

## ARTICLE / INVESTIGACIÓN

## Effect of different drying airflows and harvest periods on the quality of specialty coffee (*Coffea arabica* L.)

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**Abstract:** Coffee is one of the most consumed commercial beverages worldwide, and coffee growers are constantly seeking innovative processing techniques to improve the quality of the final product. This study evaluated the influence of four drying airflows and three harvest periods on the chemical composition of green and roasted specialty coffee beans. The samples were obtained from the Hacienda La Papaya in Loja, Ecuador. Liquid and gas chromatographic techniques characterized the chemical profile of coffee beans, and sensory analysis was performed using the Specialty Coffee Association of America methodology. In total, 49 compounds were described, 29 in green beans and 20 in roasted beans. A significant ( $p < 0.05$ ) effect of the harvest period was observed in all phenolic compounds except for chlorogenic acid. The drying type significantly affected the levels of rutin and trigonelline. In addition, samples from different harvest periods observed significant differences in the levels of the amino acids serine, arginine, phenylalanine and leucine. Similarly, the drying type significantly influenced glycine, alanine, valine and isoleucine levels. For all drying-harvest combinations, the final cupping score was higher than 85/100, as the different drying processes slightly influenced the cupping attributes. Drying with minimal airflow was characterized by a low balance and intense flavor while drying with medium airflow presented a high ratio and soft body. The harvest period and drying type cannot be used as cupping predictors since no clear trends were observed to classify specialty coffee organoleptic attributes. Therefore, other variables involved in specialty coffee processing should be explored to evaluate higher sensitivity toward flavor prediction and innovation.

**Key words:** Chromatographic analysis, *Coffea arabica* L., sensory analysis, specialty coffee.

### Introduction

Worldwide, coffee is the second most traded commodity after oil<sup>1</sup>, and drinkable coffee is one of the most significantly consumed foods<sup>2,3</sup>. Coffee aroma is the determining attribute factor that defines the quality and coffee acceptance by the consumer<sup>2,4</sup>, and it is crucial for the specialty coffee market (Arabica variety), which is very elitist<sup>5,6</sup>.

The chemical composition of green beans is very complex and plays a significant role in aroma formation<sup>7</sup>. Organic acids and certain bioactive compounds have been identified as possible coffee sensory quality descriptors<sup>8,9</sup>. Trigonelline and chlorogenic acids are precursors to other volatile compounds produced during coffee roasting and contribute directly to the coffee aroma<sup>10</sup>. Trigonelline is an alkaloid in green coffee beans that is degraded considerably into pyridines and pyrroles<sup>11</sup>. Caffeine is associated with an undesirable bitterness that, depending on its concentration, can make the drink worthless<sup>8</sup>. Other aroma precursors in green coffee beans are sugars, proteins and free amino acids<sup>12</sup>. The protein content of dry green coffee beans is about 8-12%, and it is mainly degraded to amino acids during maturation, which is accelerated by chlorogenic acids and their derivatives<sup>13</sup>. Coffee beans contain several free amino acids such as alanine, arginine, asparagine, cysteine, glutamic acid, histidine, glycine, isoleucine, leucine, lysine, methionine, phenylalanine, proline, serine, tyrosine, threonine, and valine<sup>14</sup>. Those compounds could contribute

to the character and even the acceptability of the beverage since it has been suggested that the amino acid profile influences the yield of specific volatiles during roasting<sup>15</sup>.

Coffee bean processing is essential to obtain a high-quality product<sup>16</sup>. Roasting, washing and drying processes are the major stages that may influence coffee's chemical composition and, consequently, its aroma<sup>17,18</sup>. Drying is one of the post-harvest steps with the most significant influence on coffee quality<sup>19,20</sup>. The coffee drying process reduces the bean's moisture content and prevents the microbial action responsible for spoilage during storage<sup>21</sup>. The drying process of green coffee beans may be accompanied by changes in the physical, chemical, and organoleptic properties of heat-sensitive components<sup>20</sup>. Thus, the quality of the beverage could be defined while controlling the drying process<sup>22</sup>. The need for lower production costs and mitigating environmental damage has led to the development and research new drying techniques<sup>21</sup>.

Coffee aroma generation occurs predominantly during roasting through a complex series of Maillard reactions, in which nitrogenous heterocyclic compounds (pyridines, pyrazines and pyrroles) are formed. In addition, caramelization products generated by the thermal degradation of polysaccharides and simple sugars present in green coffee beans contribute to the development of roasting characteristics<sup>4,23,24</sup>.

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Besides pre and post-harvest practices, the sensory attributes can be influenced by the coffee variety and environmental/climatic factors (soil, altitude, sun exposure, rainfall, temperature)<sup>25-27</sup>. According to the Specialty Coffee Association of America (SCAA), the sensorial attributes evaluated in coffee cupping are fragrance/aroma, flavor, aftertaste, acidity, body, balance, sweetness, uniformity, clean cup and overall cupping<sup>28</sup>. Limited literature relates the composition of green coffee beans to the coffee cup quality<sup>22,29</sup>. Thus, more studies are required to support coffee to identify quality parameters at the green coffee bean stage, which is the most widely used maturity stage for purchasing and trading<sup>30</sup>.

The objective of this study was to evaluate the influence of the combination of different drying processes and harvest periods in aroma related-compounds and its relation with the sensory quality of the final product in green beans of an Ecuadorian specialty coffee.

## Materials and methods

### Coffee samples

Green coffee beans samples of *Coffea arabica* L. typica variety were obtained from the Hacienda La Papaya, located in Saraguro, Loja province, Ecuador, at 1700 m.a.s.l. This farm produces specialty coffee, and all process parameters are controlled until parchment coffee is obtained. Coffee beans were manually collected as mature fruits (17 - 22 °Brix). Ripe coffee beans were subjected to wet fermentation for 15 hours, followed by drying for 7 - 10 days in greenhouse-type experimental rooms, where temperature, relative humidity and aeration were controlled. Grains were homogenized four times a day using a wooden paddle. Grains were dried until reaching a humidity level between 10 - 12% to prevent fungal contamination. After drying, the samples were stored in a controlled temperature chamber at 10°C and 60% relative humidity for 30 days. Experimental models of 250 g of dried green grains were collected and processed in July-August 2019. Samples were packed in hermetic metalized bags until analysis.

### Experimental design

The study was based on an unbalanced 3x4 factorial design. Experimental variables were the harvest period (mid-July, early August and mid-August) and drying airflow: i) minimum (internal air movement produced by forced air from fans); ii) medium (high and low windows closed 50%, doors closed during the day and night); iii) zero airflow (high and low windows closed at 100%, doors closed during the day and night) and iv) maximum airflow (high and low windows open 100%, doors open during the day and closed at night). The experimental drying rooms were of the greenhouse type, built with wood and plastic. The drying rooms were rectangular, approximately 7 x 3.5 m<sup>2</sup>; the front and back parts correspond to the windows, and these were divided in half so that when the upper windows were opened, the lower ones could remain closed and vice versa. The lateral sides correspond to the doors of the greenhouses. In total, 36 samples were collected (12 conditions in triplicate). The response variables were the contents of some polyphenols, amino acids and volatile compounds involved in coffee aroma development.

## Chemical analysis

### Materials and reagents

Methanol and acetonitrile HPLC grade, fluorenyl-methoxycarbonyl chloride (FMOC; > 99%), and ortho-phthalaldehyde (OPA; > 99%) were supplied by Sigma Aldrich (St. Louis, MO, USA). Dibasic sodium phosphate is anhydrous by Mallincrodt AR (Phillipsburg, USA), glacial acetic acid and fuming hydrochloric acid by Merck KGaA (Darmstadt, Germany). Borate buffer and 3-mercaptopropionic acid, and ultrapure water were obtained from a NANOpure Diamond system (Barnstead, USA). Standards 3-O-caffeoylquinic acid, 4-O-caffeoylquinic acid, 5-O-caffeoylquinic acid, caffeine, trigonelline, caffeic acid, gallic acid, ferulic acid, apigenin, epicatechin, luteolin, rutin, DL-norvaline supplied by Sigma Aldrich (St. Louis, MO, USA) and quercetin by ROTH Carl (Karlsruhe, Germany). A stock solution of the amino acid standards (L-alanine, ammonium chloride, L-arginine, L-aspartic acid, L-cysteine, L-glutamic acid, L-leucine, L-lysine, L-serine, L-threonine, L-tyrosine, L-valine, L-histidine, L-isoleucine, L-methionine, L-phenylalanine, L-proline and glycine) were provided as a mixed solution at a concentration of 2500 pmol/μl was supplied by Sigma Aldrich (St. Louis, MO, USA).

