

Physicochemical characterization of a composite *Coffea arabica* L. sample from Alto Noroeste Fluminense under three roasting profiles¹

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ABSTRACT

Coffee production in Alto Noroeste Fluminense has gained regional relevance, although information on the physicochemical characteristics of locally produced beans under different roasting conditions remains limited. This study characterized a composite sample of naturally processed *Coffea arabica* L. from the 2022 and 2023 harvests produced in Bom Jesus do Itabapoana, Rio de Janeiro, Brazil, under light, medium, and dark roasting conditions. Moisture, ash, lipids, protein, pH, titratable acidity, water-soluble extract, alcohol-soluble extract, and caffeine were determined using official analytical procedures. Each roasting profile was applied to a single portion of the composite sample, and the subsequent triplicate determinations represented analytical replicates rather than independent experimental replicates. Therefore, the results were interpreted descriptively. Moisture values decreased from 3.51% in the light roast to 1.76% in the dark roast, while titratable acidity decreased from 221.67 to 178.37 g L⁻¹. Higher ash and lipid proportions were observed with increasing roasting degree, whereas caffeine showed comparatively limited variation, ranging from 0.876 to 0.918 g 100 g⁻¹. These findings provide an initial physicochemical reference for the evaluated composite sample of Arabica coffee from Alto Noroeste Fluminense and describe distinct physicochemical patterns among the three roasting profiles.

Keywords: thermal processing, titratable acidity, extract yield, proximate composition.

INTRODUCTION

Coffee production has historical and socioeconomic relevance in the state of Rio de Janeiro, with the Northwestern region representing its main coffee-producing area. In recent years, initiatives involving producers, extension services, research institutions, and local organizations have promoted quality-oriented production and the development of specialty coffee systems in Alto Noroeste Fluminense, particularly in municipalities such as Bom Jesus do Itabapoana, Porciúncula, Natividade, and Varre-Sai.^(1,2)

This process has increased the visibility of regional coffees and reinforced the need for scientific information capable of supporting their technological characterization and value addition. Among the processing operations applied to coffee, roasting is one of the most important determinants of the physicochemical and sensory characteristics of the final product. Progressive thermal treatment promotes moisture loss and complex transformations involving carbohydrates, proteins, lipids, organic acids, phenolic compounds, and other components of the coffee matrix.^(3,4)

These transformations are associated with changes in acidity, color, aroma, extractability, and the relative proportions of nonvolatile constituents, making roasting control an important factor in coffee quality and processing consistency.⁽³⁻⁵⁾ Previous studies have demonstrated that roasting degree affects several physical and chemical properties of *C. arabica*, including moisture, pH, titratable acidity, color, and composition.^(5,6)

Despite the increasing recognition of Alto Noroeste Fluminense as an emerging coffee-producing region, peer-reviewed information on the physicochemical characteristics of coffees produced in this area remains limited, particularly regarding samples evaluated under different roasting conditions. Establishing such information is relevant for understanding the behavior of regional coffee during processing and for providing an analytical basis for future studies on its chemical, technological, and sensory characteristics. Therefore, this study aimed to characterize the physicochemical properties, extract yields, and caffeine content of a composite *Coffea arabica* L. sample from Alto Noroeste Fluminense after light, medium, and dark roasting.

MATERIALS AND METHODS

Plant material and experimental design

Naturally processed *Coffea arabica* L. beans from the 2022 and 2023 harvests were obtained from a

coffee-producing property located in Bom Jesus do Itabapoana, Rio de Janeiro State, Brazil, within the Alto Noroeste Fluminense coffee-producing region. The specific cultivar was not documented; therefore, the plant material is reported at the species level. The identity of the producer is withheld for confidentiality reasons. Following harvest, the green coffee beans were stored in specialized bags suitable for green coffee storage under laboratory conditions at AlQualis Junior, Instituto Federal Fluminense (IFF), Campus Bom Jesus do Itabapoana, Rio de Janeiro, Brazil. Beans from the 2022 and 2023 harvests were combined in a 1:1 proportion and homogenized to obtain a single composite sample. Harvest year was therefore not considered an experimental factor. The composite sample was subsequently divided among the three roasting profiles evaluated in this study. Analytical determinations were performed in triplicate for each roasting profile; these triplicates represented analytical replicates and not independent experimental replicates.

