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Ion beam analysis of ground coffee and roasted coffee beans

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ABSTRACT

The way that coffee is prepared (using roasted ground coffee or roasted coffee beans) may influence the quality of beverage. Therefore, the aim of this work is to use ion beam techniques to perform a full elemental analysis of packed roasted ground coffee and packed roasted coffee beans, as well as green coffee beans. The samples were analyzed by PIXE (particle-induced X-ray emission). Light elements were measured through RBS (Rutherford backscattering spectrometry) experiments. Micro-PIXE experiments were carried out in order to check the elemental distribution in the roasted and green coffee beans. In general, the elements found in ground coffee were Mg, P, S, Cl, K, Ca, Ti, Mn, Fe, Cu, Zn, Rb and Sr. A comparison between ground coffee and grinded roasted beans shows significant differences for several elements. Elemental maps reveal that P and K are correlated and practically homogeneously distributed over the beans.

1. Introduction

Coffee is one of the most popular and consumed beverages worldwide [1]. Basically, coffee processing starts from green coffee beans submitted to a thermal procedure, leading to roasted coffee beans. The composition of green coffee beans depends on several factors such as variety, origin, field practices, processing, climate and soil. Nevertheless, the chemical composition of roasted coffee beans seems to play a major role in defining the quality of this beverage. During the roasting process, the beans suffer physical changes and go through several chemical reactions which change, generate and degrade several substances [2] which are responsible for the flavor and aroma of coffee [3]. Different techniques like nuclear magnetic resonance (NMR) [3] and synchrotron X-ray microtomography [4] have been employed in the study of the effects of the roasting procedure on green coffee beans.

The elemental composition of ground coffee has been widely investigated through different types of techniques such as inductively coupled plasma atomic emission spectroscopy (ICP-AES) [5], flame atomic absorption spectroscopy (FAAS) [6,7] and neutron activation analysis (NAA) [8]. One of the drawbacks of these methods is that the sample preparation is somewhat complex. Indeed, this procedure requires extra preparation steps involving chemical products, with the risk of incomplete dissolution, contamination and losses. These problems can be overcome by techniques like particle-induced X-ray emission (PIXE) [9] which deals mainly with solid samples. No reports on the investigation of elemental composition of ground coffee as well as whole green and roasted coffee beans by PIXE technique are available in the literature. Moreover, no attention has been paid regarding the elemental maps of minerals present in whole green and roasted coffee beans. In this case, micro-PIXE is the technique of choice since a proton beam of few micrometers in diameter is scanned over the region of interest, thus providing information on the distribution of elements. Micro-PIXE has proved to be a powerful technique to determine the distribution of important minerals present in all kinds of beans [10].

For practical reasons, most consumers buy coffee in the form of packed roasted ground coffee, which is ready for drip brewing. However, it is well known that grinding roasted coffee beans right before brewing leads to a better beverage richer in flavor and aroma. It has been suggested that one of the reasons for this difference might be related to the non-selectivity of the raw material used by manufacturers during harvesting, roasting and grinding procedures which may include not only beans but leaves and twigs. Indeed, the study of other beverages like mate tealeaves (*Ilex paraguariensis* A. St.-Hil.) has shown that tuigs are rich in elements like P, Cl, K, Ca, Fe and Zn [11] which, in turn, may have an impact on the basic features of the final beverage.

The aim of this work is to use ion beam techniques in order to study the elemental composition of coffee. In particular, a full elemental analysis of packed roasted ground coffee and packed roasted coffee beans ground right before the analysis is carried out in order to establish the differences between these types of coffee. To that end, a popular Brazilian brand of coffee which sells

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packed ground coffee and packed roasted coffee beans was chosen. The samples were analyzed by PIXE for the elemental concentrations of elements heavier than Na. Light elements like C, O and N were measured through Rutherford backscattering spectrometry (RBS) [12] experiments. Whole green and roasted coffee beans were analyzed. Finally, in order to check the elemental distribution in whole roasted and green coffee beans, micro-PIXE experiments were carried out as well.

2. Material and methods

2.1. Samples

A popular Brazilian brand of coffee (Melitta) which sells packed roasted ground coffee and packed roasted coffee beans was chosen for the present study. The roasted coffee bean samples were prepared by grinding the roasted beans with a pestle and mortar right before the analysis. The resulting ground coffee was then homogenized and pressed into pellets of 25 mm diameter and 3 mm thick. A total of 20 samples were prepared in this way. Moreover, 20 pellets of packed roasted ground coffee were prepared as well.

Whole green and roasted beans were also analyzed by PIXE. In this case, 6 green beans and 6 roasted beans were analyzed. Finally, for the micro-PIXE measurements, green and roasted beans were cut and sectional areas of them were scanned by the proton beam.

