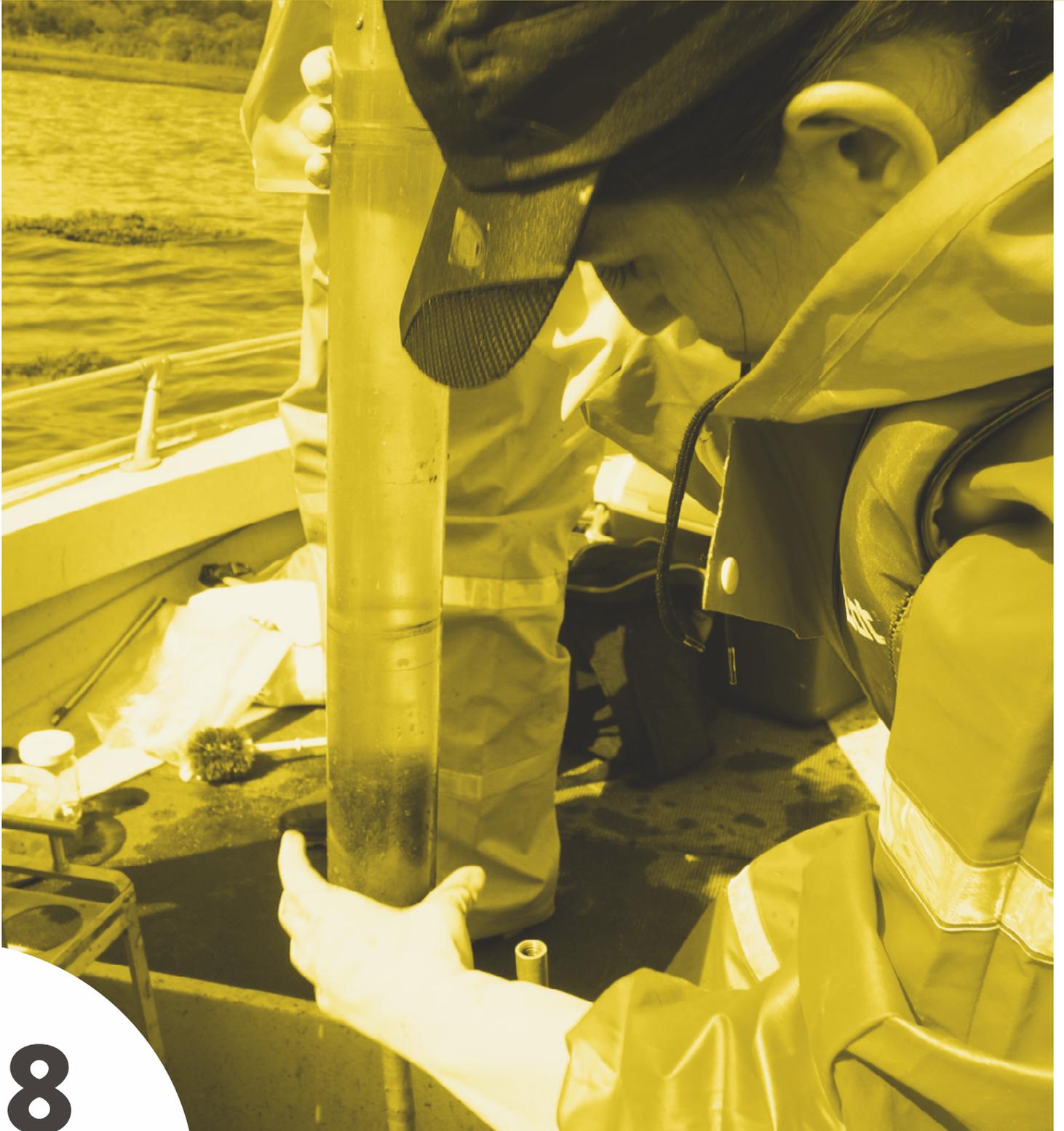


Environmental Science and Technology



Introduction

The Environmental Science And Technology Program was structured as a consequence of the continuous growth of environmental activities on areas related to nuclear programs of IPEN/CNEN-SP. Moreover, it was an answer towards the society concern with the climate changes and biodiversity preservation; aspects like pollution & sustainable, Waste Management and Environmental Risk, etc. Conducting the environmental management in a strategic way, thinking about the sustainable development of the institution. The program comprehend five main areas: Environmental analysis; Chemical Technology; Polymer technology nucleus; Chemical characterization of materials using spectrometric techniques and Analytical Chemistry for the Nuclear Fuel Cycle. If we look at the evolution of our research lines, it is clear that IPEN/CNEN-SP has built up solid grounds on environmental technology.

This is a result of many actions deeply linked to our recent activities, to highlight just a few:

- Chemical Metrology:

Development, validation and use of moderns analytical technology for the characterization of pollutants (metals, trace elements, organic compounds, rare earth, uranium, etc.) and endocrine disruptors (PAH's, hormones, plasticizer and human pharmaceuticals) in order to evaluate water, sediment and soil quality.

- Environmental Chemistry and Water Science:

Characterization of pollutants and contaminated áreas (areas): Environmental monitoring of emerging pollutants, PAHs, pharmaceutical, endocrine disruptors and organic markers in water for public supply on the Guarapiranga dam, Brazil;

factors affecting the occurrence of *Dolychospermum solitarium* bloom (Cyanobacteria, Nostocaceae) in Lajeado reservoir in Palmas-Tocantins state, Brazil

- Studies of trace gases and greenhouse gases of urban areas and remote areas:

Modifications by Anthropogenic Pollution of the Natural Atmospheric Chemistry and Particle Microphysics of the Tropical Rain Forest During the GoAmazon (Green Ocean Amazon);

the Amazon Tall Tower Observatory (ATTO): overview of pilot measurements on ecosystem ecology, meteorology, trace gases, and aerosols

characterization of São Paulo contaminated areas by persistent organic pollutants (POP's) and development of a safe decomposition process.

- Chemical Safety; chemical waste management, environmental Risk and Preventing risk:

Study on municipal management of construction and demolition wastes and illegal dumping of chemical waste in the metropolitan region of

São Paulo, its social and environmental impacts - a proposal for public policy for coping emergency situations.

- Studies and development of recycling technologies reuse of materials:

Development of value-added materials from wastes:
Environmental characterization and application.

- Studies and developments in biodegradable polymers, polyolefins and advanced methods for polymer and rubber destruction etc.;

- Clean Technologies:

Development of electrocatalysts for application in fuel cells.

- Microwave technology in:

Hydrotreating process intensification of middle distillates;

production of biodiesel from vegetable oils and additional purification of Mo-99 produced from LEU targets and

development of units of reaction with microwave, for operation in high pressure and high temperature;

Clean Technologies

- **Eletrocatalysts**

Development of electrocatalysts for application in fuel cells

- **Microwave technology**

Hydrotreating process intensification of middle distillates;

Production of biodiesel from vegetable oils and additional purification of Mo-99 produced from LEU targets;

Development of units of reaction with microwave, for operation in high pressure and high temperature;

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 of the guidelines of drinking water standards; Chemical emergencies and waste management - minimization of environmental damage caused by accidents involving Brown-fields. These achievements were conducted with support from national and international funding agencies – FAPESP, CNPq, FINEP, CAPES, AIEA, NOAA, NERC, NASA as well as cooperation with partners and clients, such as SABESP, CETESB, INPE, INPA, USP, Petrobras, Botanic Institute / SP; Federal University of Tocantins-UFT.

Cooperation in studies of trace gases and greenhouse gases with: Harvard University, Scot Martin, Lead Principal Investigator (PI); University of Colorado, Boulder, Jose Jimenez; University of California, Irvine,

Saewang Kim, with subcontract to James Smith and Alex Guenther, University Center for Atmospheric Research (UCAR); University of São Paulo (Universidade de São Paulo; USP), Henrique Barbosa (PI); Federal University of São Paulo (Universidade Federal de São Paulo; UNIFESP), Luciana Rizzo, Theotonio Pauliquevis; University of the State of Amazonas (Universidade do Estado do Amazonas; UEA); Biogeochemistry, Multiphase Chemistry, and Air Chemistry Departments, Max Planck Institute for Chemistry, Mainz, Germany; Scripps Institution of Oceanography, University of California San Diego, La Jolla, CA 92037, USA; Universidade Federal Santa Maria, Dept. Física, Santa Maria, RS, Brazil,

Empresa Brasileira de Pesquisa Agropecuária (EMBRAPA), Belém-PA, Brazil and Instituto de Física, Universidade de São Paulo (USP), São Paulo, SP, Brazil.

