SPECTROSCOPIC ANALYSIS OF SOLUBLE COFFEE USING NUCLEAR AND ATOMIC TECHNIQUES

Cibele B. Zamboni, Vera A. Maihara, Frederico A. Genezini, *Leonardo D. Jr, Ana H. Bressiane and **Sonia P. Camargo

Instituto de Pesquisas Energéticas e Nucleares/CNEN
Caixa Postal 11049
05422-970

*Promon Engenharia
São Paulo, Brasil

**Universidade Santo Amaro
Caixa Postal 55201
04829-300, São Paulo, Brasil

ABSTRACT

This paper describes the application of two spectroscopic methods: Gamma-Spectroscopy and Energy Dispersive Spectroscopy (EDS) employed to qualitative identification of some available commercial soluble coffee. The concentration of the elements present in the samples, when it was possible, was calculated using Neutron Activation Analysis (NAA).

Key Words: coffee, nuclear spectroscopy, electron microscopy, neutron activation.

I.INTRODUCTION

The importance of essential elements to human health has stimulated the analysis of their occurrence in a wide variety of foodstuffs. Particularly, the coffee analysis is very important due to its high consumption by Brazilian population and also for its medical and nutritional use. Furthermore, a recent bibliographic review shows a lack of information about the coffee elemental composition.

In this study, the application of the nuclear analysis is appropriate due high sensitivity in the identification and quantification of the radioactive products (with Z>20), without destruction of the samples. In the atomic analysis, using the electron microscopy with EDS, it is possible qualitative information for elements with atomic number greater than boron (Z >5).

To perform this spectroscopic determination, various samples of commercial available soluble coffee, from different origin, were analyzed and the elements present in them were identified by γ-spectroscopy and EDS techniques.

II. METHODS

Four brands of coffee (classified by: A, B, C and D) that are consumed by local population were obtained from supermarket of the city of São Paulo. Each coffee sample was mixed and homogeinezeid in a domestic blender coated with teflon equipped with titanium blades. This was necessary to avoid any contamination from metallic parts during this step. Following this procedure it is possible to obtain samples with an adequate granulometry for performing all the analysis.

The nuclear analysis consist of the irradiation of coffee samples in a thermal and fast neutron flux, in the IEA-R1m Reactor at IPEN-São Paulo. Two independent γ-spectrometer systems with HPGe detectors of high resolution were used. The detectors were calibrated for energy and efficiency through the measurements of standards sources [1]. These samples were irradiated with neutrons in the nuclear reactor for different periods, from minutes to days, in order to identify and to quantify short and long lived isotopes present in the samples. The nuclear properties, such as energy of gamma transitions and half-life, of the radioactive products provide information about its elemental composition.

In the atomic analysis (EDS) the samples are placed in a Scanning Electron Microscopy (SEM) and were bombarded with electrons which causes the emission of

characteristic X-rays. Monitoring the wavelength of these X-rays, it is possible to identify the elements present in the samples. The detection limit of this method is ~ 0.5 wt % if the element is finely dispersed in the sample.

Measurements. In the γ-spectroscopy experiment the single spectra were taken with a 89cm^3 HPGe coaxial detector and an Ortec 671 amplifier, in pile-up rejection mode. The FWHM was 1.79keV for the 1332keV gamma ray of ^{60}Co. The background radiation was reduced by employing the iron shield described by Vanin et al. [2]. Various measurements of one minute each were taken within a period of 4 hours, for each sample of soluble coffee. The analysis of the γ– ray spectrum for short-lived isotopes revealed the presence of Mg, Al, Mn, K, Na, Cl, and Ca in all the samples and Cu, in the samples A and D, and V, only in the sample A. Single spectra were taken for long irradiation, too. In this case, measurements of one hour were done within a period of hours as well as day. Systematically, this procedure was repeated during three months and the radioactive isotopes of Br, Fe, Co (samples A and D), Sb (only in sample B) and Y (only in the sample A) were identified. Finally, after five months, the coffee samples were γ-counted again and only the presence of ^{60}Co and ^{124} Sb has been identified.