2.2. Measurements

All measurements were carried out at the Ion Implantation Laboratory of the Physics Institute of the Federal University of Rio Grande do Sul using a 3 MV Tandetron accelerator. The PIXE experiments were performed with a 2 MeV proton beam. The pressure inside the chamber was about 10^{-5} mbar. Typically, each target was irradiated with an average current between 3 and 5 nA during 400 s, and the respective count rates varied from 1000 to 1500 counts per second. The charge was collected in the reaction chamber itself which works as a Faraday cup. The beam spot size on the samples was 9 mm^2. Since the samples were insulators, an electron flood gun [13] was used in order to neutralize the samples during the proton bombardment. The X-rays induced in the samples were detected by a Si(Li) detector placed at 135° with respect to the proton beam direction. The energy resolution of the Si(Li) detector was 160 eV at 5.9 MeV.

The RBS measurements were carried out with 1.2 MeV alpha particles with typical currents ranging from 10 to 20 nA. Backscattered particles were detected by two Si surface barrier detectors placed at + 15° and −15° with respect to the alpha beam direction. The energy resolution of both detectors was 12 keV for alpha particles.

Micro-PIXE measurements were carried out using the ion microprobe system from Oxford Microbeams consisting of three quadrupole lenses and a scanning system. Green and roasted coffee beans were scanned by a 3 MeV proton beam focused to a 2.5×2.5 μm^2 spot size. Typical currents ranged from 25 to 60 pA. Moreover, the scan size varied from 500×500 to 2000×2000 μm^2 depending on the sample. Qualitative elemental images were obtained for green and roasted beans at two different spots, namely the surface and the inner part of a transverse section.

2.3. Data Analysis

The PIXE data was analyzed using the GUPIXWIN software developed at the University of Guelph [14]. All X-rays peaks appearing in the X-ray spectra are fitted simultaneously according the least-square fitting procedure. Thanks to the extensive atomic physics database included in the software (ionization cross sections, stopping powers, fluorescence yields, etc.), peak areas are converted to elemental concentrations. In order to obtain the detector's solid angle, an apple leave standard from National Institute of Standards and Technology (NIST material 1515) was used as a standard. This standard was chosen due to the similarity of matrix elements like C, O and N.

The RBS spectra were analyzed with the SIMNRA software [15], which simulates backscattering spectra obtained with MeV ions. A database consisting of non-Rutherford cross sections, nuclear reactions and stopping powers allow the simulation of backscattering spectra of a great variety of ion-target combinations. Energy loss straggling, multiple and plural scattering are taken into account as well in the simulations.

All elemental concentrations presented in this work are represented by the mean and the respective standard deviation. The statistical analysis made use of the Fisher and student's T tests for the comparison between two sets of data. The level of significance was 5%.

Finally, the processing of the elemental maps was carried out with the OMDAQ++ software from Oxford Microbeams.

3. Results and discussion

Through the RBS experiments it was possible to determine the matrix composition of coffee. The matrix composition was found to be compatible with 84% of carbon, 11.5% of oxygen and 4.5% of nitrogen when normalized to 100%. These data serve as primary input to the analysis of elemental concentration with PIXE.

3.1. Packed roasted ground coffee and packed roasted coffee beans

The elemental compositions of packed roasted ground coffee and packed roasted coffee beans were measured by PIXE. Fig. 1 shows the average spectrum of roasted coffee beans. Elements such as Mg, P, K, Ca, Mn, Fe, Cu, Rb and Sr are clearly seen in the spectrum. The mean recovery of the elemental concentrations is (97 ± 2)%. Typical limits of detection for Mg, P, K, Fe and Zn were 96, 43, 27, 1.4 and 2 μg/g respectively. The quantitative results

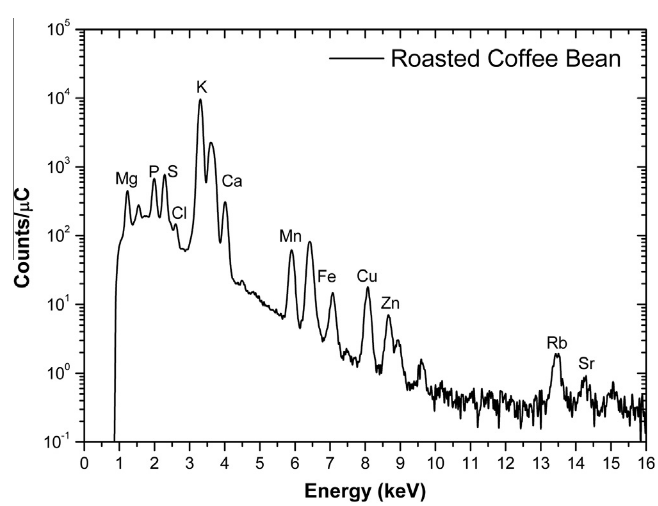

Fig. 1. Typical PIXE spectrum of roasted coffee bean ground right before the experiment. The X-ray yield was normalized by the total charge accumulated during the experiment. The energy of the proton beam was 2 MeV with an average current of about 5 nA. The irradiation time was 400 s. See text for further details.