### Instrumentation

For the analysis of phenolic compounds and derivatives, as well as amino acids, a high-performance liquid chromatography (HPLC) equipment (Agilent 1200 series) equipped with a quaternary pump, diode array (DAD) and fluorescence (FLD) detectors (Agilent Technologies, USA) was used. For volatile compounds, a gas chromatograph (GC) (7890A series) coupled to a mass spectrometer (MS) (5975C series) (Agilent Technologies, Santa Clara, CA) was used.

### Analysis of phenolic compounds and alkaloids

The extraction was carried out from 250 mg of dry and ground green coffee beans with 10 mL of an aqueous solution of 70% methanol. The solution was stirred for 1 minute with a vortex homogenizer, followed by sonication (BRANSONIC 3510, Mexico) for 1 hour with vigorous shaking every 10 minutes. Then, it was centrifuged at 6000 rpm for 10 minutes (Hettich MIRKO 220R, Germany). One mL of the supernatant was diluted with 9 mL of ultrapure water. The extract was filtered through 0.45 μm PVDF membrane filters before analysis.

Chromatographic separation of chlorogenic acids (5-CQA, 3-CQA and 4-CQA), caffeine, caffeic acid, gallic acid, ferulic acid, apigenin, epicatechin, luteolin, quercetin, rutin and trigonelline was performed by HPLC-DAD based on the method described in Saquicela (2018)<sup>31</sup> with some adaptations to include alkaloids. Separation was achieved with a Zorbax Eclipse C18 column (250 x 4.6 mm; 5 μm) (Agilent Technologies, USA), set at 30 °C and using a gradient elution at 1 mL/min of flow rate. The mobile phases were A: water acidified at 0.3% with acetic acid, B: acetonitrile: water/mobile phase A, 50:50 v/v, and C: acetonitrile 100%. Elution started with 10% B for 2 min, then increased to 55% B until 27 min and remained until 37 min. At 39 min, 100% C was reached and kept until 42 min (to wash the column). Finally, the column was re-equilibrated until 43.5 min. The injection volume was 10 μl. Detection was performed at 254 nm (trigonelline, rutin, luteolin and quercetin), 280 nm

(gallic acid, caffeine and epicatechin), and 320 nm (5-CQA, 3-CQA, 4-CQA, caffeic acid, ferulic acid and apigenin). The analytical parameters of the HPLC method are shown in Supplementary material (S1).

### Analysis of amino acid

The extraction was performed according to the method described in Murkovic & Derler (2006)<sup>32</sup>. Briefly, 200 mg of dry and ground green coffee beans were mixed with 10 mL of 0.1 N HCl solution. The solution was shaken for 1 min with a vortex homogenizer and sonicated for 15 min. Finally, the extract was filtered with a 0.45 µm PVDF membrane filter prior to analysis.

The extracts were subjected to programmed pre-column derivatization in the HPLC autoinjector. Primary amino acids were derivatized with OPA (5.12 mg in 1 mL methanol, adding 4 mL 0.4 M borate buffer) and 10 µl 3-MPA. Secondary amino acids (particularly proline) derivatization was performed with FMOC (2.5 mg/mL in acetonitrile)<sup>33</sup>.

Derivatized amino acids were separated with a Zorbax Eclipse AAA column (4.6 x 150 mm; 5 µm) (Agilent Technologies, USA), set at 40 °C. A flow rate of 2 mL/min was applied. Mobile phases were A) 40 mM Na<sub>2</sub>HPO<sub>4</sub>, pH 7.8, and B) methanol/acetonitrile/water 45:45:10 v/v/v. Elution started with 100% A from 0 to 1.9 min, followed by an increase in B from 0-57% until 18.1 min and then increased to 100% at 18.6 min, maintained until 22.3 min, to finally re-equilibrate until 26 min. Compounds eluted up to 10 min were detected at 340 nm excitation and 450 nm emission, while eluates up to 15 min were detected at 266 nm excitation and 305 nm emission<sup>34</sup>. The analytical parameters of the HPLC method are shown in S1.

### Analysis of volatile compounds

Volatile compounds were evaluated in the experimental coffee samples after a standardized roasting process carried out in 50 g-roaster (Ikawa V2 model, London). The roasting process begins with a preheating phase of the Ikawa toaster until it reaches a temperature of 159.8 °C. In the meantime, 50 grams of green coffee beans were weighed and introduced into a doser of the roasting equipment. When the Ikawa equipment was preheated, the doser was opened, and the coffee beans fell through the hole into the roasting chamber. Then the roasting process begins at minute zero at 158.2 °C; this process lasts approximately 5 minutes and reaches a temperature of 205 °C. After roasting, Ikawa entered a cooling phase. Coffee was cool down inside of Ikawa, this took about 2 minutes more, and the roasted coffee was obtained ready for analysis in GC-MS.

Roasted grains were ground and kept in liquid nitrogen. Volatile compounds were extracted by solid phase headspace microextraction (SPME). Approximately 1 g of ground material was transferred to 50 mL vials of SPME. The samples were equilibrated at 50 °C for 30 minutes in a water bath. Finally, they were placed on a 50/30 µm SPME fiber of Divinylbenzene / Carboxen / Polydimethylsiloxane (DVB/CAR/PDMS) (Supelco, Bellefonte, PA, USA) in the headspace of each sample vial for 30 min at 50°C. SPME fiber received initial conditioning at 270°C for 1 hour and daily maintenance conditioning at 240°C for 10 min.

The SPME fiber was removed from the vial and injected in Split mode at 240°C into the GC-MS. Chromatographic separation was performed with a DB5-MS column (30m x 250µm x 0.25µm). The oven temperature ramp started at 70°C and rose to 310°C at a rate of increase of 7°C/min.

Helium was used as carrier gas at a flow rate of 0.8 mL/min. The MS was set to maximum sensitivity in electron impact mode, positive polarity, and the total ion current was recorded for a mass range of 50 to 550 amu.

For the identification of metabolites, the MZmine 2 software was used. First, the data from the analysis of aromatic compounds obtained from the chromatography equipment were imported. The presence of peaks (metabolites) in each sample was then checked, and low-quality samples (chromatograms with few peaks) were discarded. The aromatic compounds that are present in coffee were searched in the literature according to the peaks found in each sample obtained after discarding the low-quality samples. The baseline height and the height of the smallest peak in each sample were defined. The following parameters were determined: the average baseline, the average value of the small peaks and the minimum value of the peaks. Parameters that were later used for mass detection and construction of chromatograms. Then deconvolution, normalization and alignment of the peaks were performed. Subsequently, the process known as gap filling was carried out, making it possible to search for peaks that could have been erroneously eliminated in the previous steps. The data obtained in the MZmine 2 software was exported to an Excel file to identify the metabolites. The metabolites were defined by comparing the retention times obtained in the MZmine 2 software and the retention times of the data obtained from the chromatography equipment, as well as with the metabolites described in the literature and the probability percentage for each compound that was obtained from the chromatograph data.

