COFFEE SCIENCE
ISSN: 1984-3909

eISSN: 1984-3909
Coffee Science, e182136, 2023

Doi: https://doi.org/10.25186/.v18i.2136

Physicochemical comparison of aqueous extracts from Peruvian coffee husks (*Caturra*, *Catimor* and *Geisha*) *varieties*

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Received in May 25, 2023 and approved in August 23, 2023

ABSTRACT

The study aimed to determine and compare the physico-chemical characteristics of coffee husks aqueous extracts corresponding to three Peruvian varieties of Arabica coffee: *Caturra*, *Catimor* and *Geisha*. Each sample was dehydrated at 45 °C for 10 hours, ground, sieved and dissolved in 2.5% w/w water. Aqueous extracts were obtained and analyzed for pH, total solids (TS), whiteness index (WI), reducing sugars (RS), total phenols (TP) and reducing power (RP) for the antioxidant activity The influence of coffee varieties, particle sizes (0.097, 0.033, and 0.0139in) and extraction temperaturas (60 and 80 ºC) on the physicochemical characteristics of their corresponding extracts was determined by factorial analysis. Correlation between TP and RP was significant while RS and TP showed an inverse relationship. Cluster values indicate similarity between *Geisha* extracts at 60 and 80 °C and the corresponding *Catimor* extracts at 80 °C. Differences were observed between the *Catimor* extracts at 60 °C and *Caturra* at 60 and 80 °C. Based on these results, the extracts exhibit distinctive intrinsic characteristics. The *Caturra* extracts at 60 °C showed the highest antioxidant capacity, while the Catimor extracts at 60 °C or 80 °C exhibited the highest WI (water holding capacity) and pH values. Additionally, the Geisha extracts displayed the highest amount of RS (reducing sugars). These findings are of utmost significance for the development of novel products using Peruvian coffee husks, encompassing beverages, concentrated juices, and the enhancement of coffee quality offered in coffee shops. These developments can be guided by the physicochemical characteristics obtained in this study.

Key words: Antioxidant capacity; Coffee husks; Reducing sugars; Total phenols; Particle sizes.

1 INTRODUCTION

Coffee is one of the most widely consumed beverages in the world, an important cash crop for many countries and the second most traded commodity worldwide (Klingel et al., 2020). World coffee production is based on two major species, *Arabic*a coffee and its varieties (*Typica, Bourbon*, *Caturra*, *Geisha*, Blue Mountain, among others) and the *Robusta* coffee species (*Coffea canephora*) which includes varieties such as *Comilon*, *Kouilloi, Niaoulli* and *Uganda* (Minagri, 2020a, 2020b). Coffee bean extract is appreciated for its bitter taste characteristics as well as for its sugars and polyols content that provide coffee body to the beverage in addition to the compounds generated during roasting (Setyaningsih et al., 2022).

The main coffee producing countries are Brazil, Vietnam, Colombia, Indonesia and Ethiopia, which together account for more than 60% of total world coffee production. Starting in 2021, Peru ranked as the 9th largest coffee producer in the world with an estimated annual production around 267,000 metric tons of coffee beans.

According to the criteria and aptitudes of coffee varieties (Midagri, 2022b) the cup quality rates of the Geisha and Caturra genotypes are considered excellent and Catimor as good. Díaz and Willems (2017) report that 88% of national production is destined for trade as blends for either certified or gourmet specialty coffee. The social impact of coffee production in Peru is very important, 20% of exported certified coffee is managed by producer organizations representing 232 thousand families in Junín, San Martín, Cajamarca, Cuzco, Amazonas, Huánuco and Pasco.

Coffee cherry measures between 1 and 6 mm, its color varies from light to dark brown with reddish hues, its main components are digestible carbohydrates, dietary fiber and water (EFSA; NOVEL FOODS AND FOOD ALLERGENS, 2022). Coffee cherry processing generates various residues, such as parchment (endocarp) and also the husk (pericarp) comprising the skin (exocarp), pulp (mesocarp) mucilage (pectin), parchment (endocarp) and tegument (silver skin) (Pérez; Saldaña, 2017).

It is estimated that the amount of coffee waste generated is significant, most of the coffee husks is used as a source of energy (pyrolysis), composting, biogas production, and a lesser extent in animal feed without represent a major value addition (Rijo et al., 2021) but generating environmental and health concerns (Duangjai et al., 2016). In this regard, the effort of many coffee producers is aimed at reducing the amount of waste generated during coffee processing and producing new ways to recycle it; however, the cost of drying, storage and

transportation does not favor economic margins. Murthy et al. (2012) suggest the use of these wastes as new additives or supplements with higher nutritional value. Nowadays coffee husks are already marketed in the form of infusions as a healthier alternative to traditional coffee due to their lower caffeine and acidity content.

