

Elemental Characterization and Speciation

CEEFI

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The Elemental Characterization and Speciation Group of the LFI (CEEFI), carries out work of research, development and application of ion beam based nuclear analytical techniques for the characterization of samples elemental composition, aiming also at speciation methodologies. So far, the main focus was put on particle induced x-ray emission (PIXE) and airborne material. Atmospheric environment related samples like airborne particulate matter and/or biomonitoring samples being different faces of this focus. A tuning of this working line is presently being undertaken in order to cover a broader but more concise focus on small mass samples such as particulate matter (airborne or not), nanoparticles, macromolecules or thin film samples,

Strategically, the group assumes that it is important not to depend exclusively on collaborations neither for sampling nor for data handling processes. Therefore, a strong R&D effort is put on airborne particle sampling and data handling methods. Data handling R&D includes both spectra analysis, as well as environmental data analysis (namely inverse methods for source apportionment).

Taking into account that PIXE is already a matured analytical technique, services are provided to the community in general, and to the scientific community in particular. In this cases, analysis of samples other than small mass ones is carried out, and it is not rare that important spin offs associated to details or specific developments of the PIXE technique, do emerge in this framework.

Within the organics of the Ion Beam Laboratory (LFI), CEEFI is responsible for the maintenance and improvement of PIXE facilities, and assures that, at least, are no losses on the installed capacity occur.

In 2007, the groups' activity was focused on the implementation of the Laboratory for Characterization and Speciation of Aerosols (LCEA), a major equipment upgrade based on a large re-equipment project. Equipment acquisition and installation were tasks using a fair chair of the time. The project contributed to the acquisition of a refurbished Tandemtron accelerator where the new PIXE setup for High Energy and High Resolution PIXE is under installation. The first High Energy spectra were obtained and presented at the International PIXE Conference that took place in Mexico.

Apart from this the major lines of work involve: (1) x-ray lines relative intensity studies, these have been shown to present an apparent dependence on the beam energy, which origin is under study and is subject of a PhD thesis in ion beam analysis speciation; (2) improvements on the existing data handling software, including holistic approaches and the recovery of some lines of work that had been abandoned in the middle of the 1990 decade (eg: ion ionization cross-sections) which are again becoming an important subject due to the recent developments in both software and hardware (namely fact that a 10eV resolution x-ray EDS detector is about to be installed at ITN as one of the major outcomes from the LCEA project). Finally, the database on airborne element content measurements in Portugal dating back to January 1995 was completed and is being formatted for publication during 2008.

Researchers (*)

M.A. REIS, Aux. (Coordinator)

Students

P.C. CHAVES, Ph.D. student, FCT grant

Technical Personnel

R. PINHEIRO

Collaborators

G. DIAS, Graduated (until July 2007)

R. MALVEIRO, Graduated (April to August 2007)

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Micro and Nano-particles Characterization and Speciation

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The characterization and speciation of small mass samples like airborne particles, is the basis and central issue of the CEEFI group work. Each of the specific activities of the group therefore converges towards this aim, or emerges from it. During 2007, the main activity besides the operation of an airborne particle sampling unit at ITN campus, was the installation of the Laboratory for the Characterization and Speciation of Aerosols (LCEA), the development of software and the study of Mo and Gd compounds X-ray lines relative intensity as function of the incident proton beam energy. All of the tasks converging to the same goal, namely using particle induced x-ray for determining chemical and/or sample size effects.

Presently almost all major installations planned for LCEA are installed, namely a SO₂, CO₂, NO, NO₂, O₃ and gas phase Hg DOAS analyser, working in continuous monitoring, a meteorological station, and the new HEHR-PIXE line, installed on the recently acquired 3MV Tandetron. In the beginning of 2008 the 10 eV resolution POLARIS EDS microbolometer x-ray detector system from Vericold Technologies GmbH, will become operational.

In respect to the study of the Gd compounds it was shown that spectra from pellets of Gd oxide powder do not present the same pattern of variation with the energy of the incident ion beam as 5nm particles of the same material, while these present sometimes patterns similar to those found during the study of pellets of Gd-DOTA, a common MRI contrast agent (see fig. 1).

In the case of Mo, it was shown that the new developed PIXE spectra software was able to provide from the deconvolution of spectra obtained using a Si(Li) detector, results similar to those obtained using a ultra high resolution detector from the JSI, in Slovenia. The same results also showed that different Mo compounds present different patterns for the variation of the x-ray line ratios with the energy of the incident ion beam.

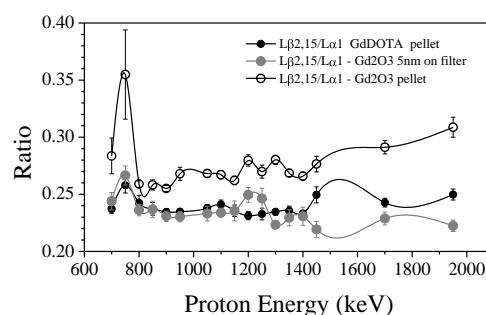


Fig. 1. Change in the ratio of intensity of lines $L_{\beta 2,15}$ and $L_{\alpha 1}$ in three samples of Gd, as function of the energy of the incident protons. Being transitions to the same L-subshell, this effect is presently unexplainable.

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