### Sensory analysis

The coffee sample cupping process was performed per the SCAA standards with the support of trained tasters. Initially, the samples were roasted (Ikawa V2, London) with a roasting profile adapted to 2500 m.a.s.l. and then ground (Coffee mill Mahlkönig Guatemalan, Germany). A sample of 12.5 g of ground coffee was weighed and placed in a 200 ml glass beaker. To reduce the subjectivity, coffee samples were coded. Aroma and fragrance were the first evaluated parameters. For this, the glass cup with the coffee was shaken and sucked by the tasters as many times as necessary to score it. Then, hot purified water was added to the coffee in the glass until it was almost complete. After 4 minutes, the foam was removed from the upper part of the glass, and the odor emanating from it was inhaled (a process known as "break cup"). The tasting was done 12 min after the contact of the coffee with the water. Coffee was absorbed with a spoon, and after a few seconds, the coffee was expectorated. With this, flavor, acidity, body, aftertaste and balance were determined. The absorption process was repeated as many times as necessary to evaluate each parameter. At the end of the tasting, a discussion and exchange of opinions on the scores were held to know each taster's points of view and observations regarding the samples evaluated without this influencing the already established scores. The samples were classified according to the scores within the following scale: 65-74.99, 75-79, 99, 80-84.99, 85-94.99 and 95-100, which corresponds to a cup quality of good, very good, specialty, excellent and exceptional, respectively.

### Statistical analysis

Chromatographic data obtained on HPLC were pro-



cessed using Chemstation software (Agilent Technologies, USA). Compounds were identified by comparison with the retention times of analytical standards. Each compound's concentration was calculated by interpolating the area values in the corresponding calibration curves.

To evaluate possible differences in the chemical coffee composition with respect to drying methods and harvest times, data was pre-processed. For the coffee samples subjected to the minimum airflow drying method, no experimental results were obtained for any of the analyzed samples. In the case of medium, zero and maximum airflow, results were obtained in some of the replicates. Considering the experimental design, data imputation before principal component analysis was performed specifically for those missing GC-MS results.

Then a normalization of the data was performed using two scaling methods: 1) the Min-max scaling method, used to scale the data in a 0-1 range, and 2) the Standard scaler method, in which the mean of the values becomes zero, and the standard deviation became one. Subsequent analyzes were performed with the values of all repetitions. Ordinary Least Squares performed linear regression to identify the compounds influenced by drying and harvest time and/or the interaction between them. Then, a principal component analysis (PCA) and heat map was constructed with the compounds that were sensitive to the study variables. The individual influence of the drying type and harvest time on the chemical composition of experimental coffee samples

was evaluated by two-way ANOVA. To determine the different experimental conditions, the posthoc Tukey's test was applied. For the evaluation of cupping data and its relation with the type of drying, the Random Forest method and Shap plots were used. In addition, a heat map was built with the significant variables. Statistical analyzes were performed with a 95% confidence level using Python software version 3.9.0 and R version 4.1.2.

## Results and discussion

### Chemical composition of specialty coffee beans (*Coffea arabica* L.)

In this study, several compounds related directly or indirectly to the organoleptic characteristics of specialty coffee were evaluated.

The quantified alkaloids and phenolic compounds in green beans of specialty coffee are presented in Table 1. Caffeine concentration ranged between 7580.04 - 9482.43 µg/g, and trigonelline ranged between 8653.24 - 10322.03 µg/g. The composition of trigonelline was comparable with other studies where concentrations ranged between 7100 - 13200 µg/g; meanwhile, caffeine composition was within a broad range of 500 - 44420 for Arabica varieties from different geographical origins, µg/g previously reported<sup>4,25,35-39</sup>. For most bio compounds, the highest concentrations were

Compound	Drying type				Harvest time
	Minimum airflow	Medium airflow	Zero airflow	Maximum airflow	
Rutin <sup>a</sup>	71.71 ± 15.37 <sup>A</sup>	33.79 ± 6.31 <sup>A</sup>	64.33 ± 10.85 <sup>A</sup>	54.60 ± 17.82 <sup>A</sup>	Mid-July
	35.28 ± 5.78 <sup>B</sup>	21.62 ± 19.02 <sup>B</sup>	29.41 ± 27.71 <sup>B</sup>	N.D	Early Aug
	35.50 ± 35.06 <sup>AB</sup>	38.64 ± 33.72 <sup>AB</sup>	26.94 ± 25.84 <sup>AB</sup>	N.D	Mid-Aug
Epicatechin	304.43 ± 8.69	288.94 ± 15.88	289.61 ± 16.24	318.96 ± 29.34	Mid-July
	333.81 ± 21.14	315.51 ± 11.79	327.24 ± 8.45	307.63 ± 14.59	Early Aug
	317.97 ± 8.85	328.48 ± 2.29	314.91 ± 24.32	331.39 ± 15.94	Mid-Aug
Apigenin <sup>a</sup>	122.36 ± 8.95 <sup>B</sup>	121.14 ± 2.88 <sup>B</sup>	121.71 ± 6.92 <sup>B</sup>	124.47 ± 2.21 <sup>B</sup>	Mid-July
	142.32 ± 6.38 <sup>AB</sup>	126.89 ± 6.77 <sup>AB</sup>	131.17 ± 10.09 <sup>AB</sup>	121.84 ± 6.74 <sup>AB</sup>	Early Aug
	122.13 ± 2.11 <sup>A</sup>	168.32 ± 20.40 <sup>A</sup>	186.35 ± 49.85 <sup>A</sup>	156.01 ± 84.02 <sup>A</sup>	Mid-Aug
Caffeic Acid	88.45 ± 0.97	86.81 ± 0.94	87.15 ± 0.18	90.90 ± 2.14	Mid-July
	92.89 ± 2.52	89.25 ± 0.46	89.60 ± 2.15	88.15 ± 1.03	Early Aug
	89.66 ± 0.88	91.35 ± 2.09	90.17 ± 0.95	92.22 ± 0.64	Mid-Aug
3-CQA	29120.97 ± 665.39	27264.53 ± 963.99	28421 ± 1287.98	29758.10 ± 1886.41	Mid-July
	29328.68 ± 1595.36	28287.42 ± 776.69	28758.13 ± 1531.08	27531.08 ± 1172.34	Early Aug
	28101.59 ± 514.63	28749.34 ± 1559.64	27483.01 ± 950.50	28989.20 ± 786.07	Mid-Aug
4-CQA <sup>a</sup>	4387.57 ± 89.23 <sup>AB</sup>	4093.97 ± 127.21 <sup>AB</sup>	4300.40 ± 259 <sup>AB</sup>	4480.51 ± 303.92 <sup>AB</sup>	Mid-July
	4180.95 ± 294.43 <sup>B</sup>	4006.46 ± 39.96 <sup>B</sup>	4267.59 ± 116.20 <sup>B</sup>	3946.99 ± 119.08 <sup>B</sup>	Early Aug
	4376.07 ± 38.91 <sup>A</sup>	4578.96 ± 430.39 <sup>A</sup>	4285.95 ± 39.21 <sup>A</sup>	4718.85 ± 65.98 <sup>A</sup>	Mid-Aug
5-CQA <sup>a</sup>	3429.42 ± 31.80 <sup>AB</sup>	3186.77 ± 113.90 <sup>AB</sup>	3342.41 ± 263.09 <sup>AB</sup>	3490.28 ± 320.91 <sup>AB</sup>	Mid-July
	3014.71 ± 239.02 <sup>B</sup>	3107.93 ± 54.25 <sup>B</sup>	3195.23 ± 80.47 <sup>B</sup>	3102.44 ± 66.32 <sup>B</sup>	Early Aug
	3413.60 ± 68.98 <sup>A</sup>	3697.03 ± 365.50 <sup>A</sup>	3350.75 ± 38.48 <sup>A</sup>	3713.19 ± 88.32 <sup>A</sup>	Mid-Aug
Trigonelline	9655.50 ± 384.06	9099.89 ± 211.46	9131.70 ± 422.03	9337.50 ± 354.15	Mid-July
	9682.93 ± 424.77	9096.21 ± 386.49	9674.59 ± 646.21	8931.38 ± 215.79	Early Aug
	9982.94 ± 311.38	9868.87 ± 310.29	9678.77 ± 173.28	9580.68 ± 247.72	Mid-Aug
Caffeine <sup>a</sup>	8070.70 ± 201.25 <sup>B</sup>	8127.50 ± 207.23 <sup>B</sup>	7806.27 ± 137.73 <sup>B</sup>	8165.46 ± 471.50 <sup>B</sup>	Mid-July
	8429.77 ± 308.93 <sup>B</sup>	7997.64 ± 382.23 <sup>B</sup>	8317.77 ± 642.36 <sup>B</sup>	7867.57 ± 277.76 <sup>B</sup>	Early Aug
	8986.72 ± 185.59 <sup>A</sup>	9006.92 ± 376.16 <sup>A</sup>	8799.83 ± 200.08 <sup>A</sup>	8984.66 ± 436.55 <sup>A</sup>	Mid-Aug

<sup>A, B</sup> Values with different capital letters in superscript indicate which treatment was different for each compound analyzed (p < 0.05).

