Hindawi
Journal of Food Quality
Volume 2018, Article ID 5908463, 7 pages
https://doi.org/10.1155/2018/5908463

Research Article

Quality of Commercial Coffees: Heavy Metal and Ash Contents

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Received 29 September 2017; Accepted 28 March 2018; Published 6 May 2018

Academic Editor: Gabriel Henrique Horta de Oliveira

This study aimed to quantify the ash content and to determine the concentration of heavy metals in roasted ground coffee and their respective infusions. The ash content was determined by incineration of the samples, and the quantification of heavy metals was performed by flame atomic absorption spectrophotometry for the following metals: cadmium, lead, copper, chromium, nickel, and zinc. According to the ash analysis, 15% of the roasted ground coffee samples were within the standards established by the legislation of the state of São Paulo, which has set an ash content of below 5%. In the roasted ground coffee samples, the Cd, Cu, Ni, and Zn contents did not exceed the limit established by Brazilian legislation. In several samples, both Pb and Cr were found in high levels, exceeding the limits established by Brazilian legislation. In the infusions of roasted ground coffee, the Cd, Cu, Cr, and Ni contents were below the detection limit of the equipment. Zn was found in all infusions and Pb was only detected in seven coffee infusion samples. Overall, the concentrations of heavy metals found in the commercially roasted ground coffee and their respective infusions are lower than the limits recommended by the official inspection agencies and, thus, are suitable for consumption.

1. Introduction

Coffee beans are one of the most widely traded commodities in the world. There are more than 100 different plant species of the *Coffea* genus in the world. Despite this great diversity, only two species have significant economic importance in the world coffee market: *Coffea arabica* L. and *Coffea canephora* Pierre ex A. Froeher, known, respectively, as arabica coffee and robusta coffee or conilon [1].

In the international market arabica coffee represents more than 70% of world production. They are finer coffees recognized for the best aroma and flavor, being more valued than conilon coffee. In addition, it is more commonly used in blends of arabica coffee, which gives more body and reduces the acidity of the beverage [2].

Roasted and ground coffee is classified as Traditional or Extraforte (arabica blended with conilon, without quantity restriction) and Gourmet (arabica only), according to the characteristics of the products identified through the coffee bean species, roasting, grinding, aroma, and flavor [3]. Coffee consumers are becoming increasingly demanding. During the purchasing process, quality factors and purity stamps are frequently sought characteristics. These factors outweigh the price factor, which demonstrates consumer demand for the best coffee [4], free of impurities and adulterants, and is a genuine product.

Normative Instruction No. 16 of the Ministry of Agriculture and Livestock (MAPA), which guarantees the quality of roasted ground coffee, establishes an acceptable level of up to 1% of impurities, sediments, and foreign matter in coffee [5]. However, there are other compounds that, when present in the grains, could cause serious damage to the health of consumers [6]; this is the case for metal contaminants.

Heavy metals are the most frequently evaluated elements in food, because of their ability to accumulate in the food chain [6–11]. Thus, their maximum levels have become quality standards around the world [10]. These elements are stable and, thus, persistent in the environment, accumulating in the soil [8] as a result of factors associated with the weathering of rocks and soil formation, the soil soluble content, pH, plant species, environmental conditions, technological practices, and use of chemical products [6, 7, 12]. Among the chemical products that contribute to increased heavy metal content in the soil are biosolids, which often contain high levels of cadmium, copper, chromium, lead, nickel, and zinc [13]. Additional sources of contaminants include the use

Table 1: Detection limits (DL) and limits quantification (QL) determined for elements quantified by atomic absorption.

	Cu (mg kg^{-1})	Cr (mg kg^{-1})	Ni (mg kg^{-1})	Cd (mg kg^{-1})	Pb (mg kg^{-1})	Zn (mg kg^{-1})
DL	0.0474	0.3105	0.0414	0.1569	0.1104	0.7262
QL	0.1438	0.9408	0.1559	0.4775	0.3347	2.2007

of phosphate fertilizers produced from sedimentary rocks [14] and the application of micronutrients from industrial byproducts [7].

First, these materials are absorbed by plants and accumulate in food, thus becoming sources of contamination for humans [7]. Magna et al. [9] detected the presence of Cd and Pb in many types of food and grasses cultivated in the municipality of Santo Amaro, Bahia. Schmidt et al. [15] observed the presence of many metals in coffee grains cultivated in the state of Paraná, some at concentrations much higher than the values established as safe for daily intake.

