business

A joint collaboration with LaserTools enterprise was concluded and resulted in transference of technology of many laser cutting and welding methods. Theses manufacturing processes has been used regularly to attend several costumers like: Zillo Lorenzetti, GM Reis, Fibra Forte / INPE and Alacer. Laser welding studies were done in copper electrical connectors used in the automobile industry to Delphi Automotive Systems. To Smar Comercial Ltda a program has been initiated to develop welds in nickel alloys foils used in chemical seals.

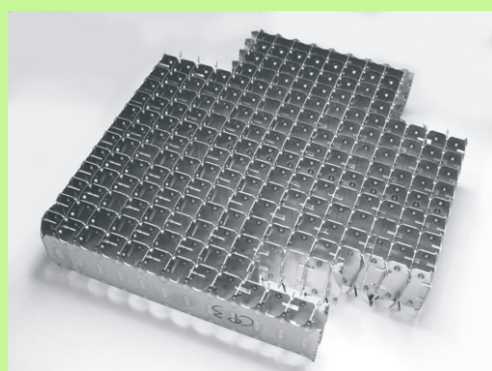


Figure 5. Spacing grating of fuel element used in Angra dos Reis nuclear reactor. Junctions are spot laser welded in thin sheets of Zircaloy-4

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. Dental caries is considered the most prevalent disease during childhood and adolescence even though its decline in the last decades, nevertheless its incidence in the development countries is still high. For this reason, more conservative restorative methods, more efficient preventive procedures and more accurate diagnostic methods are necessary. Besides caries, periodontal and

endodontical microorganism infections are a matter of concern in Dentistry, as they are closely related to other diseases such as cardiopathologies. In this sense, an extensive study of optical and thermal properties of the dental hard tissue was conducted within a scientific cooperation among IPEN CNEN / SP and the School of Dentistry - USP.

Spectroscopic and thermal characterization of enamel and dentine

Laser irradiation of hard dental tissues can promotes changes in their physical and chemical characteristics due to the temperature reached during irradiation. Temperature rise during diode, Er:YAG and Er:Cr:YSGG laser irradiation of enamel and dentin was determined using thermocouple and an infrared thermographic camera. Electron spin ressonance (ESR) and Fourier transform infrared spectroscopy (FTIR), showed that after heating the tissue to temperatures between 100 and 300°C, some water is eliminated and the hydrogen bonds, which determine collagen alpha-helix structure stabilization, are lost. After elimination, the collagen matrix is changed and electrons are probably trapped, giving rise to ESR signals and absorption bands in the ultravioletvisible spectral range.

Prevention of dental caries with lasers

Lasers can reduce the rate of subsurface demineralization in enamel. The percentage of inhibition of dental caries varies from 30 to 97.2%, and the association with fluoride enhances the inhibition of caries. Several laser wavelengths were used for this purpose. The use of TEA CO₂ laser (10.6 m) was investigated by measuring the calcium and phosphorous content after demineralization. Although CO₂ laser irradiation does not promote any surface photomodification, it was able to significantly reduce enamel demineralization. Tetracalciumphosphate was identified as a new crystalline phase formed during Ho:YLF irradiation of enamel, detected by X-ray diffraction using a synchrotron source. Er:YAG and Er,Cr:YSGG lasers changes mainly the organic matrix (collagen) and water and little the mineral matrix (OH radical). 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.

Early detection of dental caries

An optical early detection caries method studied was the fluorescence spectroscopy, which enabled to differentiate carious from natural tissue, indicating that the blue laser at 405 nm can be used clinically to carious diagnosis. The presence of three narrow fluorescence bands at 455, 582, and 622 nm indicates that the origin is from mineral matrix, while the broadband near 500 nm can be assigned to organic matrix. The optical coherence tomography (OCT) has showed to be an accurate image method that allows to detect early caries in its structure before it could be detected clinically or radiographically.