The identification of the γ-ray energies and their disintegration periods were analyzed using the IDF and the PANORAMIX programs [3,4].

An example of a gamma ray spectrum for short-lived isotopes, recorded during one hour, is shown in Fig.1(Energy in keV).

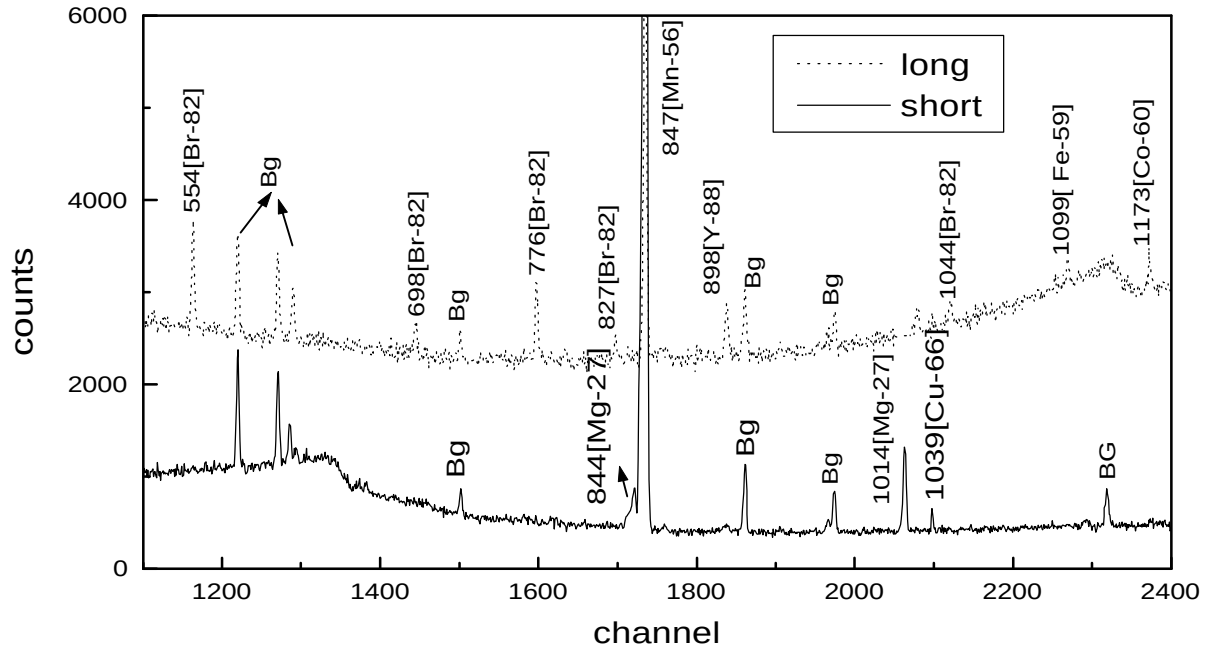

Figure 1. Partial Spectrum of Short and Long Lived Isotopes in the Soluble Coffee (Sample A).

