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The quality of roasted and ground brazilian coffee: Chemical analysis of the effect of fixed mineral residue e different types of packaging on moisture content

Qualidade de cafés brasileiros torrados e moídos: Análise química do resíduo mineral fixo e de diferentes tipos de embalagens no teor de umidade

Calidad del café brasileño tostado y molido: Análisis químico de residuos minerales fijos y diferentes tipos de envases en contenido de humedad

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Abstract
The agricultural production of national coffee faces controversy regarding the quality of roasted and ground coffee, being susceptible to quality loss because of oxygen and humidity exposure and high storage temperatures. In light of the importance of coffee to the Brazilian economy and consumption, a survey was made of the main brands of coffee sold in establishments in the city of São Luís, observing the presence or absence of the ABIC quality seal, the category (traditional, extra-strong, superior, gourmet), and the most common packaging types (pillow and vacuum-sealed bags). Then, two purity parameters established by ABIC – humidity and fixed mineral residue – were determined quantitatively in the eight representative samples. The analyses were performed following the procedures described by the Adolf Lutz Institute (2008). The survey revealed that the superior and gourmet categories of coffee are not easily found, while traditional and extra-strong coffee is very accessible, both in pillow and vacuum-sealed bags. The moisture level results showed two brands (A2 and A3) within the standards established by INMETRO for coffee in both pillow and vacuum-sealed bags. Of the samples of fixed mineral residue that were collected, brand A1 was observed to be in conformity with legislation, for coffee packaged in both pillow and vacuum-sealed bags, and brand A4 showed results for pillow bags within the given parameters. Statistical design was applied to verify the effect of packaging (pillow and vacuum-sealed bag) on moisture levels between different brands using analysis of variance (ANOVA). The results showed that despite displaying the ABIC seal attesting to the quality of the product, without fraud and free of impurities, 50% of the samples analyzed showed alterations in moisture level and 62.5% showed fixed mineral residue levels above the standards established by Brazilian legislature.
Keywords: Quality control; Varieties; Beverage; Consumers.

Resumo
A produção agrícola do café nacional encara controvérsias quanto à qualidade do café torrado e moído, sendo suscetível à perda de qualidade pela exposição ao oxigênio e umidade e pela estocagem a temperaturas elevadas. Diante da importância do café para a economia e o consumo brasileiros, foi feito um levantamento das principais marcas de café comercializadas em estabelecimentos da cidade de São Luís, observando-se a presença ou ausência do selo de qualidade da ABIC, suas categorias (tradicional, extraforte, superior e gourmet) e tipos de embalagens mais comuns (almofada e a vácuo). Em seguida, dois parâmetros de pureza estabelecidos pela ABIC - umidade e resíduo

mineral fixo - foram determinados quantitativamente nas oito amostras representativas. As análises foram realizadas seguindo os procedimentos descritos pelo Instituto Adolf Lutz (2008). A pesquisa revelou que as categorias de café superior e gourmet não são facilmente encontradas, enquanto o café tradicional e extra-forte é muito acessível, tanto na embalagem almofada quanto a vácuo. Os resultados obtidos para os teores de umidade demonstraram que duas marcas (A2 e A3) estão dentro dos padrões estabelecidos pelo INMETRO, tanto para cafés embalados em almofada quanto a vácuo. Para as amostras de teores de resíduo mineral fixo obtidos, observou-se que a marca A1 estava em conformidade com a legislação, tanto para o café acondicionado em almofada como em saquinho selado a vácuo, e a marca A4 apresentou resultados para as almofadas dentro dos parâmetros indicados. O projeto estatístico foi aplicado para verificar o efeito da embalagem (almofada e saco selado a vácuo) sobre os níveis de umidade entre as diferentes marcas por meio de análise de variância (ANOVA). Os resultados mostraram que apesar de apresentarem o selo ABIC atestando a qualidade do produto, sem fraude e livre de impurezas, 50% das amostras analisadas apresentaram alteração no teor de umidade e 62,5% apresentaram teores de resíduos minerais fixos acima dos padrões estabelecidos pela legislação brasileira.
Palavras-chave: Controle de qualidade; Variedades; Bebida; Consumidores.

