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EVALUATIVE STUDY OF COFFEE QUALITY MARKETED IN THE CITY OF CAMPOS GERAIS, MG.

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ABSTRACT

Consumed worldwide, coffee is a very popular drink, with distinctive aroma and flavor. Coffee beverage is produced from ripe fruit of known species of the genus *Coffea*, in which *Coffeaarabica, Coffealiberica and Coffearobusta* stand out. Coffee quality is defined by a set of physical, chemical, sensory and safety attributes, able to meet the taste of different types of consumers. This study analyzed six samples of coffees, three with registration: Sample A, Sample B and Sample C, and three without registration of ANVISA: Sample A. Sample B and Sample C. In the physical chemical results, values ranging from 5.91% to 6.60% for pH were obtained; 10.0% to 10.8%, total soluble solids; 1.63NaOH 0.1M.100g^{-1}to 2.60NaOH 0.1M.100g^{-1}, titratable total acidity; 0.31% to 0.46%, caffeine; 1.75% to 4.77%, moisture and 1.05 CI to 1.64 CI, coloring index.

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INTRODUCTION

According to the Brazilian Coffee Industry Association (ABIC, 2017), Brazil is currently responsible for 30% of coffee production in the international market, being consequently regarded as the world's largest producer and the second largest consumer market, second only to the United States. Coffee areas in Brazil are concentrated in the south-central regions, highlighting the states of Minas Gerais, São Paulo, Espírito Santo and Paraná, northeastern region, highlighting Bahia and the northern region such as Rondônia state. Due to the intense market demand, the coffee industry has been adapting in recent years. Coffee quality has been more and more valued every day, in relation to the growing expansion of specialty coffees consumption. In order to maintain this quality, high levels of technology have been required for the coffee sector. As a consequence of this technology, there have been increased productivity, reduced costs and restriction to the use of agrochemicals (Mendonça *et al.,* 2007). Coffee has become one of the most important species in the agricultural market, marketed and consumed worldwide. Harvest and post-harvest processes influence its quality. Production and processing of coffee beans influence its chemical composition. Roasting process becomes the most important for coffee taste and aroma. Companies that carry out the process of roasting the coffee beans must be licensed and registered in the National Agency of Sanitary Surveillance (ANVISA), which is responsible for ensuring products quality. Thus, the objective of this work was to analyze the quality of the coffee marketed in the city of Campos Gerais - MG, with and without registration of ANVISA.

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MATERIALS AND METHODS

Raw material: In this study, samples of coffee sold in supermarkets of the city of Campos Gerais, MG were used. The methodology for sampling, collecting, packaging, transport and adequate analysis of each type of food obeyed the provisions of ANVISA - National Health Surveillance Agency - Ministry of Health, by DRC Nº 12 of January 2, 2001 (Brazil, 2001). Samples were purchased in supermarkets and analyzed in the laboratory. Samples with and without ANVISA registration were analyzed and the results compared. The completely experimental process was carried out in the Laboratory of Bromatology of the Faculty of Sciences and Technologies of Campos Gerais.

Experimental design: A completely randomized design (CRD) was used, of 6 coffee samples (3 with registration and 3 without registration) with 4 replicates for each analysis.

Statistical analysis: Results were subjected to Analysis of Variance, by means of the statistical software Sisvar (Ferreira, 2011). In addition, a tukey test at 5% probability was applied.

Preparation of samples for chemical and physical chemical analysis: Coffee samples were purchased at the supermarket ready for the analysis. After the purchase, they were transferred to amber packaging, hermetically sealed and kept in a dry, ventilated and dark place.

Preparation of aqueous extracts: 2g coffee powder were weighed in 25ml distilled water, for 48 hours under refrigeration, according to the technique of Silva *et al.* (2009). Then, blends were filtered on filter paper and the insoluble parts soaked in another 25ml distilled water over the same period of time. Resulting blends were filtered and liquids obtained from this filtration were joined, leading to the extracts that were studied. Twelve extracts were made, of which 6(six) were obtained from registered coffee powder samples, and 6 obtained from samples of coffee powder without registration. From these extracts, pH, total solids and titratable acidity were evaluated.