Environmental Chemistry

Environmental chemistry and water science

Environmental monitoring of emerging pollutants, PAHs, pharmaceutical, endocrine disruptors and organic markers in water for public supply on the Guarapiranga dam, Brazil (Fig. 1).

This research aims to the development and validation of methods to use on investigation of some endocrine disruptors and others organic compounds present in the environmental, which may affect the water quality of public supply. Scientific cooperation programs, supported by CNPq and FAPESP, were developed in partnership with SABESP and the Botany Insti-

tute. In this research, the solid phase extraction (SPE) technique was used for extraction and concentration of samples, followed by gas chromatography coupled to mass spectrometry, GC / MS, for the identification and quantification of organic compounds. A large and diverse group of organic compounds such as personal care products, plasticizer, pharmaceutical and other products classified as potential endocrine disruptors get to the market every day. Hormones and drugs stand out because they are compounds potentially active in biological systems and are related to the origin of various types of cancers. After ingestion, the majority of human pharmaceuticals are excreted in urine and feces via the sewage treatment network. The occurrence of these substances in aquatic environment is due primarily to inefficiency of conventional procedure (coagulation, and



Fig. 1 Guarapiranga reservoir;
São Paulo/SP-Brazil



Fig 2. Collection of sediment samples in the Guarapiranga reservoir; São Paulo/SP-Brazil

filtration) of the STPs that are unable to totally remove them. As a result, in the last decades, many researchers have reported the presence of pharmaceuticals in sewage treatment plant (STP) influent/effluent, sea water, surface water, and, exceptionally, in the drinking water. A national program to assess impacts needs to be implemented, because the effects of these endocrine disruptors on human health are not fully known.

Guarapiranga reservoir, located in an urban region, has suffered environmental impact on water quality due mainly to the release of untreated sewage from the disorderly occupation of their surroundings. It is one of the largest producer of drinking water in the metropolitan region of São Paulo and supplies more than four million inhabitants. Applying the validated analytical procedures on the samples from Guarapiranga reservoir, some compounds have been detected at raw and drinking water, principally during drought season. In this research, it was used solid phase extraction technique (SPE) for extraction and concentration of samples, followed by gas chromatography coupled to mass spectrometry (GC/MS), high performance liquid chromatography (HPLC) and liquid chromatography electrospray ionization-high performance mass spectrometry (LC-ESI-MS/MS), for identification and quantification of organic

compounds. The analysis results have shown two areas of greatest impact that coincide with the region of highest occupancy around the dam. The higher concentration of some organics marker compounds in these areas suggests the occurrence of direct discharge of domestic sewage into the dam waters.

The developed methods are also being applied in the area destined to the construction of the Brazilian Multipurpose Reactor, in order to establish a basic monitoring before the implementation of the project.

Chemical Composition of the Guarapiranga Water System

Water is an essential life resource, and it must be present in adequate quantity and quality for consumption. This research aimed to evaluate the environmental quality of Guarapiranga reservoir, classified as Class 1. This is located

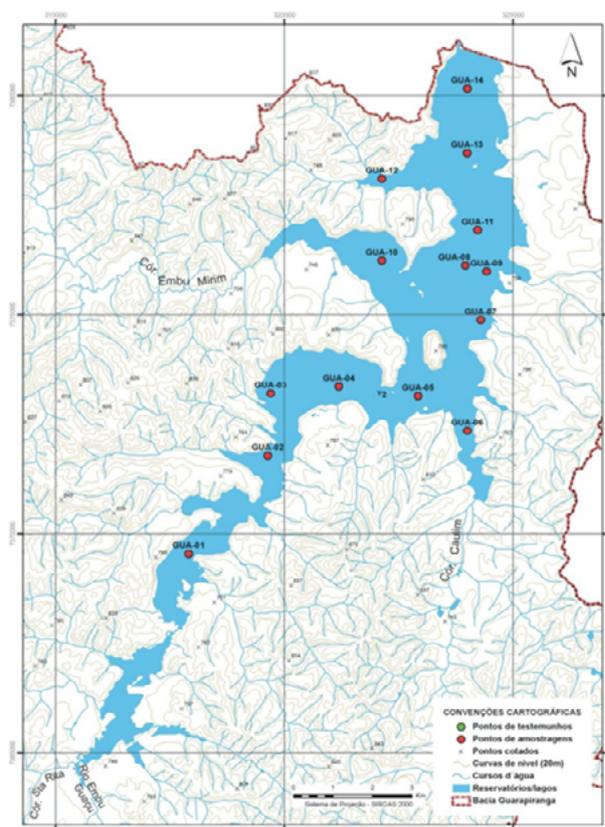


Figure 3 – Guarapiranga Reservoir, São Paulo- SP; Sampling sites

in parts of the municipalities of Cotia, Embu, Itapeverica da Serra, Juquitiba, São Lourenço da Serra and São Paulo and in the Embu Guaçu's total area. This reservoir has significant degradation in the quality of water due to domestic and industrial wastewater contribution, which affects the cost of treatment and makes it difficult to access drinking water. The study presents the results of 26 metals, 7 anions, limnology parameters: pH, conductivity, temperature, dissolved oxygen, water clarity, obtained from samples collections between the years 2011-2013, These are water samples on the surface, middle and bottom on 14 points and bottom sediment in these same points being evaluated 26 metals. The sediment results were compared with TEL (Threshold Effect Level) and PEL (Probable Effect Level) values, water results were compared with the established limits by the resolution of CONAMA 357/2005. Limnological parameters, conductivity, pH and dissolved oxygen were outside the framework of CONAMA's resolution. All Ionic species responded to established by CONAMA, however it is noted an increase in concentrations between samples collected. Nitrogen series as well as Fe:P ratio showed important contribution of control parameters of the organic load in reservoir. Of the 26 elements analyzed, only Al, Cu, Mn concentrations were observed outside of the CONAMA's resolution. In the sediments analyzed, the chemical

elements Cu, Cr, Zn showed values above the TEL and PEL. The results indicated significant environmental impacts from urban invasion and sewage waste, all of which seriously affect water quality and, consequently, the sediment quality.

Distribution of Nitrogen Species in the Guarapiranga Dam, São Paulo – SP

Guarapiranga Dam is responsible for about 20 % of the water supply of the Metropolitan Region of São Paulo (RMSP). This reservoir started to be used as public supply source to the city of São Paulo in 1928, however, since the 60s, there has been an irregular and disordered occupation around its region. As a result, the reservoir has significantly been altered due to the multiple environmental impacts caused by anthropic activities. These hydrodynamic changes in the water body are involved in the eutrophication processes, in other words, in the distribution of nutrients in the resource, as presented by several studies. Eutrophication is a natural process that occurs in water bodies, nevertheless, when associated with anthropic activities, it produces changes in aquatic ecosystems, increasing nutrients amount, mainly nitrogen and phosphorus, and through contributions from wastewater, it may interfere in the water quality and use. As nitrogen is an important nutrient and ammoniacal species

