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# Physicochemical and sensory characteristics of dehydrated coffee pulp in function of drying temperature[1]

## Características físico-químicas e sensoriais da polpa de café desidratada em função da temperatura de secagem

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*HIGHLIGHTS:*

*High drying temperatures influence the degradation of chlorogenic acids in coffee pulp beverage.*
*Coffee pulp prepared in single-dose capsules has good sensory characteristics.*
*Using dehydrated coffee pulp for beverages is a viable alternative to reduce environmental impact.*

**ABSTRACT:** Currently, the use of coffee pulp to prepare infusions is being studied based on its antioxidant properties. The objective of this study was to evaluate the effect of drying temperature on the chemical properties of dehydrated coffee pulp to characterize the coffee pulp beverage in single-dose capsules physically and sensorially after being subjected to three thermal treatments (CT, natural drying; T50, oven drying at 50°C; T60, oven drying at 60°C). Chemical characterization of the dehydrated pulp was performed using Fourier transform infrared spectroscopy analysis (ATR-FTIR) and liquid chromatography (HPLC). Next, physical and sensorial characterization of the beverage was performed to determine the soluble solids (SS), pH, titratable acidity, and color. On the other hand, this beverage was evaluated sensorially. Principal component analysis was performed on the data from the FTIR spectral ranges of 1,800-650 $cm^{-1}$. Physicochemical and sensory results were analyzed using ANOVA. The chemical, physical, and sensory behavioral results allowed the identification of T60 as a viable processing treatment.

**Key words:** spectroscopy, chlorogenic acids, caffeine, by-products coffee

**RESUMO:** Atualmente, uma das opções da utilização da polpa de café é a preparação de infusões com base na mesma, cujas propriedades antioxidantes foram avaliadas. O objetivo deste estudo é avaliar o efeito da temperatura de secagem nas propriedades químicas da polpa de café desidratada, bem como caracterizar física e sensorialmente a bebida da polpa de café em cápsulas de dose única, sujeitas a três tratamentos térmicos (CT: secagem natural, T50: secagem em estufa a 50 °C, e T60: secagem em estufa a 60 °C). A caracterização química da polpa desidratada foi realizada através da análise espectroscópica por infravermelhos da transformada de Fourier (ATR-FTIR), e cromatografia líquida (HPLC). Em seguida, foi realizada a caracterização física e sensorial da bebida, determinando sólidos solúveis (SS), pH, acidez titulável, e a cor. Por outro lado, a bebida foi avaliada sensorialmente. Foi realizada uma análise de componentes principais nos dados do espectro FTIR com intervalos de 1.800-650 $cm^{-1}$. Os resultados físico-químicos e sensoriais foram analisados pela ANOVA. Os resultados do comportamento químico, físico e sensorial permitiram identificar o T60 como o tratamento viável para o processamento.

**Palavras-chave:** espectroscopia, ácidos clorogênicos, cafeína, subprodutos do café

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## Introduction

The generation of organic and inorganic wastes contributes to pollution. Wastes generated in the household or countryside are likely to be treated for later use (Murthy & Naidu, 2012). Organic wastes from the agricultural sector are produced in enormous quantities worldwide, which is a major contributor to pollution, as agricultural waste is not limited to a specific place, but is broadly distributed, affecting natural resources (Doula & Sarris, 2016).

Coffee is one of the most consumed beverages worldwide and is highly important for the Colombian economy. However, the processes involved in coffee production generate approximately 784.000 tons per year of waste biomass, including mucilage, pulp, husk, and spent coffee grounds, among others (Serna-Jiménez et al., 2018). The coffee industry uses only 5% to prepare beverages (Janissen & Huynh, 2018).

One of the options for using coffee pulp is as dehydrated pulp-based infusion, which has shown favorable characteristics such as antioxidant capacity due to the presence of chlorogenic acids. The consumption of a coffee pulp-based beverage represents up to 64% of antioxidants consumed by adults (Pérez-Hernández et al., 2013).

Dehydration is a unitary operation that aims to reduce the water present in food and reduce and inhibit both chemical and enzymatic reactions and microbial growth, all of which lead to a deterioration in food quality (Gonçalves et al., 2017). Using different thermal treatments during dehydration could generate different physicochemical and sensory characteristics in the coffee pulp.