Chemical analysis

Determination of Ph: pH was determined by glass electrode potentiometry, using a Digital QUIMIS pH meter. Based on the technique of the Association of Official Analytical Chemistry – AOAC (2005). The technique consists in measuring the apparatus with solution pH 7.0 and then with pH 4.0. After each measurement, the electrode was washed with distilled water.

Total Titratable Acidity (TTA): It was determined by filtrate titration, with a standard solution of NaOH 0.1M, according to the technique of A O A C (2005). The results obtained were expressed in g of total acids by $100g^{-1}$ of coffee powder. For the preparation of the solution to be titrated, 5mL of extract was used and completed to 50mL with distilled water in erlenmeyer flask. Then, 3 drops of 1% phenolphthalein dye was added to the solution. Solution was titrated to the turning point (pink color) and the spent amount of NaOH was recorded for each sample.

Determination of Humidity: Moisture content was performed according to the gravimetric method of the Association of Official Analytical Chemistry – AOAC (2005), which consists of temperatures from 100° C to 105 ° C, water loss by dehydration. For humidity determination, 2 g of coffee powder were weighed, in crucibles previously submitted to the oven at 150 ° C for about three hours, removed with tweezers aid, packed in a hermetically sealed desiccator for about 30 minutes, and tared. Capsules with the respective samples were taken 60+/-5 °C oven. After 6 hours, they were removed from the oven and stored in a hermetically sealed desiccator for 30 minutes to cool, and then they were weighed. Integral sample is the name given to the product used prior to moisture removal, after moisture removal, it is called a dried or desiccated sample. The following variables will be used to determine humidity:

Humidity (%) = {[(capsule + integral sample) - (capsule + dry sample)] / integral sample} x 100

Total Soluble Solids (TSS): Total soluble solids were determined using a digital refractometer, Atago brand, and model PR-100 Palette, with automatic temperature adjustments, thus, results were expressed as percentage of soluble solids per 100g of coffee powder, according to AOAC methodology (2005).

Determination of Caffeine

Preparation of extract by acid extraction: This step was performed by acid extraction, that is, there is a selective carbonization of sample organic matter with sulfuric acid, releasing caffeine, which is then extracted using chloroform, according to the method described by A O A C (2005). 1g of coffee powder was weighed in 100 mL beaker, and then carefully added 4mL of sulfuric acid, avoiding the formation of groats with the aid of a glass stick. Mixture after homogenization was heated in a water bath at 60 ° C for 15 minutes. 50 mL of distilled water was added at 60 ° C. It was then heated in a water bath for another 15 minutes. It was hot-filtered to a 250 mL beaker through filter paper moistened with distilled water. Soon after, filter and the beaker were washed with 3 portions of 10 mL hot distilled water acidulated with 3 drops of sulfuric acid. Filtrate was placed in a 500 mL separatory funnel (decantation), to which 30 mL of chloroform were added, then, stirring is continued for a further 2 minutes. Subsequently, the separation was carried out, filtering the chloroform mixture, which was in the lower layer, taking care not to let the residue of caffeine pass by. Extraction was repeated with a further 30 mL chloroform portion. Chloroform extract obtained was evaporated in water bath at 100 ° C.Residue was then diluted with hot distilled water, filtering to a 100 mL volumetric flask.Finally, samples were allowed to cool and the absorbance was measured at 320nm in a spectrophotometer.

Determination of caffeine by spectrophotometry: Caffeine quantification was performed according to the method described by A O A C (2005). Anhydrous caffeine was oven dried at 150 ° C for one hour and then cooled in a desiccator. Soon after, a stock solution of caffeine was prepared with 100mg per 100 mL^{-1} of distilled water. Using a 10 mL burette, aliquots of 2, 3, 5, 7, 8, 10, 15 mL were transferred into 100 mL volumetric flasks. Volume was then filled with distilled water and homogenized. Absorbance was measured at 320nm using a blank distilled water for spectrophotometer calibration. With values obtained, standard curve was constructed by linear regression of the absorbance values obtained (y-axis) and caffeine concentrations (x-axis) expressed in mg of caffeine per $100g^{-1}$ solution.