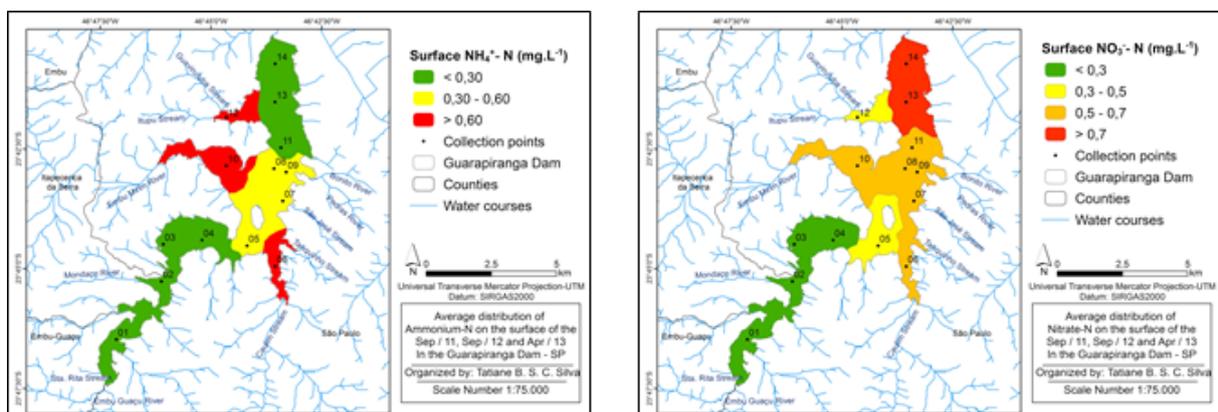


Figure 4 (a,b) - Nutrient and ammoniacal species distribution at Guarapiranga Dam.

are the initial product of the decomposition of nitrogenous organic residues, its oxidation processes (where it slowly converts to nitrate) favor the flowering of algae, being a contaminant with difficult removal and the ammonia consumption of dissolved oxygen from natural waters (to be biologically oxidized), the amount of ammoniacal nitrogen is an important parameter for the classification of natural waters. Nitrite is an intermediate in the ammoniacal oxidation process (nitrification). Nitrate is the final product of the aerobic stabilization of organic nitrogen, indicating old contamination. High concentrations of nitrate also indicate inadequate release of domestic and industrial wastewater or the use of nitrogen fertilizers in agriculture, as well. Therefore, this study aims to assess the distributions of nitrogen species in the Guarapiranga Dam, due to its use, irregular occupation of its around and the increasing eutrophication in this water body, in order to understand the contribution of these nutrients to the quality of Guarapiranga Reservoir.

Factors affecting the occurrence of *Dolychospermum solitarium* bloom (Cyanobacteria, Nostocaceae) in Lajeado reservoir in Palmas-Tocantins state, Brazil

This work was performed with the goal of identifying the conditions to the emergence of a bloom of cyanobacteria *Dolychospermum solitarium*, registered in June 1, 2012, in Lajeado reservoir hydroelectric power plant (HPP) in Palmas – Tocantins, Brazil, (S10°17,215'/10°11,284' the W48°18,729'/48 21,874'). Four campaigns to collect and analyze the water were carried out between June 2013 and 2014. Seventeen limnological variables were evaluated upstream and downstream the site of the bloom. The trophic state index proposed by Lamparelli (2004) and analysis of correlation between total phosphorus con-

centrations were applied. The study allowed recognizing the contribution of phosphorus effluent of the Aurenly wastewater treatment plant (WWTP), in conjunction with the contributions from the basin of the Taquaruçu Grande stream, part of watershed streams Machado and Tiúba, provided to the impacted area an environment for the potential growth of cyanobacteria. The study also showed that concentrations of total phosphorus in the point near the WWTP (S10°17,304'/W48°19,968') were inversely proportional to the levels observed in effluents of the WWTP. It was also noted that climatic factors, like an extemporaneous rain, certainly contributed to the thermal destabilization of the environment and enabled the species *D. solitarium* the temporary rate domain.

Studies of trace gases and greenhouse gases of urban areas and remote areas

Modifications by Anthropogenic Pollution of the Natural Atmospheric Chemistry and Particle Microphysics of the Tropical Rain Forest During the GoAmazon (Green Ocean Amazon)

The GoAmazon campaign seeks to quantify and understand how aerosol and cloud life cycles in a particularly clean background in the tropics are influenced by pollutant outflow from a large tropical city, all in the context of addressing the susceptibility of cloud-aerosol-precipitation interactions to present-day and future pollution in the tropics. Within this context of GoAmazon, the overall goals of the proposed research are (i) to measure and mechanistically understand the factors affecting the number-diameter distribution $n(d)$ of the atmospheric particle population over a tropical rain forest (especially the effects of anthropogenic pollution as a perturbation to natural state) and (ii) to develop and implement an upscaling analysis from this new data set and knowledge of $n(d)$ to prognosticate possible climatic impacts of present-day urban pollution and possibly greater pollution in the future.

The Amazon Tall Tower Observatory (ATTO): overview of pilot measurements on ecosystem ecology, meteorology, trace gases, and aerosols

The Amazon Tall Tower Observatory (ATTO) has been set up in a pristine rain forest region in the central Amazon Basin, about 150 km northeast of the city of Manaus. Two 80m towers have been operated at the site, since 2012, and a 325m tower is nearing com-

pletion in mid-2015. An ecological survey including a biodiversity assessment has been conducted in the forest region surrounding the site. Measurements of micrometeorological and atmospheric chemical variables were initiated, in 2012, and their range has continued to broaden over the last few years. The meteorological and micrometeorological measurements include temperature and wind profiles, precipitation, water and energy fluxes, turbulence components, soil temperature profiles and soil heat fluxes, radiation fluxes, and visibility. A tree has been instrumented to measure stem profiles of temperature, light intensity, and water content in cryptogamic covers. The trace gas measurements comprise continuous monitoring of carbon dioxide, carbon monoxide, methane, and ozone at five to eight different heights, complemented by a variety of additional species measured during intensive campaigns (e.g., VOC, NO, NO₂, and OH reactivity). Aerosol optical, microphysical, and chemical measurements are being made above the canopy as well as in the canopy space. They include aerosol light scattering and absorption, fluorescence, number and volume size distributions, chemical composition, cloud condensation nuclei (CCN) concentrations, and hygroscopicity. In this paper, we discuss the scientific context of the ATTO observatory and present an overview of results from ecological, meteorological, and chemical pilot studies at the ATTO site.

Characterization of São Paulo contaminated areas by persistent organic pollutants (POPs) and development of a safe decomposition process

Pesticides banned, obsolete or discarded constitute a serious environmental risk around the world, especially in developing countries. Pesticides are among the compounds that constitute the group of so-called POPs, or

persistent organic pollutants that are regulated internationally by the Basel Convention. Among the major POPs, one could cite pesticides, dioxins and PCBs that represent, according to

the United Nations Industrial Development Organization - UNIDO, one of the most serious and urgent problems to be faced, because of, on one hand, its wide dissemination in environment and, secondly, because of its properties and characteristics, which determine its persistence in soil and water. The United Nations Environmental Protection - UNEP, for example, launched a global action for the establishment of an international treaty to reduce and / or eliminate emissions and discharges of 12 specific POPs, also known as “dirty dozen” (Aldrin,

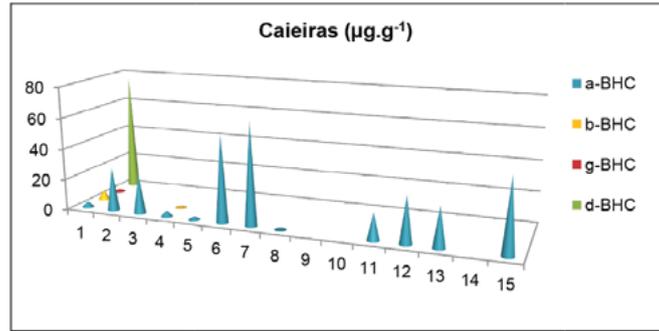


FIGURE 5 – Results from the Caieiras sampling site

Chlordane, Mirex, Dieldrin, DDT, Dioxins, Furans, PCBs, Endrin, Heptachlor, Toxaphene and BHC), besides the adoption of scientific criteria for the possible inclusion of others. The HCHS,

or Hexachlorcyclohexanes also called BHC or Lindane, are organochloride insecticides that have been banned in most countries in the 70s and 80s. These compounds are highly stable and toxic to the environment and living organisms, among which are addressed in the work as chlordane cis/trans ($C_{10}H_6C_{18}$), heptachlor ($C_{10}H_5C_{17}$), heptachlor epoxide cis/trans ($C_{10}H_5C_{17}$) and the isomers α , β , γ and δ benzene hexa-chloride BHC (C_6C_{16}).