Taking into account the research carried out on coffee pulp, the objective of this study was to evaluate the effect of drying temperature on the chemical properties of dehydrated coffee pulp, and to characterize physically and sensorially the coffee pulp beverage in single-dose capsules subjected to three thermal treatments.

## Materials and Methods

A total of 180 kg of cherry coffee variety Colombia (*Coffea arabica*) was harvested in the central zone of the Department of Huila, Colombia, at a harvesting altitude of 1650 masl. Samples were taken to the pilot plant at the Universidad Surcolombiana in Neiva-Colombia (2°56'34.35" N, 75°18'03.48" W) for processing. The coffee cherry was selected based on its optimum maturity. Then, it was washed with potable water, submerged to overflow, and shaken by hand, and the procedure was carried out thrice. Finally, the cherries were submerged in 0.2% water containing sodium hypochlorite for 30 min. The coffee pulp was obtained using a coffee pulping machine (INGESEC, Gaviota GV300, Colombia). Finally, 78.95 kg of coffee pulp was obtained from the different treatments.

Three drying processes were carried out: sun drying (control treatment; CT) and stove drying at 50°C and 60°C in a Mermmet stove (UF55) of 53 L volume. In all three treatments, the pulps were brought to a moisture content of 7-8% on a wet basis (NCT 2698). The drying process for the CT was eight days; the stove drying lasted for two days. The moisture loss of the pulps was measured using a moisture analyzer (RADWAG, MA X2. A, Poland, EU).

Fourier transform infrared spectroscopy analysis (ATR-FTIR) of the dried pulp samples was performed according to Barrios-Rodríguez et al. (2021). An AFTIR Cary 630 (Agilent, USA) spectrometer with a DLATGS detector and ATR sampling accessory was used. FTIR-ATR measurements were carried out under ambient conditions of 60% relative humidity and a temperature of 23°C. Approximately 1 g of ground, dehydrated coffee pulp was placed in the sampling accessory. All spectra were recorded within the range of 4,000-600 $cm^{-1}$, 16 $cm^{-1}$ resolution, and 16 scans. The final spectrum was obtained as the average of three independent sample readings.

The extracts were prepared according to the methodology described by Heeger et al. (2017). A 1 g sample of dehydrated coffee cherry pulp previously ground in an electric grinder (Hamilton beach), added to 10 ml distilled water, was taken to a Bain-marie (Labnet, W1106) at 85°C, and constantly stirred for 15 min. Subsequently, the mixture was centrifuged at 3,220 rpm for 7 min in a centrifuge (HETTICH, EBA 200). This process was repeated twice, and the obtained supernatants were mixed at a ratio of approximately 2:3 (supernatant/solid). Subsequently, 10 mL of the extract was passed through 0.22 µm filters and then transferred to 2 mL vials for reading in an HPLC (Agilent technologies, LC 1260 infinity II, EE.UU) using a C18 column. The mobile phase was methanol: water (25:75), 1 mL $min^{-1}$ flux, 2-µL injection volume. The results are expressed as mg of caffeine and chlorogenic acid per gram of coffee sultana (mg $g^{-1}$). Standard curves were obtained for caffeine and chlorogenic acid (Sigma-Aldrich) for 25-800 mg $L^{-1}$ ranges. Run times were 20 min. The separation of caffeine and chlorogenic acid occurred at minutes 16 and 12, respectively.

The samples used for determining physicochemical variables were prepared with 8 g of dehydrated coffee pulp ground and 170 mL of water, which were placed in reusable keurig k-cup pod coffee filters. These were carried out in a KEURING single-serve coffee machine (K40, CHINA). The analyses were performed in triplicates.

pH was determined using a previously calibrated digital potentiometer (Starter 5000, EE.UU.). The refractive index of the beverage extract was measured using a digital refractometer (ATAGO, PR-201α, USA) calibrated with distilled water.

The titration was carried out with 0.1 N sodium hydroxide until pH reached the value 6.5, and 20 mL of the prepared beverage was taken to a beaker with 10 mL of distilled water. The volume spent in the burette was recorded and the percentage of acid was calculated using Eq. 1.

$$A = \frac{B \cdot C \cdot 2 \cdot 354.31 \cdot R \cdot H}{D} \quad (1)$$

where:

A - chemical titratable acidity of dehydrated coffee pulp sample (mg chlorogenic acid $g^{-1}$ of dehydrated coffee pulp);

B - volume of hydroxide (mL);
C - concentration of Sodium hydroxide (0.1 N);
2 - dilution factor (dimensionless);
354.31 - molecular weight of chlorogenic acid (g $mol^{-1}$);
R - 1/1000 conversion factor (L $mL^{-1}$);
H - 1000/1 conversion factor (mg $g^{-1}$); and,
D - grams of pulp exactly weighed (g).

