

Bromatological properties of *Coffea canephora* peels and grains

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ABSTRACT

Post-harvest handling through processing of coffee beans tend to generate high waste value, particularly hulls. These materials are often discarded or misused, resulting in no added value or additional income for coffee farmers. The objective of this study was to characterize through physicochemical analyses the hulls, green beans, and roasted beans of coffee genotypes grown in Western Amazonia, Brazil. The association between traits was interpreted using simple correlation estimates, clustering of means, and joint analysis of variance. The interaction between coffee fruit products and byproducts was significant, indicating differences in chemical composition between hulls, green beans, and roasted beans of genotypes grown in Western Amazonia. Ash content, aqueous extract, soluble solids, acidity, soluble sugars, reducing sugars, and non-reducing sugars were higher in the hulls. The pH value and concentrations of protein, lipids, phenolic compounds, and caffeine were higher in the beans. The ratio between the concentrations of total soluble solids/total titratable acidity was not significant. The husks of Amazonian Robusta coffees present physical and chemical characteristics suitable for their use, both for the extraction of chemical compounds and to produce beverages.

Key words: Coffee residues; chemical physical analysis; adding value; Western Amazon.

1 INTRODUCTION

The structure of coffee fruits is generally composed of the epicarp (skin), mesocarp (pulp or mucilage), endocarp (parchment), and the silver skin, in addition to the beans. During coffee processing using the natural fermentation method, the amount of by-products can reach up to 50% of the total production (Blinová et al., 2017). This can be significant especially in Brazil, where in 2024/2025, approximately 55 million bags of processed coffee were produced. In the Western Amazon, the estimated production for that same period was 2.3 million processed bags (CONAB, 2025).

Coffees cultivated in the Western Amazon showed an average post-pulping weight of approximately 53.0%, resulting in about 47% of waste production (Lourenço et al., 2022; Silva et al., 2025). In other words, for each 60 kg bag of processed coffee produced in the state of Rondônia, approximately 28 kg consist of by-products that are often underutilized (Manasa, Padmanabhan & Anu-Appaiah, 2021) or improperly managed. Furthermore, the husks can cause severe soil contamination when used improperly (Alves et al., 2017; Murthy & Madhava-Naidu, 2012; Oliveira & Franca, 2015) or mixing them with animal feed can induce carcinogenic effects (Arya et al., 2022). Therefore, strategies to add value to these by-products are essential to improve the environmental, economic, and social sustainability of the coffee industry.

In addition to caffeine and phenolic compounds, coffee husks contain minerals, proteins, lipids, cellulose, pectin, and sugars. They have been recognized as a novel food by the

European Union (EU) (Lachenmeier et al., 2022) due to the presence of substances responsible for high human biological activity, making them suitable for use as a high-value dietary supplement (Kristanti et al., 2022).

A study found that post-processing coffee by-products can be converted into promising and safe foods and/or food ingredients, showing no visible signs of toxicity, abnormal behavior, or mortality after ingestion (Iriondo-Dehond et al., 2020).

Several products derived from coffee husks have been studied and, in some cases, are already used in certain regions of Brazil. One example is coffee husk tea, commonly known as *cáscara*, which has been described as having highly appreciated sensory notes by consumers—aromatically sweet, floral, and with hints of dried fruits (Zeckel, Susanto & Erfiani, 2019), and has been well accepted in terms of sensory attributes (Thagunna et al., 2024).

In more in-depth studies on the beverage produced from coffee husks—specifically from the husks of *Coffea arabica* fruits—it was found that the antioxidant capacity of the aqueous extract was higher than that of the beans. The beverage showed good acceptance among tasters, emerging as a new alternative for the beverage industry (Neves et al., 2019). In *cáscara* samples originating from Switzerland, caffeine content was reported at 226 mg/L, with an antioxidant capacity of 8.9 mmol TE/L (Heeger et al., 2017).

In addition, coffee husk flour has potential for use in human nutrition, as it contains high concentrations of carbohydrates, proteins, caffeine, tannins, and polyphenols.

When combined with traditional flours, it contributes to the production of energy-rich and protein-rich foods with enhanced antioxidant activity (Damat et al., 2019).

Although studies reporting data on the chemical composition of the main coffee by-products can be found in the literature, information regarding this residue produced in the Western Amazon remains scarce, particularly concerning comparisons between husks and their respective beans. The genotypes cultivated in this location are hybrid plants (Conilon and Robusta varieties), selected by the farmers themselves or developed through controlled pollination by Embrapa - RO (Teixeira et al., 2020), also called Amazonian Robustas, and differentiated from coffees produced in other locations in Brazil (Reinicke; Rocha; Espindula, 2024).

Therefore, in order to define appropriate processes for adding value to coffee husks, our objective was to chemically characterize the coffee husk meal obtained from different genotypes and compare it between raw and roasted beans.

2 MATERIAL AND METHODS

2.1 Plant material and experimental location

The *C. canephora* genotypes studied in this work are publicly available, selected by coffee growers in the state of Rondônia, and are among the most widely cultivated clones in Rondônia's plantations (Table 1). These genotypes are characterized as inter-varietal hybrid plants (Espindula et al., 2022). The experimental samples were kindly provided by various coffee growers maintaining plantations within the geographical indication of the Matas de Rondônia region for Amazonian Robusta, classified as a designation of origin.

Table 1: List of *Coffea canephora* genotypes used for the evaluation of physicochemical attributes of husks, green beans, and roasted beans cultivated in the Western Amazon.

Genotype	Post-harvest
LB10	FN
GJ8	FN
LB102	FN
GJ25	FN
GJ25-f2	FI with the addition of components
GJ25-f1	FI
RE90-f1	FI
RE22-f1	FI
VR1	FN

FN: natural fermentation (dry method). FI: wet fermentation using semicarboxylic maceration for 20 days under anaerobic fermentation conditions (Alves et al., 2020).

The experimental procedures were carried out at the Food Science and Technology Laboratory (LCTA) of the Federal Institute of Education, Science, and Technology of Rondônia, Jaru Campus. For the experiment, the fruits were dried and pulped, separating the husks from the green beans.

2.2 Sample preparation and measurements

A portion of the green beans was roasted following the recommendations of the Fine Robusta cupping protocol. The roast color ranged from medium to medium-dark, measured using a roast color disk and confirmed with a Delta Coffee colorimeter, showing an approximate Agtron scale value of #45 for the whole bean and #75 for the ground coffee (UCDA, 2010). The husks, green beans, and roasted beans were ground to a particle size of 20 openings per inch (20 mesh), equivalent to a 0.85 mm opening. The green beans were ground using a knife mill, and the husks and roasted beans were ground using an electric disc grinder.

For the measurement of physicochemical characteristics, the coffee fruit products and by-products were analyzed for total ash, aqueous extract, lipids, caffeine, total soluble solids, and the ratio between total soluble solids and total titratable acidity following standard protocols (Instituto Adolfo Lutz, 2008). The total soluble solids content was determined by direct reading using a bench-top ABBE-type refractometer with minor modifications. One gram of each sample was weighed and dissolved in 10 mL of boiling water (92–94°C), and the samples were centrifuged at 4,000 rpm for 10 minutes. Results were expressed in °Brix.

The pH was measured by weighing 5 g of each sample and diluting it in 50 mL of boiling distilled water, followed by agitation for 1 hour at 150 rpm on a shaker table. The sample was then filtered through Whatman No. 4 paper, and the pH was measured using a digital potentiometer. Total titratable acidity was determined by weighing 1 g of each sample and diluting it in 100 mL of boiling distilled water, followed by agitation for 1 hour at 150 rpm on a shaker table. The sample was filtered through Whatman No. 4 paper and titrated with 0.1 mol·L⁻¹ NaOH until reaching pH 8.1–8.2 at a temperature of 25°C. Results were expressed as the volume of 0.1 mol·L⁻¹ NaOH solution per 100 g of sample (Association of Official Agricultural Chemists - AOAC, 2000).