Resumen
La producción agrícola de café nacional enfrenta controversias en cuanto a la calidad del café tostado y molido, siendo susceptible de pérdida de calidad por exposición al oxígeno y humedad y por almacenamiento a altas temperaturas. En vista de la importancia del café para la economía y el consumo brasileño, se realizó una encuesta de las principales marcas de café vendidas en los establecimientos de la ciudad de São Luís, observando la presencia o ausencia del sello de calidad ABIC, sus categorías (tradicional, extra fuerte, superior y gourmet) y los tipos de envasado más habituales (cojín y vacío). Luego, se determinaron cuantitativamente dos parámetros de pureza establecidos por ABIC - humedad y residuo mineral fijo - en las ocho muestras representativas. Los análisis se realizaron siguiendo los procedimientos descritos por el Instituto Adolf Lutz (2008). La encuesta reveló que las categorías de café premium y gourmet no se encuentran fácilmente, mientras que el café tradicional y extrafuerte es muy asequible, tanto en envasado al vacío como en cojín. os resultados del nivel de humedad mostraron dos marcas (A2 y A3) dentro de los estándares establecidos por INMETRO para café tanto en bolsas tipo almohada como en bolsas selladas al vacío. Para las muestras de contenido de residuos minerales fijos obtenidas, se observó que la marca A1 cumplía con la legislación, tanto para café envasado en cojín como en bolsa sellada al vacío, y la marca A4 arrojó resultados para los cojines dentro de los indicados parámetros. Se aplicó el proyecto estadístico para verificar el efecto del empaque (cojín y bolsa sellada al vacío) sobre los niveles de humedad entre las diferentes marcas mediante análisis de varianza (ANOVA). Los resultados mostraron que a pesar de exhibir el sello ABIC que acredita la calidad del producto, sin fraude y libre de impurezas, el 50% de las muestras analizadas presenta alteraciones en el nivel de humedad y el 62,5% presenta niveles fijos de residuos minerales por encima de los estándares establecidos por la legislatura brasileña.
Palabras clave: Control de calidad; Variedades; Bebidas; Consumidores.

1. Introduction

Due to the diversity of the regions where coffee is cultivated, the variety of climates, terrain, altitude, and latitude, Brazil produces varied types of beans, which allows it to serve the different demands of palate and price of Brazilian and foreign consumers (MAPA, 2018). Coffee growing is of great importance in the Brazilian economy, in addition to generating jobs for 10 million people directly and indirectly, from production to commercialization (Ferraz *et al*., 2017; Medeiros & Rodrigues, 2017).

The two species of coffee tree of great commercial importance are *Coffea arabica* L. and *Coffea canephora* Pierre ex A. Froeher, known, respectively, as arabica coffee and robusta coffee or conillon. The species are different in their prices, quality, and acceptance by consumers. These species can also present differences in chemical composition and in the behavior of their components during the roasting process (De Conti *et al.*, 2013; Ribeiro *et al*., 2014; Scholz *et al*., 2011).

The Coffee Quality Program (PQC), created in 2004, revolutionized the way in which the traditional roasted bean is produced and sold in our country. Through strict and rigorous tests, ABIC classifies each Brazilian coffee with a Global Quality (QG) score, dividing them into four categories: Traditional, Extra-Strong, Superior, and Gourmet (ABIC, 2008).

Besides the four certifications described above, ABIC also performs purity and sustainability tests on coffee sold in Brazil. The presence of a seal on the packaging certifies the purity and trustworthiness of a product found in the market. In

other words, the consumer is buying a coffee with no mixture. The seal does not guarantee better or worse flavor, as this is a subjective characteristic and inherent in the palate of each individual (ABIC, 2008).