It was determined using the CIE L*a*b* system, which defines each color from the coordinates L* (luminosity), a* (varies between red and green), and b* (varies between yellow and blue). A previously calibrated colorimeter (Konica Minolta CR-410, NJ, USA) was used. A portion of the coffee pulp was placed on a white surface, and the colorimeter lens was placed 5 cm from the sample to take the color registers.

The sensorial properties of the aromatic beverages obtained from each treatment were measured through qualitative descriptive analysis. Beverages were prepared as described for physicochemical analysis.

The evaluation was conducted by a panel of 20 judges with prior knowledge of coffee beverage analysis. The analysis was conducted in two sections, each with 10 persons. Each judge was provided with 30 mL of the three beverages coded and organized randomly, corresponding to each treatment.

The judges evaluated the attributes of color, aroma, flavor, body, and aftertaste and ultimately gave an overall acceptance value to the beverage. The rating scale used was a verbal hedonic scale, in which the lowest-rated score corresponded to a value of one point, and the best-rated to a value of nine points (Martinez-Saez et al., 2013).

A principal component analysis (PCA) was performed in ranges from 1,800-650 $cm^{-1}$, following the methodology used by Barrios-Rodríguez et al. (2020), with a matrix of 9 × 155. Physicochemical and sensory results were analyzed using analysis of variance. Means were compared using Tukey's test ($p \leq 0.05$). PCA was performed for physicochemical and sensory information. Statistical analysis was performed using the Statgraphics Centurion XVI-Version 16.1.1 statistical software and R-statistical software version 3.6.3.

## Results and Discussion

ATR-FTIR analysis showed that the control treatment (CT) yielded the highest absorbance values of the spectrum (Figure 1). The peaks of interest are associated with different chemical compounds present in coffee, such as caffeine, carbohydrates, water, and proteins (Ribeiro et al., 2011; Reis et al., 2013). The absorbance ranges reported by some authors for proteins are wavelength numbers 1,550-1,567 and 1,653 $cm^{-1}$, due to the flexion of NH in amide II and vibrations of $NH_2$ in amide I groups, respectively (Craig et al., 2014). In the 3,280 $cm^{-1}$ range, attributed to water, drying at 50°C and 60°C showed absorbance values similar to those of the control treatment, which showed higher absorbance, indicating high water activity in this sample (Barrios-Rodríguez et al., 2020).

Paradkar & Irudayaraj (2002) reported that the absorbance values related to the presence of caffeine in a product are the wavelength numbers 2,920 and 2,850 $cm^{-1}$, which have also

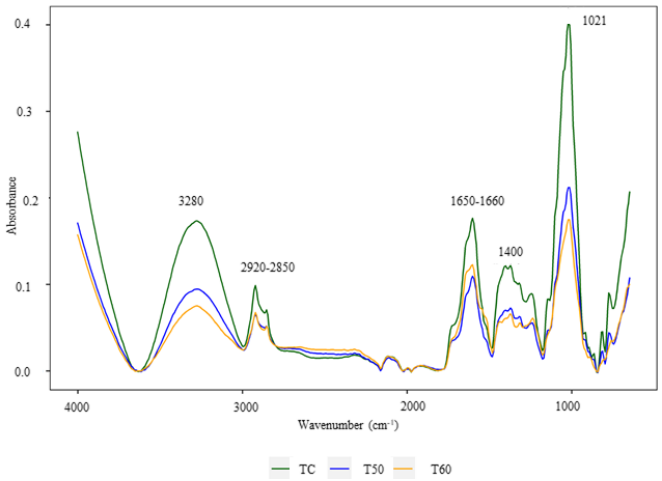


**Figure 1.** Identification of chemical compounds in dehydrated coffee pulp using infrared spectroscopy, obtained using Fourier transform infrared spectroscopy analysis (ATR-FTIR), subject to standardization and baseline correction.

been associated with the C-H bond vibration of the methyl group of the caffeine molecule (Craig et al., 2012). Reis et al. (2013) also reported that the caffeine content in coffee husk is similar to that in coffee beans; therefore, the peaks expressed in the wavenumber range 2,922-2,855 $cm^{-1}$ are associated with the C-H bond vibration of the methyl group of the caffeine molecule (Craig et al., 2012). Wavenumber ranges reported by the authors are related to those found in this research. Other authors mention the presence of caffeine in the range of 1,650-1,600 $cm^{-1}$ in the mid-infrared spectrum, associated with absorption by cyclic amides (Craig et al., 2014). They also reported that this region was related to the presence of lipids (Cremer & Kaletunç, 2003).