The following formula was used for the calculation:

$\{(A-b)xV\} / (axPx1000)$

That is:

A = sample absorbance;
b = linear coefficient of the line obtained in the standard curve;
a = absorbance (the angular coefficient of the line obtained in the standard curve;
V = volume in mL of the dilution of the caffeine residue;
P = mass of the sample in g.

Coloring Index: 2 g of each ground coffee sample were weighed and placed in Erlenmeyer flask, adding 50 ml of distilled water, stirring for one hour in a horizontal shaker. The samples were then filtered on filter paper. From this filtrate, 5

ml were withdrawn and 10 ml of distilled water added, leaving it to stand for 20 minutes. Reading was performed on spectrophotometer at 425 nm absorbance, with blank the distilled water according to methodology described by Silva *et al.* (2009). Results were expressed as CI (color intensity) at 425 nm.

RESULTS AND DISCUSSION

Physicochemical analysis

pH determination: pH values of ANVISA registered coffees obtained in the research are described in Table 1. It can be observed that these vary from 5.78 to 6.34. Table 2, below, shows pH results for coffees without registration. These range from 6.27 to 6.60. According to Siqueira and Abreu (2006), coffee, undergoing roasting changes in its pH, which may interfere with its acceptability by the consumer. For these authors, the ideal pH should be from 4.95 to 5.20, which makes it good without excesses of acidity or bitterness. According to pH values indicated in Tables 1 and 2, all samples of roasted and ground coffee analyzed are above ideal values (4.95 a 5.20). Among those with registration, the sample with the highest pH was Sample A (6.34) and sample with lower pH was of Sample C (5.78). Among samples of coffee without registration, sample A presented a lower pH (6.27) statistically equal to sample B, and sample C presented higher pH (6.60). Fernandes *et al.* (2003) when analyzing the chemical constituents and aqueous extract contents of arabica (*Coffeaarábica*) and conilon (*Coffeaconilon*) roasted coffees obtained values of pH 5.87 to 6.03, a value similar to that found in Sample B coffee with a registration (5.91). According to table 1, the 3coffee samples with registration were statistically different, and obtained a mean Coefficient of variation (CV) of 0.07%. In the 3coffee samples without registration sample A and sample B were statistically the same and different from sample C. Mean CV among the unregistered samples was 1.08%.

Total Titratable Acidity (TTA): Acidity has an inverse relationship with the beverage quality, the higher the acidity, the worse the coffee quality (Martinez *et al.,* 2013). Titratable total acidity value found in the samples with registration shown in Table 3 is from 1.74 NaOH 0.1 M. $100g^{-1}$ to 2.60 NaOH 0.1 M. $100g^{-1}$. Statistically, samples B and C are the same, and different from sample A. Mean CV among the samples with registration was 9.89%. In analyzed samples without registration (Table 4) the values were from 1.63 NaOH 0.1 M. $100g^{-1}$ to 1.84 NaOH 0.1M. $100g^{-1}$. Statistically, all unregistered samples (sample A, B, and C) are the same. Mean CV among the samples was 6.88%. Values were lower than those reported by Filho *et al.* (2015) in six coffee beverage patterns and submitted to two degrees of roasting, whose contents ranged from 2.80% to 3.50%. Fernandes *et al.* (2003) found values ranging from 1.62% to 1.72%, similar to that found in the search for Sample C of unregistered coffee (1.63 NaOH 0.1 M. $100g^{-1}$). Coffee bean roasting severity reduces the acidity of the beverage by destroying chlorogenic acids that are attached to the grain matrix.Due to roasting, the amount of carboxylic acids present in coffee also decreases. Due to carbohydrates degradation, volatile acids during roasting process are increased, reaching a maximum, which then decrease with increasing roasting due to volatilization (Fernandes *et al.,* 2001).