Concerned about the importance of the prob-



FIGURE 6 – Sampling sites of Caieiras, São Paulo. The numbers represent the sites in figure 4.

lem, researchers of CQMA have performed a study that aimed to develop and validate a method to identify and quantify persistent organic pollutants, known as POPs in soil of industrial regions Caieiras and Franco da Rocha municipalities in São Paulo by gas chromatography coupled with Mass Spectrometry (GC/MS) and Electron Capture Detector (GC/ECD), as it can be observed in the figures 1 and 2. To ensure reliability of the analysis carried out, tests were carried out validation method. The extraction technique used was QuEChERS, achieving recovery in the range 70 to 120% for most of the compounds, acceptable for complex matrices. The limits of detection and quantification of the method comprises the range of 0.0002 and 0.01 $\mu\text{g}\cdot\text{g}^{-1}$, respectively. The samples analyzed were contaminated by compounds hexachlorobenzene α , β , γ and δ , many of which are above the maximum allowable in accordance with national legislation and international law (FIG. 5; FIG 6).

Besides the development of characterization methods, IPEN has also developed advanced processes for safe decomposition of POPs. The technology developed at IPEN is applicable to intrinsically safe disposal of hazardous organic wastes, particularly the organochlorides, whose degradation has presented problems when using the most common methods, such as incineration. The molten salt oxidation is a process which promotes a more complete and safer decomposition of wastes considered critical, such as POPs, as can be observed schematically in the figure 7.

Organochloride compounds can be decomposed in a safe way in the molten salt equipment because the chlorine present in the wastes reacts with elements present in the bath salt, such as sodium, for example, forming the sodium chloride which is retained in the salt. It is important to mention that the retention of chlorine has greater importance

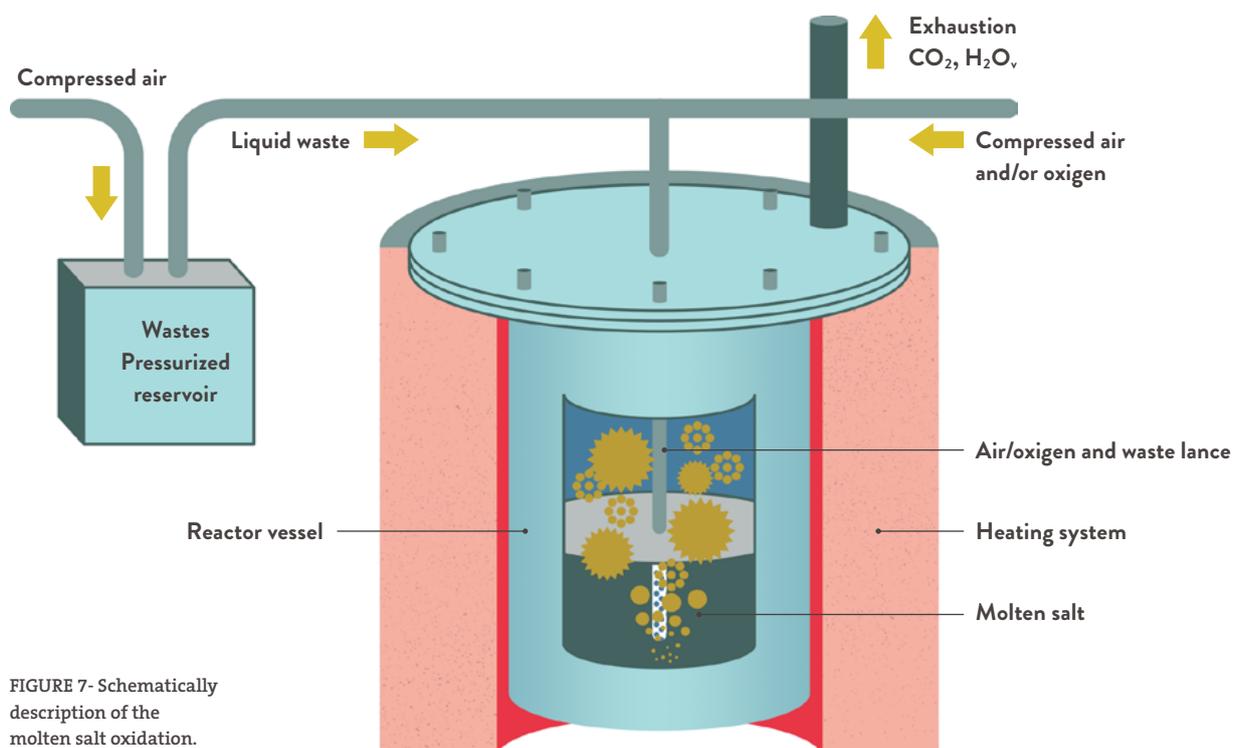


FIGURE 7- Schematically description of the molten salt oxidation.

than the oxidation of other constituents of organic waste, such as carbon and hydrogen. The saline bath which was being investigated consisted of pure sodium carbonate, whose melting temperature is 851°C.

It was assembled a molten salt decomposition equipment constituted by two reactors. The waste (BHC in powder) was introduced with air in the first reactor and the gaseous products of the decomposition reactions were introduced in the second reactor. The results have proved that chlorine can be retained at low temperatures, as mentioned above, by forming the corresponding chlorine salts, i.e. lithium, sodium and potassium chloride. These innocuous chlorides salts are retained in the molten salt bath (FIG.7).

Chemical Safety, chemical waste management, environmental Risk and Preventing risk

Study on municipal management of construction and demolition wastes

The main problems faced by the cities related to construction and demolitions waste (CDW) are associated to the disposal of huge quantities. This paper presents the results obtained by Guarulhos City Hall with the implementation of waste reception from construction and demolition in 17 voluntary delivery points in the 2010 to 2014 period. In 2014, 54,793.10³ kg of waste were received, enabling savings of R\$ 16,167,770.60 in the collection of illegal discharges. After separation of this material, it was sent, to the Plant Waste Recycling, the amount of 32,015.00 10³ kg mineral residue grinding. Whereas all this material was turned into sand (the cheapest of households purchased by the city), after processing, a total of R\$ 1,152,540.00 was saved in buying fine aggregate. As an example of using this recycled material, the experience of PROGUARU public company responsible for paving appears. 23,964 10³ kg in base and sub-base of flexible pavements were used.

Illegal dumping of chemical waste in the metropolitan region of São Paulo, social and environmental impacts

In the State of São Paulo (Brazil), public institutions, such as the Fire Department, the Civil Defense and the Environmental Agency, have been involved in a high number of cases in which chemical waste has been discarded on land, highway margins and access roads as well as on streets and avenues. This harmful practice is a threat to public health and the

environment. According to the Environmental Agency of São Paulo State (Companhia Ambiental do Estado de São Paulo - CETESB), the fourth-leading cause of chemical emergencies in the state is the illegal dumping of chemical waste. In this context, State Decree No. 59263/2013, which regulates Law No. 13577/2009 on the protection of soil quality and the management of contaminated areas, created the State Fund for the Prevention and Remediation of Contaminated Sites (FEPRAC) to identify and remedy these so-called “orphan areas”. Addressing the complexities of chemical waste disposal when the source of the waste cannot be identified, FEPRAC presents itself as an economic instrument that can eliminate the danger in such cases by supporting the proper removal, storage, characterization and final disposal of such waste based on Contaminated Sites Management procedures.

Leaching tests on columns to evaluate the potential of pollutants in sludge and sludge/soil mixtures

Water treatment plant sludge is a residue from the production of drinking water generated in large quantities in Brazil. Most of this sludge has been disposed in river which can alter its quality and cause risks to human health and aquatic organisms. Some examples of beneficial uses of this sludge are cement and brick making, plant cultivation. Geotechnical and environmental studies, involving sludge and soil mixtures to enable the use of this sludge in landfills, have been coordinated by a researcher at the Escola Politécnica of the University of São Paulo in partnership with researchers from the Nuclear and Energy Research Institute (IPEN) and the Technological Institute of Aeronautics (ITA). These studies have received financial support from the São Paulo Research Foundation (FAPESP) and the Basic Sanitation Company of the State of São

Paulo (SABESP). At IPEN, column tests with sludge and sludge/soil mixtures have been established to simulate field conditions and to evaluate the potential of leachate pollutants from sludge and mixtures.