<sup>a</sup> Compound with significant differences due to the harvest time

<sup>b</sup> compound with significant differences due to the drying type

**Table 1.** Alkaloids and phenolic compounds composition in green coffee beans (µg/g).

obtained at zero and maximum airflow and in samples of the last harvest (mid-August).

Chlorogenic acids are the most abundant phenolic compounds in coffee samples<sup>40–42</sup>. In general, the contents of 3-CQA (26250.39 - 31912.57 µg/g), 4-CQA (3861.64 - 5044.66 µg/g) and 5-CQA (2744.60 - 4054.43 µg/g) did not differ considerably among experimental samples. The results for 3-CQA and 4-CQA were within the broad ranges reported in another study (4930 - 149200 µg/g and 7020 - 101900 µg/g, respectively), while our results for 5-CQA were markedly lower than previous reports (57330 - 263600 µg/g)<sup>4,39,43–45</sup>. The significant variations among the concentrations of chlorogenic acids in coffee beans have been attributed to the application of different post-harvest processing<sup>46</sup>, environmental factors and genetic variability between coffee species<sup>45</sup>. Consequently, it has been suggested that more studies are required to elucidate the correlation between these factors and grain chemistry<sup>46</sup>.

The highest concentrations for alkaloids and phenolic compounds quantified corresponded to the last harvest time (mid-Aug), except for rutin and acid 3-CQA whose highest concentrations were for the first harvest time (mid-July). Concerning the drying method, the highest concentrations of compounds were obtained in samples dried at zero and maximum airflow.

The concentration of amino acids in green coffee samples is shown in Table 2. In total, 14 amino acids were quantified, from which proline was the most abundant (4931.48 - 8704.18 µg/g), followed by glutamic acid (852.10 - 2377.78 µg/g) and aspartic acid (274.22 - 1279.43 µg/g). The highest concentrations quantified for the amino acids corresponded to the samples harvested during mid-July and early August; meanwhile, the highest concentrations were obtained when samples were dried at zero airflows, except for glycine, phenylalanine, lysine and proline, whose highest concentrations corresponded to the drying at maximum airflow.

Different amino acid profiles have been previously reported. Wei & Tanokura, (2015)<sup>47</sup> reported 29 types of free amino acids in green coffee bean extracts, where aspartic acid, glutamic acid, serine, glycine, valine, phenylalanine and lysine were similar to our results. In contrast, the concentrations of histidine, threonine and alanine were higher, while arginine, isoleucine, leucine and proline were lower to those determined in the present study. In Casal *et al.*, (2003)<sup>48</sup>, the concentrations of glycine and proline were lower, whereas leucine concentrations were higher than our results. In Lee *et al.* (2017)<sup>49</sup>, lower concentrations of glutamic acid, glycine and proline, and high amounts of serine and valine were reported. Other studies reported similar proline, aspartic acid and glutamic acid concentrations but higher concentrations for the other amino acids<sup>19,50</sup>. Dong *et al.* (2017)<sup>20</sup> suggest that composition variations in coffee can be attributed to factors such as coffee variety<sup>50,51</sup>, geographical origin<sup>51</sup> or analytical methods<sup>52</sup> used. Regarding drying methods, Kulapichitr *et al.*, (2019)<sup>19</sup> evaluated different drying methods in Thai coffee beans. Samples subjected to heat pump drying presented slightly higher levels of most amino acids and no differences at higher temperatures. Tray drying showed a moderate effect on amino acids compared to sun drying, possibly due to shorter drying times.

The volatile compounds identified in the coffee beans after standardized roasting are presented in Table 3. A total of 20 volatile compounds, including pyridines, pyrazines, alcohols, pyrroles, cyclohexanes, aldehydes, furans, and ke-

tones, were identified, similarly as reported elsewhere<sup>24,53</sup>. The highest concentrations of volatile compounds were obtained for samples harvested during mid-August and dried under zero airflow. No significant differences among experimental treatments were determined for the aromatic compounds.

Volatile compounds profile depends on a series of factors such as the species and variety grain, geographical origin, soil conditions and grains storage, as well as the time and temperature of the roasting process, among others<sup>20,54</sup>. Cheong *et al.* (2013)<sup>53</sup> determined volatile sulfur compounds in roasted coffee, which impacted the sensory evaluation. Additionally, furans, pyrazines, pyridines, pyrroles and furanone were the main contributors to the roasted coffee aroma. Amanpour & Selli (2016)<sup>54</sup>, mainly furans and lactones, followed by pyrazines, pyridines, acids, cyclopentane, pyrroles, furanone, ketones, thiols, alcohols, aldehydes, among others, were identified. Lee *et al.* (2017)<sup>49</sup> evaluated the effect of the reverse process of grinding and roasting coffee on volatile compounds profiles, identifying 50 compounds in coffee roasted with the conventional method and 39 using the reverse method of grinding and roasting. To Laukaleja *et al.* (2019)<sup>55</sup>, main volatile compounds were furans, pyrazines, aldehydes and ketones, attributing to the last three an association with a pleasant aroma and flavor in specialty coffees. Heo *et al.* (2020)<sup>56</sup> identified 36 volatile compounds, highlighting that the extraction method and temperature could influence the volatile compound profiles.

#### **Influence of drying processes and harvest time on coffee beans chemical composition**

In this study, the content of several aroma-related compounds from specialty coffee samples harvested in different periods and subjected to other drying processes was assessed (Table 4). The harvest time significantly influenced all phenolic compounds' composition ( $p < 0.05$ ), except chlorogenic acid (3 CQA). Harvest time also influenced on the content of the amino acids serine ( $p = 0.003$ ), arginine ( $p = 0.009$ ), phenylalanine ( $p = 0.0004$ ) and leucine ( $p = 0.00006$ ) and the only one aromatic compound cyclohexane ( $p = 0.04$ ). Drying type significantly influenced on the concentration of rutin ( $p = 0.03$ ), trigonelline ( $p = 0.04$ ) and the amino acids glycine ( $p = 0.04$ ), alanine ( $p = 0.01$ ), valine ( $p = 0.02$ ) and isoleucine ( $p = 0.01$ ).

Experimental treatments that significantly differed were identified (Tables 1 & 2). For polyphenols, the quantified concentrations of rutin, 4-CQA and 5-CQA of the coffee beans collected in early August were statistically lower than the other harvest periods. For apigenin and caffeine, concentrations in the coffee beans collected in mid-August were significantly higher than in the other harvest periods. For amino acids, concentrations of aspartic acid were quite different among all harvest periods, obtaining the highest concentration in samples collected mid-July. Serine and arginine concentrations in samples collected in early August were significantly lower than those obtained from other harvest periods. For phenylalanine, statistically lower concentrations were observed in samples collected in mid-August. For histidine and threonine, significantly lower concentrations were obtained in coffee samples subjected to minimal aeration.