Caries removal and cavity preparation with lasers

Enamel and dentin removal can be achieved using lasers with wavelengths that are strongly absorbed by these tissues. In the infrared, the strongest absorption bands occur in the 2-3 μm and 9-11 μm regions, that are readily accessible by holmium, erbium and CO_2 lasers. Usually the morphological pattern of Er:YAG laser irradiated enamel and dentin has a rough aspect with a clear exposition of the prisms and dentinal tubules. The melted surfaces covering the CO_2 laser irradiated enamel and dentin, occlude the dentinal tubules and the enamel prisms. The rough pattern after Er:YAG laser irradiation, which originates from the micro-explosion of water, does not occlude the dentinal tubules, whereas the surface morphology after CO_2 laser irradiation, which originated from the temperature rise above hydroxyapatite melting point, shows dentinal tubules occlusion and tissue melting. These changes influence the tissue properties such as increase of acid resistance and may benefit the bond strength between the tissue and composite resin used for restorations. An optical coherence tomographic system verified the quality of restorations by measuring the distance of restorative material and normal tissue.

Lasers in Periodontology and Endodontics

Thermal and photodisruptive laser effects result in the elimination of pathogenic microorganism in periodontology and endodontics. Although the Er:YAG laser did not interfere in the adhesion of blood components, it caused more changes on the root surface, whereas the diode laser inhibited the adhesion. The Nd:YAG laser

associated with scaling and root planning in class II furcation defects in patients with chronic periodontitis treatment promoted significant bacterial reduction immediately after irradiation. The thermal effects at the external root surface and the morphological changes on the root canal walls after continuous (CW) and pulsed diode laser root canal irradiation at 810 nm was investigated. The temperature will not increase above the safety limit (10°C) for the periodontal tissues; however, after each treatment a 20-second resting time should be considered to prevent an excessive temperature rise in the tissue when the laser is operating in either continuous or pulsed mode.

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 (LED) to provide a nonthermal, noninvasive, environmental safe treatment that can be useful in the Health Sciences.

Photobiomodulation

Low-power laser therapy (LPLT) is a treatment modality that is becoming more useful in medicine and dentistry although mechanisms underlying light effects are still not completely understood. Studies were carried out in vitro and in vivo to investigate the influence of LPLT on cell cultures, wound healing, temporomandibular disorders, thyroid gland and caries prevention.

- Cell cultures: LPLT increases epithelial cell growth rate in stressed cells by growth under nutritionally deficient states. This cell growth improvement is directly proportional to the number of irradiations; however, it was not enough to reach the full cell growth potential rate of Vero epithelial cell line observed when growing under nutritional regular condition.

- Wound healing: In vivo researches show that LPLT may accelerate cutaneous wound healing and the recovery of dental structures involved in cavity preparation at the pre-dentine region even if a single laser treatment is performed. Besides, polarized red laser radiation affects skin repair by reepithelization stimulation, fibroblast differentiation, protein synthesis regulation, inflammatory process reduction and collagen bundle organization. In a clinical trial, LILT was an effective adjunctive treatment that appeared to promote healing following

gingival surgery.

- Temporomandibular disorders: LPLT was more effective than Transcutaneous Electrical Neural Stimulation (TENS) in improving the amplitude of mandibular movement in patients with temporomandibular disorders.

- Thyroid gland: LPLT of the thyroid gland may affect the level of thyroidal hormones. A statistically significant hormonal level alteration between the first day and 7 days after the last irradiation was found.

- Caries prevention: Although LPLT before acidulated phosphate fluoride application appeared to diminish the caries progression, LPLT did not present any additional benefit compared with fluoride on the prevention of induced-dental caries in rats.

- Photosensitization: Photodynamic Antimicrobial Therapy (PAT), which involves the use of light sources with appropriate wavelength to kill cells previously treated with a photosensitizer drug, is an athermal, selective and new alternative to replace antibiotics, antimicrobials and chemical agents against microorganisms. PAT has been studied as a promising approach to eradicate oral pathogenic bacteria that cause endodontic diseases, periodontitis, and peri-implantitis.

- Peri-implantitis: In vivo studies indicate PAT as a more efficient, noninvasive method that could be used to reduce periodontal bacteria in peri-implantitis when compared to conventional therapy, with flaps elevated of mucoperiosteal mucosa for scaling the implant surface. It is a potential application since PAT in this study was as effective as a surgery, therefore avoiding this invasive and painful treatment.

- Periodontitis: *Aggregatibacter actinomycetemcomitans* is an important periodontal pathogen that can be photosensitized in vitro by red laser combined with malachite green, a dental plaque disclosing agent. Besides, the dye is photodegraded following irradiation and new non toxic photoproducts were formed in suspension.