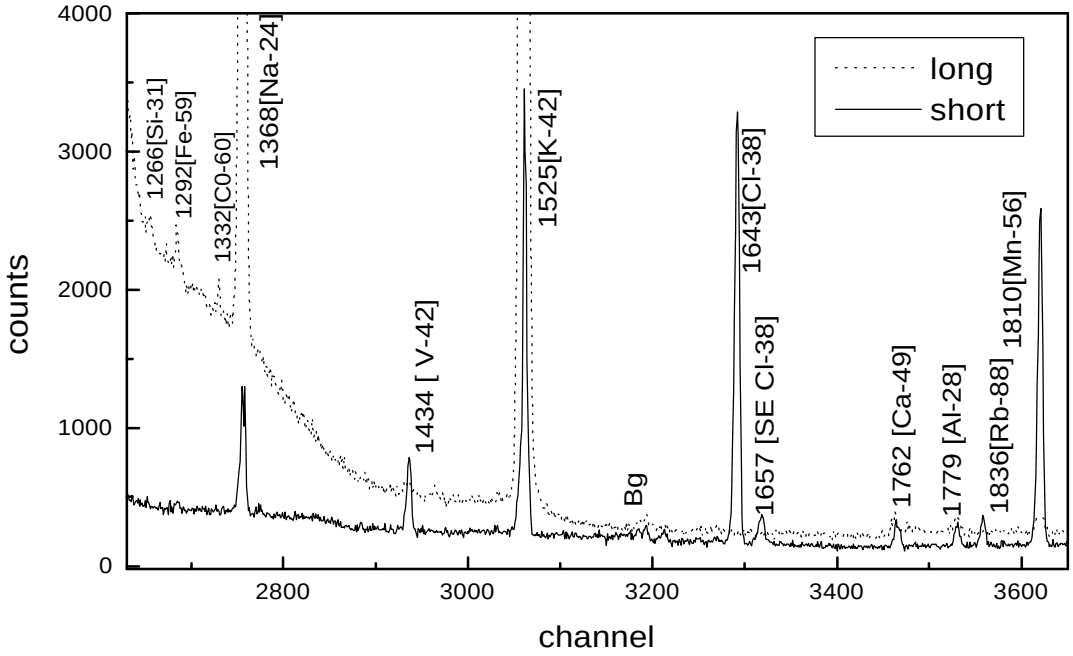

Figure 1. Partial Spectrum of Short and Long Lived Isotopes in the Soluble Coffee (Sample A).

In the atomic experiment, samples for EDS analysis must be able to withstand a vacuum environment and must be conductive (this can be accomplished by coating a non conducting specimen with a thin metal film or carbon film). This analysis generates a spectrum with qualitative elemental information for elements with atomic numbers greater than boron. Following this procedure, the results of the atomic experiment are presented in Fig. 2.The elements identified by γ-spectroscopy and EDS analysis are show in Table 1.

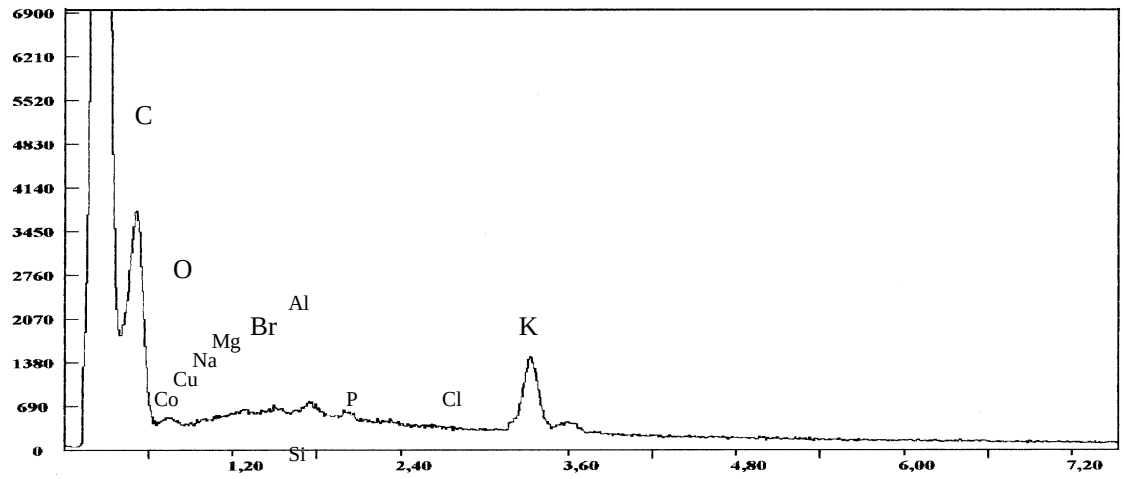

Figure.2 Representative SEM Spectra (numbers of counts by electron energy) from the Atomic Analysis to the Sample A

TABLE 1. Atomic Elements Identified in the Samples of Soluble Coffee in these Spectroscopic Analysis.