Chlorogenic acids are an ester family that forms between certain trans-cinnamic acids and chemical acids (Nemzer et al., 2021), and are associated with absorption peaks in the region of 1,450-1,000 $cm^{-1}$ (Barrios-Rodríguez et al., 2021a). For the three treatments, a high absorbance was observed at 1,021 $cm^{-1}$, with CT being the highest, associated with the presence of the ester functional group C=O=C (Silverstein et al., 1962). These peaks confirm the presence of quinic acid, which belongs to the chlorogenic family, a product of chlorogenic acid hydrolysis (Reis et al., 2013).

Figure 2 shows the results of PCA, where the first two components explain 70.3% of the total variance.

Figure 2 shows three groups separated by quadrants, each corresponding to a thermal treatment: CT (PC1 negative, PC2 negative), T50 (PC1 negative, PC2 positive), and T60 (PC1 negative, PC2 negative–negative). PC2 allowed separation between CT and T50, while T60 was located to the right of PC1, which was attributed to the differences observed in absorbance.

Figure 3 shows the wavelength distribution of each component.

In Figure 3, greater contributions from wavelength numbers 1,400 and 1,100 $cm^{-1}$ to CP1 and from wavelength numbers 850 and 900 $cm^{-1}$ to CP2 were observed.

Ribeiro et al. (2011) reported that the range of 1,700-1,600 $cm^{-1}$ is closely related to the chlorogenic acids and

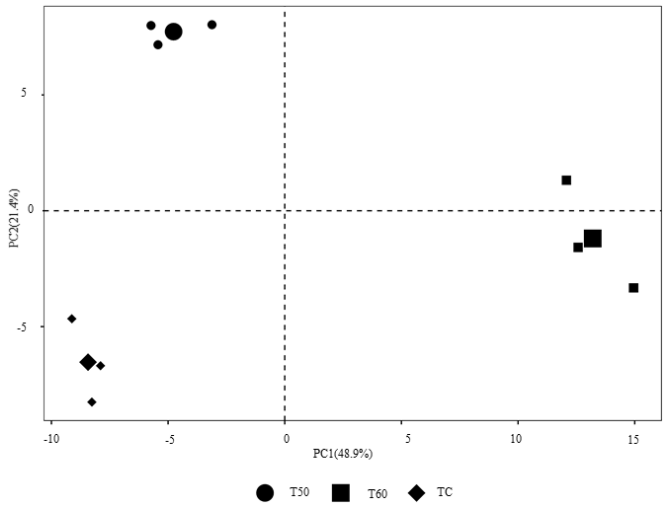


**Figure 2.** Dispersion in the first (PC1) and second (PC2) principal components of dehydrated coffee pulp in the ATR-FTIR spectra, subject to baseline normalization and correction

