Summary

Habilitation Thesis

Contributions in the development of miniaturized instrumentation and advanced analytical spectral methods using plasma sources

The Habilitation Thesis reviews the research activity, main results and future projects of the author. After obtaining the PhD degree in chemistry in 1996 at Babes-Bolyai University of Cluj-Napoca, my research activity has been focused primarily on the development of miniaturized instrumentation, study of microplasma sources and development of advanced spectral methods based on these plasma sources. The thesis comprises two parts:

(1) Research activity and main results;
(2) Future projects

Research activity and main results

Chapter I. Multielemental analysis methods for liquid samples by capacitively coupled plasma microtorch optical emission spectrometry. This chapter presents the original miniaturized instrumentation based on a capacitively coupled plasma microtorch of low power (10 – 30 W) and low argon consumption (100 – 300 ml min^{-1}) interfaced with low resolution microspectrometers aimed for elemental analysis by atomic emission in liquid samples after pneumatic nebulization or electrothermal vaporization from a Rh filament. The instrumentation was design and constructed by our research group. The analytical performances and applications related to environmental samples, as well as comparative studies with traditional analytical systems are presented.

Chapter II. Arsenic and antimony determination using hydride generation capacitively coupled plasma microtorch optical emission spectrometry. The chapter presents the main results and contributions that have made possible the simultaneous determination of As and Sb by optical emission spectrometry in a capacitively coupled plasma microtorch and detection with a microspectrometer, after derivatization to hydride with low reagent consumption. Both the instrumentation and method were characterized in terms of analytical performance and then applied on real samples of soil and non- or biodegradable materials. The statistical evaluation revealed that the miniaturized instrumentation including a capacitively coupled plasma microtorch provides results similar to the traditional method based on hydride generation and detection by optical emission spectrometry in inductively coupled plasma.

Chapter III. Mercury determination using cold vapour generation capacitively coupled plasma microtorch optical emission spectrometry. The main achievements related to Hg determination at extremely low concentration in different samples, such as water, soil, sediment, non- and biodegradable materials, and food using miniaturized equipment including a capacitively coupled plasma microtorch and a microspectrometer for optical emission measurements are presented. The main devices designed and realized in order to comply with the analytical demands associated to mercury determination are described. The proposed analytical method was compared with those traditionally used for Hg determination by atomic fluorescence spectrometry and atomic absorption spectrometry after direct desorption from solid sample. The method was also verified in terms of compliance with requirements of the European legislation for mercury determination in food. The conclusion was that the miniaturized instrumentation equipped with a capacitively coupled plasma microtorch could be successfully used for mercury determination given that the performances are similar to those of classical instrumentation and meet the legislation demands in terms of analytical capability.

Chapter IV. Optical emission and fluorescence spectrometry in medium power radiofrequency capacitively coupled plasma source. This chapter presents the results relative to the opportunity to use a capacitively coupled plasma torch of medium power (275 W) as excitation source in optical emission and fluorescence spectrometry. The investigated plasma source provided excellent results in optical emission for multielemental analysis and especially in atomic fluorescence for the determination of Cd, Pb and Zn. An original method is presented for quenching of the OH and nitrogen molecular emission by methane addition as collision/reaction gas, which enables determination of Pb determination by atomic fluorescence spectrometry free from OH interference, using a low resolution microspectrometer. Both atomic emission and fluorescence spectrometry were applied for the analysis of various samples (multimineral supplements, supraconductive materials, soil, water) and compared to inductively coupled plasma atomic emission spectrometry. On the basis of preliminary results it has been demonstrated that the capacitively coupled plasma can be a source of ions for mass spectrometry.

Future projects

The atomic spectrometry using microplasmas/microtorches is a relatively new and dynamic field of and I intend to develop it further in the future. The instrumentation developed up to now, although at miniature scale, was used only for methods applied in laboratory. Worldwide the trend is the development of on-site methods applicable on miniaturized instrumentation supplied from rechargeable batteries. Such new methods are particularly necessary as they eliminate the need to preserve samples after collection during transportation to laboratory, which is the major source of errors. The instrumentation realized at miniature scale can be supplied from rechargeable batteries thus opening the door to the development of on-site methods.

The research directions I want to continue are:

1. Embedding of the existing miniaturized capacitively coupled plasma source in portable equipments;
2. Development of on-site analytical technologies as alternative to traditional methods used in laboratory;
3. Development of green methods for ultrasensitive determination of elements in natural samples using optical emission spectrometry in capacitively coupled plasma source.

Expected results

- Generating new knowledge in the field of applied research of analytical microsystems as alternatives to the classical laboratory approaches;
- Analytical technologies for laboratory, on-site or a combination of them, with deep innovative character;
- Valorization of results through publication in ISI journals and patents;
- Development of a research group in the field of non-conventional analytical methods;
- Increasing human resource competence by participating PhD students in the projects.

The added value of research results at national and international level

- Improvement of the scientific research within the Department of Chemistry, Faculty of Chemistry and Chemical Engineering by involving PhD students in research of current interest;
- Development at national level of a new research field of high technology meeting the current demands and expectations in terms of chemical analysis and monitoring;
- Progress in evaluation of environmental and food quality using rapid methods of analysis and miniaturized laboratory/on-site instrumentation.