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The study of the influence of industrial processing on the elemental composition of mate tealeaves (*Ilex paraguariensis*) using the PIXE technique

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ABSTRACT

The influence of the industrial processing on the elemental composition of mate tealeaves was investigated using the Particle Induced X-ray Emission (PIXE) technique. Considerable changes were observed after the main steps of the industrial process. The concentration of Mg, P, S, K and Ca increased after the roasting (rapid contact of the leaves with wood flames and fog) and the drying processes, possibly due to the presence of contaminants in the hot gases (originated from burning wood) used in both steps, biomass burning and evaporation of water. Elements like Al, Si, Mn and Rb increase in concentration after the roasting but decrease after the drying process, while Fe and Ti were not affected by the drying step. Differences in concentration for several elements were observed due to the stationing period (waiting between the drying and the packaging of the leaf material). The mineral contents of leaves and twigs were also compared and results show that most of the elements are present in greater amounts in the leaves, except for Ca, Ti and Zn. These results highlight the importance of understanding the influence of the industrial process on the mineral content of mate tealeaves for the development of new products with controlled characteristics.

1. Introduction

Among tea like beverages consumed in South American countries, mate tealeaves (*Ilex paraguariensis* A. St.-Hil.) is by far the most popular. The benefits of mate tealeaves to human health have been extensively studied. Indeed, several aspects of mate tea infusions including health benefits, chemical composition ant technological issues have been recently reviewed (Heck & de Mejia, 2007), which shows an ever growing interest on the properties of this beverage.

In view of the great competitiveness among industries that process mate tealeaves, the knowledge of the influence of the industrial process on the final product gains new impetus since it provides an extra tool to improve quality standards by having control over desired specifications like taste and smell. Prominent efforts have been directed to the evaluation of existing procedures as well as the development of new and more efficient methods of processing mate tealeaves, regarding mostly the drying stages of the process (Schmalko & Alzamora, 2005; Schmalko, Morawicki, & Ramallo, 1997; Zanoelo, Abitante, & Meleiro, 2008). Alternative drying procedures like microwave radiation (Esmelindro, Girardi, Mossi, Jacques, & Dariva, 2004; Passardi, Schvezov, Schmalko, & Gonzalez, 2006) and superheated steam-drying (Zanoelo, Cardozo-Filho, & Cardozo, 2006) were also investigated, revealing differences in the extraction of compounds according to the chosen method and offering promising and more efficient substitutes to conventional processes. Regarding the chemical composition of unprocessed and roasted leaves, Esmelindro, Toniazzo, Lopes, Oliveira, and Dariva (2005) reported an increase in squalene contents and a decrease in caffeine, steroids and terpene contents after the roasting. According to Schmalko and Alzamora (2001), a sharp decrease in chlorophyll contents is observed with substantial losses, possibly as a result of the high water activities and temperatures involved in the process. Furthermore, Bastos, Fornari, Queiroz, and Torres (2006) reported that moisture content in the leaves varies during the processing stages and affects the soluble solids as well as the concentration of bioactive compounds in the aqueous extraction. Finally, Manzocco and collaborators (Manzocco, Anese, & Nicoli, 1998) reported that processing and storage conditions of tea might affect the antioxidant properties of tea extract.

Despite all the progress made as far as compounds are concerned, information about the influence of industrial processing of mate tealeaves over the mineral content of the commercial product is still very scarce. Jacques et al. (2007) carried out an experiment with mate tealeaves under controlled conditions like

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light exposure, fertilizers type and age of the leaves (agronomic variables) as well as temperature. Among several interesting results, they showed that the minerals have a key role in the decomposition and thermal stability of the leaves in the drying process. Therefore, the changes in the mineral profile of mate tealeaves during the processing stages could provide important data concerning nutritional aspects as well as contamination issues.

Recently, Giulian et al. (2007) reported on the elemental characterization of commercial mate tealeaves before and after hot water infusion using Particle Induced X-ray Emission (PIXE) technique. This technique is based in the emission of characteristic X-rays induced by energetic ions (Johansson, Campbell, & Malmqvist, 1995), thus providing elemental concentrations independently of any form of mixture or compound that makes up the sample. PIXE is a non-destructive technique and has multi-elemental capabilities. Moreover, its sensitivity is compared to that provided by optical-based techniques like ICP-MS and ICP-AES (Saitoh, Sera, Gotoh, & Nakamura, 2002), in the range of a few parts per million (ppm), with the advantage that its sensitivity varies smoothly with atomic number. The sample preparation for solids does not require any sophisticated handling or any chemical treatment, drastically reducing any chance of contamination. PIXE has been successfully used in the characterization of a great variety of materials, including biological (Franke et al., 2006) and environmental (Braga et al., 2005) samples.