Several studies refer the high fiber content and the bioactive compounds contained in the pulp and husks, approximately 18 odorant compounds in the pulp as well as sugars (7), organic acids (6), methylxanthines (3) and polyphenols (35) involved in its fruity and black tea flavor (Pua et al., 2021, Amorocho-Cruz et al., 2021). A beverage from aqueous extracts of roasted coffee parchment rich in caffeine, chlorogenic acid, melanoidins and total dietary fiber was elaborated by Tores de la Cruz et al. (2019). Also Iriondo-DeHond et al. (2020) demonstrated that coffee husk is a potential source of phenolic compounds for the preparation of antioxidant beverages. (EFSA; NOVEL FOODS AND FOOD ALLERGENS, 2022) considers coffee husk as a traditional food marketable in Europe for human consumption, highlighting that its nutritional contribution favors the reduction of environmental impact.

Since products formulation is based on the characteristics of the raw materials, this study aims to report on the physicochemical characterization and comparison of extracts prepared from Peruvian coffee husks, specifically those derived from the Caturra, Catimor, and Geisha varieties.

2 MATERIAL AND METHODS

2.1 Raw material

Peruvian coffee husks of the *Caturra* (A), *Catimor* (R) and *Geisha* (G) varieties from the Villarrica area, department of Pasco were obtained by the CITE[1]-Agroindustrial Oxapampa, dried at 45 °C/10h were vacuum-packed and sent to the Research, Innovation and Technology Transfer Department Laboratories of the Instituto Tecnológico de la Producción del Perú (ITP). The husks (Figure 1) were ground and sieved using 0.0937, 0.033 and 0.0139 in diameter mesh, coded as 1, 2 and 3 fractions respectively. The larger fractions showed lighter shades than the smaller ones with powdery appearance which result from the pulp adhered to the husk and had a darker shade accentuated by drying (Figure 2). The yield percentages obtained for all husks variety after dry milling and sieving were 70%, 15% and 15%.

2.2 Preparation of the aqueous extracts

Hot water was added to the fractions (2.5% w/w) at two temperatures (60 and 80 °C), stirring at 200 RPM for 30 min and filtering on white biodegradable corn fiber mesh (6x8 cm). The extracts obtained from fractions at both temperatures and particle size were prepared in triplicate.

2.3 Physicochemical análisis

1. Total solids (TS) were determined in duplicate by gravimetry on 10 g of extract in previously tared pesafilter and subjected to oven drying at 100 °C for 12 h**.**

2. pH was measured in 10 g of extracts diluted in 100 mL distilled water using a Thermo Scientific® digital potentiometer.

3. Whiteness Index (WI) was determined in a Konica Minolta® CM-5 colorimeter, operating in CIE L*a*b* coordinates. Values were obtained by applying the following Equation 1:

$$IB_1 = 100 - \sqrt{\left(100 - L^*\right)^2 + a^{*2} + b^{*2}} \quad \textbf{(1)}$$

4. Antioxidant activity / Reducing Power (RP): was performed on the extract centrifuged at 5000 RPM for 2 min under refrigeration conditions. 50uL of the sample was mixed with phosphate buffer (0.2 M, pH 6.6) and 250 uL of 1% $K_3Fe(CN)_6$ and incubated at 50 °C for 20 min. After cooling the mixture 250mL of 10% trichloroacetic acid was added. 80uL of ferric chloride and 100 mL of distilled water were mixed with 100 uL aliquots of the supernatant. The results were obtained by spectrophotometer reading at 700 nm using gallic acid (1mg/mL) as the oxidizing compound and expressing the values as equivalent activity mg gallic acid/mL of sample (Zou; Lu; Wei, 2004). All analyses were performed in duplicate per extract.

5. Reducing Sugars (RS)÷ were determined by the DNS method. 100 µL of extracts, 200 µL of deionized water and 1800 µl of DNS reagent were shaken for mixing in test tubes, after heating in a water bath at 85 ºC for 15 min, 600 µl of sodium and potassium tartrate (40%) were added heating for 5 additional minutes. Quantification was performed by reading at 540 nm in a spectrophotometer using a 0.1% glucose standard curve. All analyses were performed in duplicate per extract (Bello; Carrera; Díaz, 2006).

6. Quantification of phenolic compounds: were determined according to the Folin-Ciocalteau method using 400 ppm gallic acid (100 mg gallic acid/25 ml of deionized water), 50 uL Folin Ciocalteau reagent 2 N and a sodium carbonate solution (75 g/L) prepared at the time of analysis due to the instability of the reagent (Singleton et al., 1965). For quantification, a calibration curve was prepared using 400ppm gallic acid as a standard. Total phenols (TP) were analyzed as gallic acid equivalents (GAEs). All analyses were performed in duplicate per extract.