Roasting treatments and sample preparation

A total of approximately 700 g of the composite green coffee sample was divided into three portions corresponding to the light, medium, and dark roasting profiles. Roasting was performed using a Stratto coffee roaster (Carmomaq, Brazil).

Roasting cycles lasted approximately 13–15 min, depending on the targeted roasting degree, with temperatures reaching approximately 230 °C during the final stage of the process. At the end of each roasting cycle, the beans were immediately transferred to the cooling tray and allowed to cool to room temperature.

Approximately 150 g of coffee from each roasting profile was subsequently ground in a disc mill and homogenized to obtain representative samples for physicochemical analyses.

Physicochemical analyses

Physicochemical determinations were performed according to official analytical procedures described by the Instituto Adolfo Lutz (IAL).⁽⁷⁾ All determinations were conducted in analytical triplicate. Moisture content was determined gravimetrically by drying the samples in an oven at 105 °C until constant mass. Ash content was determined by incineration in a muffle furnace at 550 °C. Total lipid content was quantified by Soxhlet extraction using petroleum ether.

Crude protein was determined by the classical Kjeldahl method, involving digestion, distillation, and titration of total nitrogen. Protein content was calculated using a nitrogen-to-protein conversion factor of 6.25, as recommended by the IAL for foods without a specific conversion factor.⁽⁷⁾ The pH was measured using a digital benchtop pH meter (PHS3BW, BEL Engineering, Italy), with a manufacturer-stated accuracy of ± 0.01 pH unit. Before each analytical session, the instrument was calibrated at 25 °C using standard buffer solutions at pH 4.00 and 7.00. This pre-analysis calibration constituted the routine quality-control procedure for pH determination. Measurements were performed in analytical triplicate for each roasting profile.

Titrateable acidity

Titrateable acidity was determined by acid-base titration according to the IAL procedure.⁽⁷⁾ The prepared sample was titrated with standardized 0.1 mol L⁻¹ sodium hydroxide solution using 1% phenolphthalein as the endpoint indicator. The endpoint was identified by the development of a persistent pale-pink coloration. Titrateable acidity was calculated from the volume of NaOH consumed, its correction factor, and the sample amount, with the final values converted and expressed as g L⁻¹.

Water-soluble extract

Water-soluble extract was determined according to the IAL procedure.⁽⁷⁾ One gram of ground coffee was weighed into a 125 mL Erlenmeyer flask, followed by the addition of 100 mL of hot distilled water. The mixture was maintained in a boiling water bath for approximately 1 h.

The suspension was quantitatively transferred to a 250 mL volumetric flask, and the volume was completed with distilled water. After homogenization, the solution was filtered, and a 25 mL aliquot was transferred to a previously weighed 50 mL beaker. The aliquot was evaporated to dryness in a water bath. The residue was subsequently dried in an oven at 105 °C for 1 h, cooled in a desiccator, and weighed. Drying, cooling, and weighing were repeated until constant mass. Water-soluble extract was expressed as a percentage of the original sample mass.

Alcohol-soluble extract

Alcohol-soluble extract was determined according to the IAL procedure.⁽⁷⁾ Two grams of ground coffee were transferred to a 100 mL volumetric flask with

approximately 80 mL of alcohol. The suspension was maintained for 4 h, with agitation at 30 min intervals, and subsequently allowed to stand for 16 h. The volume was completed to 100 mL with alcohol and the suspension was filtered. A 50 mL aliquot of the filtrate was transferred to a previously weighed 100 mL beaker. The solvent was evaporated in a water bath, and the remaining residue was dried at 105 °C for 1 h, cooled in a desiccator, and weighed until constant mass. Alcohol-soluble extract was expressed as a percentage of the original sample mass.

Caffeine determination

Caffeine was determined according to the IAL spectrophotometric method.⁽⁷⁾ One gram of ground coffee was weighed into a 100 mL beaker, and 4 mL of sulfuric acid were carefully added. After homogenization, the mixture was maintained in a water bath for 15 min. Fifty milliliters of hot distilled water were subsequently added, and the suspension was heated for an additional 15 min.

The hot suspension was filtered through water-moistened filter paper into a 500 mL separatory funnel. The beaker and filter were washed with three 10 mL portions of acidified hot water, and the washing fractions were combined with the filtrate. After cooling, caffeine was extracted with 30 mL portions of chloroform. The extraction was repeated, and the combined chloroform fractions were evaporated using a rotary evaporator. The residue was dissolved in hot distilled water, filtered, and quantitatively transferred to a 1,000 mL volumetric flask.