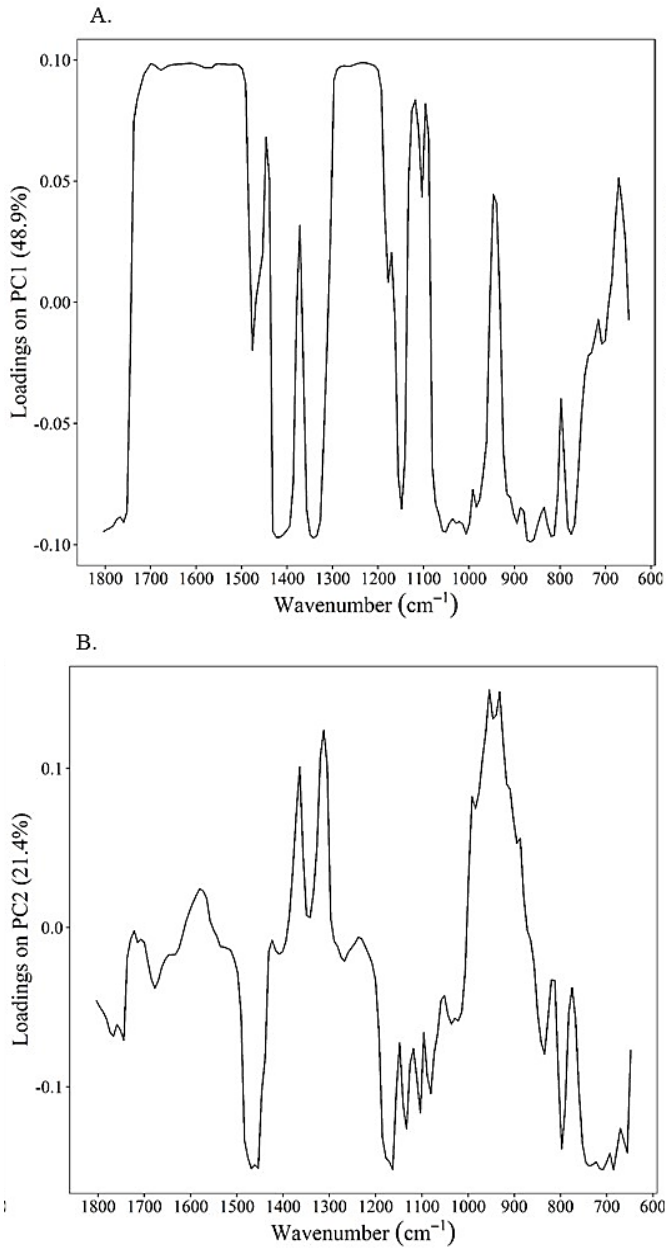


**Figure 3.** Wavelength loading 1,800-600 $cm^{-1}$ for first (PC1, A) and second (PC2, B) principal components

caffeine concentration, as shown in the graph corresponding to PC1. A high contribution in the positive region for the wavelength of 1,450 $cm^{-1}$ was observed, which is associated with the presence of chlorogenic acids pursuant to (Lyman et al., 2003) reports. Ranges of 1,381-1,376, 1,161-1,153, and 1,053 $cm^{-1}$ could also be associated with chlorogenic acids (Barrios-Rodríguez et al., 2020). The wavelength range of 1,400-900 $cm^{-1}$ is characterized by the vibrations of various types of bonds, including C=H, C=O, C=N, and P=O (Assis et al., 2019), both for PC1 and PC2, with high contributions from these wavelengths in the positive region.

In the graph corresponding to PC2 (3 B), a high contribution was observed in the positive region at 1,300 $cm^{-1}$, and according to Kemsley et al. (1995), chlorogenic acids show strong absorption at 1,300-1,150 $cm^{-1}$.

These acids correspond to a large family of esters formed between quinic acid and one to four residues of certain trans-cinnamic acids, most commonly caffeic, p-coumaric, anbarid, and feluric acids (Barrios-Rodríguez et al., 2021b). The ester bond C=O=C absorbs at 1,300-100 $cm^{-1}$ (Silverstein et al., 1962).

In the 1,400–900 $cm^{-1}$ range, contributions to PC2 were observed, characterized by vibrations of various types of bonds, including C=H, C=O, C=N, and P=O (Wang et al., 2009).

Thermal treatments affected caffeine and chlorogenic acid concentrations (Table 1).

The highest values of caffeine concentration occurred in the control treatment (CT), showing values between (8.87-9.28 mg caffeine $g^{-1}$ of dehydrated coffee pulp), followed by drying at 60°C (7.97-8.15 mg caffeine $g^{-1}$ of dehydrated coffee pulp); finally, the drying treatment at 50°C showed the lowest caffeine concentration (5.76-5.93 mg caffeine $g^{-1}$ of dehydrated coffee pulp). Pacheco et al. (2018) reported higher caffeine concentrations in coffee pulp (35.68 ± 1.57 mg caffeine $g^{-1}$ coffee pulp). The differences might be due to the coffee variety used in each study, and also due to factors such as the time of harvest, maturity condition, and altitude of harvesting, among others (Cheng et al., 2016), which could affect the final caffeine concentration.

In other studies, caffeine concentrations range from 0.22 to 12 mg caffeine $g^{-1}$ in dehydrated coffee pulp (Heeger et al., 2017; Janissen & Huynh, 2018). Heeger et al. (2017) also reported caffeine values in samples of coffee cherry between 3.4 and 6.8 mg $g^{-1}$ of dry husk, equivalent to the values obtained for the treatment at 50°C.