The combined influence of the experimental variables was explored by constructing a PCA based on the biocompounds whose simple linear regressions were statistically significant. Those compounds were one polyphenol (apige-

Compound	Drying Type				Harvest time
	Minimum airflow	Medium airflow	Zero airflow	Maximum airflow	
Aspartic acid <sup>a</sup>	773.34 ± 81.96 <sup>A</sup>	727.73 ± 27.38 <sup>A</sup>	885.46 ± 341.29 <sup>A</sup>	691.61 ± 46.64 <sup>A</sup>	Mid-July
	432.16 ± 137.59 <sup>C</sup>	472.54 ± 20.74 <sup>C</sup>	463.04 ± 28.64 <sup>C</sup>	510.59 ± 54.87 <sup>C</sup>	Early Aug
	563.79 ± 113.41 <sup>B</sup>	595.94 ± 51.10 <sup>B</sup>	661.28 ± 40.21 <sup>B</sup>	623.46 ± 41.47 <sup>B</sup>	Mid-Aug
Glutamic acid	1468.28 ± 153.40	1441.29 ± 17.25	1773.14 ± 525.93	1509.35 ± 68.51	Mid-July
	1314.31 ± 402.06	1315.07 ± 27.40	1475.98 ± 63.29	1261.42 ± 102.67	Early Aug
	1478.32 ± 228.86	1494.14 ± 104.87	1458.12 ± 138.63	1571.22 ± 77.67	Mid-Aug
Serine <sup>a</sup>	199.35 ± 16.79 <sup>A</sup>	201.47 ± 8.39 <sup>A</sup>	252.17 ± 72.90 <sup>A</sup>	202.10 ± 13.80 <sup>A</sup>	Mid-July
	153.29 ± 41.44 <sup>B</sup>	171.99 ± 5.81 <sup>B</sup>	187.21 ± 2.57 <sup>B</sup>	172.79 ± 14.35 <sup>B</sup>	Early Aug
	184.89 ± 35.56 <sup>AB</sup>	214.72 ± 11.23 <sup>AB</sup>	205.70 ± 15.59 <sup>AB</sup>	223.15 ± 22.17 <sup>AB</sup>	Mid-Aug
Histidine <sup>b</sup>	53.64 ± 1.68 <sup>B</sup>	58.78 ± 5.71 <sup>AB</sup>	71.18 ± 24.51 <sup>A</sup>	55.22 ± 6.29 <sup>A</sup>	Mid-July
	39.37 ± 34.54 <sup>B</sup>	58 ± 3.53 <sup>AB</sup>	65.61 ± 9.41 <sup>A</sup>	65.23 ± 6.43 <sup>A</sup>	Early Aug
	42.13 ± 36.69 <sup>B</sup>	63.85 ± 3.42 <sup>AB</sup>	60.30 ± 3.83 <sup>A</sup>	71.10 ± 11.56 <sup>A</sup>	Mid-Aug
Glycine	831.82 ± 79.48	981.23 ± 19.45	921.54 ± 81.50	918.38 ± 11.68	Mid-July
	829.50 ± 282.14	898.31 ± 130.54	1067.46 ± 117.47	1008.13 ± 75.54	Early Aug
	566.69 ± 494.58	963.98 ± 146.08	946.59 ± 37.51	1034.10 ± 174.27	Mid-Aug
Threonine <sup>b</sup>	12.34 ± 10.76 <sup>B</sup>	16.93 ± 1.44 <sup>AB</sup>	26.77 ± 6.99 <sup>A</sup>	20.27 ± 1.84 <sup>AB</sup>	Mid-July
	12.94 ± 11.23 <sup>B</sup>	23.08 ± 1.26 <sup>AB</sup>	24.77 ± 2.33 <sup>A</sup>	23.26 ± 2.04 <sup>AB</sup>	Early Aug
	2.017 ± 21.63 <sup>B</sup>	12.95 ± 11.24 <sup>AB</sup>	16.68 ± 1.12 <sup>A</sup>	17.06 ± 1.03 <sup>AB</sup>	Mid-Aug
Arginine <sup>a</sup>	182.24 ± 17.72 <sup>AB</sup>	242.87 ± 5.10 <sup>AB</sup>	294.33 ± 117.60 <sup>AB</sup>	227.13 ± 20.16 <sup>AB</sup>	Mid-July
	229.43 ± 85.08 <sup>B</sup>	221.62 ± 29.74 <sup>B</sup>	244.66 ± 29.88 <sup>B</sup>	207.65 ± 37.31 <sup>B</sup>	Early Aug
	265.73 ± 35.24 <sup>A</sup>	308.85 ± 21.06 <sup>A</sup>	278.13 ± 72.15 <sup>A</sup>	345.76 ± 85.97 <sup>A</sup>	Mid-Aug
Alanine	206.91 ± 14.22	227.48 ± 3.28	296.57 ± 95.80	222.55 ± 5.71	Mid-July
	203.93 ± 54.86	276.76 ± 15.05	304.15 ± 10.96	272.08 ± 19.45	Early Aug
	240.20 ± 40.45	277.48 ± 10.32	239.69 ± 16.90	288.61 ± 38.15	Mid-Aug
Valine	54.28 ± 3.09	60.25 ± 0.50	72.07 ± 24.75	57.21 ± 2.87	Mid-July
	52.67 ± 14.36	67.81 ± 2.65	77.95 ± 1.66	69.64 ± 2.45	Early Aug
	61.17 ± 7.68	68.37 ± 3.20	61.28 ± 2.73	70.40 ± 7.72	Mid-Aug
Phenylalanine <sup>a</sup>	143.56 ± 14.19 <sup>A</sup>	141.12 ± 21.42 <sup>A</sup>	172.20 ± 69.90 <sup>A</sup>	153.59 ± 6.12 <sup>A</sup>	Mid-July
	138.05 ± 43.99 <sup>A</sup>	172.39 ± 20.13 <sup>A</sup>	170.91 ± 5.20 <sup>A</sup>	185.80 ± 4.60 <sup>A</sup>	Early Aug
	116.97 ± 23.80 <sup>B</sup>	124.41 ± 17.17 <sup>B</sup>	106.39 ± 6.80 <sup>B</sup>	116.88 ± 7.11 <sup>B</sup>	Mid-Aug
Isoleucine	43.21 ± 2.08	47.44 ± 0.94	57.58 ± 19.34	45.31 ± 2.23	Mid-July
	42.39 ± 12.04	55.67 ± 2.51	64.18 ± 2.45	58.42 ± 1.57	Early Aug
	48.56 ± 6.79	55.50 ± 2.74	49.60 ± 1.54	55.28 ± 5.51	Mid-Aug
Leucine	29.55 ± 0.77	35.70 ± 0.43	41.74 ± 12.91	34.29 ± 3.22	Mid-July
	34.92 ± 10.12	47.00 ± 2.50	57.50 ± 2.79	49.10 ± 2.32	Early Aug
	39.10 ± 3.25	44.28 ± 2.33	37.15 ± 2.18	44.51 ± 4.78	Mid-Aug
Lysine	27.41 ± 47.47	80.89 ± 6.72	26.77 ± 46.37	87.96 ± 6.85	Mid-July
	67.77 ± 59.43	67.07 ± 58.09	105.28 ± 8.20	91.27 ± 5.22	Early Aug
	32.41 ± 56.13	68.65 ± 59.52	89.11 ± 13.64	96.64 ± 11.34	Mid-Aug
Proline	7510.47 ± 1035.40	7169.54 ± 416.42	7248.46 ± 1090.10	6838.75 ± 1452.58	Mid-July
	6393.97 ± 1295.46	6652.94 ± 427.68	6717.53 ± 1031.98	6540.74 ± 1053.70	Early Aug
	5675.32 ± 735.74	7079.78 ± 583.75	6874.80 ± 1289.95	7847.51 ± 848.53	Mid-Aug

<sup>A,B,C</sup> Values with different capital letters in superscript indicate which treatment was different for each compound analyzed (p < 0.05).

<sup>a</sup> Compound with significant differences due to the harvest time

<sup>b</sup> compound with significant differences due to the drying type

**Table 2.** Amino acid composition in green coffee beans (µg/g).

nin), five amino acids (arginine, alanine, valine, isoleucine, and leucine), and 10 aromatics (2,3-pentanedione, carbon monoxide, butane, dihydro-2-methyl 3(2H)-furanone, 2-methylpyrazine, 2-furancarboxaldehyde, 2,6-dimethylpyrazine, 2-ethylpyrazine, 2-ethyl-6-methylpyrazine, and 2-ethyl-3-methylpyrazine). Three main components were defined and explained 73% of the variation of the data based on the harvest time (PCA1 39% and PCA2 24%).