Studies and development of recycling technologies reuse of materials

Development of value-added materials from wastes: Environmental characterization and application

Brazilian coals are richer in ash (~ 50 wt.%) and poorer in carbon when compared to worldwide coal and coal ash disposal is a serious environmental concern. Only 30% of fly ash is applied as raw material for cement and concrete production. The remaining solid wastes are disposed in on-site ponds, nearby abandoned or active mine sites, or landfills. Reuse of coal ash may provide a new source of revenue for coal-fired power plants and may offset expenses associated with its disposal. In the environmental aspect, the increased use of coal ash can reduce energy consumption, reduce the need for additional landfill space, and conserve natural resources.

In our group, we have been developing nanoporous zeolites synthesized from coal fly ash by conventionally hydrothermal treatment and fusion method. Pure zeolites X and A, surfactant-modified zeolites and zeolitic materials were synthesized and characterized. In addition, zeolites were used as low-cost adsorbents. The results obtained in the project showed that zeolites are highly efficient materials for the removal of dyes and toxic metals from aqueous solution.

A simple synthetic route to the formation

of Al-substituted tobermorite, katoite, hydrocalumite and zeolite hydroxysodalite by hydrothermal treatments of semi-dry flue gas desulfurization, a waste generated from the desulfurization system of coal-fired power station, has been demonstrated. All synthesized materials present ionic exchange capacity and could be used as low-cost adsorbent.

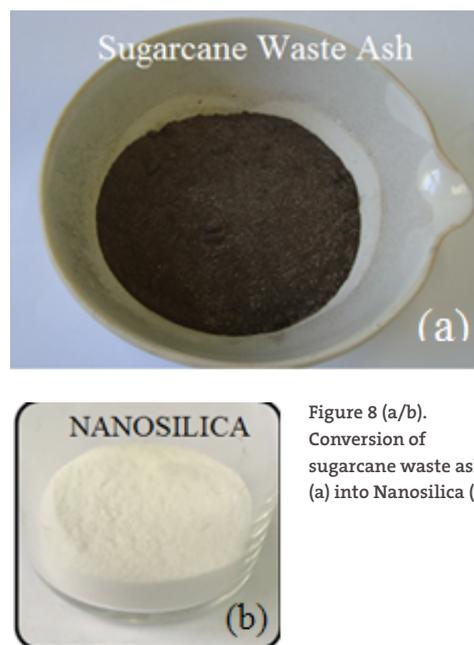


Figure 8 (a/b). Conversion of sugarcane waste ash (a) into Nanosilica (b)

Brazil is the largest producer of sugarcane in the world. In the harvest 2015/16, 665.6 million of tons of sugarcane were produced. The processing of sugarcane to produce sugar and ethanol generates various agricultural wastes, especially straw and bagasse. About 50% of these residues are used as a source of energy and the burning generates 1-4% ash. Another line of research has been developed focusing the conversion of sugarcane waste ash into nanosilica (Fig. 8). Pure amorphous silica was successfully extracted with 99.1% yield from sugarcane waste ash by a two-stage heating process followed by precipitation. The results suggest that sugarcane waste ash could be converted into a value-added product, minimizing the environmental impact of disposal problems.

Adsorption and desorption studies of dyes and pharmaceutical compounds using biocarbon ceramics

The agriculture business knows Brazil and their products around the world. The fertilizer industry has performed high yield in food production with tendency to increase daily. The study of fertilizers desorption is essential to modelling and predict its nutrients releasing capacity, mostly to improve its use benefits and avoid the environmental impacts of high nutrients discharge in surface water resources. The project aims to the use of Brazilian biocarbon as slow release fertilizer in the early steps to control the nutrients input to the environment by desorption processes improvements.

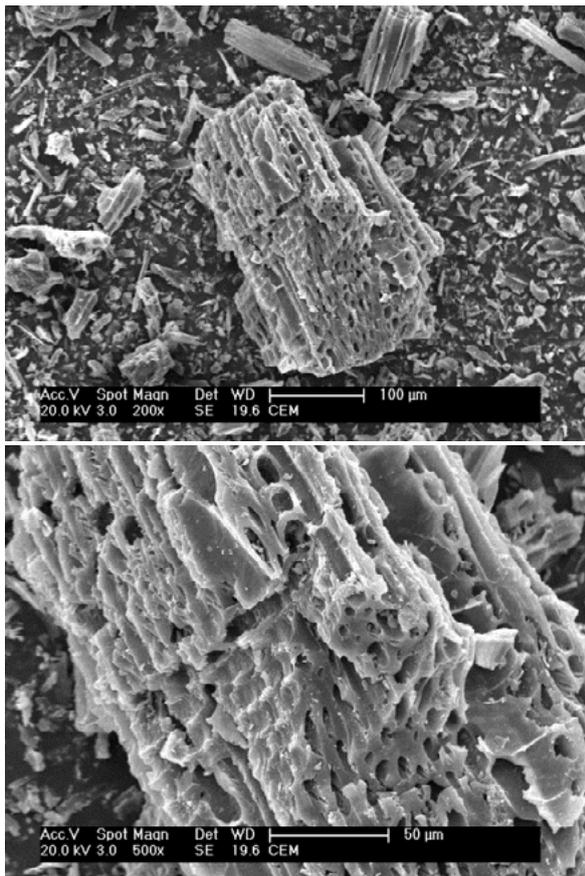


Figure 9 (a/b): SEM Micrography of biocarbon sample with 200x and 500x of magnification

Raw domestic sewage discharges are the primary sources and the most responsible

contamination for pharmaceutical and endocrine disruptors in water courses. Nowadays, it is widely known that the pharmaceuticals compound present in water pollution is considered the greater responsible for the increasing bacterial resistance. Micronized biocarbon and ceramic sorbents as magnetite, diatomite, and water treatment plant sludge (WTP sludge) have desirable properties for environmental conservation. The uses of such adsorbent material with chemical stability, high surface area, low toxicity and especially for magnetite, the magnetic response enhance the water treatment efficiency with low cost (Fig. 9).

Analytical Chemistry & Metrology

Quality system based on ABNT ISO/IEC 17025 Norm

In order to align to the requirements of Nuclear Fuel Cycle, a Quality System based on the ABNT ISO/IEC 17025 norm is being implemented in the analytical laboratories. As required, laws and rules compliances, personnel training, documentation, processes and environment monitoring and controlling are examples of what kind of issues are to be done. Existing analytical procedures are being configured and new ones are in course according to that norm. As the system will be implemented, many benefits are expected not only about organization, but also in the quality of results. Procedure validation and the estimative of measurement uncertainties allowed increasing the knowledge of what is being done. In a broader view, this local system is part of the institutional integrated quality system management policy which includes other norms of conformity, such as the ABNT NBR ISO 9001. As the system is implemented, higher levels of standards are

met showing the way for total conformity assessment.

Analytical chemistry for environmental diagnosis

The Laboratories of Chemistry and Environmental Diagnosis Center – CQMA have established methodologies for evaluation of physical-chemical, chemical and toxicological parameters to support several research projects in development for environmental diagnosis. All methodologies adopted are established in the standard methods (ASTM, EPA) or specified by the clients. The classic methods and instrumental techniques analysis such as atomic absorption spectrometry (AAS), inductively coupled plasma emission spectrometry (ICP-OES), ion chromatography (IC), gas chromatography (GC), gas chromatography mass spectrometry (GCMS), high performance liquid chromatography (HPLC);, X-ray fluorescence (WD-XRFS) have been used. The Center has been participating in several international and national interlaboratory programs sponsored by: IAEA (International Atomic Energy Agency), INTI (Instituto Nacional de Tecnología 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).