The drying treatment in a stove at 50°C showed the highest chlorogenic acid concentration (4.63-4.76 mg $g^{-1}$). Other studies obtained values of 128.9 mg $g^{-1}$ (Heeger et al.,

**Table 1.** Caffeine and chlorogenic acid concentration in dehydrated coffee pulp

| Compounds | CT | T50 | T60 | CV (%) |
|---|---|---|---|---|
| Chlorogenic acid (mg $g^{-1}$) | 3.18 b | 4.71 a | 2.86 c | 1.97 |
| Caffeine (mg $g^{-1}$) | 9.04 a | 5.87 c | 8.04 b | 1.89 |

CT, natural drying; T50, oven drying at 50°C; T60, oven drying at 60°C; different lowercase letters in the same row indicate significant differences between the thermal treatments by Tukey's test ($p \leq 0.05$)

2017; Janissen & Huynh, 2018). Values in the range 17.06-8.41 mg $g^{-1}$ have also been reported, which are higher than those found in this research. This is probably due to only the content of 5-O-caffeoylquinic acid being reported; usually, the total GCA concentration is reported, the values of which are around 37 mg $g^{-1}$ (Clifford et al., 2006).

Physicochemical characterization showed significant differences ($p < 0.05$) in all the physicochemical variables evaluated (Table 2).

Interestingly, different percentages of soluble solids with different concentrations were obtained from each extraction. The differences could be attributed to the drying conditions. An increase in the drying temperature causes an increase in the product temperature, and simultaneously increased the water diffusion coefficient, as was evident from the speed of water and sugar removal from the product (Torres-Valenzuela et al., 2019). On the other hand, convection drying could lead to the permanent loss of humidity and sugar, because the temperature is kept steady during the process; during natural drying, the humidity and temperature are variable.

The oven-drying treatments showed higher values than the natural-drying treatment. Common pH values for coffee were between 4.9 and 5.2 (Valencia et al., 2015). The values obtained for coffee pulp did not match the reported values despite belonging to the coffee sub-products and having many characteristics regarding its composition. However, the relationship between pH and titratable acidity, as mentioned by Gloess et al. (2013), shows that the lower the pH value, the higher the acidity.

The titratable acidity, expressed as a percentage of chlorogenic acids, showed significant differences when subjected to natural treatment (CT) and drying in a stove at 50 and 60 °C (Table 2). Although the samples did have the same percentage of humidity, the highest content of titratable acidity was obtained in the samples of the control treatment (37.5 ± 6.36 mg chlorogenic acid $g^{-1}$ of dehydrated coffee pulp). This was related to the changes in temperature to which it was subjected to during the control treatment, which did not exceed 40 °C, conserving many properties of fresh pulp.

In the CieLab space, L* indicates the tone luminosity or obscurity (0-100), a* varies between green and red (-a, +a), and b* oscillates between yellow and blue (+b, -b). Table 2 shows that sun exposure affected pulp characteristics to a lesser degree than heat exposure in a stove, where it takes a brownish color; when exposed to the sun, the pulp retained some natural tone of the coffee cherry (red). The highest L* value was observed during drying in a stove at 50 °C (57.9 ± 0.24), and the lowest value was observed during the control treatment (35.2 ± 0.32). This is different from what has been reported in another study, where dry samples showed a darker color (Torres-Valenzuela et al., 2019). These differences may be related to browning processes (Wang & Lim, 2015) catalyzed by the temperature used for drying the pulp. The highest value for the reddish tone (a*) was observed in the natural treatment (CT). Regarding the b* coordinate, only CT showed significant differences compared to drying at 50°C and 60°C, with the values being more positive and the samples showing a yellow tone (Table 2).

Considering the values obtained for the coordinates, we obtained a characteristic orange-reddish color for the infusion, which in turn is the characteristic color of the dehydrated coffee pulp.

The results of the sensorial analysis show that the judges had a general aceptation, and due to these attributes, treatment at 60°C was preferable. The color and aroma showed statistically significant differences between treatments (Table 3).

Differences in color were observed for the treatments carried out in an oven (50 and 60 °C), with the treatment at 50 °C being the one that was given the lowest score by the tasters.

According to the scores given by the tasting panel, the treatments did not affect the flavor or body of the beverages. Regarding the body, no beverage scored high values, considering that it is a light-body beverage. Finally, the residual flavor did not show significant differences ($p > 0.05$); however, it was emphasized that the pulp treated at 60 °C obtained the best qualification.