Figure 1 shows a heat map of the variation of coffee bean composition according to the drying type and harvest time. Two groups were distinguished, one for amino acids and another for aromatic compounds, except for butane and

carbon monoxide. This suggested assembling early formed precursors and roasting products, discarding intermediate processes, such as ripening and drying, that were not significant when constructing the PCA model. These results may support the observed more considerable influence of the harvest period compared to the evaluated drying processes.

Several studies refer to the interaction of the chemical composition of coffee with genotype<sup>10</sup>, environment<sup>10</sup>, geographical origin<sup>97</sup>, climatic factors<sup>58</sup>, and processing<sup>10</sup>, among others. Kulapichitr *et al.* (2019)<sup>19</sup> evaluated the influence of heat pump drying, tray drying and sun drying on



Compound	Functional Group	Drying type			Harvest time
		Medium airflow	Zero airflow	Maximum airflow	
2,3-Pentanedione	Ketone	19.49 ± 16.65	18.39 ± 21.64	2.78 ± 0.12	Mid-July
		28.22 ± 5.93	2.53 ± 0.18	13.23 ± 12.94	Early Aug
		8.91 ± 6.20	2.01 ± 0.96	17.70 ± 20.83	Mid-Aug
Carbon monoxide	Carbonyl	10.41 ± 7.85	35.41 ± 39.48	63.82 ± 5.41	Mid-July
		5.80 ± 0.24	59.17 ± 7.37	15.80 ± 20.17	Early Aug
		26.36 ± 27.23	68.22 ± 1.74	13.96 ± 14.70	Mid-Aug
Butane	Ketone	17.97 ± 15.65	5.62 ± 3.54	1.21 ± 0.61	Mid-July
		4.34 ± 2.99	1.96 ± 0.87	7.49 ± 7.41	Early Aug
		3.68 ± 2.18	1.50 ± 0.82	3.86 ± 2.25	Mid-Aug
Pyridine	Pyridine	0.71 ± 0.19	1.16 ± 0.66	0.44 ± 0.03	Mid-July
		1.42 ± 0.29	0.54 ± 0.11	1.14 ± 1.39	Early Aug
		0.85 ± 0.03	0.45 ± 0.22	0.96 ± 0.38	Mid-Aug
Dihydro-2-methyl 3(2H)-Furanone	Furanone	0.64 ± 0.44	1.31 ± 0.82	0.62 ± 0.06	Mid-July
		1.70 ± 0.32	0.64 ± 0.04	0.67 ± 0.13	Early Aug
		1.42 ± 0.24	0.54 ± 0.23	1.00 ± 0.30	Mid-Aug
2-methyl pyrazine	Pyrazine	4.54 ± 0.27	4.57 ± 2.82	2.56 ± 0.48	Mid-July
		7.60 ± 0.19	2.26 ± 1.10	6.23 ± 3.20	Early Aug
		2.84 ± 0.38	1.75 ± 0.31	4.04 ± 0.97	Mid-Aug
2-Furancarboxaldehyde	Pyrazine	23.01 ± 23.43	7.72 ± 1.51	11.60 ± 6.70	Mid-July
		12.33 ± 10.82	15.07 ± 7.99	20.55 ± 5.66	Early Aug
		24.96 ± 13.28	10.43 ± 5.94	33.25 ± 14.71	Mid-Aug
Methylpyrazine	Furanone	0.33 ± 0.23	0.55 ± 0.32	0.17 ± 0.01	Mid-July
		0.83 ± 0.05	0.23 ± 0.09	0.90 ± 0.75	Early Aug
		0.48 ± 0.04	0.24 ± 0.08	0.44 ± 0.24	Mid-Aug
2-Furamethanol	Alcohol	1.81 ± 0.10	2.62 ± 1.26	1.59 ± 0.048	Mid-July
		3.93 ± 0.46	1.59 ± 0.03	2.36 ± 0.50	Early Aug
		2.60 ± 1.09	1.49 ± 0.40	2.83 ± 0.79	Mid-Aug
2-Propanone, 1-(acetyloxy)	Ketone	4.51 ± 0.91	4.02 ± 1.24	3.08 ± 0.69	Mid-July
		7.01 ± 2.29	4.49 ± 0.03	7.84 ± 3.87	Early Aug
		7.89 ± 7.71	3.01 ± 0.80	4.41 ± 2.10	Mid-Aug
2,6-Dimethyl pyrazine	Furan	4.12 ± 0.17	5.70 ± 2.92	2.62 ± 1.25	Mid-July
		7.14 ± 0.80	2.84 ± 0.95	3.17 ± 1.27	Early Aug
		5.13 ± 0.14	2.02 ± 0.09	4.50 ± 1.74	Mid-Aug
1-(2-furanyl)-ethanone	Pyrazine	4.82 ± 1.26	1.13 ± 0.37	1.58 ± 1.14	Mid-July
		5.07 ± 5.39	0.92 ± 0.003	6.93 ± 7.22	Early Aug
		3.18 ± 3.17	1.84 ± 1.55	1.33 ± 0.17	Mid-Aug
2-Ethylpyrazine	Pyrazine	0.49 ± 0.51	1.12 ± 0.87	1.48 ± 1.50	Mid-July
		0.76 ± 0.85	0.58 ± 0.12	3.19 ± 0.90	Early Aug
		0.73 ± 0.19	0.41 ± 0.01	0.65 ± 0.77	Mid-Aug
5-Methyl 2-Furancarboxaldehyde	Aldehyde	3.35 ± 0.35	5.14 ± 2.01	3.22 ± 0.87	Mid-July
		6.61 ± 0.72	3.45 ± 0.33	5.00 ± 2.59	Early Aug
		5.15 ± 0.37	2.81 ± 0.75	5.32 ± 0.97	Mid-Aug
2-Furamethanol, acetate	Alcohol	1.77 ± 0.36	2.93 ± 0.83	1.80 ± 0.32	Mid-July
		3.86 ± 0.99	1.94 ± 0.16	2.52 ± 0.86	Early Aug
		2.94 ± 0.25	1.75 ± 0.31	2.83 ± 0.98	Mid-Aug
2-Ethyl-6-methylpyrazine	Pyrazine	0.34 ± 0.07	0.39 ± 0.19	0.16 ± 0.10	Mid-July
		0.51 ± 0.09	0.27 ± 0.02	0.39 ± 0.06	Early Aug
		0.40 ± 0.03	0.23 ± 0.03	0.48 ± 0.06	Mid-Aug
2-Ethyl-3-methylpyrazine	Pyrazine	1.16 ± 0.12	1.54 ± 0.85	0.76 ± 0.09	Mid-July
		1.80 ± 0.52	0.99 ± 0.06	1.67 ± 0.78	Early Aug
		1.63 ± 0.18	0.89 ± 0.03	1.59 ± 0.81	Mid-Aug
1-methyl-4-(1-methylethenyl)-cyclohexane	Cyclohexane	0.11 ± 0.03	0.16 ± 0.05	0.12 ± 0.02	Mid-July
		0.26 ± 0.10	0.08 ± 0.02	0.19 ± 0.03	Early Aug
		0.17 ± 0.07	0.05 ± 0.04	0.20 ± 0.04	Mid-Aug
2-Ethyl-3,5-dimethylpyrazine	Pyrazine	0.25 ± 0.03	0.39 ± 0.15	0.21 ± 0.03	Mid-July
		0.45 ± 0.12	0.29 ± 0.03	0.43 ± 0.22	Early Aug
		0.40 ± 0.07	0.21 ± 0.01	0.44 ± 0.16	Mid-Aug
Pyrrole N-furfuryl	Pyrrole	0.19 ± 0.01	0.13 ± 0.09	0.18 ± 0.01	Mid-July
		0.37 ± 0.09	0.17 ± 0.01	0.30 ± 0.18	Early Aug
		0.27 ± 0.01	0.15 ± 0.001	0.21 ± 0.06	Mid-Aug