7. The yield was calculated based on total solids extract and expressed in total extractable solids with respect to the husk (g of extractable solids in aqueous conditions/100 g of coffee husk)

[1]CITE is a Center for Productive Innovation and Technology Transfer that is part of the ITP Cite network.

Figure 1: Dried coffee husks.

Figure 2: Milling fractions of the Caturra variety obtained after sieving.
A: Coffee husk fraction 1, particle size > 0.0937 in
B: Coffee husk fraction 2, 0.0937 in0>particle size>.0330 in
C: Coffee husk fraction 3, 0.0330 in0>particle size>.0139 in

2.4 Statistical análisis

To test significative differences among groups based in factors, results were subjected to a factorial design ($p<0.05$) for three categorical factors. The first factor was extraction temperature at two levels (60 °C and 80 °C), the second factor variety at three levels (*Caturra, Catimor* and *Geisha*) and the third factor: particle size at three levels (0.0937, 0.033 and 0.0139 in) coded as 1, 2 and 3 respectively.

A correlation analysis of the average mean data ($p < 0.05$) was performed to determine the correlation among physicochemical characteristics. Additionally, a clustering test using principal components analysis (PCA) was applied to reduce the multiple physicochemical analyses into new variables (principal components) and obtain clusters for different types of coffee husks and their extraction conditions.

3 RESULTS

As the objective of this study was to extract components from the fractions (husks ground, and sieved) that may be scaled up when developing products aqueous, extracts at 60 or 80 °C were prepared by shaking fractions in water for 30 min. Three fractions sizes were evaluated for each husk variety (A, B, C). It could be observed that fractions obtained by sieves A and B were similar, but not fraction C which was different in shape and color as shown in Figure 2.

In the Tables 1 and 2 present the chemical and physicochemical analyses evaluated in coffee husk extracts. For better understanding, the results are presented in mg/mL of extract or mg/g of coffee husk husk in Table 2.

The average data set (mg/g) analyzed by Pearson's correlation looks for association and direction among the variables characterized in the coffee husk extracts in the present study (Table 3). The significant correlations obtained in the analyses indicate WI - pH ($r = 0.5739$, p-value = 0.0128), WI- St ($r = -0.63$, p-value = 0. 0051), WI-Yield ($r = -0.6344$, p-value = 0.0047), pH - St ($r = -0.8999$, p-value = 0.00), pH - Yield ($r = -0.8982$, p-value = 0.00), TP-RP ($r = 0.9582$, p-value = 0.000). The low extracts WI values derive from the TS extracted amount and would indicate the extraction of melanoidins present in the husks but which would be different from those generated by the roasting process. It was found that the antioxidant capacity of the extracts evaluated by their RP was correlated with the amount of TP ($r = 0.9582$).

Analyzing by multiple ANOVA the factors evaluated were Variety - Particle size - Extraction temperature with Variety Levels of *Caturra*, *Catimor* and *Geisha,* particle size level 1, 2 and 3 (0.097 in, 0.033 in, 0.0139 in) and 60 and 80 °C temperatures respectively. The results allow us to determine whether the factors have a significant effect on the physicochemical characteristics of the coffee husk extracts and what interaction of factors is responsible for these characteristics. Each one of the effects (Temperature, Variety and Particle size) resulted significant on the physicochemical characteristics, thus each factor interacted in these multiple interaction for the RS, WI, TP and RP ($p<0.05$), except for TS y pH values ($p>0.05$).

The PCA statistical analysis applied to the results obtained allows identifying patterns of relationship in a set of data. This grouping technique is used in coffee beans and wine quality in order to present the name of origin by using molecular markers, chemical, physicochemical, biological, sensory, bioactive and techno-functional properties. For the present case, the physicochemical variables involved in the study are pH, WI, TS, RS, TP and antioxidant capacity through RP and yield. In the Figure 3, clusters three occur: pH-IB, Sugars-Yield, Phenols-Capacity Antioxidant (Reductor Power).

Table 1: Physicochemical characteristics of coffee husk extracts.