- Endodontic diseases: PAT was effective for reducing *Enterococcus faecalis* in root canals and can be an adjunct to endodontic treatment. Our results suggest that the use of PAT as an adjuvant to the conventional endodontic treatment leads to a statistically significant further reduction of bacterial load and in particular reduces the amount of bacterial regrowth after 24 h compared to PAT or endodontic treatment alone. This result indicates that the combined therapies may reduce the need of antibiotic therapy on root canal treatment, overcoming microbial resistance, a major problem nowadays.

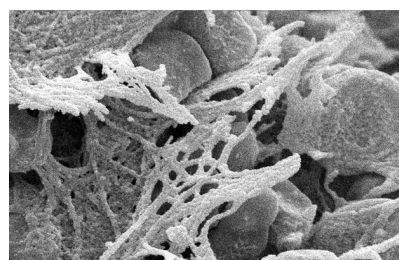


Figure 6. Electron-micrograph of the implant surface showing bacterial cells

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 an OCT system, based in an open air Michelson interferometer, using a femtosecond laser running at 830 nm as central wavelength and with 40 nm of bandwidth. This configuration provides 10 μm of longitudinal and lateral resolution and 3,000 μm of maximum scan depth in air. This system can be improved to be polarization sensitive, allowing measuring birefringent samples. As examples of possible applications, (where was possible to measure) simulated caries disease process (demineralization process by acid etching, and by bacteria process, Figure 7) were measured, making it possible the study of the caries dynamics.

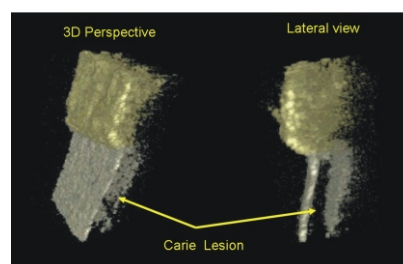


Figure 7. 3D reconstruction of human teeth for 11 days of caries disease process

This development resulted in a collaboration with a cosmetic company (NATURA) that has been supported this research. This collaboration allow us to have a commercial system working at 930 nm with longitudinal and lateral resolution of 6 μm , maximum penetration depth of 1,600 mm in air, and real time data acquisition (3 frames/sec) in maximum resolution (2000 x 512 pixels), up to 8 frames/sec are possible with lower resolution.

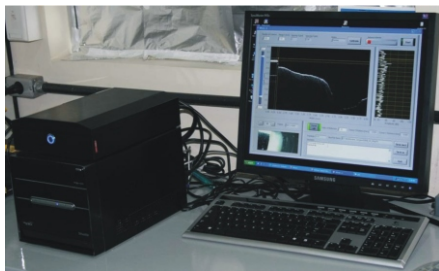


Figure 8. OCT system from Thorlabs Inc

Laser Remote Sensing of the Atmosphere

The LIDAR technique is based on the emission of a collimated laser beam in the atmosphere and on the detection of the backscattered laser light by the suspended atmospheric aerosols and atmospheric molecules. The LIDAR technique, through its high temporal (from seconds to minutes) and spatial (less than 30 m) resolutions is a powerful tool to visualize in real time, the structure of the Planetary Boundary Layer (PBL) using the aerosols as passive tracers of the atmospheric dynamic processes. A backscattering LIDAR can thus provide information on the PBL's mixed layer depth, entrainment zones and convective cells structure, aerosol distribution, clear air layering, cloud-base altitudes, cloud statistics, atmospheric transport processes and other inferences of air motion. A ground-based elastic backscatter Lidar system was setup in the Laboratory of Environmental Laser Applications at the Centre for Laser and Applications (CLA). This system is operational since 2001 and has since then routinely collected data to monitor aerosols in the atmosphere as well the planetary boundary temporal evolution as an indication of pollutant dispersion conditions. Besides, beginning in 2007 the LIDAR system has been upgraded and two additional channels were added in order to achieve both Nitrogen and Water Vapor Raman signals in the atmosphere up to 4 km in altitude, this new feature provides this system an unique capability of not only qualitatively assessing these gaseous species but also quantitatively. Currently our group has collaborations with Lidar and atmospheric sciences groups in Greece (National Technical University of Athens), United States (Goddard Space Flight Center - NASA) and in Brazil (Insituto Nacional de Pesquisas Espacial INPE, Instituto de Pesquisas Meteorológicas IPMET/UNESP and University of São Paulo.