As for the type of coffee, it is more common to find traditional and extra-strong coffee in pillow bag packaging, but difficult to find gourmet and special types in pillow bags.

The Brazilian Coffee Industry Association (ABIC, 2008) works daily to guarantee that coffee produced and sold in Brazil meets all the quality standards demanded by the final consumer. The Purity Seal appeared in 1987 when a survey showed that, to the Brazilian consumer, "*all coffee is the same", "most of them are blends"* and *"the best products are exported".* This reality was a direct consequence of price fixing and a program to increase internal consumption developed in the 1960s by the now extinct IBC – Brazilian Coffee Institute – which resulted in the proliferation of roasters who, besides not meeting minimum quality requirements, also adulterated their products (INMETRO, 1998).

In a national quality survey performed by ABIC in 2008, 583 frauds were identified from a total of 2,400 brands of roasted and ground coffee sold in Brazil surveyed – practically 25% of the samples. The results confirmed the presence of impurities such as coffee husks, corn, rye, caramel (sugar), açai seeds, and pieces of wood (Fagioli, 2010). Similarly, Da Silva *et al* (2021) conducted a study in which he found adulterants in samples of *Coffea* sp., the main adulterants found were corn grains and vegetable residues.

The tests defined by legislation in Decree #377, on April 26, 1999, (BRASIL, 1999) to determine the physio-chemical characteristics of roasted and ground coffee set forth eight parameters. Among these are Humidity and Fixed Mineral Residue (RMF) in g/100g with maximum permitted values (VMP) of 5.0%.

Brands presenting RMF quantities above what is permitted by legislation demonstrate a lack of commitment to the purity of their product through the addition of materials of mineral origin, such as sand, for example (INMETRO, 1998).

The humidity of coffee grounds is related to their storage, which must be done in adequate ways and places to maintain their properties (Pinto *et al.*, 2019). The coffee beans are harvested with a high water content, ranging from 50% to 65% on a wet basis (bu.), and therefore they must be immediately processed and dried after harvest (Alves *et al.*, 2017; Borém & Shuler, 2014; Isquierdo *et al.*, 2013; Siqueira *et al.*, 2016) in view of how drying can significantly interfere with coffee quality (Alves *et al*., 2013; Borém *et al*., 2018; Kulapichitr *et al*., 2019) since, elevated moisture levels in beans with uncontrolled fermentation/overpopulation of microflora are the cause of ethyl esters of short-chain fatty acids (overproduction of acetic, butyric, and propionic acids) and short-chain acid production considered responsible for overfermented, acidic, and musty flavor defects (Hameed *et al.*, 2018).

The quality of a coffee beverage is a key factor to reach the markets which pay the best price for the product, and there is always an increasing demand in the international market for special coffee that stands out from common coffee, mainly because of the better quality and complexity of the beverage (Giomo & Borém, 2011).

In light of the importance of coffee to the Brazilian economy and consumption, this project evaluated the purity of roasted and ground coffee packaged in pillow and vacuum-sealed bags, produced in different regions of Brazil, in the traditional and extra-strong categories using two purity parameters established by ABIC.

2. Methodology

2.1 Sampling

Firstly, a survey was made in commercial establishments in the city of São Luís of the main brands of coffee with or without the ABIC quality seal, as well as their categories (traditional, extra-strong, superior, gourmet) and the most common types of packaging (pillow and vacuum-sealed bags). Then, eight samples from different producers, categories, packaging and ABIC quality seal status were selected and obtained. The representative samples were taken to the Chemical Analysis and

Laboratory for Seeds of the Maranhão State University (*Universidade Estadual do Maranhão*) to be submitted to triplicate analysis of two purity parameters established by ABIC: moisture level and fixed mineral residue.