Content crude protein determination was performed according to the official methodology with modifications. For the analysis, 1g of the dry sample was weighed, 7 mL of sulfuric acid and 2.5g of catalytic mixture (copper sulfate + potassium sulfate, in a 1:10 ratio) were added. The results were expressed as a percentage.

For the determination of total soluble sugars, total reducing sugars, and total phenolic compounds, the extract was prepared following standard methodology (Costa et al., 2014). Specifically, 2.5 g of the sample was weighed and dissolved in 50 mL of boiling distilled water, stirred in a Dubnoff water bath for 2 hours at 40°C, and then filtered through Whatman No. 4 paper.

The concentration of total soluble sugars was determined using the Antrona method, with results expressed as a percentage of glucose at a wavelength of 630 nm. Total reducing sugar content was determined using the Somogyi-Nelson method (Maldonado et al., 2013), with values expressed as a percentage of glucose at 540 nm. Total phenolic compounds were measured using the Folin-Ciocalteu method (AOAC, 2000), with results expressed as a percentage of gallic acid at a wavelength of 760 nm. The concentration of non-reducing sugars was calculated as the difference between total soluble sugars and total reducing sugars, and the results were expressed as a percentage.

2.3 Data analyses

In the data analysis, the significance of the genotype effect was tested individually, with the aim of comparing means between groups belonging to a single experimental factor, considering each set of treatments independently (Cruz, 2013), according to equation 1:

$$Y_{ijk} = m + G_i + e_{ijk} \quad (1)$$

where Y_{ijk} refers to the observation of the i^{th} genotype in the j^{th} measurement, m is the experimental mean, G_i is the effect of the i^{th} genotype and e_{ijk} is the experimental error.

Additionally, a joint analysis of variance was performed to simultaneously evaluate the effect of genotype and the influence of treatments and the genotype $\times$ treatment interaction (Cruz, 2013), according to equation 2:

$$Y_{ijk} = m + G_i + Y_j + GY_{ij} + e_{ijk} \quad (2)$$

where Y_{ijk} refers to the observation of the i^{th} genotype in the j^{th} measurement, m is the experimental mean, G_i is the effect of the i^{th} genotype, Y_j is the effect of the j^{th} measurement, GY_{ij} is the interaction effect between the i^{th} genotype and the j^{th} measurement, and e_{ijk} is the experimental error.

The associations between the variables were interpreted using Pearson correlation estimates (Cruz, Carneiro & Bhering, 2021), and the genetic diversity among the genotypes was evaluated through the dispersion of the first two principal components (PCA) derived from the genotypic values of the evaluated characteristics. The PCA was performed using the correlation matrix, considering that the evaluated variables presented different measurement scales. Before the analysis, the data were standardized.

Of the thirteen parameters evaluated in this study, only eleven were used for correlation and PCA analyses, excluding the non-reducing sugars variable and the ratio between total soluble solids and total titratable acidity. These were excluded because their values are derived from the subtraction and/

or division of other variables, creating inherent correlations between the factors.

All laboratory analyses were performed in triplicate, and the statistical processing of the data was performed using the GENES software (Cruz, 2013).

3 RESULTS

Some chemical compounds were observed to be present in higher amounts in the husks than in the green beans, such as minerals, represented by total ash content (ASH); organic acids, quantified as total titratable acidity (TTA); total reducing sugars (RS); non-reducing sugars (NRS); and a higher content of water-soluble compounds, characterized by aqueous extract (AE) and total soluble solids (TSS). In contrast, the concentrations of proteins (CPC: crude protein), caffeine, lipids (EE: ether extract), and total phenolic compounds (TPC) are higher in the green beans. The pH value was lower in the husks than in the green beans (Table 2).

The combined analysis of variance showed that the genotype $\times$ treatment (G $\times$ T) interaction was significant for all variables, indicating that the husks, green beans, and roasted beans from the same coffee fruit cultivated in the Western Amazon exhibit distinct values for physicochemical characteristics.

Regarding the effect of genotype on physicochemical characteristics, the variables pH, protein, and ether extract were significant. A significant effect of treatments (fruit parts and processing) was observed for all variables, except for the ratio between total soluble solids and total titratable acidity (Table 2).

Some of the physicochemical variables underwent changes in concentration when green coffee beans were subjected to the roasting process, including total soluble solids, acidity, proteins, caffeine, lipids, and phenolic compounds. Others, such as total sugars and non-reducing sugars, decreased in concentration. Aqueous extract, ash, pH, reducing sugars, and the ratio between total soluble solids and total titratable acidity maintained their constant values (Table 2).

Table 3 presents the data from the combined analysis of variance relating pairs of characteristics: G $\times$ T1 (husks vs. green beans); G $\times$ T2 (green beans vs. roasted beans); and G $\times$ T3 (husks vs. roasted beans).

Unlike the combined analysis of variance with all three treatments, the paired ANOVA (Table 3) showed a significant effect for the variables including pH (Treatment 1 – husks x green Beans), CPC and EE (Treatment 2 - green beans x roasted Beans), and pH, CPC, and EE (Treatment 3 - husks x roasted Beans). The type of treatment also indicated detectable statistical differences, with the exception of the variables NRS and ratio (treatment 1), pH, caffeine, and ratio (treatment 2), and AE, TSS, and ratio (treatment 3). The G $\times$ T interaction was significant for all variables, indicating that the responses of fruit parts from the genotypes vary according to the type of treatment.

Table 2: Summary of the combined analysis of variance for the physicochemical characteristics evaluated on a dry matter basis in husks, green beans, and roasted beans of genotypes cultivated in the Western Amazon, Brazil.

SV	DF	AE	TSS	ASH	pH	TTA	CPC	
Genotypes	8	0.99ns	0.75ns	1.93ns	4.33**	1.90ns	3.68*	
Treatment (By-product/Product/Processing)	2	5.46*	26.42**	43.61**	119.92**	14.60**	178.33**	
GxT	16	81.55**	34.22**	272.45**	597.43**	161.12**	7.97**	
Residue	54							
Total	80							
Overall average		33.66	35.27	5.76	4.76	224.69	12.91	
Husks		36.84a	41.11a	7.58a	3.94b	288.82a	8.55c	
Green grains		31.08b	27.88c	4.64b	5.10a	167.53c	14.62b	
Roasted beans		33.07b	36.82b	5.06b	5.25a	217.73b	15.56a	
CVe		2.14	3.30	1.31	0.29	2.90	4.05	
SV	DF	Caffeine	EE	TPC	SS	RS	NRS	r
Genotypes	8	1.19ns	4.36**	0.17ns	0.84ns	0.97ns	0.55ns	2.04ns
Treatment (By-product/Product/Processing)	2	245.42**	551.21**	92.27**	21.23**	13.85**	4.87**	0.63ns
GxT	16	10.96**	6.97**	34.96**	239.73**	1064.35**	48.09**	65.34**
Residue	54							
Total	80							
Overall average		1.50	5.52	3.83	8.52	4.46	4.06	0.16
Husks		0.34c	1.21c	1.22c	16.55a	10.76a	5.8a	0.15a
Green grains		2.01b	5.23b	4.67b	6.08b	1.54b	4.53b	0.17a
Roasted beans		2.15a	10.13a	5.59a	3.39c	1.08b	2.30c	0.17a
CVe		6.70	6.77	5.50	5.72	5.22	13.16	4.81

*Significant at 5% probability. **Significant at 1% probability. ns, not significant. SV: source of variation; DF: degrees of freedom; CVe: experimental coefficient of variation. AE: aqueous extract (%); TSS: total soluble solids (°Brix); ASH: total ash (%); pH: hydrogen potential; TTA: total titratable acidity (mL NaOH 0.1 mol·L⁻¹·100 g⁻¹); CPC: crude protein content (%); caffeine (%); EE: ether extract (%); TPC: total phenolic compounds (g gallic acid eq./100 g of sample); SS: total soluble sugars (%); RS: total reducing sugars (%); NRS: non-reducing sugars (%); r: ratio between total soluble solids and total titratable acidity (% of total soluble solids / mL of NaOH). Means followed by the same letter in a column do not differ according to the Scott-Knott test at 5% probability.