Samples of Coffee	Atomic Elements
Sample A: Lyophilized Coffee	C, O, Na, Mg, Al, Si, P, Cl, K, Ca* , V*, Mn, Fe*, Co, Cu, Br, Y*
Sample B: Decaffeinated Coffee	C, O, Na, Mg, Al, Si, P, Cl, K, Ca, Mn, Br, Sb*
Sample C: Traditional Coffee	C, O, Na, Mg, Si, P, Cl, K, Ca, Mn
Sample D: Traditional Coffee	C, O, Na, Mg, Al, Si, P, Cl, K, Ca, Fe, Co, Cu, Br.

C, O, Si and P only by EDS.
* only by γ-spectroscopy

The concentration of the elements Mg, Ca, Mn, Na, K, Cl, Cu and V were determined by NAA. The samples of coffee and aliquots of standard solutions, with well-known concentration, were used. The identification of the elements present in the samples was carried out by comparing with standards.
The samples were weighed and sealed in plastic envelopes. Aliquots of standard solutions of these elements were pipetted onto $1cm^2$ of Whatman N0 42 filter paper, evaporated to dryness under an infrared lamp. Samples and standards were irradiated at a thermal neutron flux of about $5\ 10^{11}$ $n/cm^2.s$ at the nuclear reactor. The γ-ray spectra were processed by using the VISPECT program [5].
To evaluate the concentrations of the elements present in the samples, the counting rates (area of photopeaks) corresponding to the γ-rays of the radioisotopes from samples and standards were compared. The results are presented in Table 2.

Table 2. Concentration of Elements in the Samples of Coffee by NAA.

Elements	Sample A (Lyophilized coffee) μg/g	Sample B (Decaffeinated coffee) μg/g	Sample C (Traditional coffee) μg/g	Sample D (Traditional coffee) μg/g
Mg	3118 ± 351	3530 ± 301	3673 ± 174	3345 ± 111
Ca	1397 ± 107	1502 ± 65	1491 ± 145	1551 ± 115
Mn	38 ± 2	34 ± 2	24.2 ± 1.7	24 ± 2
Na	291 ± 32	179 ± 15	1220 ± 42	1434 ± 107
K	55128 ± 2419	45595 ± 6896	46965 ± 2340	39593 ± 2661
Cl	1333 ± 128	1671 ± 143	801 ± 59	857 ± 81
Cu	31 ± 10	17 ± 11	nd	nd
V	0.20 ± 0.01	nd	nd	nd

nd: not determined

III. Discussion

Basically, the elemental composition of the samples of soluble coffee analyzed are given by: Al, Mn, Mg, K, Na, Ca, Si, Co, Cu, Fe, Br, and V identified both nuclear and atomic techniques, and by C, O and P identified only by EDS, due low number atomic (Z< 20). The elements Y and Sb, observed only in γ−spectroscopy experiment, are not present in all the samples and their concentrations, from NAA, are not calculated due to interference from other radioisotopes with γ- transitions of same energy and also to low statistic.

These spectroscopic techniques allowed qualitative identification of soluble coffee with compatibility among the results. These information can be useful in another research fields such as health and nutrition areas.

Financial Support: FAPESP, UNISA and CNPq

REFERENCES

[1] IAEA- **International Atomic Energy Agency, X-Ray and Gamma-Ray standards for Detectors Calibration**, IAEA - TECDOC-619, Viena, 1991.

[2] Vanin, V.R., A.Passaro, A ,and Passaro, A.M.P., **Decay of $^{101}Rh^{m}$ and $^{101}Rh^{g}$**. Phys. Rev. C32, p.1349-53, 1985.

[3] Gouffon, P., **Manual do programa IDEFIX**, Universidade de São Paulo, Instituto de Física, Laboratório do Acelerador Linear, 1982.

[4] Vanin, V.R., **Manual do programa PANORAMIX**, Universidade de São Paulo, Instituto de Física, Laboratório do Acelerador Linear, 1988.

[5] Piccot, D. Private communication, IPEN, 1989.