2.2 Analytical definition of the purity parameters

Of the eight tests set forth by Brazilian legislature (INMETRO, 2002) to determine the physio-chemical characteristics of roasted and ground coffee, two were performed according to the procedures laid out by the Adolf Lutz Institute (Zenebon & Pascuet, 2008).

2.2.1 Humidity

A greenhouse was used to determine moisture levels, where the samples were subjected to heat until reaching constant weight. The analyses were performed in triplicate. Four grams of each sample were weighed in a previously weighed porcelain capsule, then heated for three hours in a greenhouse with a regulated temperature of 105 ºC. Next, the samples were placed in a desiccator until returning to room temperature. Finally, the samples were weighed again to verify moisture levels (Zenebon & Pascuet, 2008).

The determination of humidity level was calculated using Formula 1:

$$\frac{100 \times N}{P} = \text{humidity or volatile substances at 105 ºC per hundred m/m (Formula 1)}$$

Where:

N = number of grams of moisture (loss of mass in g)

P = number of grams of sample

2.2.2 Fixed Mineral Residue (RMF)

A muffle furnace was used to determine RMF levels, in which the samples were subjected to incineration until reaching constant weight. The analyses were performed in triplicate. Four grams of the sample were weighed in a previously weighed porcelain capsule and heated in a muffle furnace at 550 °C for eight hours until the ashes became white or slightly grey. Next, the ashes were placed in a desiccator until returning to room temperature and, finally, (Zenebon & Pascuet, 2008).

The determination of RMF level was calculated using Formula 2:

$$\frac{100 \times N}{P} = \text{ashes per hundred m/m (Formula 2)}$$

Where:

N = number of grams of ashes

P = number of grams of the sample

All the determinations were done in triplicate for the precision of the analysis.

2.3 Analysis of the results obtained and Statistical design

The results obtained of the levels of moisture and RMF were compiled in an Excel spreadsheet, followed by the calculation of the average result for each representative sample analyzed. The quality of the coffee was evaluated next to see if the average results for the two parameters analyzed were in line with what is required by Brazilian legislation.

Statistical design was applied to verify the possible effects of type of packaging (normal and vacuum-sealed) on the level of moisture of the various roasted and ground Brazilian coffees analyzed. Therefore, to determine whether the moisture level differs significantly depending on the packaging used, a comparison was made of the moisture averages between different brands, using analysis of variance (ANOVA) comparing the averages by means of Tukey's test, with a 95% confidence level.

3. Results and Discussion

3.1 Sampling

In the survey of the main brands of coffee sold in establishments in the city of São Luís, most were observed displaying the ABIC seal. As for the categories, coffee of superior and gourmet quality of the same brands as other qualities was not found available for analysis. Because of this, all the representative samples obtained possessed the ABIC seal, belonged to traditional or extra-strong categories and were in pillow bags or vacuum-sealed packaging.

Table 1 shows the eight selected and obtained samples of coffee with the ABIC quality seal, of different brands, categories, and packaging methods.

Table 1 – Coffee producers sold in supermarkets in the city of São Luís, MA.

Samples	Producers	Categories	Packaging	ABIC Seal
A1- pillow	**1**	Traditional	Pillow Bag	✔
A1 – vac			Vacuum-Sealed	
A2 – pillow	**2**	Traditional	Pillow Bag	✔
A2 – vac			Vacuum-Sealed	
A3 – pillow	**3**	Extra-Strong	Pillow Bag	✔
A3 – vac			Vacuum-Sealed	
A4 – pillow	**4**	Extra-Strong	Pillow Bag	✔
A4 – vac			Vacuum-Sealed	

Source: Authors.

The representative samples were sent to two laboratories of Maranhão State University – São Luís Campus: Laboratory of Chemical Analysis and Seed Laboratory. The analyses of moisture levels were performed in the Laboratory of Chemical Analysis and the analyses of fixed mineral residue levels in the Seed Laboratory.