Code	Temp.	Var.	Size	pH	Whiteness Index (WI)	L*	a*	b*	Total Solids (TS) g/100 g extract	Yield g extract /100 coffee husk
60A1			1	3.95 ± 0.01	36.87 ± 0.36	81.19 ± 0.34	10.14 ± 0.36	59.40 ± 0.77	1.15 ± 0.01	45.97 ± 0.27
60A2	60 ºC	Caturra	2	3.91 ± 0.01	25.01 ± 0.43	75.62 ± 1.04	15.68 ± 0.75	69.16 ± 0.61	1.13 ± 0.01	45.15 ± 0.31
60A3			3	3.89 ± 0.00	22.06 ± 0.20	74.56 ± 0.47	16.85 ± 0.38	71.72 ± 0.28	1.14 ± 0.01	45.46 ± 0.41
60G1			1	4.02 ± 0.00	30.17 ± 0.22	78.22 ± 0.34	12.41 ± 0.34	65.18 ± 0.45	1.32 ± 0.01	52.85 ± 0.42
60G2	60 ºC	Geisha	2	3.92 ± 0.01	10.94 ± 0.18	69.43 ± 0.32	22.77 ± 0.32	80.49 ± 0.32	1.27 ± 0.00	50.88 ± 0.06
60G3			3	3.88 ± 0.01	5.20 ± 0.09	65.15 ± 0.38	26.85 ± 0.23	83.98 ± 0.08	1.25 ± 0.01	50.13 ± 0.40
60R1			1	4.78 ± 0.01	43.45 ± 0.55	81.34 ± 0.96	9.21 ± 0.75	52.58 ± 0.98	0.92 ± 0.01	36.91 ± 0.26
60R2	60 ºC	Catimor	2	4.78 ± 0.00	38.09 ± 0.25	78.73 ± 0.33	11.06 ± 0.29	57.08 ± 0.49	0.89 ± 0.01	35.69 ± 0.50
60R3			3	4.80 ± 0.00	22.19 ± 0.18	70.26 ± 0.48	18.81 ± 0.33	69.40 ± 0.23	0.94 ± 0.01	37.56 ± 0.25
80A1			1	3.93 ± 0.02	27.63 ± 0.02	76.67 ± 0.12	14.64 ± 0.16	66.92 ± 0.3	1.27 ± 0.01	50.64 ± 0.23
80A2	80 ºC	Caturra	2	3.60 ± 0.16	17.12 ± 0.17	70.47 ± 0.59	20.54 ± 0.38	74.66 ± 0.26	1.27 ± 0.01	50.91 ± 0.54
80A3			3	3.86 ± 0.00	16.95 ± 0.17	70.72 ± 0.38	20.15 ± 0.35	75.06 ± 0.40	1.29 ± 0.02	51.43 ± 0.81
80G1			1	4.04 ± 0.01	24.84 ± 0.31	75.84 ± 0.63	14.98 ± 0.59	69.58 ± 0.67	1.31 ± 0.02	52.30 ± 0.83
80G2	80 ºC	Geisha	2	3.91 ± 0.01	5.93 ± 0.23	64.98 ± 0.92	26.64 ± 0.66	83.14 ± 0.20	1.34 ± 0.01	53.71 ± 0.52
80G3			3	3.89 ± 0.01	3.19 ± 0.20	61.85 ± 1.42	28.86 ± 0.70	84.16 ± 0.23	1.27 ± 0.05	50.93 ± 1.81
80R1			1	4.71 ± 0.00	38.25 ± 0.41	78.80 ± 1.05	11.30 ± 0.60	56.88 ± 0.82	0.98 ± 0.00	39.40 ± 0.11
80R2	80 ºC	Catimor	2	4.74 ± 0.00	30.8 ± 0.23	75.15 ± 0.49	14.34 ± 0.35	62.97 ± 0.49	0.99 ± 0.01	39.42 ± 0.25
80R3			3	4.75 ± 0.00	20.26 ± 0.31	70.04 ± 1.14	19.65 ± 0.67	71.23 ± 39	0.99 ± 0.01	39.70 ± 0.33

Factor: Temperature (60ºC y 80 ºC). Factor: Variety (*Caturra* "A", *Catimor* "R", *Geisha* "G"). Factor: Particle Size (0.0937 in "1"; 0.033 in "2" y 0.0139 in "3").

Table 2: Chemical characteristics of coffee husk extracts.