After cooling, the volume was completed with distilled water. Absorbance was measured at 274 nm using an ultraviolet-visible (UV-Vis) spectrophotometer. Caffeine concentration was determined using an external calibration curve prepared with anhydrous caffeine and was expressed as g caffeine 100 g⁻¹ coffee.

Caffeine calibration curve

Anhydrous caffeine was previously dried at 105 °C for 1 h and subsequently cooled in a desiccator. A stock solution containing 10 mg of caffeine per 100 mL of distilled water was prepared. Aliquots of 2, 3, 5, 7, 8, and 15 mL of the stock solution were transferred to separate 100 mL volumetric flasks and completed to volume with distilled water, producing six calibration levels of 0.2, 0.3, 0.5, 0.7, 0.8, and 1.5 mg 100 mL⁻¹, corresponding to a concentration range of 2–15 mg L⁻¹. Absorbance at each calibration level was measured in duplicate at 274 nm

against distilled water as the analytical blank, and the mean absorbance of the duplicate readings was used for linear regression. Linear regression between absorbance and caffeine concentration yielded the equation $A = 0.5689C + 0.00373$, where A represents absorbance and C represents caffeine concentration in mg 100 mL⁻¹, with a coefficient of determination of $R^2 = 0.9998$. Caffeine concentrations in the coffee samples were calculated from the regression equation and corrected according to the sample mass and dilution employed in the analytical procedure.

Statistical treatment

Results were expressed as mean $\pm$ standard deviation of three analytical determinations for each roasting profile. Analytical triplicates were used to assess measurement precision and were not considered independent experimental replicates. Therefore, no inferential statistical comparisons among roasting profiles were performed. For caffeine determination, analytical repeatability was additionally expressed as relative standard deviation (RSD), calculated as (standard deviation/mean) $\times$ 100.

RESULTS AND DISCUSSION

Physicochemical changes associated with roasting

Differences in the physicochemical profile of the evaluated composite coffee sample were observed among the three roasting profiles (Table 1).

Moisture content decreased progressively from 3.51% in the light roast to 2.94% in the medium roast and 1.76% in the dark roast. In contrast, ash content increased from 4.10 to 4.46%, while lipid content increased from 11.78 to 13.92%. Protein content showed comparatively smaller variation, ranging from 13.82% in the light roast to 14.00% in the medium and dark roasting treatments. The progressive reduction in moisture is consistent with the increased thermal exposure occurring during roasting. Water loss constitutes one of the major physical transformations of coffee beans during this process and contributes to changes in mass, density, structure, and other physicochemical characteristics of roasted coffee.^(4,6) Previous studies have also shown that roasting degree affects moisture and other physical characteristics of *C. arabica*.⁽⁶⁾ The increases observed in ash and lipid contents should be interpreted primarily as relative concentration effects rather than absolute formation of these constituents. As roasting progresses, the loss of water and volatile substances

decreases the total mass of the coffee matrix, consequently increasing the proportional representation of comparatively less volatile components. Similar behavior was observed by Alamri *et al.*,⁽⁸⁾ who reported lower moisture and higher ether extract values with increasing roasting intensity in Arabica coffee.

Therefore, these results should not be interpreted as evidence of increased nutritional density resulting from roasting. Instead, they reflect changes in the relative composition of the coffee matrix following thermal processing. This distinction is important because roasting simultaneously promotes mass loss and numerous chemical reactions, meaning that an increase in the percentage of a constituent does not necessarily indicate an increase in its absolute amount.^(3,4)

Changes in acidity and pH

The acid-base characteristics of the evaluated samples differed among the three roasting profiles. The pH values were 4.98, 5.02, and 5.23 for the light, medium, and dark roasts, respectively, whereas titratable acidity values were 221.67, 205.70, and 178.37 g L⁻¹. The inverse pattern between pH and titratable acidity is consistent with chemical transformations known to occur during coffee roasting. Progressive thermal processing can alter several classes of compounds, including organic acids and phenolic constituents, while also promoting Maillard reactions, caramelization, and thermal degradation pathways.^(3,5)