The repeated measures analysis among the evaluated treatments, represented here by the correlation estimate (Table 4), showed a significant and correlated effect for some variables, namely total ash (ASH), pH, and total phenolic compounds (TPC) in treatment 1; pH and total soluble sugars (TSS) in treatment 2; and crude protein (CPC) and total reducing sugars (RS) in treatment 3.

Due to the predominance of complex G×T interactions for most variables in this dataset, individual analysis of variance must be considered. It was observed that all evaluated physicochemical characteristics, for husks, green beans, and roasted beans, were significant, indicating differences among the genotypes and reflecting the genetic variability present in the materials (Table 5). This is explained by the fact that in the joint analysis of variance, the data are evaluated considering simultaneously different parts of the fruit (husks and green beans) and the roasted coffee beans.

Thus, the variability introduced by the combined effect of the materials and their interactions increased the experimental error, which reduced the significant differences between genotypes for the physicochemical variables. In the individual analysis of variance, the data were analyzed separately for each type of material, reducing experimental heterogeneity and allowing the isolation of the genotypic effect within each specific matrix.

The experimental coefficient of variation (CVe), estimated based on the mean performance of the characteristics and the experimental error estimate, was evaluated to assess the precision of the experiments. Most characteristics showed low CVe values, below 10%, indicating good experimental precision, with values for husks ranging from 0.41% (pH) to 31.88% (EE); green beans ranging from 0.28% (pH) to 8.05% (NRS); and roasted beans ranging from 0.19% (pH) to 6.63% (caffeine) (Table 6).

Table 3: Summary of the paired combined analysis of variance for the physicochemical characteristics evaluated on a dry matter basis of genotypes cultivated in the Western Amazon, Brazil.

Treatment 1 – husks x green Beans								
SV	DF	AE	TSS	ASH	pH	TTA	CPC	
Genotypes	8	0.95ns	0.85ns	1.82ns	4.88*	1.93ns	1.77ns	
Treatment (Husks/Green Beans)	1	6.99*	37.34**	55.75**	157.76**	24.68**	131.72**	
GxT	8	86.17**	72.90**	286.30**	487.02**	196.32**	11.72**	
Residue	36							
Total	53							
SV	DF	Caffeine	EE	TPC	SS	RS	NRS	r
Genotypes	8	1.30ns	2.19ns	0.11ns	0.92ns	0.96ns	0.48ns	2.43ns
Treatment (Husks/Green Beans)	1	458.26**	280.47**	55.22**	16.06**	13.03**	0.37ns	0.92ns
GxT	8	16.30**	5.48**	46.44**	237.61**	1096.78**	54.84**	100.62**
Residue	36							
Total	53							
Treatment 2 - green beans x roasted Beans								
SV	DF	AE	TSS	TA	pH	TTA	CPC	
Genotypes	8	2.16ns	0.49ns	2.27ns	1.87ns	0.47ns	5.75*	
Treatment (Green Beans vs. Roasted Beans)	1	67.55**	59.86**	14.65**	1.43ns	7.16*	9.47*	
GxT	8	18.33**	11.33**	90.93**	1271.89**	138.53**	5.52**	
Residue	36							
Total	53							
SV	DF	Caffeine	EE	TPC	SS	RS	NRS	r
Genotypes	8	2.40ns	2.45**	1.16ns	2.47ns	0.33ns	1.84ns	0.53ns
Treatment (Green Beans vs. Roasted Beans)	1	3.43ns	249.17**	13.00**	106.90**	17.15**	51.44**	0.0002ns
GxT	8	5.83**	9.55**	14.30**	13.33**	39.28**	18.85**	48.15**
Residue	36							
Total	53							
Treatment 3 - husks x roasted Beans								
SV	DF	AE	TSS	TA	pH	TTA	CPC	
Genotypes	8	1.02ns	0.92ns	1.41ns	5.40*	1.93ns	2.84*	
Treatment (Husks x Roasted Beans)	1	3.09ns	4.25ns	35.13**	684.65**	8.73*	431.18**	
GxT	8	80.63**	35.96**	300.04**	179.37**	148.42**	5.61**	
Residue	36							
Total	53							
SV	DF	Caffeine	EE	TPC	SS	RS	NRS	r
Genotypes	8	0.39ns	5.39*	0.83ns	0.86ns	1.00ns	0.92ns	2.03ns
Treatment (Husks x Roasted Beans)	1	264.27**	1256.99**	296.16**	25.40**	14.68**	7.54*	0.91ns
GxT	8	15.34**	5.99**	95.30**	295.72**	1085.86**	45.75**	64.44**
Residue	36							
Total	53							

*Significant at 5% probability. **Significant at 1% probability. ns, not significant. SV: source of variation; DF: degrees of freedom. AE: aqueous extract (%); TSS: total soluble solids (°Brix); ASH: total ash (%); pH: hydrogen potential; TTA: total titratable acidity (mL NaOH 0.1 mol·L⁻¹·100 g⁻¹); CPC: crude protein content (%); caffeine (%); EE: ether extract (%); TPC: total phenolic compounds (g gallic acid eq./100 g of sample); SS: total soluble sugars (%); RS: total reducing sugars (%); NRS: non-reducing sugars (%); r: ratio between total soluble solids and total titratable acidity (% of total soluble solids / mL of NaOH).

Table 4: Correlation estimates among the bromatological characteristics of coffee fruit husks, green beans, and roasted beans.

Variables	T1	T2	T3
AE	-0.14 ^{NS}	0.38 ^{NS}	0.06 ^{NS}
TSS	-0.11 ^{NS}	0.34 ^{NS}	-0.07 ^{NS}
ASH	0.68*	0.38 ^{NS}	0.41 ^{NS}
pH	0.77*	0.52 ^{NS}	0.80**
TTA	0.43 ^{NS}	-0.35 ^{NS}	0.46 ^{NS}
CPC	0.28 ^{NS}	0.83**	0.50 ^{NS}
Caffeine	0.15 ^{NS}	0.41 ^{NS}	-0.47 ^{NS}
EE	0.40 ^{NS}	0.56 ^{NS}	0.56 ^{NS}
TPC	-0.79**	0.15 ^{NS}	-0.16 ^{NS}
SS	-0.16 ^{NS}	0.59 ^{NS}	-0.67*
RS	-0.28 ^{NS}	-0.82**	0.11 ^{NS}
NRS	-0.59 ^{NS}	0.44 ^{NS}	-0.16 ^{NS}
Ratio	0.48 ^{NS}	-0.30 ^{NS}	0.43 ^{NS}

T1: correlation between husk and green beans; T2: correlation between green and roasted beans; T3: correlation between husk and roasted beans. Values followed by * and ** indicate statistical significance at 5% and 1% probability, respectively; NS: not significant. AE: aqueous extract (%); TSS: total soluble solids (°Brix); ASH: total ash (%); pH: hydrogen potential; TTA: total titratable acidity (mL NaOH 0.1 mol·L⁻¹·100 g⁻¹); CPC: crude protein content (%); caffeine (%); EE: ether extract (%); TPC: total phenolic compounds (g gallic acid eq./100 g of sample); SS: total soluble sugars (%); RS: total reducing sugars (%); NRS: non-reducing sugars (%); r: ratio between total soluble solids and total titratable acidity (% of total soluble solids / mL of NaOH).

When these nine materials were subjected to mean clustering analysis at a 5% probability level, the formation of different groups was observed. For husks, green beans, and roasted beans, respectively, the groups for each variable were as follows: AE – 6, 4, and 3 groups; TSS – 6, 3, and 3 groups; ASH – 6, 7, and 5 groups; pH – 7, 8, and 8 groups; TTA – 5, 4, and 5 groups; CPC – 3, 4, and 2 groups; caffeine – 5, 3, and 2 groups; EE – 3, 2, and 3 groups; TPC – 4, 2, and 4 groups; SS – 6, 4, and 5 groups; RS – 6, 4, and 2 groups; NRS – 4, 4, and 5 groups; and ratio between total soluble solids and total titratable acidity – 8, 5, and 3 groups (Table 6).