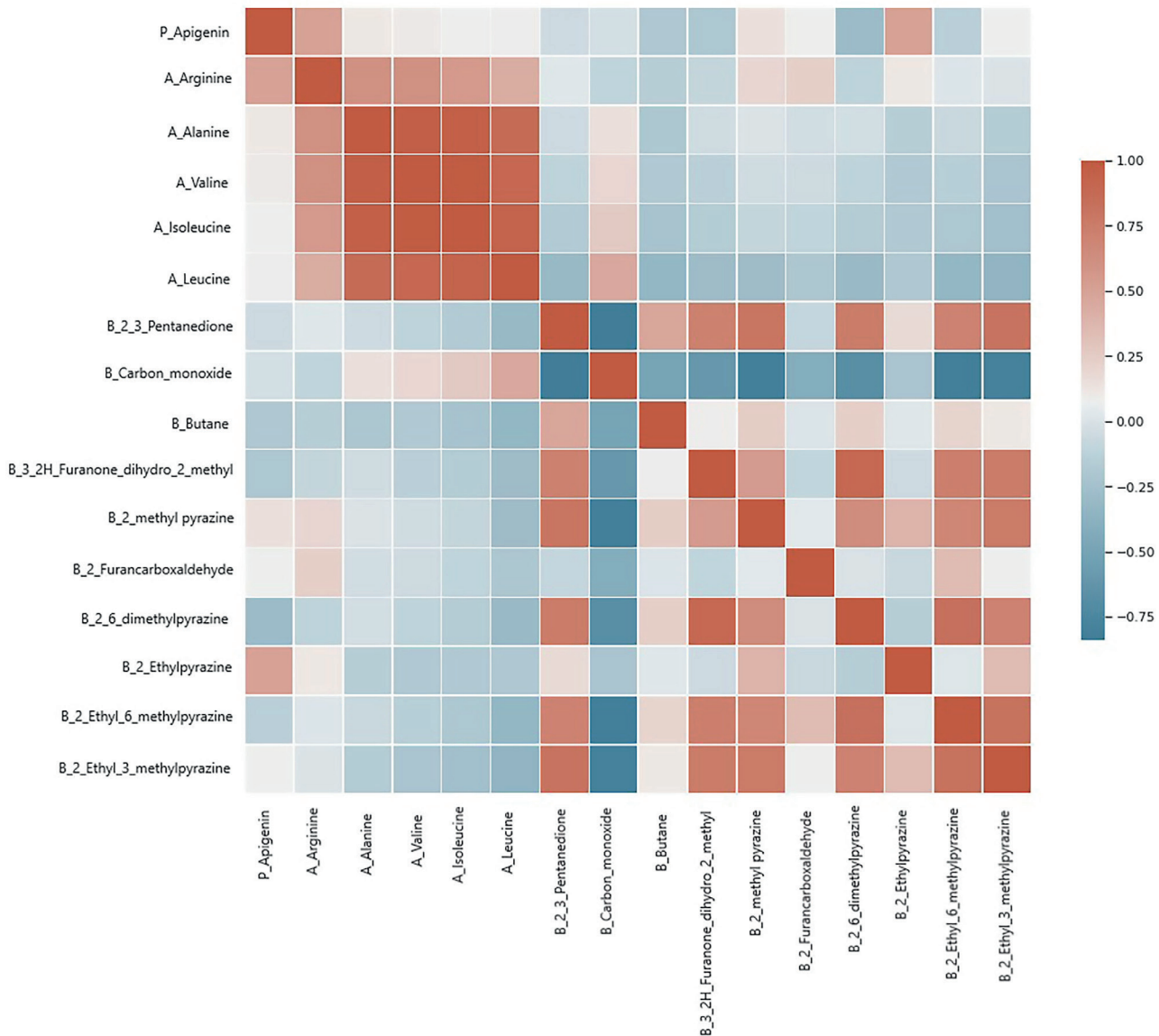
**Table 3.** Volatile compounds identified in roasted coffee beans (%).

Response Variables	p-values		
	Harvest period	Drying type	Interaction
<b>Polyphenols</b>			
Rutin	0.001*	0.031*	0.319
Epicatechin	0.004*	0.530	0.130
Apigenin	0.016*	0.637	0.377
Caffeic Acid	0.001*	0.081	0.002*
5 CQA	0.019*	0.3125	0.909
3 CQA	0.820	0.463	0.138
4 CQA	0.0004*	0.446	0.035*
Trigonelline	0.007*	0.041*	0.316
Caffeine	0.002*	0.539	0.451
<b>Amino acids</b>			
Aspartic acid	0.905	0.207	0.142
Glutamic acid	0.072	0.421	0.729
Serine	0.003*	0.098	0.542
Histidine	0.919	0.068	0.786
Glycine	0.643	0.035*	0.558
Threonine	0.489	0.306	0.539
Arginine	0.009*	0.374	0.375
Alanine	0.196	0.011*	0.098
Valine	0.260	0.020*	0.203
Phenylalanine	0.0004*	0.4584	0.5138
Isoleucine	0.092	0.011*	0.174
Leucine	0.00006*	0.244	0.227
Lysine	0.256	0.089	0.450
Proline	0.333	0.664	0.386
<b>Aromatic compounds</b>			
2,3-Pentanedione	0.765	0.332	0.317
Carbon monoxide	0.014*	0.610	0.065
Butane	0.287	0.351	0.262
Pyridine	0.295	0.366	0.058
3(2H)-Furanone, dihydro- 2-methyl-	0.092	0.749	0.045
2-methyl pyrazine	0.099	0.056	0.083
2-Furancarboxaldehyde	0.284	0.429	0.537
Methylpyrazine	0.481	0.210	0.229
2-Furamethanol	0.139	0.343	0.056
2-Propanone, 1- (acetyloxy)	0.384	0.398	0.748
2,6-dimethyl pyrazine	0.816	0.047	0.046
Ethanone	0.307	0.495	0.629
2-Ethylpyrazine	0.059	0.182	0.178
2-Furancarboxaldehyde, 5-methyl	0.274	0.341	0.086
2-Furamethanol, acetate	0.250	0.311	0.051
2-Ethyl-6-methylpyrazine	0.095	0.201	0.029
2-Ethyl-3-methylpyrazine	0.437	0.542	0.262
Cyclohexane	0.040*	0.292	0.063
2-Ethyl-3,5- dimethylpyrazine	0.512	0.321	0.181
Pyrrole N-Furfuryl	0.079	0.058	0.755

\*Significant different ( $p > 0.05$ ) by two-way ANOVA

**Table 4.** Individual influence of the drying type and harvest time on the chemical composition of green coffee beans, evaluated by analysis of variance.





**Figure 1.** Heat map for coffee samples subjected to different drying airflow types and different harvest times.

coffee's chemical composition. The drying process did not affect the caffeine content, but it influenced the concentration of histidine, as in the present study. In addition, significant differences were observed for aspartic acid and phenylalanine. Significant differences among drying methods in several compounds were described for the aromatic compounds. Green coffee beans subjected to heat pump drying presented slightly higher levels of most amino acids<sup>19</sup>. Heat pump and tray drying shared the same profile and compound content, while both differed from the composition of sun-dried coffee samples<sup>19</sup>. Tolessa *et al.*, (2017)<sup>59</sup> evaluated the influence of growing altitude, shade and harvest period on Ethiopian specialty coffee's quality and biochemical design.

It was determined that beans harvested at early and middle harvest periods were generally higher in cup quality compared to late-harvested beans. Interactions among altitude, shade and harvest periods were significant for caffeine content. The highest caffeine content (17.9 g Kg<sup>-1</sup>) was obtained in early harvested beans at middle altitude with dense shade. In comparison, the lowest range (14.5 g Kg<sup>-1</sup>) was observed in middle-harvested beans from high altitudes with the medium shade. No interactions were found for

the total chlorogenic acids content of coffee beans. Laderach *et al.* (2011)<sup>60</sup> evaluated two harvest times and their correlation with the sensory properties of coffee harvested in two Colombian states and two Mexican farms. They found significant differences in the sensory attributes of the coffee and determined that for an early harvest, most characteristics score higher except for aroma/fragrance, body and sweetness. Final cupping scores for early and late harvest coffees were 77.8 points and 72.6 points, respectively. Jeszka-Skowron *et al.*, (2016)<sup>43</sup>, evaluated the concentration of chlorogenic acids and caffeine in coffee beans from different geographical origins of the Arabica and Robusta varieties. They determined that there were no significant differences between the contents of caffeine and chlorogenic acids (3-CQA, 4-CQA and 5-CQA) of the coffee collected from different geographical origins for the Arabica variety. However, for the Robusta variety, there were significant differences in the contents of these compounds. Scarce information about associations between green coffee beans' chemical compositions and the harvest period has been published.