3.2 Moisture Level

The moisture results were expressed in percentage level (%), as well as average values of the samples and are displayed in Table 2.

Table 2 – Coffee samples, moisture level, average moisture levels, and maximum value allowed by Brazilian legislature.

Samples	Moisture Levels (%)			Average Moisture Level (%)	Maximum Level Legislation INMETRO, 2002
A1- pillow	7.0%	7.0%	7.0%	7.0%	5.0%
A1 – vac	6.7%	6.6%	6.6%	6.6%	
A2 – pillow	4.6%	4.6%	4.6%	4.6%	5.0%
A2 – vac	4.3%	4.2%	4.1%	4.2%	
A3 – pillow	4.4%	4.3%	4.3%	4.3%	5.0%
A3 – vac	4.1%	4.1%	4.0%	4.1%	
A4 – pillow	7.0%	7.0%	7.0%	7.0%	5.0%
A4 – vac	6.7%	6.7%	6.7%	6.7%	

Source: Authors.

The levels obtained varied between 4.1% and 7.0%. Two of the brands observed (A2 and A3) were within the standards established by legislation, both for pillow bags and vacuum-sealed packages and were not above 5.0% (INMETRO, 2002). However, in the other two brands, A1 and A4, the moisture levels are above the maximum allowed by legislation, both for coffee packaged in pillow bags, as well as coffee in vacuum-sealed bags. Broissin-Vargas *et al*., (2018), found that the moisture may be related to possible alteration in the packaging, stocking, or manufacturing processes.

In the study done by De Conti *et al*. (2013) arabica coffee was analyzed in five types of commercial coffee: Gourmet, Premium, and Traditional coffee from the same brand conventionally produced in Brazil and Civeta and Jacu exotics, obtaining a result between 1.66% and 4.50% moisture, in agreement with the established standard and found also in this project in samples A2 and A3.

According to Moragado, (2008), high moisture level indicates poor preservation of the product, as well as influencing aroma and flavor, and, therefore, quality. A high moisture level facilitates the activity of microorganisms and enzymes, affecting the preservation of the product.

3.3 Statistical analysis of moisture levels

To verify if the average values obtained are representative among the values obtained from the analyses of moisture levels, standard deviation (SD) was calculated. The results obtained varied between 0.000 and 0.100, indicating the consistency of the results and, therefore, good analytical precision.

The statistical analysis of the data showed that all the samples were similar, since the coefficient of variation (CV) calculated between them was small, varying between 0.000% and 0.007%.

Statistical design was applied to verify the possible effects of the different types of packaging (normal and vacuum-sealed) on the moisture levels of the various roasted and ground Brazilian coffees. To check if the moisture levels differed significantly depending on the type of packaging used, a comparison was made of the moisture averages obtained between different brands using the analysis of variance (ANOVA) comparing the averages using Tukey's test, with 95% confidence level, and Microsoft Office Excel.

The results obtained showed that the value of *F* (0,112428) is less than the value of *F* critical (*F* critical = 5,987378), indicating that there was not a significant difference between the moisture levels in the coffee samples in pillow bags and those vacuum-sealed, therefore eliminating the necessity of applying Tukey's test.

3.4 Fixed Mineral Residue Level

The results were expressed in percentage level (%), as well as the average value of the samples and are displayed in Table 3.

According the results obtained, the RMF levels varied between 4.8% and 7.3%. Brand A1 was observed to be within the standards established by legislation (INMETRO, 2002) and did not exceed 5%, both for coffee packaged in pillow bags as well as vacuum-sealed bags. Brand A4, in pillow bags, also showed results within the established range.

Table 3 – Coffee samples, fixed mineral residue (RMF) level, average mineral residue level, and maximum level allowed by Brazilian legislature.