Humidity: One of the oldest methods of preservation is drying or dehydration. Food preservation through drying is a direct consequence of water removal, without which organisms cannot grow (Jay, 2005). As shown in Table 5, humidity contents found in coffees with registration vary from 2.53% and 4.61%, all samples were statistically different, obtaining a mean CV of 5.33%. On the other hand, unregistered samples (Table 6) range from 1.75% to 4.77%, statistically all samples are different from each other with a mean CV of 8.86%. All samples with registration and without registration are in compliance with ANVISA legislation in DRC 377 of September of 2005 that recommends a maximum humidity of 5%. Silva *et al.* (2016) in a research on coffees physical chemical quality (*Coffeaarábica*) cultivated in Campos Gerais, Minas Gerais, obtained values of humidity from 1.2% and 2.0%, which are compared with humidity value of sample B without registration, obtained in this study. Silva *et al.* (2014), when evaluating the quality of coffee grown in Campos Gerais, Minas Geraishumidity values ranging from 0.24% to 1.20% were found, all below the values found in the present study (1.75% to 4.77%). According to Francisco *et al.* (2014), packaging is the guarantee that the packaged product is protected from external environment humidity. Of the analyzed samples, only one, Sample A, without registration, was not sealed, however, it did not present higher humidity value in relation to the others. Two other samples, sample B and coffee from Sample C without registration, presented non-resistant packs favorable to deterioration, like holes or violation by insects.Other brands presented packages with laminated paper that provide greater protection against moisture to the packaged product.

Total Soluble Solids: Total soluble solids contents are indicative of the amount of sugars in coffee. They provide the sweetness of coffee, thus being an important attribute in determining its flavor (Silva *et al.,* 2014). Mean Total Soluble Solids contents found in this work are described in Tables 7 and 8. In Table 7, coffee marketed with ANVISA registration, values between 10.0% and 10.8% of the total soluble solids content can be observed, and all the samples are statistically equal and have a mean CV of 4.23%.In unregistered coffees, Table 8, we can observe a variation from 10.0% to 10.5% of the total soluble solids content; all samples are also statistically the same and have a mean CV of 3.28%. Silva *et al.* (2016), when evaluating the physical chemical quality of coffee (*Coffeaarábica)* cultivated in Campos Gerais, Minas Gerais, obtained total soluble solids contents ranging from 10.3% to 29.5%, compatible with the values found in coffees Sample C with registration, of sample A without registration (10.5%), and Sample B with registration (10.8%). Silva *et al.* (2014), in a survey on coffees quality cultivated in Campos Gerais, Minas Gerais, obtained results ranging from 10.0% to 37.5%, some of which obtained in the present study (10.0% to 10, 8%). Mendonça *et al.* (2007), when studying bromatological parameters of raw and roasted grains of coffee cultivars (*Coffeaarabica*) found total soluble solids values ranging from 24.05% to 27.89%, all above the values found in the present study (10.0% to 10.8%). These values can be related to the form of roasting thebeans; high amount of Soluble Solids is desirable, since they contribute to the body and the beverage yield.

Caffeine: According to Ordinance No. 377, of September 2005 of ANVISA, caffeine minimum limit for roasted coffee is around 0.7%.

Table 1. Average values of pH in roasted traditional type coffees with registration at ANVISA

Treatment/ crop	Sample A	Sample B	Sample C
Roasted	6.34c	5.91b	5.78a
Mean CV	0.07%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 2. Average values of pH in roasted traditional type coffees without registration at ANVISA

Treatment/ crop	Sample A	Sample B	Sample C
Roasted	6.27a	6.36a	6.60b
Mean CV	1.08%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 3. Mean contents titratable total acidity (NaOH 0.1M. 100g^{-1}) in traditional roasted coffee with registration at ANVISA.

Treatment/ crop	Sample A	Sample B	Sample C
Roasted	1.74a	2.40b	2.60b
Mean CV	9.89%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 4. Mean contents titratable total acidity (NaOH 0.1M. 100g^{-1}) in traditional roasted coffee without registration at ANVISA.

Treatment/ crop	Carioca coffee	São Luís coffee	Da Serra coffee
Roasted	1.74a	1.84a	1.63a
Mean CV	6.88%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 5. Humidity percentage in roasted traditional type coffees with ANVISA registration

Treatment/ crop	Sample A	Sample B	Sample C
Roasted	2.92b	2.53a	4.61c
Mean CV	5.33%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 6. Humidity percentage in roasted traditional type coffees without ANVISA registration

Treatment/ crop	Sample D	Sample E	Samlpe F
Roasted	3.03b	1.75a	4.77c
Mean CV	8.86%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 7. Total soluble solids mean contents (%) in roasted traditional type coffees registered at ANVISA.