Therefore, this study aims to determine, by means of incineration, the ash content and the flame atomic absorption spectrophotometry, the heavy metal contents of commercial samples of roasted ground coffee, and their respective infusions.

2. Material and Methods

The coffee samples were purchased in the local markets of the cities of Rio Paranaíba and Carmo do Paranaíba in the state of Minas Gerais. Fifteen samples of commercially roasted ground coffee were analyzed, three of which were considered "Gourmet" (samples I, N, and O). Two samples of each coffee were purchased to perform the analyses. Two replicates of each sample were taken to the analysis.

For the determination of the ash content in the roasted ground samples, approximately 2 g of sample was weighed in a porcelain capsule and placed in a muffle furnace for incineration at 550°C until the organic matter had been eliminated. At the end of the incineration process, the samples were weighed. The results are expressed in percentages [16].

The coffee infusion consisted of the beverage prepared with 12 g of roasted ground coffee lixiviated by 100 mL of water heated to boiling point (95–100°C), followed by filtration [16]. Then, 25 mL of the beverage was concentrated in an oven at 60°C with air circulation until reaching a final volume of approximately 2.5 mL.

The samples of roasted ground coffee and the infusions were mineralized by the wet route, using 18 mL of the nitric–perchloric digestive mixture in a 3 : 1 ratio (nitric acid : perchloric acid) and heating at 190°C. The solution was maintained under these conditions until a translucent solution had formed [6]. The samples were transferred to 25 mL volumetric flasks, and the volume was made up with water. Then, the contents of the following elements were quantified: cadmium, copper, chromium, lead, nickel, and zinc. The measurements were made in a fast, sequential atomic absorption spectrophotometer (Varian, A240FS, Mulgrave, VIC, Australia) with atomization in an air/acetylene flame at the rate of 13.3 L min^{-1}/2.9 L min^{-1} for Cr and 13.5 L min^{-1}/2.0 L min^{-1} for the other elements. As a radiation source, a single element hollow cathode lamp (HCL) was used. The intensities of the electric currents were 7 mA (Cr), 5 mA (Mn, Pb, and Zn), and 4 mA (Cd, Cu, and Ni). The absorbances were measured at the following wavelengths (nm): Cd, 228.8; Cu, 324.7; Cr, 357.9; Pb, 217.0; Ni, 232.0; and Zn, 213.9. The procedure used and the appropriate concentrations for the standard curve were performed according to the protocols established in the equipment manual.

Deionized water was used for all analyses. The concentrations were determined by elaborating the calibration analytical curves of each element quantified. The solutions for the construction of the standard curves were obtained through stock solutions of 1000 mg/L for atomic absorption and ICP. The reagent blanks and the samples with the standards of the elements were included in the analysis.

The detection limit (DL) and the quantification limit (QL) were calculated based on the standard deviation of the response and the slope of the calibration curve (mean of 3 curves). They were obtained from the following equations [17]:

$$\mathrm{DL} = 3.3\frac{\mathrm{SD}}{S}, \qquad \mathrm{QL} = 10\frac{\mathrm{SD}}{S}, \tag{1}$$

where

SD is standard deviation of the response or instrumental target (3 curves);

S is slope or angular coefficient of the analytical curve.

The limits of detection and quantification determined experimentally for each of the evaluated elements are described in Table 1.

For optical imaging, the roasted ground coffee samples were placed on slides and humidified. Then, they were analyzed using an optical microscope and digitalized (5-megapixel resolution) using a digital camera (Samsung, WB150F, Manaus, AM, Brazil), CoolSnap Pro image system, and Image-Pro Plus software (Media Cybernetics).

The obtained data were analyzed by descriptive statistics. The heavy metal data in the roasted ground coffee samples were submitted to principal component analysis. Then, the samples were grouped by Euclidean distance, using the values of the first and second main components.

The Pearson correlation analysis was performed between the concentrations of Cd, Cu, Cr, Pb, Ni, and Zn in the samples of roasted ground coffee and their respective infusions.

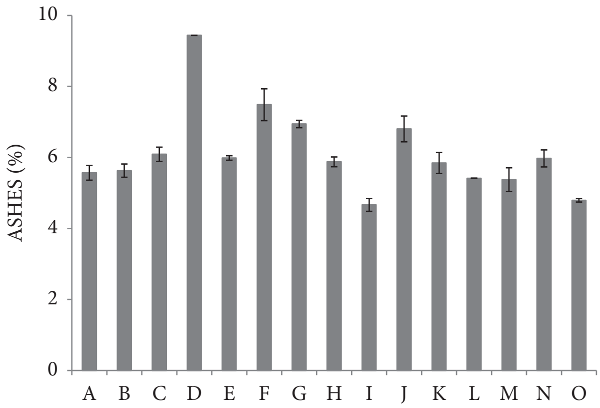


Figure 1: Ash content of the roasted ground coffee samples.