Code	Temp.	Var.	Size	Reducing Sugars (RS) (mg de glucose/ mL extract)	Reducing Sugars (RS) (mg de glucose/g husk coffee)	Total Phenols (TP) (ug gallic acid/ mL extract)	Total Phenols (TP) (ug gallic acid/g husk coffee)	Reducing Power (RP) (mg gallic acid/ mL extract)	Reducing Power (RP) (mg acid gallic/ g husk coffee)
60A1			1	4.33 ± 0.19	173.22 ± 7.42	0.27 ± 0.03	10.98 ± 1.08	0.39 ± 0.01	15.51 ± 0.45
60A2	60 ºC	Caturra	2	3.54 ± 0.05	141.45 ± 1.91	0.31 ± 0.03	12.32 ± 1.07	0.44 ± 0.01	17.52 ± 0.45
60A3			3	3.37 ± 0.06	134.95 ± 2.36	0.32 ± 0.03	12.94 ± 1.18	0.48 ± 0.02	19.23 ± 0.67
60G1			1	6.08 ± 0.20	243.48 ± 7.83	0.28 ± 0.02	11.08 ± 0.70	0.31 ± 0.01	12.26 ± 0.47
60G2	60 ºC	Geisha	2	4.39 ± 0.15	175.82 ± 5.82	0.23 ± 0.01	9.14 ± 0.45	0.47 ± 0.01	18.83 ± 0.47
60G3			3	5.29 ± 0.09	211.49 ± 3.42	0.27 ± 0.02	10.63 ± 0.65	0.55 ± 0.01	21.96 ± 0.49
60R1			1	4.81 ± 0.04	192.47 ± 1.74	0.23 ± 0.02	9.35 ± 0.81	0.36 ± 0.01	14.47 ± 0.56
60R2	60 ºC	Catimor	2	4.54 ± 0.09	181.58 ± 3.47	0.20 ± 0.03	8.11 ± 1.14	0.31 ± 0.02	12.30 ± 0.65
60R3			3	4.51 ± 0.24	180.47 ± 9.75	0.27 ± 0.01	10.66 ± 0.43	0.35 ± 0.01	14.03 ± 0.51
80A1			1	4.21 ± 0.07	168.31 ± 2.63	0.23 ± 0.02	7.40 ± 0.96	0.33 ± 0.03	13.05 ± 1.15
80A2	80 ºC	Caturra	2	4.24 ± 0.36	169.70 ± 14.28	0.27 ± 0.02	10.73 ± 0.88	0.33 ± 0.03	13.16 ± 1.18
80A3			3	3.4 ± 0.16	135.91 ± 6.21	0.18 ± 0.02	7.28 ± 0.81	0.28 ± 0.01	11.10 ± 0.56
80G1			1	6.05 ± 0.33	242.16 ± 13.11	0.22 ± 0.02	8.79 ± 0.62	0.25 ± 0.01	9.93 ± 0.58
80G2	80 ºC	Geisha	2	5.23 ± 0.25	209.14 ± 10.18	0.13 ± 0.08	6.97 ± 0.42	0.31 ± 0.02	12.53 ± 0.77
80G3			3	4.29 ± 0.17	171.56 ± 6.83	0.20 ± 0.02	7.84 ± 0.84	0.33 ± 0.03	13.13 ± 1.31
80R1			1	3.59 ± 0.13	143.75 ± 5.29	0.18 ± 0.04	8.06 ± 0.99	0.29 ± 0.02	11.75 ± 0.63
80R2	80 ºC	Catimor	2	3.5 ± 0.14	140.05 ± 5.48	0.23 ± 0.06	8.12 ± 0.94	0.26 ± 0.01	10.33 ± 0.48
80R3			3	2.74 ± 0.14	109.47 ± 5.63	0.26 ± 0.01	7.69 ± 1.88	0.30 ± 0.01	12.10 ± 0.49

Temp.: Temperature (60ºC y 80 ºC). Var.: Variety (*Caturra* "A", *Catimor* "R", *Geisha* "G"). Size: Particle Size (0.0937 in "1"; 0.033 in "2" y 0.0139 in "3").

Table 3: Pearson Correlation analysis between average physicochemical analyses.

	Reductive Sugar	Phenols	WI	pH	Antioxidant capacity	TS	Yield
Reductive Sugar		-0.3328	-0.0663	-0.2142	-0.0143	0.4162	0.4162
Phenols	-0.3328		-0.2388	-0.4159	0.9572	0.1099	0.1099
WI	-0.0663	-0.2388		0.5739	-0.3121	-0.6300	-0.6300
pH	-0.2142	-0.4159	**0.5739**		-0.3477	-0.8988	-0.8988
Antioxidant capacity	-0.0143	**0.9572**	-0.3121	-0.3477		0.1009	0.1009
TS	0.9552	0.1099	**-0.6300**	**-0.8988**	0.1009		0.998
Yield	0.9552	0.1099	**-0.6300**	**-0.8988**	0.1009	0.998	

Red numbers indicate significant correlation with $p < 0.05$. (*) Caturra, Catimor and Geisha coffee husks.