Determination of a Ca/P Molar Ratio in Hydroxyapatite (HA) by X-Ray Fluorescence Technique

Hydroxyapatite (HA) is a mineral composed of calcium phosphate employed for endodontics, restorative dentistry and other applications in orthopedics and prosthesis. Additionally, this biomaterial is an inexpensive but efficient adsorbent for the removal of heavy metals

and other unwanted species of contaminated liquid effluents. This is especially interesting when low-cost effective remediation is required. A Ca / P molar ratio of 1.667 is consistent with the theoretical Ca / P ratio for calcium hydroxyapatite with a compositional formula of $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, whose properties are well discussed in the literature. The aim of this work was implementing and validating a methodology for simultaneous determination of major and minor constituents in the hydroxyapatite (HA) as well as providing the Ca / P molar ratio. To accomplish these achievements, wavelength dispersive x-ray fluorescence spectroscopy (WDXRF) was applied. This is a non-destructive technique that requires no chemical treatment, enabling fast chemical analysis in a wide variety of samples, with no hazardous waste being generated as a result of the process of determination. A standard reference material from NIST (SRM 1400 –Bone Ash) was used to validate the methodology for the determination of magnesium, phosphorus, potassium, calcium, iron, zinc, strontium and the Ca / P ratio in HA samples by WDXRF. The Z-score test was applied as a statistical tool and showed that the calculated values were of less than 1.8 for all the measured analytes.

X-Ray Fluorescence Techniques applied to environmental, geological and biological studies Chemical characterization of nuclear and non-nuclear materials R&D activities

The X-Ray Fluorescence Laboratory (LFX) has worked in environmental, biological and nuclear areas, establishing new analytical methodologies using the X-ray fluorescence spectrometry (WDXRF, EDXRF). Different matrices, such as soils, sediments, lubricating oils, treated wood, metal environmental pollution monitoring trees, organic fluids

and nuclear and non-nuclear materials have been analyzed. The P&D activities have been supported by CNPq, FINEP and IAEA research financial organisms. The main projects, carried out and ongoing, are listed below.

Environmental area Environmental diagnostic: Metal pollution: Brazil stands out in successful biofuel technology in the automotive full flex type. Currently, full flex type cars, (83% of demand), amount to about 3 mi vehicles. The determination of wear metals in used lubricating oils is an important factor for preventative maintenance and motor engineering performance, in the automobile area, and for the concern about public health and environmental pollution. Wear metals have been determined in the used lubricating oils collected from biofuel vehicles, full flex type, to evaluate environmental impact in metal release. The project has been performed with GM (General Motors) cooperation. Wood preservation: Brazil produces around 1,2 mi m³ of treated wood to meet the annual demand of railways, electric, rural and construction sectors. The treated wood should be according to Brazilian norms; the majority of the wood preservative product used in Brazil is CCA (chromated copper arsenate). The analytical methodologies using the EDXRF (Energy dispersive X-ray Fluorescence Spectrometry) and PXRF (Portable X-ray Fluorescence Spectrometry) have been performed to Cu, Cr and As determination, in treated eucalyptus (*Eucalyptus ssp*) woods. The results will be compared with FAAS and NAA data. This study has been carried out in a joint work with IPT (Instituto de Pesquisas Tecnológicas, SP). Clinical and biological area Elements such as Na, Mg, P, Cl, S, K Ca and Fe in the whole blood of mdx humans (healthy population) and laboratory animals (Dmd /J mice, Crioula breed horse, golden hamster) were analyzed for reference interval values determination. Chemical char-

acterization of biological materials, such as rabbit urinary stone, lung tissues and saliva from *Amblyomma Cajannense* specie, has been outlined for clinical studies. Electrolytic elements, such as Ca, Cl, K, Mg, Na, S, Fe and Zn are important for athletes submitted to constant and hard physical efforts, because their health and physical performance depend on the variation and concentration of these electrolytic elements. The analytical methodology for the determination of the electrolytic elements, in whole blood samples, has been established by EDXRF (Energy dispersive X-ray Fluorescence Spectrometry) for the long distance runner; and the results will be compared with NAA (Neutron Activation Analysis) data.

Nuclear area Nuclear fuel: The chemical characterization of the nuclear fuel U₃Si₂ used in the IEA-R1 reactor has been performed by WDXRF spectrometry. U and Si content plus impurities, such as B, Mg, Al, total Ca, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, Cd, Sn and Pb have been determined using the Multivariate Calibration method. Nuclear related materials: In the fabrication of nuclear reactor core parts, Zircalloys machining scrapes are generated. Two metallurgical processes (VAR furnace and powder metallurgy) were applied to the evaluation of the scrap recycling process, at the CCTM (Centro de Ciência e Tecnologia de Materiais, IPEN). The characterization of the major constituents (Cr, Fe, Sn and Hf) and impurities, such as Al, Ca, Cu, Mg, Ni, Mn, Nb, Pb, Si, Ti, V and W, was determined by EDXRF spectrometry. Sulphur withdrawal degree in different organic solvents has been performed by EDXRF spectrometry. Eye makeup, known as eye shadow is an old and recurring custom and form in various cultures for body ornamentation and one of the earliest reports of the use of makeup dates from ancient Egypt. In Brazil, the use of cosmetics is common in several age groups, and commercially easy to

access, being the responsibility of the National Agency of Sanitary Surveillance (ANVISA) the supervision of these products as to the safety margin in the application in which they are destined, basing this index in international standards and daily dose. In the shade manufacturing process, inorganic dyes and pigments such as iron oxide, titanium dioxide, copper, chromium oxide on a talcum, cream or other base used as carrier are used to obtain the coloring. During the shade formulation process, elements such as As, Ni, Pb, Hg, Cd, Ni and others can be submerged, causing significant toxicity to the human body. In this context, the present project intends to evaluate the content of these potentially toxic elements in different shade samples using the energy scattering X-ray fluorescence spectroscopy (EDXRF) technique. The tomato is an herbaceous plant of the Solanaceae family native to Central and South America, and its culture is widespread throughout the world. In Brazil, the tomato crop is common to all states, especially those of São Paulo, Minas Gerais and Goiás, with an average production of 80 tons / year, being one of the largest productions in the world. The most common form of tomato consumption is in natura, including bark, pulp and seeds, making it the basic component of the daily diet of the population, contributing as an important source of replacement of necessary nutrients to the human being. Among them, lycopene, vitamin C, proteins, carbohydrates, fibers and the essential elements Na, Mg, Al, P, S, K, Ca, Mn, Fe, Cu, Zn and Se can be highlighted. The concentration of the essential elements in the seeds may vary depending on the variety, seasonal and geographical factors. In this context, this project intends to consider only the aspect related to the in natura consumption of tomatoes, commercialized in the metropolitan region of São Paulo, to determine the content of essential elements by EDXRF and to establish a relation as to the contribution and importance

in human consumption. The X-ray fluorescence laboratory offers an undergraduate (IPN 0014:X-ray fluorescence spectrometry) at USP (University of São Paulo) and a graduate course (TNM 5813:X-ray fluorescence spectrometry, theory and application) in Nuclear Technology program in association with the University of São Paulo, USP. Also, X-ray fluorescence short training extension courses have been offered for researchers and technicians of Brazilian and Latin American universities and industries. With quality assurance in the period, the LFX has continued the ABNT NBR ISO/IEC 17025:2005 norm establishment. Several topics of the Quality Manual were improved and adapted according to new requirements. The annual internal audit has been carried out by SGI/IPEN.

Other studies and P&D are described in the item: Nuclear reactors and fuel cycle - Chemical metrology & Quality Control in nuclear laboratories.

Implementation of an Environmental Management Program on the campus of IPEN

Implementation of an Environmental Management Program on the campus of IPEN in response to environmental agencies and IBAMA (Environmental License L.O. 1325/2016) and based on Environmental Management System ABNT NBR ISO 14000:

- > Monitoring Program to assist the non-radioactive chemical at IPEN campus;
- > Non- radioactive chemical waste disposal program;
- > Program for Monitoring and Control of Non-Radioactive Atmospheric Emissions;
- > Non-Radioactive Solid Waste Management Program;
- > Environmental Chemistry Monitoring Program (PMA-Q);
- > Report of the Social Communication Program;
- > Environmental Education Program, and
- > IPEN Annual Report on Environmental Management.