3. Results and Discussion

Figure 1 shows the mean ash content observed in the different roasted ground coffee samples. The ash content ranged from 4.48 to 9.44 g $(100\,g)^{-1}$, where the lowest mean value was obtained in one of the "Gourmet" coffee samples (sample I).

Teixeira et al. [16] analyzed different commercial coffee brands and found ash contents ranging from 3.99 to 6.10 g $(100\,g)^{-1}$, where the lowest value was also found in a "Gourmet" coffee sample. Gourmet coffee is composed only of arabica coffee, which commonly has a lower ash content. The other coffees are composed of blends and the addition of conilon coffee contributes to the increase of ash content [20]. Normative Instruction No. 16 of MAPA [5] does not specify the maximum ash content in roasted ground coffee. However, Resolutions Nos. 19 and 31 of the São Paulo State Board of Agriculture and Supply (Secretaria de Agricultura e Abastecimento do Estado de São Paulo, SAASP) define as standard the maximum fixed content of mineral residue to be 5.00% for both "Traditional" and "Gourmet" roasted ground coffee [21, 22]. Therefore, considering the São Paulo state resolutions, two samples (O and I) meet these standards.

According to a study performed by Durek de Conti et al. [20], this variation could be explained by the difference in the varieties and coffee blends, where the mean ash content in arabica ranged from 2.5 to 4.5%, whereas that of robusta coffee was 4.64%. It is also possible to associate the mineral composition of the coffee with the nutritional state of the coffee plantation and its location [23]. According to Müller et al. [24], when the ash content exceeds 5.00%, there may be a significant quantity of impurities in the sample.

The heavy metal contents found in the roasted ground coffee samples are presented in Figure 2.

The Cu contents ranged from 1.55 to 29.85 mg kg^{-1}, having a mean content of 16.49 mg kg^{-1}. For all roasted ground coffee samples analyzed, the Cu concentrations were below the maximum limit (30 mg kg^{-1}) defined by Brazilian legislation [18]. Morgano et al. [25] obtained mean Cu values higher than the results of the present study (29.86 mg kg^{-1}), considering all the samples analyzed by the authors. However, when analyzing only the samples from the Alto Paranaíba region, MG, the value found (14.17 mg kg^{-1}) is similar to that of the present study. dos Santos et al. [26] found mean Cu contents from 7.15 to 14.9 mg kg^{-1} in coffees produced in two properties in the state of Bahia. These results indicate that the mineral content is associated with the origin of the coffee, species, pesticides, and agricultural treatment, among other factors.

The roasted ground coffee samples had low Ni concentrations. We could only quantify the Ni content in six samples, and these had Ni contents ranging from 0.037 to 0.72 mg kg^{-1}, having a mean of 0.11 mg kg^{-1}. These values do not exceed the maximum limit allowed by Brazilian legislation, which establishes a maximum Ni content of 5 mg kg^{-1} [18]. In the other samples, the Ni content was lower than 0.025 mg kg^{-1}. Morgano et al. [25] determined the mean Ni content of 4.76 mg kg^{-1}, considering all raw coffee samples analyzed, and a mean value of 1.21 mg kg^{-1}, considering only the samples from the Alto Paranaíba region, MG.

The Zn contents varied between 3.31 and 25.97 mg kg^{-1}, having a mean content of 8.07 mg kg^{-1}. The determined values do not exceed the maximum Zn content allowed by Brazilian legislation of 50 mg kg^{-1} [18]. The mean Zn content found in the samples is similar to the values found by Morgano et al. [25], who determined a mean Zn content of 8.33 mg kg^{-1} in raw coffee. Mean contents higher than the results of the present study were found by dos Santos et al. [26], who obtained values from 25 to 45 mg kg^{-1} of Zn when evaluating two coffee plantation properties in the state of Bahia. In addition, Ashu and Chandravanshi [12] found a mean value of 19 mg kg^{-1} of Zn in commercial roasted ground coffee samples.