Operation and optimization of the TW peakpower laser

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° 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. Even at lower peak powers, the ultrashort character of the pulses generates nonlinear phenomena, particularly those initiated by multiphotonic processes that generates free electrons. Ultrashort pulses with 10° GW peak power were utilized to generate color centers in fluoride crystals, allowing the study of their generation phenomena. The density of the centers created is higher than what is attainable with conventional ionizing radiation, resulting in the formation of aggregates that are stable at room temperature. The multiphotonic generation of free electrons also confers a nonselective character to the ablation of solid targets, which together with the almost nonexistent thermal effects, led us to develop a new method to determine the ultrashort ablation threshold of materials, and fully describe its formalism. The reduced thermal effects were also observed in Raman studies of ablated technological materials.

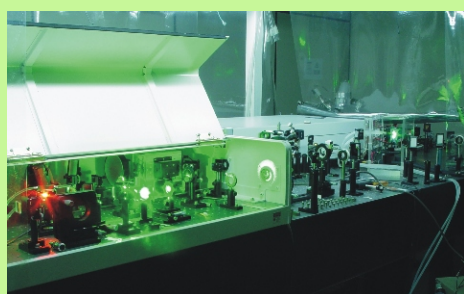


Figure 9: Hybrid Ti:Sapphire/Cr:LiSAF CPA system in operation, generating 0.2° TW peak power pulses

Color center production by high intensity ultra short laser pulses in fluoride crystals

Ultrashort pulses with peak powers in the vicinity of 10 GW were used to create color centers in fluoride crystals grown in our facilities. It was possible to determine that the centers generation begins with a multiphotonic absorption, favored by the high intensity due to the short pulse duration, that neutralizes negative ions, that are kicked out of their sites in the crystalline matrix. Once the center are formed by electrons trapped in the crystalline field, they act as a probe to study the crystal structure. Once this vacation is formed, an electron is trapped in it, originating the basic color center. 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. The probe characteristic of the centers allowed the observation of different properties in each matrix, by the measurement of the centers absorption and emission spectra.

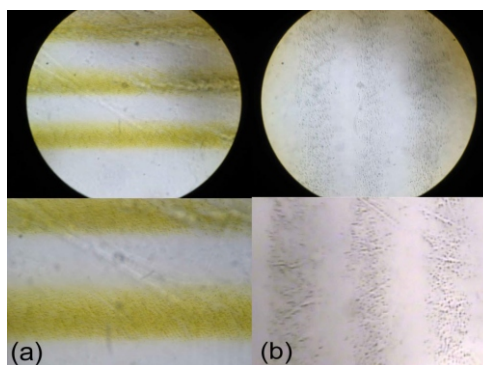


Figure 10: Optical microscopy of irradiated BLF samples with lower intensity: (a) BaLiF, grown by zone melting under HF with CCs, (b) BaLiF, grown by Czochralski under CF,

Implantation of the pump and probe optical technique to study ultrafast phenomena 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 system is excited by the pump pulse, 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. In 2007 this kind of pump and probe experiment was assembled in the Laboratory for High Intensity Lasers. To accomplish this goal it were acquired a set of Lock-in amplifiers, an ultra precise step motor, with driver, for building a delay line and a variety of first-class optomechanical components that together with the excellent infrastructure of the laboratory, will allows us to put in operation, in the Center for Lasers and Applications at IPEN-CNEN/SP a facility to do state of the art investigations of ultrafast phenomena in different systems (Figure 11). The initial tests in the setup are in progress.



Figure 11. Setup for studying ultrafast dynamic process with the pump and probe technique under development in Center of Lasers of IPEN

Another important push to this activity are in progress conjunctly by CNRS and Fapesp, allowing the interchange of researchers between the Center for Lasers and Applications and the Institut de Physique et Chimie des Materiaux de Strasbourg, CNRS/Strasbourg, França, for the study of ultrafast phenomena in different systems, metallic and magnetic materials, nanostructures and medium biological. This will be a pioneering setup to study the ultrafast magnetization dynamics in Brasil, a topic of growing interest in the international scientific community, due to the constant progress of materials science and the driving economical forces that comes from different applications that arise in the manipulation of ever more information in the ultrafast and ultrasmall scales.