Considering the combined analysis of the data, the aqueous extract content ranged from 23.52 to 44.47%, total soluble solids from 25.02 to 48.69%, total ash content from 3.97 to 9.46%, pH from 3.66 to 5.53, total titratable acidity from 134.23 to 443.17 mL of 0.1 mol·L⁻¹ NaOH per 100 g, crude protein from 7.31 to 16.92%, caffeine from 0.18 to 2.54%, ether extract from 0.22 to 12.10%, total phenolic compounds from 0.51 to 5.91 g of gallic acid equivalent/100 g of sample, and total sugars from 2.97 to 28.06%, reducing sugars from 0.90 to 19.87%, non-reducing sugars from 0.64

to 9.99%, and the ratio of total soluble solids to total titratable acidity from 0.07 to 0.27 (Table 6).

Due to the metabolic and physiological origins of the chemical constituents present in the coffee fruit, correlations between them tend to occur. For the dataset of this study, considering 11 variables, excluding NRS and ratio, twelve were significant for the husks, with seven positive correlations: AE vs SST (0.78*), AE vs SS (0.69*), AE vs RS (0.85**), SST vs RS (0.73*), pH vs SS (0.71*), pH vs RS (0.77*) and SS vs TRS (0.93**), and five negative correlations: TTA vs pH (-0.72*), TTA vs SS (-0.81**), TTA vs RS (-0.75*), pH vs CPC (-0.77*) and CPC vs RS (-0.77*).

For green beans, five correlations were significant, with two positive correlations between AE vs SST (0.68*) and SST vs RS (0.66*), and three negative correlations between ASH vs RS (-0.78*), TTA vs pH (-0.93**), and TTA vs SS (-0.66*). For roasted beans, four correlations were significant, with two positive correlations between ASH vs TTA (0.89**) and AE vs caffeine (0.83**), and two negative correlations between CPC vs TPC (-0.71*) and CPC vs RS (-0.66*).

In the scatter plot of the first two principal components, genotypes that are closer together are more similar across all evaluated physicochemical characteristics simultaneously. The projection of the variables onto this scatter, including the ideal references for maximum and minimum performance, shows that for the husks (Figure 1), there is a grouping in the upper and lower left quadrant for clones processed by wet fermentation. In the upper quadrant, the clones GJ25-f1, GJ25-f2, RE90-f1, and RE22-f1 are observed; these materials exhibit high protein and acidity levels, lower pH, low total and reducing sugar levels, and lower water solubility compared to the clones fermented via the natural method (GJ25, LB102, and GJ8). Ideal benchmarks for maximum and minimum performance were identified in the principal component scatter plot (Figure 1).

Unlike the husks, the genotypes processed by wet fermentation did not cluster in the same quadrant after the analysis of the green beans. Clones GJ25-f1 and GJ25-f2 are located in the lower-left quadrant; RE22-f1 is on the right; and RE90-f1 is in the upper-right quadrant (Figure 2).

As observed with the green beans, the genotypes processed via wet fermentation did not cluster in the same quadrant. Clone RE90-f1 is located in the lower-left quadrant; RE22-f1 and GJ25-f1 in the upper-left; and GJ25-f2 in the upper-right quadrant (Figure 3).

Comparing the PCA results with Pearson's correlation, a directly proportional relationship is confirmed between the water solubility of the husks and the total and reducing sugar content, as well as an inversely proportional relationship with the presence of organic acids and proteins (Figure 1). However, this relationship is not observed in green beans (Figure 2) and roasted beans (Figure 3).

Table 5: Summary of the individual analyses of variance for the physicochemical characteristics of husks, green beans, and roasted beans on a dry matter basis of genotypes cultivated in the Western Amazon, Brazil.

Husks								
SV	DF	AE	TSS	ASH	pH	TTA	CPC	
Genotypes	8	84.47**	112.63**	412.60**	606.24**	333.24**	10.71**	
Residue	18							
Total	26							
SV	DF	Caffeine	EE	TPC	SS	RS	NRS	r
Genotypes	8	48.32**	11.66**	142.01**	276.26**	1110.91**	43.73**	395.36**
Residue	18							
Total	26							
Green Beans								
SV	DF	AE	TSS	ASH	pH	TTA	CPC	
Genotypes	8	53.30**	19.29**	282.92**	2522.84**	170.39**	23.70**	
Residue	18							
Total	26							
SV	DF	Caffeine	EE	TPC	SS	RS	NRS	r
Genotypes	8	14.06**	5.57**	15.41**	20.19**	38.11**	24.45**	64.65**
Residue	18							
Total	26							
Roasted Beans								
SV	DF	AE	TSS	ASH	pH	TTA	CPC	
Genotypes	8	22.80**	4.60**	98.61**	491.76**	68.91**	10.92**	
Residue	18							
Total	26							
SV	DF	Caffeine	EE	TPC	SS	RS	NRS	r
Genotypes	8	8.18**	27.45**	16.64**	134.31**	6.88**	84.73**	22.65**
Residue	18							
Total	26							

**Significant at 1% probability. ns, not significant. SV: source of variation; DF: degrees of freedom. AE: aqueous extract (%); TSS: total soluble solids (°Brix); ASH: total ash (%); pH: hydrogen potential; TTA: total titratable acidity (mL NaOH 0.1 mol·L⁻¹·100 g⁻¹); CPC: crude protein content (%); caffeine (%); EE: ether extract (%); TPC: total phenolic compounds (g gallic acid eq./100 g of sample); SS: total soluble sugars (%); RS: total reducing sugars (%); NRS: non-reducing sugars (%); r: ratio between total soluble solids and total titratable acidity (% of total soluble solids / mL of NaOH).

4 DISCUSSION

Coffee fruits have three morphological structures, and after processing, the beans are separated, producing the “coffee husks.” Evaluating the physicochemical concentrations in the fruits, husks, and beans is important, particularly to support studies on the industrial potential of coffee by-products.

The joint analysis of variance considering the three treatments (husks, green beans and roasted beans) (Table 2) and the interaction between the treatments husks and green beans (1); green beans and roasted beans (2); husks and roasted beans (3) (Table 3), revealed significant differences in the physicochemical constituents for most of the variables studied, indicating that the constitution of the husks of the

same genotype is different from its green beans, and when it is subjected to increased temperature, by the roasting process, the concentration of chemical constituents is also altered.

For treatments 1, 2, and 3, respectively, 15%, 23%, and 30% of the variables could be classified as having simple interactions, with correlation values greater than 0.50 (Table 4). This indicates that most of the chemical constituents of the fruit parts (peels and green grains), processing (green and roasted grains), and peels and roasted grains have different concentrations. Some observed changes were highly complex and negatively correlated, that is, the correlation is inversely proportional, as in the case of TPC in treatment 1 and RS in treatment 2.

In a study conducted with twelve clones of *C. canephora* of the Conilon variety cultivated in the state of Espírito Santo,

Brazil, it was observed that the roasting process positively affected the presence of total organic acids and negatively affected the values of pH, soluble solids, phenolic compounds, total sugars, reducing and non-reducing sugars, and caffeine, when compared to green beans (Agnoletti et al., 2019).

However, in our study, roasting increased the levels of soluble solids, caffeine, and phenolic compounds, indicating that environmental factors and genetic variety tend to influence the physicochemical composition of coffee beans and, consequently, the post-processing chemical constituents.

Table 6: Grouping of means for the physicochemical properties on a dry matter basis of husks, green beans, and roasted beans of *C. canephora* genotypes cultivated in the Western Amazon.