Cu, Ni, and Zn are essential elements for the development of coffee plants and are applied via the soil or the leaves; therefore, they could also be present in the grains. Copper is an active ingredient of some of the pesticides used in coffee cultures, in the form of copper hydroxide, copper oxychloride, copper sulfate, or copper ethylenediaminetetraacetate (EDTA), which allows the absorption of this element by the plant, explaining its presence in the grain [11]. Furthermore, these elements could also be present in the soil, varying depending on their origin [6].

Cr was only detected in one roasted ground coffee sample, where the content was 3.27 mg kg^{-1}, a value much higher than the maximum limit allowed by Brazilian legislation, which establishes a maximum content of 0.1 mg kg^{-1} [18]. In the other samples, Cr was not detected, and all these contents were below 0.025 mg kg^{-1}. dos Santos et al. [26] did not find Cr in coffee samples from the state of Bahia.

Among the evaluated elements, Cd and Pb are considered the most toxic inorganic contaminants [8]. According to Resolution RDC No. 42 of 2013, the maximum limit of Cd in roasted ground coffee is 0.1 mg kg^{-1} and that for Pb is 0.5 mg kg^{-1} [19].

Cd was not found in the analyzed samples. Hence, the Cd content for all samples was below 0.025 mg kg^{-1}. dos Santos et al. [26] found mean Cd contents between 0.70 and 0.75 mg kg^{-1} in coffee of two different properties in the state of Bahia.