Recent chemical profiling has shown that roasting intensity modifies coffee composition and compounds associated with acidity, including chlorogenic acids and their transformation products.⁽³⁾ However, the reduction in titratable acidity observed in the present study cannot be attributed specifically to chlorogenic acid degradation because individual organic acids were not directly quantified. The decrease should therefore be interpreted more conservatively as resulting from the overall thermal transformation of acidic constituents within the coffee matrix. This interpretation avoids assigning the observed change to a specific compound without direct analytical evidence. The progressive increase in pH accompanied by decreasing titratable acidity is also technologically relevant because acidity constitutes an important characteristic of roasted coffee and is closely associated with the roasting degree and the chemical changes occurring during thermal processing.^(2,3)

Table 1. Physicochemical characteristics of the composite *Coffea arabica* L. sample under light, medium, and dark roasting profiles. Values are expressed as mean $\pm$ standard deviation of three analytical determinations

Parameter	Light roast	Medium roast	Dark roast
Moisture (%)	3.51 $\pm$ 0.01	2.94 $\pm$ 0.01	1.76 $\pm$ 0.01
Ash (%)	4.10 $\pm$ 0.01	4.20 $\pm$ 0.01	4.46 $\pm$ 0.01
Lipids (%)	11.78 $\pm$ 0.03	12.45 $\pm$ 0.03	13.92 $\pm$ 0.02
Protein (%)	13.82 $\pm$ 0.01	14.00 $\pm$ 0.01	14.00 $\pm$ 0.01
pH	4.98 $\pm$ 0.01	5.02 $\pm$ 0.01	5.23 $\pm$ 0.01
Titrateable acidity (g L ⁻¹)	221.67 $\pm$ 0.07	205.70 $\pm$ 0.05	178.37 $\pm$ 0.07
Water-soluble extract (%)	2.39 $\pm$ 0.01	2.27 $\pm$ 0.01	2.31 $\pm$ 0.01
Alcohol-soluble extract (%)	4.86 $\pm$ 0.01	5.02 $\pm$ 0.01	5.23 $\pm$ 0.01
Caffeine (g 100 g ⁻¹)	0.876 $\pm$ 0.040	0.918 $\pm$ 0.024	0.896 $\pm$ 0.023

Soluble extract behavior

The water-soluble extract showed relatively limited variation among the evaluated roasting profiles, with values of 2.39, 2.27, and 2.31% for the light, medium, and dark roasts, respectively. Conversely, the alcohol-soluble extract values increased from 4.86% in the light roast to 5.02% in the medium roast and 5.23% in the dark roast. Roasting is known to induce extensive modifications in coffee constituents and consequently influence their extractability in solvents of different polarities.^(3,4) The comparatively small variation observed for the water-soluble fraction suggests that, under the conditions evaluated in the present study, the overall proportion of compounds recovered by aqueous extraction remained relatively stable. In contrast, the pattern observed for the alcohol-soluble fraction suggests differences in the proportion and/or extractability of constituents recovered using the alcoholic solvent. Nevertheless, identification of the specific compounds responsible for these differences would require complementary chromatographic or spectroscopic analyses. Therefore, the observed alcohol-soluble extract values should not be attributed to a specific chemical class based solely on the present results.

Caffeine content

Caffeine provided an additional chemical marker for the physicochemical characterization of the evaluated composite coffee sample. Mean concentrations were 0.876 $\pm$ 0.040, 0.918 $\pm$ 0.024, and 0.896 $\pm$ 0.023 g 100 g⁻¹ for the light, medium, and dark roasting profiles, respectively. Analytical repeatability, expressed as relative standard deviation (RSD), was 4.57%, 2.61%, and 2.57% for the light, medium, and dark roasting profiles, respectively.

The comparatively limited variation observed among these values is consistent with previous studies reporting

relative stability of caffeine under conventional coffee-roasting conditions. Alamri et al.,⁽⁸⁾ for example, reported caffeine concentrations of 1.13, 1.17, and 1.08% for light-, medium-, and dark-roasted Arabica coffee, respectively. Bobková et al.⁽⁹⁾ further demonstrated that caffeine concentration in *C. arabica* can be influenced by factors other than roasting, including geographical origin, post-harvest processing, and growing conditions.

In the present sample, caffeine values varied less across the three roasting profiles than moisture, pH, titrateable acidity, ash, and lipid proportions. This pattern contributes additional information to the physicochemical characterization of the evaluated coffee while reinforcing that different components of the coffee matrix may exhibit distinct patterns under thermal processing conditions.