Temporal Comportment Assessment of metals in groundwater on the campus at IPEN/CNEN-SP

Since 2006, Nuclear and Energy Research Institute (IPEN) performs yearly the Environmental Monitoring Program of Stable Chemical Compounds (PMA-Q). Among

other parameters, metals and semi metals in groundwater, collected at Ipen's facility, are evaluated. The monitoring is conducted in nine wells, in attendance to the current Brazilian environmental legislation, which requires the monitoring of metals and semi metals in groundwater, in accordance with CETESB and CONAMA's resolutions. CETESB is the Sao Paulo State environmental regulatory agency and CONAMA is the Environmental National Council, both agencies that regulate environmental standards in Brazil and regulate Ipen's environmental activities. Besides these two environmental regulators, Ipen has to follow the request of the Term for the Adjustment of Conduct (TAC) from (IBAMA), in order to support programs to prevent and control pollution resulting from activities of Ipen's facilities. In the current PMA-Q, aluminum (Al) antimony (Sb), silver (Ag), arsenic (As), lead (Pb), chromium (Cr), cobalt (Co), zinc (Zn), boron (B), barium (Ba), calcium (Ca), iron (Fe), manganese (Mn), mercury (Hg) and nickel (Ni) are analyzed by using sensitive analytical techniques as inductively coupled plasma optical emission spectrometry (ICP-OES) and graphite furnace atomic absorption spectrometry (GF-AAS). These elements results are evaluated in this paper. Both internal and external quality controls that uses data from interlaboratory programs are discussed here. It was possible to conclude that IPEN's groundwater attends national standards and Ipen's monitoring system operates under controlled quality conditions.

Atmospheric Emission Factors and Significance Analysis applied to the Air Quality Management in the IPEN / CNEN-SP Campus

Due to the commitment to improve the environment, combined with the increasing demands of environmental agencies, and the need to identify the contribution of each activ-

ity / process developed in research institutes, as well as the impact of these on the air quality, this work aims to develop an inventory model and apply a methodology of calculation for measuring the emission of atmospheric pollutants, arising from the activities of the research and development centers of the Nuclear and Energy Research Institute (IPEN / CNEN-SP). The study was motivated by the absence of atmospheric emission calculation methodologies specific to stationary sources, such as fume hoods. For preparation of the calculations, the emission factors and the equation described in the AP-42 EPA- Environmental Protection Agency were adopted. The emission calculation methods used were: a) Direct measurement (through the inventory of air emissions); and b) Emissions estimate (using the emission rate estimate calculated from the appropriate emission factor). As the final product, an inventory of atmospheric emissions from stationary sources of the institution was obtained and the atmospheric emission of the Chemistry and Environment Center (CQMA) was estimated in order to effectively establish the Program of Monitoring and Control of Atmospheric Emissions (PMEA - IPEN), providing the basis for other Research, Development & Innovation institutions.

Clean Technologies

Development of electrocatalysts for application in fuel cells

Pt and Pd-based electrocatalysts using as co-catalysts rare earths (La, Ce, Nd and Er) and Sn for application in fuel cells have been studied. PEMFC (Proton Exchange Membrane Fuel Cell) and DAFC (Direct Alkaline Fuel Cell) were the main focuses of the development. The electrocatalysts were prepared by borohydride method and the fuels were hydrogen/oxygen, ethanol and ethylene glycol.

The PtLa/C electrocatalysts in various proportions showed that the addition of lanthanum promoted a greater power density in PEMFC fed with hydrogen and oxygen. The Koutecky-Levich diagrams suggest that PtLa/C and PtCe/Ce 80:20 for oxygen reduction reaction occurs by the 4-electron mechanism as well the commercial Pt/C. Figure 10

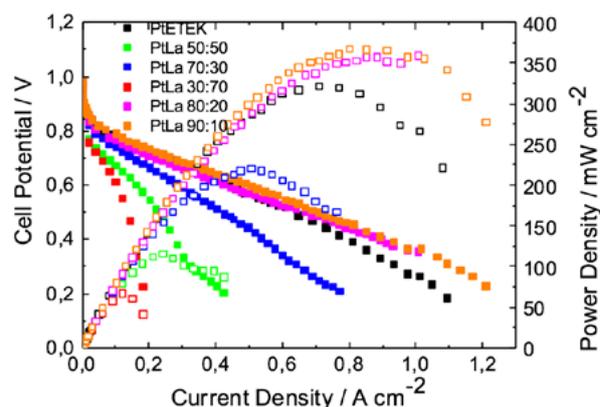


Fig. 10 Electrochemical performance of PtLa/C electrocatalysts as cathode compared to Pt/C E-TEK electrocatalyst in a fuel cell unit, fed with hydrogen and oxygen.

In the case of PtSn/C electrocatalysts in DAFC fed with ethylene glycol the results showed a better performance in the proportion Pt:Sn 70:30. These results may be associated to the selectivity to form oxalate or a bifunctional mechanism (oxygenated species from Sn). Figure 11

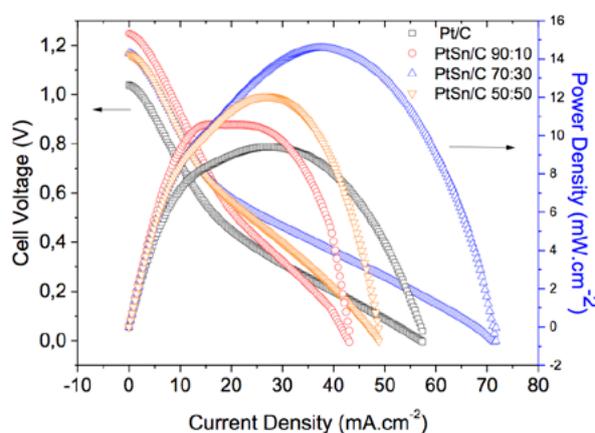


Fig. 11 Performance of the ethylene glycol fuel cell for Pt/C and PtSn/C electrochemical catalysts, at 100°C, prepared by the borohydride method, KOH treated Nafion 117 membrane, EG + KOH electrolyte (2 mol L⁻¹), flow rate of 1 mL min⁻¹.

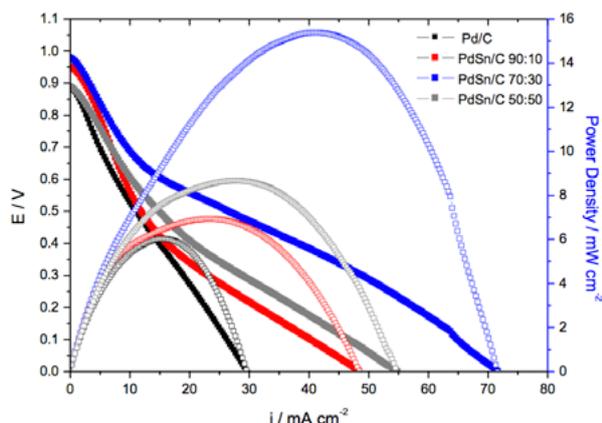


Fig 12. Performance of ethylene glycol oxidation in a single DAFC for Pd/C and PdSn electrocatalysts at 60°C.

For PdSn/C electrocatalysts, used in DAFC as anode, the 70:30 proportion also showed the lowest onset potential for EG oxidation reaction, highest current density in chronoamperometric experiments and in single cell performance test, probably due to the differentiated alcohol adsorption favoring the kinetic and the formation of most oxidized products. Figure 12

Microwave technology

Development of units of reaction with microwave, for operation in high pressure and high temperature & The use of microwave technology in: hydrotreating process intensification of middle distillates, production of biodiesel from vegetable oils and additional purification of Mo-99 produced from LEU targets.