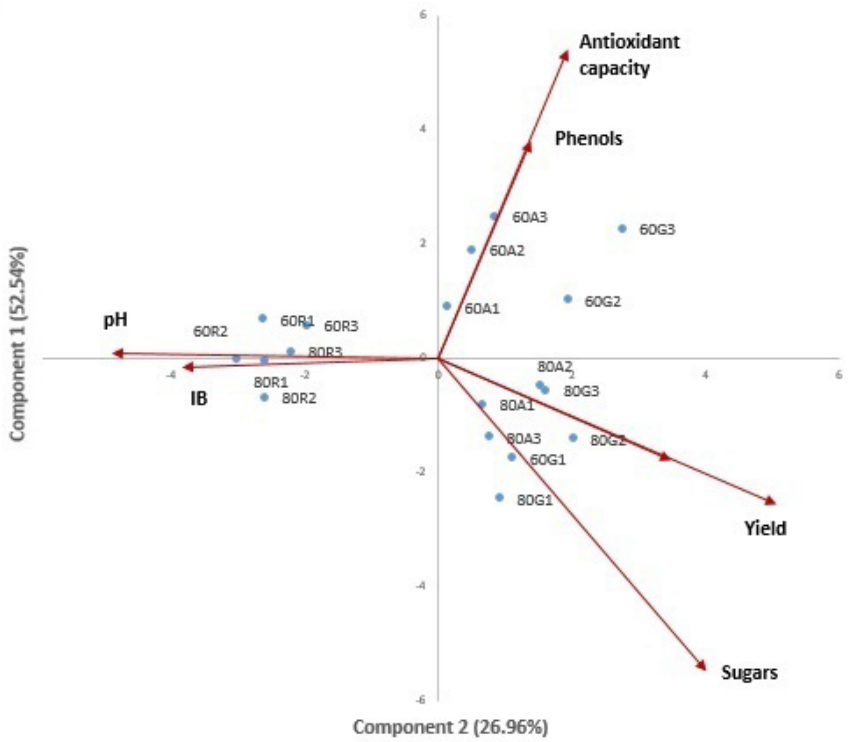

Figure 3: Analysis of Principal Components from the physicochemical analyzes of the coffee extracts*.

4 DISCUSSION

The appearance of the coffee husks−from *Caturra, Catimor and Geisha* varieties did not present major differences after drying even though commercially the coffee from these varieties present different cup qualities which could be related to their physicochemical characteristics.

Solid-liquid extraction or leaching methods to obtain coffee beverages involve the transfer of solutes from solid to fluid, therefore the quality of the extracts depends on many factors such as coffee bean variety, water/bean ratio, particle size, grind distribution, preparation times, water temperature, agitation, water quality, extraction uniformity. (Moroney et al., 2015).

Among the different extraction methods, cold extraction involving stirring coffee beans in cold water for periods exceeding 12 hours yields extracts with reduced bitterness and acidity, and a sweeter flavor profile. On the other hand, hot extraction (80 to 95 °C) provides high yields but results in extracts with higher levels of bitterness and acidity. Baristas have developed techniques to diversify drip extraction by varying factors such as the type of filter, pore size, retention time, and extraction temperature, resulting in a range of sensory profiles including acidity, bitterness, body, and aroma (Zapata; Arango; Rojano, 2019). Additionally, extracts can be obtained using supercritical fluids such as CO_2 at high pressure and temperature, resulting in a beverage with high caffeine content, low acidity and bitterness, and diverse aromatic profiles (Andrade et al., 2012).

Coffee bean extracts contain various components including caffeine, organic acids (such as chlorogenic,

malic, and citric acid), pigments (such as melanoidins and anthocyanins), diverse flavor and aroma compounds (including aldehydes, ketones, pyrazines, and furans), and also soluble fiber (such as pectin and oligocellulose) (Hoseini et al., 2021). These components contribute to the rich and complex sensory profile of coffee beverages. However, it is important to note that the extraction process and preparation methods of coffee husks produce different characteristics.

Moreover, dry and non-ground coffee husks are prepared through a short hot leaching process (90°C/30 to 60 seconds), resulting in a floral, fruity, and sweet aromatic profile with subtle hints of low bitterness (Arpi et al., 2021). While the composition of coffee husk extracts differs from that of coffee bean extracts, both play a role in enhancing the overall sensory experience of coffee consumption.

In the present study, solvents such as methanol, ethanol, and chloroform were not utilized.

Although these solvents have been shown to improve extraction yields and the diversity of extractable molecules, el crecimiento de la quimica verde pondera el uso de agua asistida por diferentes metodos fisico-quimicos-biologicos (Lajoie; Fabiano-Tixier; Chemat, 2022; Adeeyo et al., 2023).

The beverage is typically consumed as an infusion, with water-soluble molecules being the primary focus. It is important to consider the practicality and relevance of the extraction methods used in research, as they should align with the typical consumption habits and nutritional considerations of coffee consumers.

4.1 Yield

The range of yield values expressed as the percentage of extractable solids in the husks was 36.9% to 52.3% (2.5 g/100 ml/30 min/60 or 80 °C). Some yield values in Arabica coffee extracts of unroasted beans (10 g/100 ml/6 min/95 °C) fluctuated between 15.9 to 20.6 % while in roasted beans (10 g/100 ml/6 min/95 °C) a range of 16.3 - 28.8 % was obtained and in cold extracts coffee beans the yield was reported as 16.73 % (Morais et al., 2008; Violin et al., 2021, Gemechu, 2020). According to the coffee husks variety in the current study, *Geisha* extracts presented higher extraction yields, 50.1 - 52.8 % and 50.9 - 52.3 % at 60 °C and 80 °C respectively compared to *Caturra* (40 - 50 %) and *Catimor (*35 - 39 %.)