Husks													
Genotypes	AE	TSS	ASH	pH	TTA	CPC	Caffeine	EE	TPC	SS	RS	NRS	ratio
LB10	38.98c	38.18e	7.80c	4.17b	279.69c	7.50c	0.18e	1.57b	1.18b	15.54d	12.76c	2.77d	0.13e
GJ8	39.76c	46.81b	6.83e	4.26a	170.21e	7.31c	0.28d	0.22c	1.17b	28.06a	18.87b	9.19a	0.27a
LB102	44.47a	48.69a	7.53d	4.09c	219.28d	8.37b	0.24e	0.58c	1.17b	24.01b	18.86b	5.15c	0.22b
GJ25	41.73b	45.71c	9.17b	4.15b	269.78c	7.62c	0.53b	1.64b	1.16b	23.55b	19.87a	3.67d	0.16c
GJ25-f2	32.25e	34.87f	7.85c	3.69f	443.17a	8.74b	0.31d	1.30b	0.86c	8.82e	3.04e	5.77c	0.078h
GJ25-f1	23.52f	34.26f	9.46a	3.92d	281.30c	9.40a	0.37c	0.94c	0.51d	8.67e	1.31f	7.36b	0.12f
RE90-f1	34.09d	43.06d	6.95e	3.70f	384.58b	10.49a	0.61a	0.97c	1.29b	7.90e	1.45f	6.44b	0.11g
RE22-f1	34.84d	34.02f	4.79f	3.66g	273.21c	9.85a	0.36c	2.84a	0.68d	18.61c	8.62d	9.99a	0.12f
VR1	41.97b	44.40c	7.88c	3.83e	278.16c	7.68c	0.18e	0.80c	2.94a	13.76d	12.09c	1.85d	0.15d
CVe	3.29	2.30	1.54	0.41	2.66	7.07	10.76	31.88	8.31	4.71	3.66	15.85	3.38
Green Beans													
Genotypes	AE	TSS	ASH	pH	TTA	CPC	Caffeine	EE	TPC	SS	RS	NRS	ratio
LB10	31.60a	26.80b	4.74c	5.53a	150.71c	14.06c	2.26a	4.70b	4.83a	6.81b	1.32d	5.49a	0.17c
GJ8	30.37d	27.30b	4.88b	5.41d	142.66c	16.59a	2.14b	5.34a	4.35a	5.44d	1.21d	4.22c	0.19b
LB102	30.47d	27.11b	4.44f	5.45c	143.22c	15.02b	1.91c	4.66b	5.45a	5.70d	1.72b	3.97c	0.18b
GJ25	30.81c	26.89b	4.54e	5.49b	134.23d	13.37d	2.04b	5.19a	5.16a	7.18b	1.51c	5.66a	0.20b
GJ25-f2	30.74c	25.02c	4.76c	4.62g	216.82a	14.34c	1.79c	5.67a	5.01a	5.47d	1.44c	4.02c	0.11e
GJ25-f1	30.81c	27.49b	4.99a	4.55h	222.98a	13.14d	1.77c	4.54b	5.34a	5.18d	1.64b	3.54d	0.12e
RE90-f1	31.84a	31.95a	4.66d	5.12f	144.58c	15.33b	2.11b	5.39a	3.72b	7.67a	1.65b	6.01a	0.22a
RE22-f1	31.76a	31.59a	3.97g	4.57h	203.55b	16.92a	2.26a	5.96a	4.98a	5.01d	2.06a	2.95d	0.15d
VR1	31.30b	26.73b	4.80c	5.19e	149.01c	12.76d	1.77c	5.64a	3.19b	6.24c	1.33d	4.90b	0.17c
CVe	0.42	3.28	0.67	0.28	2.83	3.59	4.62	7.03	7.25	6.00	4.67	8.05	4.37
Roasted Beans													
Genotypes	AE	TSS	ASH	pH	TTA	CPC	Caffeine	EE	TPC	SS	RS	NRS	ratio
LB10	33.86a	39.27a	5.12c	5.43b	220.99c	15.02b	2.50a	9.85b	5.73b	3.73b	1.22a	2.51b	0.17b
GJ8	32.82b	38.59a	4.95d	5.46a	181.05e	16.12a	2.01b	9.67b	5.49c	2.97e	1.10a	1.87e	0.21a
LB102	32.09c	37.68a	4.96d	5.29c	191.15d	15.59b	2.03b	8.81c	5.31d	3.13d	1.05a	2.08d	0.19a
GJ25	32.90b	32.90c	5.29b	5.22e	242.27b	15.25b	2.15b	10.23b	5.67b	3.04e	1.11a	1.93e	0.13c
GJ25-f2	33.59a	37.70a	4.89d	5.06h	221.40c	14.97b	2.17b	11.67a	5.91a	3.62c	1.08a	2.53b	0.17b
GJ25-f1	32.51b	36.65a	4.87d	5.24d	198.81d	14.63b	2.05b	10.21b	5.82a	3.04e	1.12a	1.91e	0.18b
RE90-f1	32.19c	35.67b	5.24b	5.12g	252.61b	16.87a	1.78b	8.93c	5.49c	4.13a	1.06a	3.07a	0.14c
RE22-f1	33.88a	35.71b	4.62e	5.25d	180.61e	16.64a	2.54a	12.10a	5.32d	3.22d	0.90b	2.31c	0.19a
VR1	33.78a	37.23a	5.62a	5.16f	270.69a	14.91b	2.16b	9.67b	5.56c	3.59c	1.12a	2.46b	0.13c
CVe	0.79	4.15	1.00	0.19	3.11	2.72	6.63	3.63	1.59	1.76	5.17	3.21	6.05

1. Wet semi-carbonic fermentation for 20 days (F2) with addition of components. 2. Wet semi-carbonic fermentation for 20 days (F1). CVe: experimental coefficient of variation (%). AE: aqueous extract (%); TSS: total soluble solids (°Brix); ASH: total ash (%); pH: hydrogen potential; TTA: total titratable acidity (mL NaOH 0.1 mol·L⁻¹·100 g⁻¹); CPC: content crude protein (%); caffeine (%); EE: ether extract (%); TPC: total phenolic compounds (g gallic acid eq./100 g of sample); SS: total soluble sugars (%); RS: total reducing sugars (%); NRS: non-reducing sugars (%); ratio between total soluble solids and total titratable acidity. Averages followed by the same letter in the column do not differ, according to the Scott-Knott test, at 5% probability.

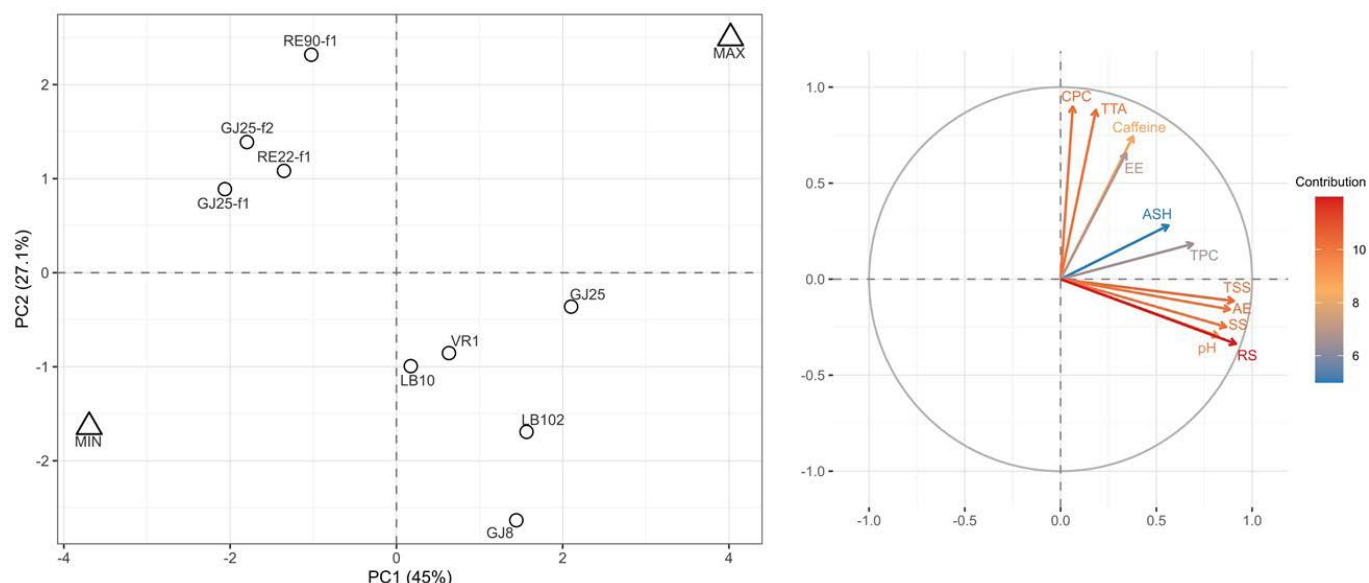


Figure 1: Scatter plot of the first two principal components for the physicochemical properties on a dry matter basis of husks from *C. canephora* genotypes cultivated in the Western Amazon.