Several types of process can benefit from microwave technique. It has been studied all over the world to identify (qualitatively and quantitatively) and define the mechanism of microwave-material interaction. The application of this process in our country is a very recent field and has been studied as a new tool in materials for hydrotreating process intensification of middle distillates, production of biodiesel from vegetable oils and additional purification of Mo-99 produced from LEU tar-

gets. 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 a 2,45GHz (which corresponds to a wavelength of 12,24cm) to avoid interference with telecommunication and cellular phone frequencies. Microwave processing offers numerous advantages in relation to conventional heating methods (convection or conduction), where the material surface heats first and then the heat moves inward. One of the most important characteristics is saving energy, because the material absorbs microwaves readily (the heat is generated from the inner parts to the surface of the material) reducing the processing time. Also, the selective energy absorption allows heating in specific points of the material. Finally, the microwave heating requires no appreciable amount of time to effect temperature changes such as conventional methods and when the microwave device is turned off the effect of these electromagnetic waves are instantaneously stopped. Two reaction systems (batch and continuous flow mode) microwave-assisted and conventional heating for processing of several materials have been used at IPEN-CNEN/SP. They operate at high temperatures (until 500°C) and under high pressures of hydrogen (until 20MPa) Figure 13 and Figure 14.



Fig. 13 – Batch reaction system microwave-assisted and conventional heating.

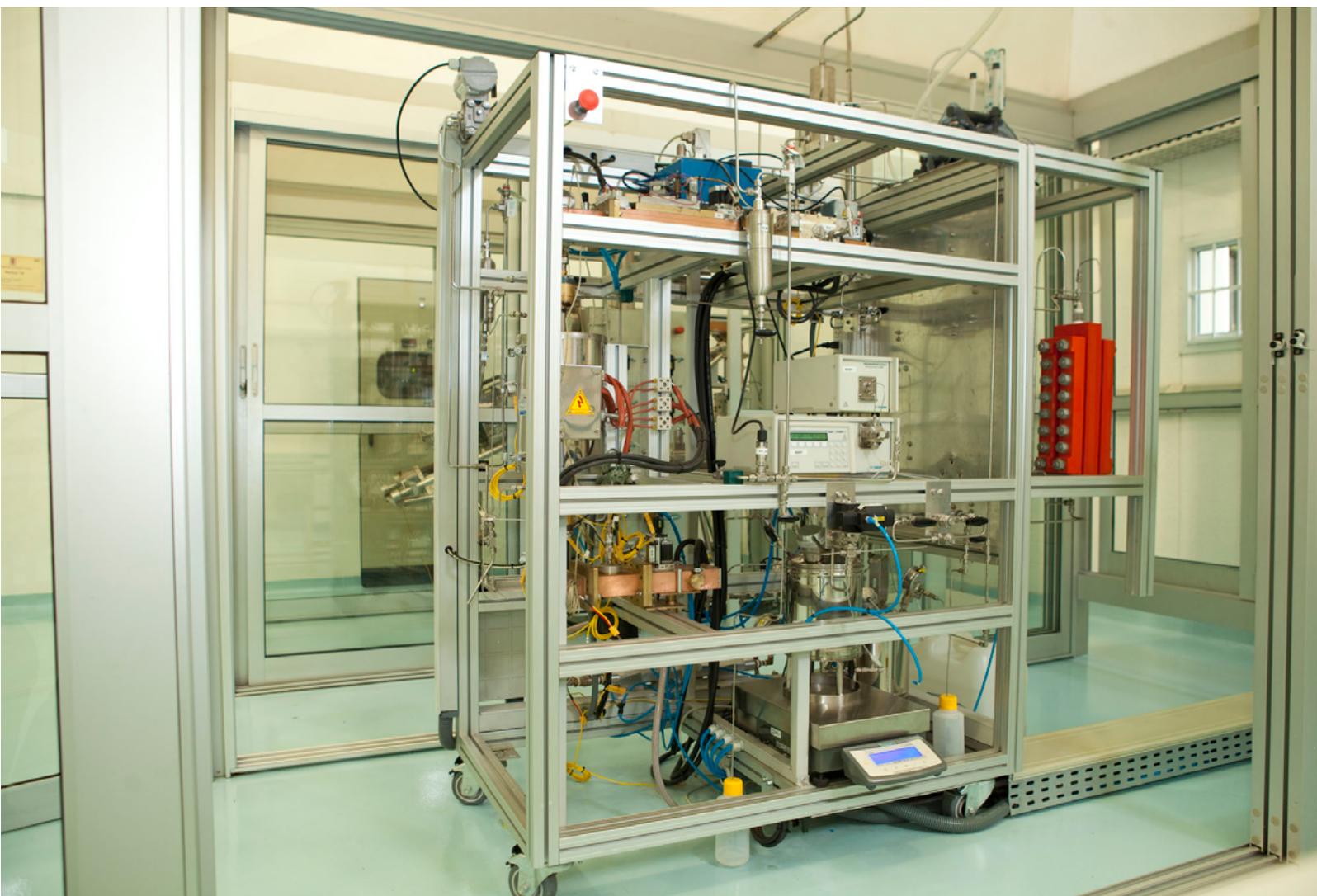


Fig. 14 – Reaction system of continuous flow microwave-assisted and conventional heating.

These apparatus are connected to an electronic control and supervision unit which enables interaction with, and measurement and monitoring of, the variables involved in the process. A Programmable Logic Controller (PLC) digitalizes and processes the signal for the microwave radiation (transmitted and reflected) identified by the wave sensors by means of a computer program. The development and

research “Development of microwave reaction units for operation in high pressure and high temperature” and “The use of microwave technology in: intensification of Hydrotreating process of middle distillates” were carried out in partnership with CENPES/Petrobras.

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HONOR MENTION AND AWARDS

- The research works “Adsorption of Rhodamine B Dye from Aqueous Solution by Surfactant Modified Zeolite from Coal Bottom Ash” and “Comparative Study of Methods for the Synthesis of Silica Gel from Biomass Residue Ash of Sugarcane” presented in 5th International Workshop on Advances in Cleaner Production, 2015, received an honorable mention.
- In 2016, Fungaro received the KURT POLITZER AWARD FOR TECHNOLOGY, in the Researcher Category, for the Project “Production of High Purity Silica and Nanosilica from Sugarcane Biomass Ash with High-Potential Target Market”.
- Received an honorable mention: in the field of chemistry of radiation and photochemistry. Polish Radiation Research Society - Marie Skłodowska Curie Memorial - 2016: Gustavo H. C. Varca, Ademar B. Lugão and Rodrigo G. Queiroz
- Excellent Reviewer, International Journal of Pharmaceutics - IJP, Elsevier (Amsterdam), 2014. Gustavo H. C. Varca
- 1° Prize Miyaky Issao award. Dental Materials/ Scientific student. 33° Meeting of the Brazilian Society of Dental Research, Brazilian Society of Dental Research (SBPqO), 2016,
- 1° Prize of Oral Presentation / Dental Materials. Brazilian Society of Dental Research (SBPqO), 2016,
- Honorable Mention in the 52° GBMD - Brazilian Group of Dental Materials: postgraduate category, Brazilian Group.
- Received an honorable mention: the best poster 2016 CBRATEC e CPANATEC, CBRATEC, 2016
- 1° Prize Effective-Aspirant Panel in the 32° Meeting of the Brazilian Society of Dental Research, Brazilian Society of Dental Research (SBPqO), 2015
- CAPES PRIZE OF 2014 for the best thesis in Engeneering II. Title : Synthesis and Characterization of nanogel and micrigel for drug release. 2013, author Maria José Alves de Oliveira, grants from FAPESP, doctorate in Nuclear Technology of Materials- IPEN/CNEN. Supervisor : PhD Duclerc F Parra.
- 1° Prize in Oral Presentation. 33° Meeting of the Brazilian Society of Dental Research, Brazilian Society of Dental Research (SBPqO), 2016,

the 1990s, the number of people in the UK who are aged 65 and over has increased from 10.5 million to 13.5 million, and the number of people aged 75 and over has increased from 4.5 million to 6.5 million (Office for National Statistics 2002).

There is a growing awareness of the need to address the needs of older people, and the need to ensure that the health care system is able to meet the needs of older people. The Department of Health (2001) has published a strategy for older people, which sets out the government's commitment to older people and the need to ensure that the health care system is able to meet the needs of older people.

The strategy for older people (Department of Health 2001) sets out the government's commitment to older people and the need to ensure that the health care system is able to meet the needs of older people. The strategy is based on the following principles:

- Older people should be able to live independently and actively in their own homes.
- Older people should be able to access the services they need to live independently and actively in their own homes.
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