4.2 Phenolic compounds

Coffee compounds contain phytochemical compounds (5 - 9 %). The main phytochemical compounds are represented by phenolic components (chlorogenic, ferulic and caffeic acid among others), tannins, alkaloids (caffeine) etc., (Hoseini et al., 2021). Coffee bean or husk polyphenols play an important role in human health by protecting against diseases related to free radical-induced damage (Geremu et al., 2016).

Iriondo-DeHond (2019) reported 15.6 mg GAE/g in 5 % aqueous extracts, 68.2 mg GAE/g in parchment and a 44.8 - 56.5 mg GAE/g values range in tegument. In this study the values in 60 °C extracts oscillated in a range of 8.11 - 12.94 mg GAE/g and at 80 °C from 7.40 - 10.73 mg GAE/g in the different 2.5 % aqueous extracts. The variety with the highest content of extractable polyphenols was Caturra extract fraction 3 at 60 °C (10.98 - 12.99 mg GAE/g); the lowest contents were observed in Geisha extract fraction 2 at 80 °C (6.97 - 8.79 mg GAE/g), in both cases these values are comparable to 9.17 mg GAE/g reported by Heeger et al. (2017) in aqueous extracts from *Bourbon* variety coffee pulp (10 %/15 min/ 85 °C).

4.3 Antioxidant capacity

Among the variety of polyphenols present in coffee bean extracts, chlorogenic acid is one of the main responsible for the antioxidant potential by hindering further oxidation of other food components due to the ability of phenolic hydroxyl groups in the ortho position to scavenge free radicals. Inhibition of DPPH, oxidation of ABTS, ORAC, CUPRAC and iron oxidation by FRAP and RP are techniques to evaluate the antioxidant capacity (Zhou et al., 2020; Munteanu; Apetrei, 2021).

The RP is an important parameter that is directly related to antioxidant properties. Table 2 shows that the highest RP values correspond to the Geisha extracts at 60 °C (fractions 2 and 3), followed by the Caturra extracts at 60 °C also in fractions 2 and 3; nevertheless no coincidence was observed with the highest values of phenolic compounds. This last would suggest that other soluble compounds such as soluble fiber with antioxidant capacity were also extracted (Cano-Muñoz et al., 2021). Other components such as melanoidins (autooxidation products of phenolic compounds) could provide antioxidant capacity (Iriondo-DeHond et al., 2021).

In contrast to the positive effect of roasting on the antioxidant capacity of coffee beans (Ormaza-Zapata; Díaz; Rojano, 2020) these oxidation processes are influenced by temperature and oxygen (Tran et al., 2020). Extraction with methanol: water (8:2) and boiling water for 4 min showed similar antioxidant capacity (based on DPPH) (Castaldo, 2018) showing that either by different techniques, the antioxidant potential of coffee can be assessed.

4.4 Reductive Sugars

Natural sugars in coffee bean extracts are a relevant aspect for the flavor profile and contribute to balance the bitterness (Constantino et al., 2020). The effect of roasting on coffee beans causes the decrease of RS which does not occur in the husks, consequently higher RS values would be expected in the latter besides of some pulpy parts containing higher sugars amounts than the bean.

The highest RS values were obtained in Geisha extracts at 60 °C and 80 °C. Hoseini et al. (2021) reported 12% RS and 14% TS in coffee husks (C. Robusta), which led to the utilization of coffee husks to produce ethanol (Gouvea et al., 2009). In the Geisha extracts RS oscillated from 16 to 24 %, these results are comparable with the 15.6 to 20.6 % RS range obtained in silverskin coffee extracts (Arabiga and Robusta varieties) at 25 - 80 °C and 10.7 to 13.7% RS range at 210 - 270 °C (Mesías et al., 2014).

4.5 Color

The color of extracts can vary depending on many factors such as bean type, roasting process and extraction method. Typically coffee extracts can vary in the range of light to dark brown and sometimes black. During the roasting coffee beans undergo chemical browning reactions producing various aromatic components and changes of color. The higher the degree of roasting the darker and more intense the color and flavor (Yeager et al., 2022, Bekedam, 2008).