The first two principal components of the physicochemical and sensory characteristics explained 99.9% of the total variance (Figure 4).

PCA allowed the identification of differences between the treatments. pH and chlorogenic acid contributed to PC2 in the negative region; simultaneously, the variables were mostly associated with T50. For PC1, the contribution of acidity in the positive region was observed to be associated with CT, and for the negative region, the most contributing parameters were L*, flavor, and residual flavor, with the latter showing an association with T60. Sensory attributes, such as color, body, residual flavor, and general acceptance, were related to T60, ranking the treatment as the best scored in general terms.

**Table 2.** Physicochemical variables of coffee pulp beverage dehydrated at different temperatures

| Variables | | CT | T50 | T60 | CV (%) |
|---|---|---|---|---|---|
| SS (°brix) | | 2.16 a | 0.60 c | 1.43 b | 12.14 |
| pH | | 4.47 b | 4.79 a | 4.39 c | 0.24 |
| Acidity (mg CGA $g^{-1}$ CF) | | 37.50 a | 14.9 b | 12.90 b | 17.18 |
| Colour | L* | 35.20 c | 57.9 a | 56.40 b | 0.69 |
| | a* | 29.10 a | 20.4 b | 18.60 c | 1.76 |
| | b* | 19.58 c | 49.9 b | 50.41 a | 3.95 |

CT, natural drying; T50, oven drying at 50 °C; T60, oven drying at 60 °C; different lowercase letters in the same row indicate significant differences between the thermal treatments by Tukey's test ($p \leq 0.005$); SS: Soluble solids; CGA: Chlorogenic acid; L* - coordinate of luminosity; a* - Coordinate of variation of colors between red and green; b* - Coordinate of variation of colors between yellow and blue

**Table 3.** Statistical summary of the sensory analysis by attributes

| Variables | CT | T50 | T60 | CV (%) |
|---|---|---|---|---|
| Color | 6.23 ab | 5.73 b | 7.09 a | 20.94 |
| Aroma | 6.64 a | 4.73 b | 6.64 a | 25.32 |
| Flavor | 5.34 a | 5.64 a | 5.82 a | 31.02 |
| Body | 5.64 a | 5.64 a | 6.00 a | 24.37 |
| Residual Flavor | 5.73 a | 5.82 a | 6.23 a | 26.79 |

CT, natural drying; T50, oven drying at 50 °C; T60, oven drying at 60 °C; different lowercase letters in the same row indicate significant differences between thermal treatments by Tukey's test ($p \leq 0.005$)

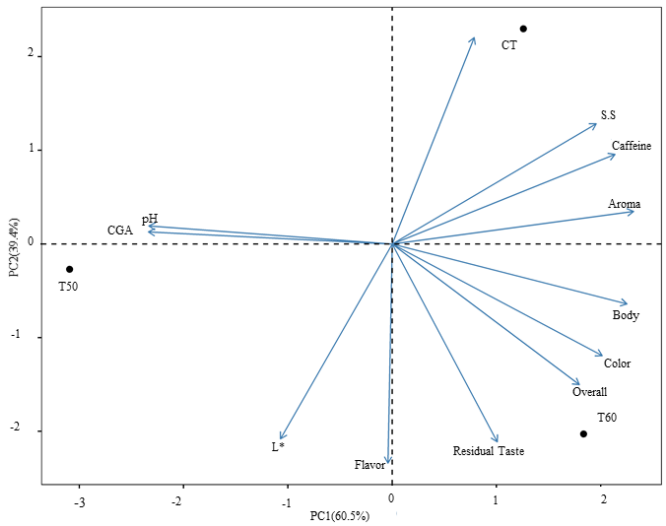


**Figure 4.** Dispersion in first (PC1) and second (PC2) principal components of physicochemical and sensorial variables of three temperature treatments (CT: natural drying, T50: oven drying at 50°C, and T60: oven drying at 60°C)

## Conclusions

1. The drying temperature had a significant effect on the chlorogenic acid concentration of the coffee pulp. A drying temperature of 50°C resulted in 39% more chlorogenic acid compared to treatment at 60°C, and approximately 32% more with respect to natural drying.

2. Based on the physicochemical and sensory behavior of the dehydrated pulp-based infusion, the most viable thermal treatment was drying in an oven at 60°C.

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