1. Wet semi-carbonic fermentation for 20 days (F2) with addition of components. 2. Wet semi-carbonic fermentation for 20 days (F1). AE: aqueous extract (%). TSS: total soluble solids ($^{\circ}$ Brix). ASH: total ash (%). pH: hydrogen potential. TTA: total titratable acidity ($\text{mL NaOH } 0.1 \text{ mol} \cdot \text{L}^{-1} \cdot 100 \text{ g}^{-1}$). CPC: content crude protein (%). caffeine (%). EE: ether extract (%). TPC: total phenolic compounds ($\text{g gallic acid eq./100 g}$ of sample). SS: total soluble sugars (%). RS: total reducing sugars (%).

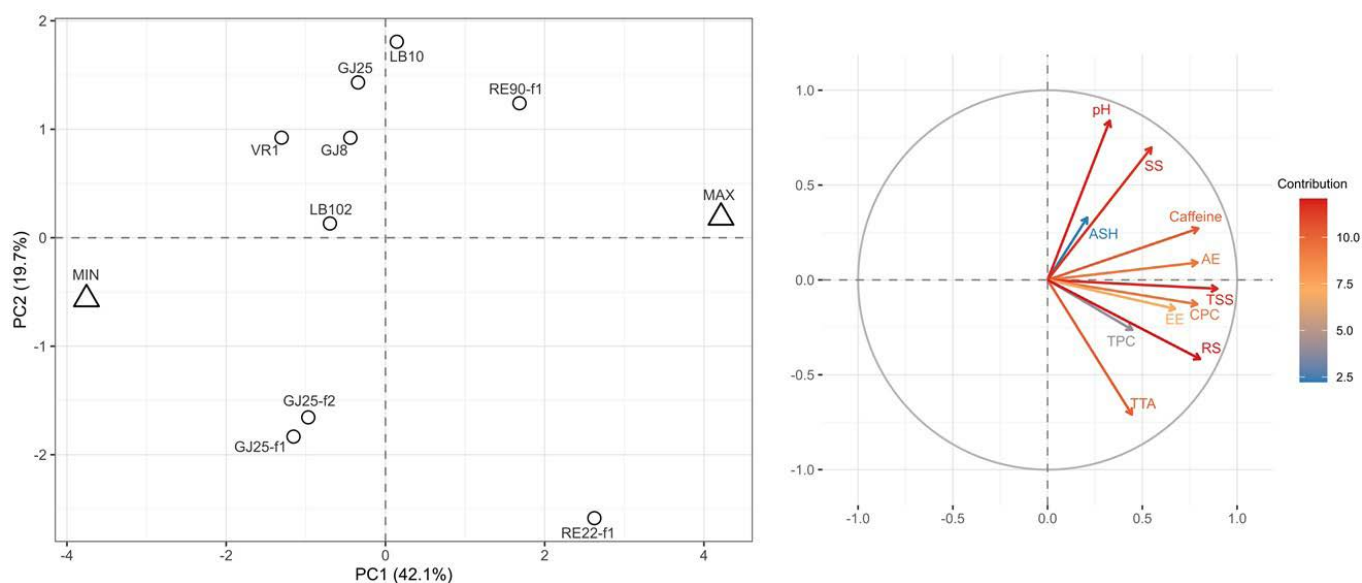


Figure 2: Scatter plot of the first two principal components for the physicochemical properties on a dry matter basis of green beans from *C. canephora* genotypes cultivated in the Western Amazon.

1. Wet semi-carbonic fermentation for 20 days (F2) with addition of components. 2. Wet semi-carbonic fermentation for 20 days (F1). AE: aqueous extract (%). TSS: total soluble solids ($^{\circ}$ Brix). ASH: total ash (%). pH: hydrogen potential. TTA: total titratable acidity ($\text{mL NaOH } 0.1 \text{ mol} \cdot \text{L}^{-1} \cdot 100 \text{ g}^{-1}$). CPC: content crude protein (%). caffeine (%). EE: ether extract (%). TPC: total phenolic compounds ($\text{g gallic acid eq./100 g}$ of sample). SS: total soluble sugars (%). RS: total reducing sugars (%).

The high genetic variability of clones observed in this study, as demonstrated by individual ANOVA (Table 5), is due to two main factors. First, the self-incompatibility (SI) of *C. canephora*, a physiological condition present in its reproductive

system, naturally favors cross-pollination, resulting in allogamous populations that transmit their alleles to subsequent generations, thereby increasing genetic diversity (Ferrão et al., 2021; Souza et al., 2021). This explains the significant differences among

naturally fermented materials. Second, the type of processing also contributes, as the dry or wet fermentation method tends to alter the chemical composition of the coffee beans (Vaz et al., 2023), consequently affecting the characteristics of the fruit by-products and the final product after roasting.

This observed variability is supported by similar studies reported in the literature. A study evaluated 16 genotypes of *C. canephora* cultivated in the Brazilian Amazon and identified variability in nutrient accumulation between husks and grains (Schmidt et al., 2023). In green beans, a study involving 86 coffee genotypes cultivated in Rondônia also reported high variability in physicochemical characteristics (Junior et al., 2024). Similarly, variability has been observed in the chemical composition of roasted beans of *C. canephora* from the Western Amazon (Rocha et al., 2023; Tadeu et al., 2024; Costa et al., 2024), which in turn tends to influence beverage quality (Reinicke, Espindula & Rocha, 2023).

Table 6 allowed for the individual characterization of the clones and variables studied. It was observed that the coffee fruits (husks + green beans) of the GJ25-f1 genotype presented the highest total ash content, also called total minerals. The value found in the husks was 64.9% higher than that of the beans of the same genotype, showing that the husks accumulate more nutrients than the beans. Other authors, conducting studies with coffee from the Brazilian Amazon, observed that the husks accumulate more K, Ca, S, Cu, Fe, and B compared to the beans (Schmidt et al., 2023).

The water solubility of coffee fruits was interpreted considering the values of aqueous extract and total soluble

solids. These two variables studied showed a high positive and significant correlation, both in the husks and in the green beans. Among the genotypes, the highest solubility in coffee fruits was found in clone LB102. This characteristic for this clone is due to the higher concentration of soluble chemical compounds in its husks. When observing the water solubility in green beans, genotypes LB10, RE90-f1, and RE22-f1 showed higher values (Table 6).

Coffee husks have a higher concentration of chemical constituents that favor their solubility in water when compared to green and roasted beans (Table 2). This is a factor of great importance for the production of new products. An example of this is the infusion prepared from these husks, commonly known as cascara. This beverage is being studied for its market potential. In a sensory analysis carried out with coffee husks of the Arabica and Canephora species, it was observed that the Arabica coffee samples stand out for their high aftertaste and acidity, and the Canephora coffee samples for their bitterness; however, none of the samples obtained an acceptability index higher than 70% (Poláková et al., 2023).

The highest concentration of organic acids was found in the fruits of the genotype GJ25-f2, with acidity in the husks being 100% higher compared to the beans of the same genotype (Table 6). It is evident that the wet fermentation process promotes the accumulation of organic acids in the husks. This clone exhibited higher acidity in both the husks and the beans. The genotype with the lowest fruit pH was RE22-f1, showing elevated values in both husks and beans.