The extracts of coffee husks in this study were reddish to dark brown in color. In the *Geisha* fraction 2 at 80 ºC, fraction 3 at 80 ºC and fraction 3 and 60 ºC, the WI values were 5.93, 3.49 and 5.2 and the L* values 64.98, 61.85 and 65.15 correspondingly. On the contrary, the extracts with the highest WI were the *Caturra* extracts at 60 ºC fraction 3 (43.45) and fraction 2 (38.09), *Catimor* extracts 80 ºC fraction 1 (38.25), L* values were 81.34, 78.73 and 78.80 respectively.

Fractions 1 of all varieties were observed lighter and higher in RS content probably because of polyphenoloxidases activity on melanoidin-type pigments in addition to the Maillard reaction, this last induced by the drying process in the husk. In fractions 3, the powdery material, produced darker extracts with less RS (Table 1).

Color evaluation in the current coffee husks extracts at 60°C shows 65 – 81 en L*, 9.2 - 22.7 a*, 52.58 - 80.49 b* value ranges while in fractions extracted at 80°C results indicated 61.98 - 78.8 L*, 11.3 - 28.86 a* and 56.88 - 84.16 b*. In an *Arabica* and *Robusta* coffee beans blend, Wongsa et al. (2019) found 38.57-41.1 L*, 7.23-7.86 a* and 10.42-13.35 b* ranges. It is interesting to note that comparision of all these values suggest that extracts prepared with beans present a darker tonality in contrast to the reddish colors of coffee husks.

4.6 pH

This physicochemical characteristic reflects organic acids content (citric, acetic chlorogenic and formic) as well as organic bases (trigonelin, phosphates, carbonates, etc) (Ormaza-Zapata et al., 2020).

The pH range in coffee husk extracts has been reported 5.35 to 6.63 by Hoseini et al. (2021). In Arabica coffee extracts (10%/6 min/96 ºC) with and without roasting this value oscilated from 5.15 to 5.84, with the highest values in the more intense roasting samples (Morais et al., 2008). In coffee bean extracts (10 %/4 min or 24 h/95 ºC or 15 ºC) Violin (2021) obtained pH values of 5 and 5.1 and higher darkening in the extracts of hot extracted samples. Cold extractions in *Arabica* coffee beans showed 5.15 to 5.20 pH and in *Robusta* coffee beans 10% extracts 5.32 - 5.37 (Portela et al., 2022).

4.7 PCA

Seven variables could be reduced to two components representing the accumulated variability up to 79.495 % (Comp.1: 52.539 % and Comp.2: 26.957 %) reaching 92.286 % with a third component (Comp.3: 12.791%). The groups formed were pH-WI, TS-Yield and TP-RP, among which the coffee husk extracts are regrouped to represent its relevant characteristics. In the pH-WI cluster we found *Catimor* extracts at both 60 and 80 °C with the three types of fractions. The cluster RP -TP indicates the highest antioxidant capacity in *Geisha* extracts at 60 °C (fraction 2) and Caturra at 60°C (fractions 1, 2 and 3).

TS-Yield cluster showed extracts at 80 °C *Geisha*, *Caturra* (fractions 1, 2 and 3 for both) and *Geisha* extracts 60 °C, fraction 1, with the highest values. It is important to remark that the particle size and temperature were determining factors for the differences in the physicochemical characteristics of extracts.

In summary, dry fractionating the ground husks allows obtaining different extracts from the same sample with the same yield since they contain the same amount of TS with differences in the RS, RP and TP contents. Relevant physical aspects such as color could be differentiated from particle size and variety. Dry fractionation opens up possibilities for new processes for this raw material enabling new and diverse types of products with different own basic characteristics. The results indicate that the extraction methods, the coffee husks variety and the particle size influence the variability of the physicochemical characteristics of the samples, especially antioxidant activity

5 CONCLUSIONS

The physical sieving process achieved an effective separation of ground coffee husks into different sizes, enabling the extraction of diverse types of extracts, each with well-defined physicochemical characteristics, depending on the coffee variety, extraction temperature, and grinding degree.

The Geisha variety extract stood out for presenting the highest RS content and the lowest WI value. On the other hand, the Caturra and Geisha extracts were characterized by having the highest TP (Total Antioxidant Capacity) and RP (Reactive Phenols) values, while the Catimor variety exhibited the highest WI value and the lowest acidity (pH).

Regarding the effect of the extraction temperature, it was observed that TP and RS values decreased at 80°C, which also affected the RP and WI values.

These results provide valuable and detailed information about the unique characteristics of the different extracts obtained from the studied coffee varieties. They have a significant impact on the industry as they provide a solid foundation for the development of new products and the optimization of quality in coffee shop offerings, delivering a unique and satisfying experience for coffee lovers worldwide.

6 AUTHORS' CONTRIBUTION

MPY, GCP and DPV performed the experiment, MPY and DPV wrote the manuscript and MEA co-work the manuscript, DPV conducted all statistical analyses.

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