Relevance to the characterization of coffee from Alto Noroeste Fluminense

The present study contributes physicochemical information on a naturally processed Arabica coffee sample originating from Alto Noroeste Fluminense, a Brazilian coffee-producing region for which analytical information remains comparatively limited. The evaluated material consisted of a single composite sample of *C. arabica* beans prepared by combining the 2022 and 2023 harvests in a 1:1 proportion.

The main limitation of the present study is pseudoreplication at the roasting-treatment level, because each roasting profile was applied to only one portion of a single composite sample. The three determinations performed for each profile represented analytical rather than independent experimental replicates. Consequently, variability among independent coffee lots, harvests, farms, and roasting processes could not be estimated. The observed

differences among roasting profiles should therefore be interpreted descriptively within the evaluated composite sample and should not be extrapolated as population-level estimates for coffees produced throughout Alto Noroeste Fluminense.

Accordingly, these findings should not be interpreted as establishing a definitive physicochemical fingerprint for all coffees produced in Alto Noroeste Fluminense. Such characterization would require a broader sampling design encompassing independent farms, lots, harvest years, altitudes, identified cultivars, and post-harvest conditions. Instead, the present study provides an initial analytical baseline for the evaluated composite regional coffee sample. Geographical origin, production environment, genetic background, and processing conditions may independently influence coffee composition.^(4,5)

Sensory characterization of specialty coffees from Alto Noroeste Fluminense has already identified relevant sensory attributes in coffees produced in the region.⁽¹⁰⁾ However, sensory characterization by trained or qualified assessors does not replace consumer-based evaluation. Future studies integrating independent coffee lots, replicated roasting experiments, physicochemical characterization, and consumer acceptance and preference tests would therefore be valuable for determining whether physicochemical differences among roasting profiles correspond to perceptible differences and consumer preferences.

The present study provides an initial assessment integrating conventional physicochemical parameters, caffeine determination, and extractability measurements in a composite Arabica coffee sample from Alto Noroeste Fluminense. The results expand the analytical information available for this emerging coffee-producing region and provide a basis for future investigations involving independent regional sampling, replicated roasting experiments, consumer acceptance and preference, bioactive compounds, and chemical markers of geographical origin.

Overall interpretation

Overall, the evaluated composite Arabica coffee sample showed distinct physicochemical patterns among the light, medium, and dark roasting profiles. Lower moisture and titratable acidity and higher pH, ash, and lipid proportions were observed with increasing roasting degree, whereas caffeine showed comparatively limited variation among the three profiles.

Together, these findings provide an initial physicochemical reference for a composite coffee sample from a still poorly characterized Brazilian producing region. Because the roasting conditions were not independently replicated, the observed patterns should be interpreted descriptively rather than as estimates of a generalized roasting effect. The results nevertheless provide a scientific basis for future investigations involving independent experimental replication and broader evaluation of the chemical, sensory, technological, and consumer-related characteristics of coffees produced in Alto Noroeste Fluminense.

CONCLUSIONS

Distinct physicochemical profiles were observed in the composite *Coffea arabica* L. sample after light, medium, and dark roasting. Increasing roasting degree was accompanied by lower moisture and titratable acidity and by higher pH, ash, and lipid proportions, whereas protein content showed comparatively limited variation. Caffeine concentration remained comparatively stable among the evaluated roasting profiles.

These findings provide an initial physicochemical reference for the evaluated composite sample of naturally processed Arabica coffee from Alto Noroeste Fluminense. Because the roasting treatments were not independently replicated, the results should be interpreted descriptively and should not be generalized to the regional coffee population. Future studies involving independent coffee lots, different harvests and farms, identified cultivars, replicated roasting experiments, and consumer acceptance and preference assessments are required to establish a broader regional profile.

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DATA AVAILABILITY STATEMENT

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

DECLARATION OF THE USE OF ARTIFICIAL INTELLIGENCE TOOLS IN WRITING

During the preparation of this work, the authors used Google Gemini and ChatGPT (OpenAI) to assist with language enhancement, technical translation, structural organization, and editorial refinement of the manuscript. After using these tools, the authors reviewed and edited the content as necessary and take full responsibility for the scientific accuracy, integrity, and content of the manuscript.

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