Therefore, by exploring the capabilities of the PIXE technique, the purpose of this research is to investigate changes in the elemental composition of mate tealeaves at different stages of the industrial processing of the material collected in an operational plant, thus providing useful information about the changes occurring in the material till it is ready for consumption. Moreover, the elemental characterization of processed mate tealeaves and twigs was carried out using the same technique.

2. Materials and methods

2.1. Industrial process

Prior to consumption, the mate tealeaves must be processed in order to preserve the characteristics of the plant and prevent its degradation after harvesting. Despite a continuous development of processing techniques, some industries are still based on primitive ways of processing mate tealeaves. Although some details of the procedures may vary among industries, the procedure is basically the same and details described here were the ones employed for the samples analyzed in this work. The overall processing technique can be divided in five main stages: harvesting, roasting, drying, grinding and stationing (Valduga, Finzer, & Mosele, 2003). Harvesting consists of cutting of the thin branches of the trees and the delivery of the material to the processing plant. As soon as they arrive in the factory, the leaves are rapidly toasted in wood flames, in a procedure known as roasting, which by driving moisture out prevents the leaves from becoming dark and bad tasting. This process can take about 3 min with temperatures in the order of 500 °C. The final quality of the product relies on the rapidity and efficiency of this first process. After that, the leaves are dried in a furnace by convection of gases originated from wood burning. This process takes up to 30 min with temperature around 100 °C, causing the moisture of the leaves to decrease to about 5 g/100 g of its total mass weight. Next, the dried material is ground and then stored for several days, depending on the demand of the product. Finally the product is minced and separated into leaves and twigs. According to product regulations and specifications, a proper mixture of leaves and twigs is undertaken and then it is packaged and commercialized. Usually, the final product is a mixture of 70 g/100 g leaves and 30 g/100 g other parts of the plant, including twigs (Giulian, 2005).

2.1.1. Samples

The mate tea leaf samples were collected directly from a factory located in the Rio Grande do Sul state, Brazil, all in the same day, after the main steps of the industrial process, which are defined here as steps A, B and C. Step A consists of the leaves as they arrive at the factory after being harvested. Step B stands for the leaf material after the roasting, and step C represents the leaf material after being dried and ground. The collection of material was carried out under strict supervision during short periods of time due to safety regulations since the processing plant was operational at the time. Finally, soil samples were collected at an organic mate tea garden in the proximities of the industry.

2.1.2. Sample preparation

After steps A and B, the mate tealeaves still preserve, to some extent, their original shape. In order to avoid any possible interference in the material due to extra handling, an effort was made to keep the samples as they are at the end of each step. For this reason, 10 leaves for steps A and B were randomly selected from different batches and the respective samples were prepared by just cutting representative parts of the leaves and attaching them to a special support. Both sides of the leaves were analyzed and averaged to give the results presented in this paper.

As the mate tealeaves corresponding to step C were already ground, two sets of 10 samples were collected, powdered and pressed into pellets of 25 mm diameter and 3 mm thick approximately. To simulate the stationing of the leaf material, the two sets of samples were collected in different dates and then stored in atmospheric pressure for different periods of time. One set was analyzed 3 and 23 days after collection, while the other set was analyzed 13 and 33 days after collection. In this particular case, each sample could be analyzed twice as PIXE is non-destructive, which allows several measurements of the same sample.

The separation of leaves and twigs was done after step C. Again 10 samples of leaves and twigs were powdered and pressed to form pellets as described above. Finally, the same procedure was adopted for the soil samples although, in this case, only three different samples were prepared.

2.1.3. Instrumentation

The measurements were carried out at the Ion Implantation Laboratory of the Physics Institute (UFRGS) using a 3 MV Tandetron accelerator. All PIXE measurements were performed with a 2 MeV proton beam and an average current of 1 nA on target. The characteristic X-rays induced by the proton beam were detected by a lithium-doped silicon detector, with an energy resolution of about 155 eV at 5.9 keV, which was positioned at an angle of 135° with respect to the beam direction.

2.2. Data analysis

Experimental data were analyzed using the GUPIX code (Campbell, Hopman, & Maxwell, 2000; Maxwell, Campbell, & Teesdale, 1989; Maxwell, Teesdale, & Campbell, 1995). The standardization procedure was carried out using apple leaves standard from NIST (reference material 1515) for the mate tealeaves samples and a river bed sediment standard also from NIST (reference material 8704) for the soil samples. The quantitative PIXE analysis of a sample in a thick target approximation (pellets) requires the knowledge of its matrix composition. For organic materials such as *I. paraguariensis* the matrix consists basically of C, O and H, which cannot be detected through their characteristic X-rays in the present setup and, therefore, are considered invisible elements