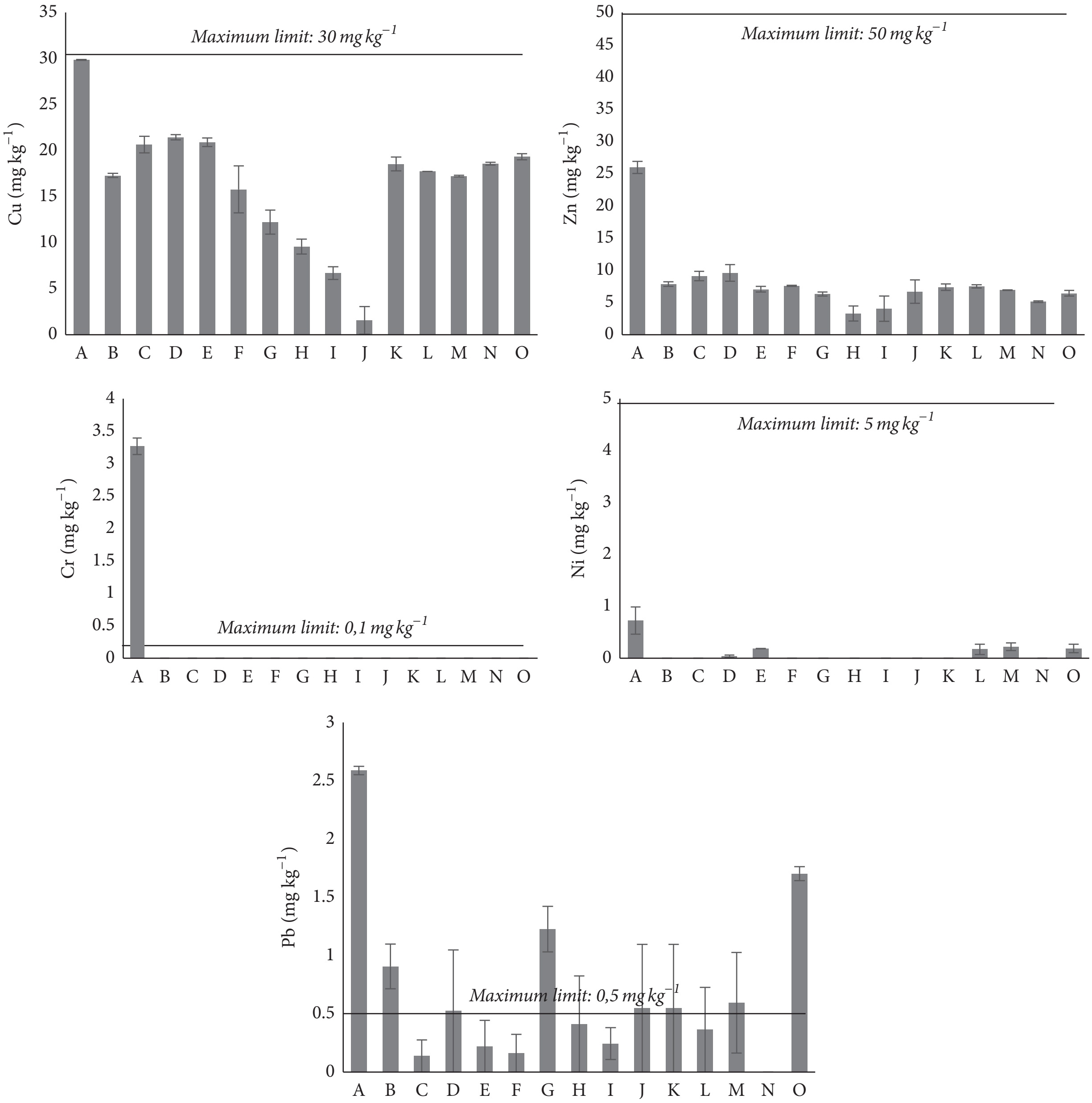


Figure 2: Mean content of heavy metals in roasted ground coffee samples and maximum contents establish by Brazilian legislation [18, 19].

Pb was undetectable only in one sample. The Pb contents ranged from 0.14 to 2.59 mg kg^{-1}, having a mean of 0.69 mg kg^{-1}. Among the analyzed samples, eight had Pb contents above the maximum limit allowed by Brazilian legislation (0.5 mg kg^{-1}) [19]. Most of the samples in the present study originate from local production (Alto Paranaíba region, MG), which indicates that a large part of the coffee produced in this region contains Pb at high levels, indicating that, most likely, this element is found in high concentration in the soils of the Alto Paranaíba region, MG [6]. Pb can act on the neurological system, causing damage to the central and peripheral nervous systems, interfering in the conversion of vitamin D and the homeostasis of calcium, and inhibiting hemoglobin synthesis, in addition to being a potential carcinogen [27].

In the analysis of main components (Figure 3), the first main component explains 76.61% of the variation of the contents of heavy metals of the analyzed samples, while the second main component explains 12.37%.

The distance between the samples suggests the existence of a difference in the heavy metal content of the samples. By calculating the Euclidean distance, it is concluded that the samples differ in three groups.

Sample A showed a positive correlation with the first main component and a great distance from the other samples. It was the sample that presented a greater content of heavy woods. During the acid digestion, precipitate was observed

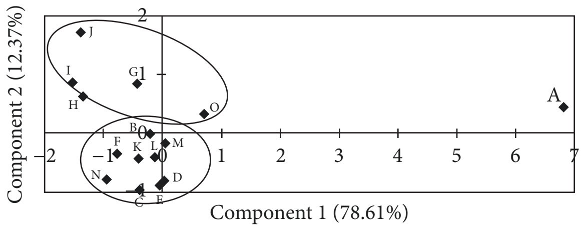


Figure 3: Analysis of major components of heavy metal content in the roasted ground coffee samples. Distinguished groups across Euclidean distance.

in sample A. The presence of precipitate in acidic solution and the high content of heavy woods compared to the other samples suggests the presence of inorganic contamination by material that contains silica, which is an insoluble mineral in acid.

The other samples were divided into two other groups, and the samples of gourmet coffee analyzed (I, N, and O) are distributed in both groups, showing that the fact that they are better classified sensorially is not related to the content of heavy metals. The variations in the contents of heavy metals in the roasted ground coffee can be explained by the chemical composition of the soil and the management and cultivation of the plants and coffee [15]. Arabica and conilon coffee have many distinct characteristics, namely, of botanical, agronomic, and morphological nature. These species can be blended into the commercial coffee industry, which comes from a wide variety of geographical areas. These characteristics may also have contributed to variations in the heavy metal content [28].

In the coffee infusions (50 mL), Cd, Cu, Cr, and Ni were not detected. Zn was detected in all samples, having a maximum content of 15.472 μg 50 mL^{-1} and a minimum of 4.998 μg 50 mL^{-1}, having a mean value of 9.63 μg 50 mL^{-1} (Table 1). Noël et al. [29] found the mean value for Zn for a coffee beverage of 14.25 μg 50 mL^{-1} in the coffee infusions, a value higher than the mean and similar to the maximum value found in the present study. Pb, which was present in almost all roasted ground coffee samples (Figure 2), was only detected in seven coffee infusion samples, having contents ranging from 0.129 to 2.835 μg (50 mL)$^{-1}$ (Table 2).

The maximum acceptable intake of Pb is 25 μg kg^{-1} body weight per week [9], which represents an intake of 250 μg per day based on an adult male weighing 70 kg. Hence, the maximum daily intake of four cups of coffee represents 0.21% to 4.54% of the maximum Pb intake.

The infusion process used to prepare the coffee has a strong influence on the concentration of metals. The lower metal concentration in the infusions is most likely due to the release of all elements in the form of their simple ions with the coffee matrix [30].