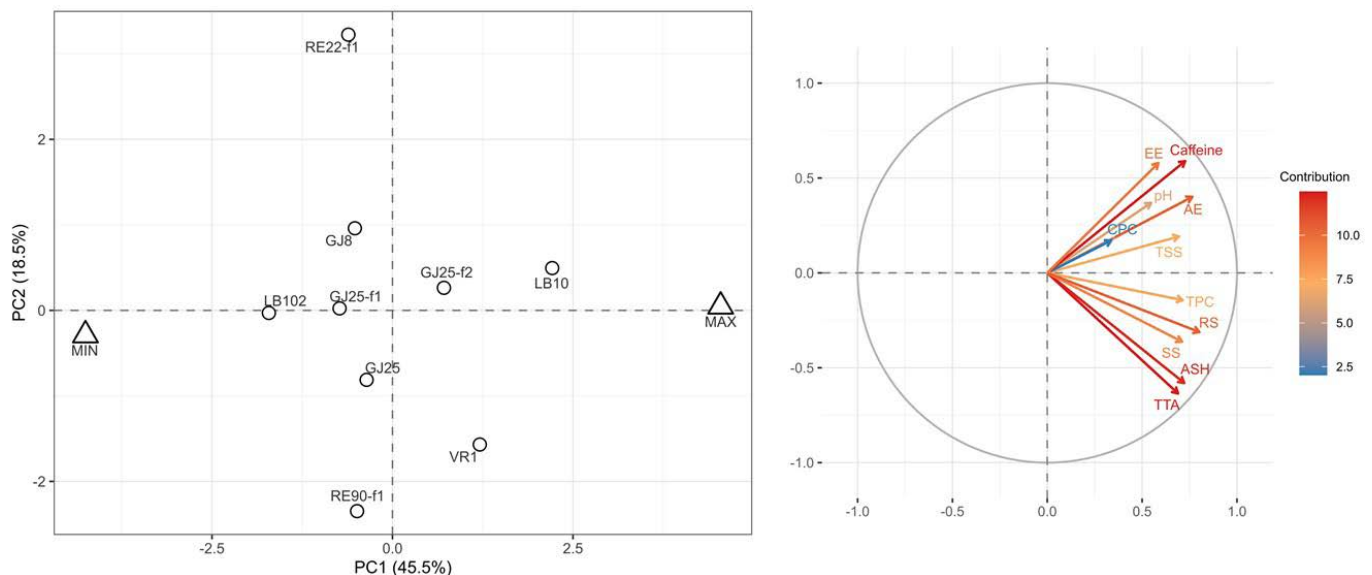


Figure 3: Scatter plot of the first two principal components for the physicochemical properties on a dry matter basis of roasted beans from *C. canephora* genotypes cultivated in the Western Amazon.

1. Wet semi-carbonic fermentation for 20 days (F2) with addition of components. 2. Wet semi-carbonic fermentation for 20 days (F1). AE: aqueous extract (%). TSS: total soluble solids (°Brix). ASH: total ash (%). pH: hydrogen potential. TTA: total titratable acidity (mL NaOH 0.1 mol·L⁻¹·100 g⁻¹). CPC: content crude protein (%). caffeine (%). EE: ether extract (%). TPC: total phenolic compounds (g gallic acid eq./100 g of sample). SS: total soluble sugars (%). RS: total reducing sugars (%).

The ratio between total soluble solids and total titratable acidity in coffee fruits was higher in clone GJ8. This clone also presented low acidity and a higher content of water-soluble compounds, mainly due to the concentration of sugars in its husks. For this genotype, this ratio was higher only in the husks; in green beans, clone RE90-f1 presented a higher value (Table 6). The ratio between the soluble fraction, specifically total soluble solids and total titratable acidity, is associated with the perception of sweetness. When this ratio is unbalanced, it can create the sensation that the product is “diluted” or “too acidic” (Junior et al., 2024), which can effectively affect quality, such as the production of a sweet beverage with low acidity, as is the case with the husks of clone GJ8, or with high acidity and low sweetness, as with clone GJ25-f2.

The highest protein content in the fruits was found in the genotype RE22-f1, with values in the husks being 41.78% lower than those in the beans of the same genotype. The prominence of this clone in terms of total crude protein (CP) content is due to its high protein levels in the beans, which, together with the GJ8 clone, were 16% higher than the other materials. In the husks, the clones RE90-f1 and GJ25-f1 showed values above 9% compared to the other genotypes (Table 6). For the beans, protein content is closely related to the flavor and aroma of coffee, as during roasting, proteins contribute to the formation of various chemical compounds through specific reactions, notably the Maillard reaction, in which amino groups react with reducing sugars to produce numerous precursors highly desirable for high-quality coffee (Buffo & Cardelli-Freire, 2004).

The fruits of the genotype RE22-f1 also showed the highest lipid concentrations, with values in the husks being 52.84% lower than those in the beans of the same genotype. This clone exhibited higher lipid content in both husks and beans (Table 6). Lipid content influences the sensory properties of coffee, contributing to the formation of compounds responsible for flavor, aroma, texture, and color (Wang et al., 2025).

The physicochemical variability observed among the evaluated genotypes is consistent with previous sensory evaluations conducted using the same germplasm cultivated in the Western Amazon. In that study, beverage quality ranged from 74.77 to 84.21 points, with 25 of the 50 evaluated genotypes classified as Fine Robustas (>80 points). Furthermore, principal component analysis demonstrated that the highest-quality beverages were positively associated with sensory descriptors such as Nutty/Cocoa, Sweet, Floral, and Fruity, highlighting the strong influence of genotype on beverage quality (Junior et al., 2026).

Considering that compounds such as sugars, proteins, lipids, organic acids, and phenolic compounds act as important precursors of aroma and flavor during roasting, the marked physicochemical variability identified in the present study provides a biochemical explanation for the sensory differences previously reported among these genotypes.

Regarding the phenolic compound content in the fruits, the LB102 clone stands out, showing higher values compared to the other genotypes in this study. This elevated concentration is due to the higher levels of this compound in its beans, while in the husks, the VR1 clone is prominent, with a value of 2.94 g gallic acid equivalent per 100 g (Table 6). Phenolic compounds, especially chlorogenic acids and their degradation products (caffeic, ferulic, and p-coumaric acids), have health-promoting properties, not only due to their potent antioxidant activity but also as hepatoprotective, hypoglycemic, and antiviral agents (Farah & Donangelo, 2006). The presence of phenolic compounds supports the development of functional foods.

In a study with *C. canephora* bark from Panama, a phenolic compound value of 2.12 g gallic acid equivalent per 100 g was found, with an antioxidant activity of 0.29 CE/100 g ps (Machado et al., 2023). According to the literature, the main phenolic compounds found in aqueous extracts of coffee husks are chlorogenic acid, protocatechuic acid, kaempferol-3-O-galactoside, and gallic acid (Rebollo-Hernanz et al., 2021).

The fruit belonging to the GJ8 genotype showed the highest content of soluble and non-reducing sugars, accumulating approximately 33% of total sugars, of which 28.06% are present in the husks. In the beans, the RE90-f1 clone presented the highest value for this variable, totaling 6.67%. Of the sugars accumulated in the fruits of the GJ8 clone, approximately 20% are reducing sugars and 13.4% are non-reducing sugars (Table 6). Among 68 coffee genotypes cultivated in Western Amazonia, this clone presented the highest content of reducing sugars (Junior et al., 2024). Sugars are responsible for the sweet taste; in the case of green beans, the sugars present are precursors of flavor and aroma, reacting in various ways (fragmentation, caramelization, or interaction with amino acids) after the roasting process (Liu et al., 2022).

The GJ25 genotype showed the highest accumulation of reducing sugars (RS), with an overall concentration of approximately 20%, of which 18.87% is present in the husks. In the beans, the RE22-f1 clone exhibited the highest value (Table 6). Regarding non-reducing sugars (NRS), higher values were found in the husks (GJ8 and RE22-f1 clones), while in the beans, the highest values were observed in the LB10, GJ25, and RE90-f1 clones. High concentrations of these compounds in the fruits and their respective products and by-products contribute to a sweeter food. In the case of beans, elevated TRS content tends to promote the formation of chemical compounds favorable to beverage quality during roasting.