Treatment/ crop	Amostra A	Amostra B	Amostra C
Roasted	10.0a	10.8a	10,5a
Mean CV	4.23%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 8. Mean values of total soluble solids (%) in roasted traditional type coffees without registration at ANVISA.

Treatment/ crop	Sample A	Sample B	Sample C
Roasted	10.5a	10.0a	10.0a
Mean CV	3.28%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 9. Average caffeine contents (%) in roasted traditional type coffees registered at ANVISA

Treatment/ crop	Sample A	Sample B	Sample C
Roasted	0.36b	0.33a	0.46c
Mean CV	2.13%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 10. Average caffeine contents (%) in roasted traditional type coffees without registration at ANVISA

Treatment/ crop	Sample A	Sample B	Sample C
Roasted	0.34a	0.30a	0.31a
Mean CV	0.91%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 11. Coloring index at 425nm (CI) in roasted traditional type coffees with registration at ANVISA

Treatment/ crop	Sample A	Sample B	Sample C
Roasted	1.64b	1.05a	1.07a
Mean CV	1.66%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Table 12. Coloring index at 425nm (CI) in roasted traditional type coffees without registration at ANVISA

Treatment/ crop	Sample A	Sample B	SampleC
Roasted	1.63c	1.33b	1.26a
Mean CV	2.23%		

* Means followed by lower-case distinct letters in the lines differ from each other, by Tukey test, at 5% probability.

Caffeine value found in the samples with registration shown in Table 9 are between 0.33% and 0.46%. Samples A, B and C with registration were statistically different from each other and presented a mean CV of 2.13%. Measured caffeine values in the unregistered samples shown in Table 10 ranged from 0.30% to 0.34%, we can observe that all samples without registration are statistically equal, having a mean CV of 0.91%.

Coloring Index: Characteristic color intensity of roasted coffee is influenced by roasting point. Color is the main controller of end roasting point. In most industries through a standard sample, coffee color being roasted is continuously monitored (Siqueira and Abreu, 2006). According to table 11, coloring index of registered coffee samples ranged from 1.05% to 1.64%, samples B and C are statistically equal and different from sample A, and the mean CV among samples was 1.66%. Coloring index of unregistered coffee samples varied from 1.26% to 1.63% (Table 12), all samples (samples A, B and C) are statistically different from each other. Mean CV among unregistered samples was 2.23%. According to Siqueira and Abreu (2006) in an evaluation on the physical chemical composition of coffee submitted to two types of roasting and different forms of processing obtained results on coloring index in the natural processing in light roast of 1.13 CI, and medium roasting of 2.00 CI. Values obtained in this work are among the values obtained by Siqueira and Abreu (2006). Lucciardi *et al.,* (2005) evaluating the chemical composition of roasted and ground coffee of different brands marketed in southern Minas Gerais, obtained coloring index results ranging from 1.28 CI to 2.08 CI, values similar to those found in the present study.

Conclusion

Coffees with and without registration at ANVISA obtained higher pH values than indicated by law. pH is a quality indicator, and high values may be indicative of coffee bitterness and acidity. Total titratable acidity and humidity content of the studied coffees (with and without registration at ANVISA) are within the parameters established to obtain quality for consumption. Total soluble solids values obtained from studied coffees did not have significant differences among them. Concentration of soluble solids are desirable for coffee quality. Coloring index establishes the type of roasting that was held in coffee. Therefore, with the results of this study we can verify that they are quite similar. Caffeine concentration found in the studied coffees are lower than the limit set by law. Caffeine levels found in this study can be consumed by people with caffeine sensitivity, since substance can cause restlessness, anxiety, headache, insomnia, also causes contraction of the veins and arteries which hinders blood circulation and accelerates the heartbeat. In pregnant women, it is recommended to have a little or even cut coffee as it may cause fetal deformities. Therefore, it can be reported that, in view of the results obtained, all coffees studied with and without ANVISA registration are fit to be marketed and consumed by the population.

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