The Pearson correlation coefficients were estimated for the Pb and Zn concentrations in the samples of roasted ground coffee and their respective infusions. For Pb, the Pearson correlation coefficient was 0.1204, and for Zn it was 0.0649, indicating that there is no correlation between the content of metals in the roasted ground coffee and their respective infusions. In addition to the differences in the blends and edaphoclimatic differences in the production of the grains, because these are commercial samples, the grain size of the coffee powder could influence the extraction ratio for these metals, making their extraction easier or more difficult (Figure 4).

Table 2: Mean contents (μg 50 mL^{-1}) of heavy metals present in a cup (50 mL) of coffee infusion.

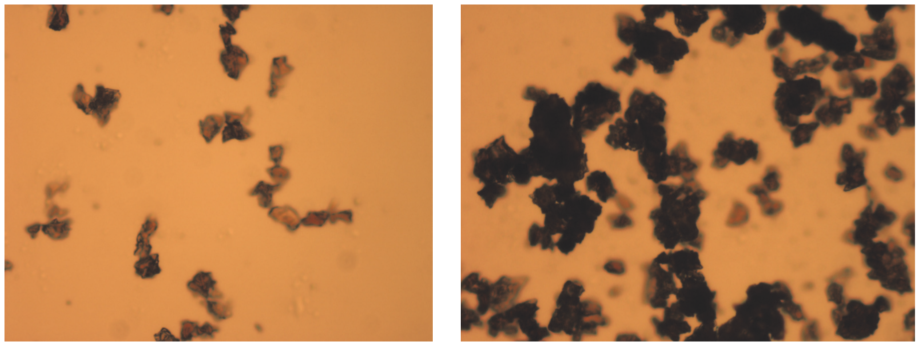
Samples	Zn	Pb
A	10.952 ± 2.963	0.838 ± 0.218
B	12.375 ± 1.401	0.129 ± 0.039
C	9.105 ± 0.1086	nd
D	9.202 ± 1.575	0.322 ± 0.086
E	4.998 ± 3.237	nd
F	7.584 ± 0.239	nd
G	7.116 ± 2.649	nd
H	9.626 ± 0.217	1.289 ± 0.211
I	10.430 ± 2.756	nd
J	9.822 ± 3.322	2.835 ± 0.172
K	9.833 ± 2.901	1.160 ± 0.179
L	9.452 ± 1.043	0.258 ± 0.038
M	7.736 ± 0.043	nd
N	14.635 ± 2.727	nd
O	6.486 ± 0.206	nd
Mean	9.626	0.455

nd: not detected.

The mean extraction of Pb in the "Traditional" and "Extra Strong" coffee infusions, which have smaller grain sizes (Figure 4(a)), was 16.96%. For the coffees of higher quality, named "Gourmet," of larger grain size (Figure 4(b)), Pb was not detected in their infusions, even when high Pb contents were found in the roasted ground coffee.

When comparing the data of the present study with that of the literature, differences are observed in the concentrations found in some elements. Rocks are natural sources of all chemical elements found on Earth [6]. Released by weathering, the elements in the rocks are made available in the soil and then are carried to rivers and groundwater, interacting with the ecosystem where humans live. However, the natural occurrence of these elements is not equally distributed on the surface of the Earth and problems may arise when their concentrations are too low (deficiency) or too high (toxicity) [6].

Metal contamination could also be caused by human activities, such as waste from mining, the steel industry, the cosmetics industry, or agricultural activities. The contamination that affects the agricultural areas currently represents a significant problem, given that many pollutants play an essential role in economic activities, for example, pesticides and fertilizers [6, 31], and many of these products can remain in the soil or water, thus contaminating food [11].



(a) (b)

Figure 4: Microscopy images of grains of the "Traditional" roasted ground coffee sample (a) and a "Gourmet" coffee sample (b).

4. Conclusions

The roasted and milled coffees quantified in the present study present high levels of ash indicating that products available on the market can contain high amounts of impurities.

For most of the quantified samples, the concentrations of heavy metals found in the commercial roasted ground coffees are below the limits recommended by the official inspection institutions and, thus, are suitable for consumption.

Some roasted ground coffee samples have heavy metal contents above the allowed levels. Parameters such as the species and the origin of the coffee beans can contribute to variations in the contents of heavy metals.

There is no correlation between the Pb and Zn contents in the roasted ground coffee and their respective infusions and, consequently, the beverages can be considered uncontaminated by these metals.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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