Green coffee beans, in addition to being roasted for beverage production, are also used for caffeine extraction. Considering the fruit of the genotypes in this study, the clone RE90-f1 showed the best potential for caffeine extraction. In the husks, this clone presented the highest value (0.61%). In the green beans, clones LB10 and RE22-f1 showed higher values (Table 6). Even though the main raw material for

extracting this constituent is green coffee beans, the husks of *C. canephora* cultivated in Western Amazonia emerge as an alternative for recovering this bioactive compound.

Some of the green beans of the genotypes in this study have been characterized by other authors (Junior et al., 2024) over two harvests in Porto Velho, Rondônia, including GJ25, GJ8, and LB10. Comparing the data from that study with the results of the present work, it is observed that some variables remained consistent regardless of the environment, such as aqueous extract, ash, and protein content. In a study with 14 *C. canephora* genotypes cultivated in two distinct environments also in Western Amazonia, among twelve physicochemical variables evaluated, only two (aqueous extract and proteins) resulted in a simple genotype x environment interaction, with small alterations in the performance of the genotypic classification; other parameters, such as soluble solids, pH, acidity, lipids, phenolic compounds, and sugars, resulted in a complex type of interaction (Lopes Junior et al., 2025).

It is known that fruit filling is affected by both genetic and environmental factors, resulting in changes in the chemical composition of the beans after the drying process (Lourenço et al., 2022; Lopes Junior et al., 2026). Factors such as soil, altitude, management practices, harvesting, and post-harvest procedures, such as drying, peeling, and storage, are the main factors that influence the expression of the physical characteristics of the fruit and coffee beans (Höfig et al., 2024).

Regarding the husks versus roasted beans, physicochemical characteristics such as mineral content, aqueous extract, soluble solids, acidity, and sugars are higher in the husks (Table 2). This favors the production of a beverage with higher mineral content, a fuller body, and greater sweetness, while its lower pH results in a beverage with higher acidity compared to coffee prepared from roasted beans.

The roasting process tends to significantly influence the levels of certain chemical compounds present in coffee beans. For example, total chlorogenic acids (CQAs) undergo substantial degradation with increasing roast intensity, free amino acid concentrations decrease sharply, while caffeine remains stable (Ali et al., 2025). In another study, it was observed that roasting at different color classes (light, medium, and dark) generally maintains total protein content, due to the method used, which quantifies total nitrogen rather than individual degradable components. Other variables, however, were significantly affected: aqueous extract, total ash, and pH increased during roasting, whereas titratable acidity (TTA) decreased (Lopes Junior et al., 2022).

The repeatability of the genotypes in maintaining their characteristics after the roasting process tends to be low, indicating that the individual traits of each genotype are generally not correlated post-roasting. There are some exceptions, such as protein content ($r = 0.83$), calculated from total nitrogen, total soluble sugars ($r = 0.60$), and total reducing

sugars ($r = -0.83$). For proteins, the clones GJ8, RE90-f1, and RE22-f1 showed higher levels in both green and roasted beans. The RE90-f1 clone, which exhibited the highest TSS in green beans, also maintained high levels after roasting, although with a decrease of approximately 46.15%. Conversely, reducing sugars showed an inverse pattern: the RE22-f1 clone, which had the highest value in green beans, experienced a loss of approximately 56.31% after roasting (Table 6).

Coffee fruit products and by-products tend to have higher sugar concentrations when acidity is low and pH is higher, thereby favoring greater total soluble sugars (TSS) and resulting in a higher TSS/TTA ratio. This indicates elevated sugar levels in both husks and beans (Table 6). After roasting, with increased acidity and reduced sugar content, a positive correlation is observed between TTA and TSS, which contrasts with the relationship seen in husks and green beans.

The correlation among the bromatological characteristics of the husks indicates that water solubility positively influences both the TSS/TTA ratio and sugar content, while negatively affecting lipid levels. In addition, high acidity proportionally reduces the ratio and sugar content, resulting in a more acidic and less sweet beverage. Elevated ratio and pH values are positively influenced by sugar content.

In the husks (Figure 1), wet fermentation resulted in lower concentrations of aqueous extract (EA), total soluble solids (TSS), pH, total phenolic compounds (TPC), total sugars (TSS), and reducing sugars (RS), while increasing titra table acidity (TTA). This indicates that natural fermentation of the fruits enhances the water solubility of the husks and reduces organic acidity, while positively influencing the concentration of phenolic compounds and soluble sugars.

Similarly, in the green beans (Figure 2), wet fermentation also resulted in lower water solubility of compounds and higher acidity. Other variables were however, not affected, indicating that the influence of fermentation differs between the fruit components. In roasted beans (Figure 3), unlike husks and green beans, the type of processing did not directly affect the concentrations of the measured variables.

It was also observed from the scatter plots (Figures 1, 2, and 3) that some genotypes clustered within the same quadrant regardless of treatment, indicating similarity in their physicochemical characteristics. This was the case for genotypes GJ25-f1 and GJ25-f2, which grouped in the same quadrant regardless of processing, suggesting that wet fermentation and wet fermentation with added components do not significantly influence the physicochemical traits of this material. Additionally, genotypes LB10, GJ25, and VR1 also remained clustered in the same quadrant, indicating similarity in their characteristics.

Analyzing the data on the chemical composition of the husks reported in the literature, the values are very close to those found in this study, such as the concentration of

extractive phenolic compounds, which in a study with two samples of Arabica coffee husks, ranged from 983 to 196 mg GAE/100g. In the same study, the caffeine content in the husks ranged from 696 to 418 mg/100g (Cangussu et al., 2021).

Regarding the physicochemical properties, the literature shows values for Arabica coffee husks that differ from those in this study, such as protein concentration (10.03%), lipids (5.01%), and ash (5.20%) (Cangussu et al., 2021). When our data are compared with *C. canephora* husks, the values become more similar, such as ash content (7.86%), protein (8.77%), and lipids (1.06%) (Machado et al., 2023).

For robusta coffee husks, it has been reported that the beverage exhibits a low pH usually below 5.1, high °Brix values above 0.6, and a total phenolic content of 0.64 mg/100 mL (Murlida et al., 2021). Other studies reported a phenolic content of 20 mg/100 mL, indicating that the chemical composition of the husks directly influences the beverage characteristics (Kristanti et al., 2022). For husks derived from Arabica coffee, values of pH 4.16, °Brix 4, acidity 1.24%, caffeine 220 mg/L, and tannins 45.7 mg/L were observed, with high overall acceptability (Thagunna et al., 2024).

Thus, we conclude that a beverage produced from the different components of coffee fruits tends to present distinct flavor and aroma characteristics. A beverage made with the husk will have a higher mineral content and greater water-soluble constituents, resulting in a more full-bodied beverage. Furthermore, it will exhibit higher acidity, lower astringency, reduced bitterness, and greater sweetness compared to coffee made with roasted beans

4 CONCLUSIONS

Coffee husks, green beans, and roasted beans differed in physicochemical composition. Husks contained higher levels of ash, aqueous extract, soluble solids, acidity, and sugars, whereas beans were richer in proteins, lipids, phenolic compounds, and caffeine. High genetic variability was observed among the clones, with GJ8 and VR1 showing the most favorable profiles for husks and beans, respectively. These results highlight the potential of coffee husks from Western Amazonia as raw material for functional foods, beverages, and biobased products.

5 AUTHOR CONTRIBUTION

Conceptual idea: Lopes Junior, H. and Alves, E.A.; Methodology design: Lopes Junior, H. and Rocha, R.B.; Data collection: Lopes Junior, H., Mountim Gama, M.B., Carvalho, C.G., and Marmentini, R.P.; Data analysis and interpretation: Rocha, R.B., Lopes Junior, H., and Alves, E.A.; Writing and editing: Lopes Junior, H., Alves, E.A., Rocha, R.B., and Marmentini, R.P.

6 DATA AVAILABILITY STATEMENT

Data available upon request to authors.

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