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Honor Mention and Awards

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Homenagem à Profa. Denise M. Zezell pelo trabalho pioneiro em Terapia Fotodinâmica de Tumores no Brasil. Workshop em "Terapia Fotodinâmica: Integração dos Aspectos Moleculares, Tecnológicos e Aplicações na Área de Saúde", São Pedro-SP, 2007

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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. This Program completed 30 years on March 2006. Some highlights of the Program since its creation are summarized below:

1975 - Prof. Rui Ribeiro Franco was nominated by the superintendent at that time, Prof. Rômulo Ribeiro Pieroni, to elaborate a proposal for a graduate program for IPEN in association with the University of São Paulo. At that moment Prof. Orlando Marques Paiva was the dean of the University. Prof. Paschoal Ernesto Américo Senise, responsible for the Graduate Program at USP was very supportive during the creation of the Program at IPEN.

1975 - Professors Fausto Walter de Lima, Alcídio Abrão, José Moura Gonçalves, together with Rui Ribeiro Franco conceived the master and doctoral program in nuclear technology.

17/11/1975 - The superintendent Prof. Rômulo Ribeiro Pieroni, settled the first Graduate Committee constituted by Rui Ribeiro Franco, Wladimir Sanchez, Alcídio Abrão, Fausto Walter de Lima and Roberto Fulfaro. Claudio Rodrigues, Constância Pagano Gonçalves da Silva, Laura Tognoli Atalla, Maria José Coutinho Nastasi and Achilles Alfonso Soares were their substitutes.

16/03/1976 - Resolution 905 from University of São Paulo was published approving the Graduate Program Statute.

01/04/1976 - The Nuclear Technology Program Basic Nuclear Technology Concentration Area was officially implemented for Master and Doctoral Degree.

01/03/1977 - Another concentration area was implemented Nuclear Power Reactor and Nuclear Fuel technology.

12/12/1997 - A new terminology for the Graduate Program was approved, including the following areas:

Nuclear Technology - Applications

Nuclear Technology - Materials

Nuclear Technology - Reactors

Since its creation 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. Along its 30 years the Nuclear Technology Program awarded 833 Master's Degrees and 387 Doctor's Degrees.

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 2005-2007. IPEN has maintained this grade since 2003. Until 2007 the program awarded 1395 titles: 937 master's degrees and 458 doctor's degrees. The performance during this period is shown in Table 1.

TABLE 1. Nuclear Technology Graduate Program

	2005	2006	2007
Master degrees awarded	42	58	56
Doctor degrees awarded	51	59	28
Offered disciplines	66	79	87
Total graduate enrollment	384	390	421

IPEN is also responsible for the Professional Master Degree Course - Lasers in Odontology, 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

	2005	2006	2007
Master degrees awarded	15	14	17
Offered disciplines	8	8	8

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

	2005	2006	2007
Offered disciplines	17	16	17
Enrolled students	234	213	282

The institute has a research 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 Research 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	DOCTOR DEGREE		
	2005	2006	2007
CNPq	35	35	36
CAPES	17	17	17
FAPESP	13	10	11
CNEN	-	6	5
IPEN	-	-	3
Total	65	68	72

Funding agency	MASTER DEGREE		
	2005	2006	2007
CNPq	22	23	24
CAPES	15	15	15
FAPESP	8	12	13
CNEN	-	10	20
IPEN	-	-	14
Total	45	60	86

On 2005 it was approved the postdoctoral program regulating the activity at the institution. During the considered period around 30 professionals enrolled the program.

To accomplish all the activities mentioned above, IPEN has an Education Directory and a dedicate building, having facilities such as: one hall for events, two auditoriums (70 people approximately), two computer labs and 10 classrooms.

Concerning scientific information support, it is available a central specialized library, which offers, beyond traditional collections and services, a comfortable studying place for the students and the IPEN community, nice equipped with network wireless and stations to support search and consult to all the information resources.

There is a local reference and technical information desk support provided by the staff. Access to all the main electronic information resources in the country, as to the electronic CAPES and USP data bases and journals are assured and managed through collaborative arrangements.

The library also manages the institutional repository with 40% full text records authored by IPEN scientific community; 1800 digital dissertations and theses added in 2008.

The nuclear science libraries network was implemented, last year, in order to improve the nuclear science literature access and the interlibrary loan.

Scientific and Technical Production



Journal Articles	148
Thesis and Dissertations	182
Patents Pending	194

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Radiopharmacy

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