Samples	RMF Levels (%)			Average RMF Level (%)	Maximum Level Legislation INMETRO, 2002
A1- pillow	4.9%	4.8%	5%	4.9%	5.0%
A1 – vac	4.8%	4.9%	4.9%	4.9%	
A2 – pillow	7.1%	7.2%	7.2%	7.2%	5.0%
A2 – vac	7.2%	7.3%	7.3%	7.3%	
A3 – pillow	7.1%	7.0%	7.1%	7.1%	5.0%
A3 – vac	7.2%	7.1%	7.2%	7.2%	
A4 – pillow	4.9%	4.8%	4.7%	4.8%	5.0%
A4 – vac	5.2%	5.2%	5.3%	5.2%	

Source: Authors.

For the other two brands, A2 and A3, in both coffees packaged in pillow bag as well as in vacuum-sealed bags, and in vacuum-sealed brand A4, the RMF levels obtained are above the maximum allowed by current legislation.

In the study by Pinto *et al.* (2019), all the samples evaluated showed levels of ashes above the maximum limit permitted, varying between 8% and 16.9% - behavior similar to what this study observed in samples A2 and A3.

The brands which present RMF quantities above what is permitted by legislation demonstrate a lack of commitment to the purity of the product through the addition of material of mineral origin, such as sand, for example (INMETRO, 1998).

Due to its high commercial value, roasted and ground coffee has been the subject of frequent adulterations (DE MORAIS *et al.*, 2019). In the study of Mendes *et al.* (2016), 70 samples of roasted and ground coffee were evaluated, totaling 420 determinations. The purity results indicated a concentration above 5%, with an average of 7.18% concentration of impurities.

3.5 Statistical analysis of the Fixed Mineral Residue level

The values obtained for standard deviation (SD) of the samples in the analyses of RMF levels are relatively small, varying between 0.0058 and 0.100, thus confirming the good precision of the triplicate analysis.

Statistical analysis of the data demonstrated that all the samples showed similarity amongst themselves, since the calculated coefficient of variance (CV) was small, with a maximum value of 0.007%.

All the packages analyzed displayed the ABIC seal. In general, coffee producers use the seal of purity to certify the quality of the product to the consumer, without fraud and free of impurities (husks, corn, barley, caramel, etc.). However, after obtaining the results above, it was possible to observe that despite displaying the ABIC seal, the samples presented adulterations in the characteristics that define the purity, excellence, and quality of the coffee.

4. Conclusion

The categories of superior and gourmet coffee are not easily found in commercial establishments in São Luís, while the traditional and extra-strong categories are very accessible, both in pillow bag and vacuum-sealed packaging.

As for the purity parameter, 50% of the samples analyzed conform to the quality standards set forth by the current legislature, taking into account both pillow bag and vacuum-sealed packaging. When considering that this figure is related to the stability, quality, and composition of the product, the conclusion is that brands A2 and A3 may have suffered alteration in the packing, stocking, or manufacturing processes.

In the levels quantified in this study, the coffee samples analyzed demonstrated results that did not have significantly different levels of moisture, indicating the same level of protection offered by pillow bag and vacuum-sealed packaging.

Of the samples analyzed for their level of fixed mineral residue, 37.5% were approved for presenting results in line with current legislation. The others exceeded the maximum level permitted, both for coffee packaged in pillow bags, as well as that in vacuum-sealed bags. Since this figure is related to the presence of impurities in the samples, the conclusion is that brands A2 and A3 in both pillow bag and vacuum-sealed packaging as well as brand A4 with vacuum-sealed packaging presented problems regarding their quality or suffered alterations in the production process.

Despite displaying the ABIC seal, which certifies the quality of the product to the consumer, without fraud and free of impurities, 50% of the samples analyzed indicated alterations in the moisture level and 62.5% presented fixed mineral residue levels above the standard set forth by Brazilian legislature.

This study considers itself to be of great importance for providing information to the population of Maranhão about the products consumed and sold in supermarkets in the city of São Luís and for warning that industrialized products do not always guarantee good quality, as may be described on their packaging.

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