Other studies have evaluated the relationship between coffee chemical composition and drying conditions. In Dong

*et al.* (2017)<sup>20</sup>, five drying conditions were assessed, and, by PCA, it was determined that both hot air-dried and cold-dried samples were located in the positive direction of PC1. In contrast, the models treated with the heat pump drying, solar drying, and room temperature drying methods were close to each other and in the negative PC1 direction, indicating that the different drying methods influence coffee chemical compounds' content. In addition, solar drying significantly influenced caffeine and trigonelline content; meanwhile, freeze-drying and heat-pump drying significantly influenced the concentration of amino acids and volatile compounds, respectively<sup>20</sup>.

Other factors before harvest, such as the species, cultural practices, fertilization, temperature and altitude, can influence the quality of the coffee cup. According to Bastian *et al.*, (2021)<sup>61</sup>, the quality of coffee beverages is affected by the ripening time of the fruit, which is also related to geographic and climate conditions<sup>62</sup>. Velásquez & Banchón, (2022)<sup>63</sup> mentioned that climatic changes where there are heat waves and droughts directly affect the production of Arabica coffee due to its greater sensitivity to climatic changes. Overall, the association of different variables throughout the pre-and post-harvest coffee processing should continue being explored towards defining predictor variables for coffee classification, particularly for high-quality coffee due to its sensitiveness.

### Sensory analysis results

The drink quality, given by its sensory attributes, is the main characteristic that differentiates specialty coffee from regular coffee<sup>46,64</sup>. In this study, drink quality was subjected to sensorial panels only considering the drying type since it was the less sensitive variable related to the coffee chemical composition. The sensory scores given by professional tasters are shown in Table 5. According to the SCAA, specialty coffee must present a final sensory score greater than or equal to 80 out of 100<sup>65</sup>. This narrow scoring is scale-based, which reduces the possibility of quantitative analysis. A proposal to match sensory attributes with the drying type was modeled by Random Forest analysis, through which the drying type could be predictable only within a cupping range between 8 and 9.5.

The primary tasting descriptors were the balance and the general tasting value—figure 2. A shows the results obtained for samples dried under minimum airflow, in which the most representative descriptors were balance, flavor

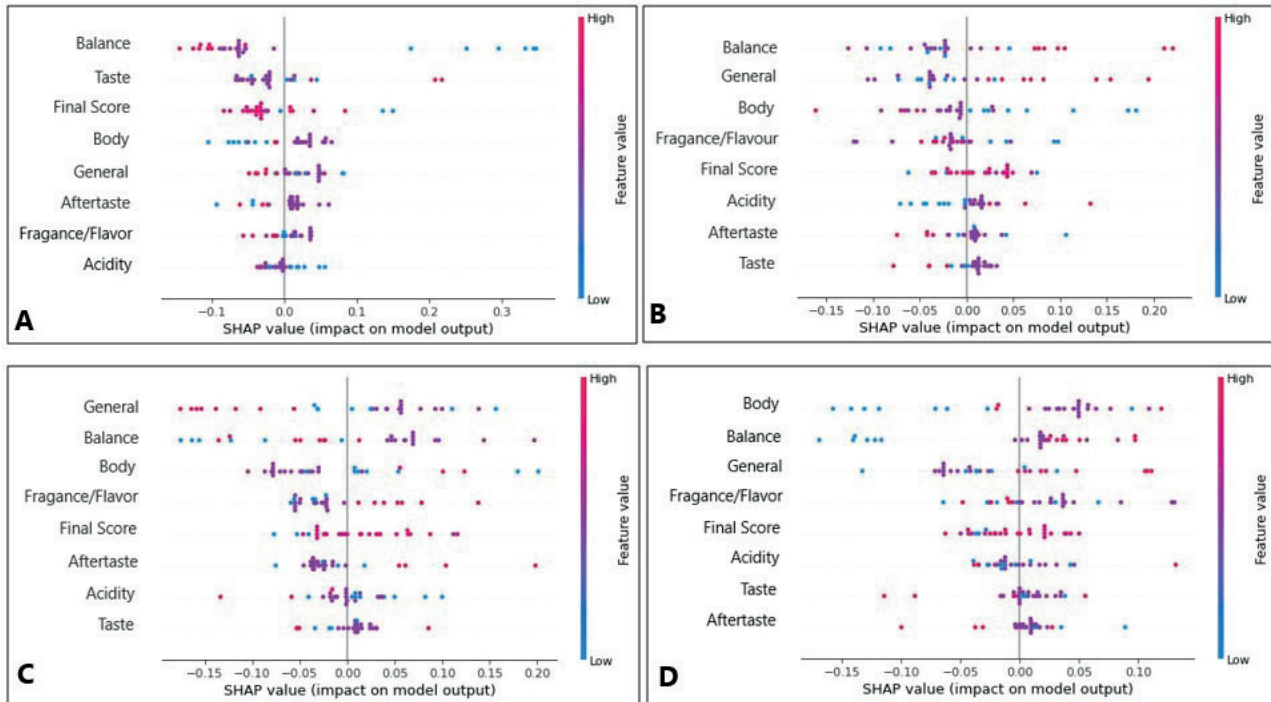
and final score. A low balance and high flavor values characterized drying at minimum airflow. Figure 2.B presents the model for drying at medium airflow. This drying type was defined as a tasting result with low body values, high balance and high general tasting values. For drying at zero airflows, balance, available tasting value and body were the most relevant descriptors without defining any trend since the medium and low values were mixed (Figure 2.C). This drying type could only be defined with high aroma values. Similarly, for drying at maximum airflow, no clear trend of cupping descriptors was observed (Figure 2.D).

These results suggested an association between the drying at a minimum and medium airflow with the final tasting. In contrast, extreme drying conditions, i.e., zero and maximum airflow, could trigger diverse metabolic processes that result in a mixture of tasting characteristics.

Previous studies evaluate the sensory analysis relationship of coffee with the processing type. Wet processing requires large amounts of water<sup>66</sup> and involves the mechanical depulping of coffee cherries, which removes most of the bean flesh<sup>61</sup>. What is obtained is parchment coffee surrounded by mucilaginous residues, which are degraded through fermentation in water pools that cover the coffee beans entirely for a certain period; the final product is a "washed" or "parchment" coffee<sup>67</sup>. This method is widely used in Arabica coffee<sup>61</sup>. Pinto *et al.*, (2013)<sup>68</sup> established that for the sensory attributes evaluated in their study (drink clarity, acidity, body, flavor, aftertaste, balance, general value and final score) there was a significant difference between the treatments, being the wet superior to dry processing coffees with final scores between 82.93-82.95 and 78.12-75.65, respectively. Rodríguez *et al.* (2020)<sup>69</sup> determined that post-harvest coffee processing did not affect the total cup score, obtaining a mean value for the semi-dry processing method of  $85.94 \pm 0.57$ , while for wet processing, it was  $84.13 \pm 0.42$ . In addition, none of the attributes analyzed individually for final tasting was significantly different between both processes; however, they noted slightly better values for fragrance/aroma, aftertaste, acidity, and body attributes for the wet processing. They also observed that uniformity, balance, clean cup, and sweetness parameters increased the overall rating. Ribeiro *et al.* (2016)<sup>10</sup> obtained results contrary to the studies mentioned above, where the significantly highest average values final score were observed in dry-processed coffee, with a value of 85.57 vs. 84.61 obtained with the wet method.

Attributes	Drying Method (Mean ± SD)			
	Minimum airflow	Medium airflow	Zero airflow	Maximum airflow
<b>Fragrance/Flavor</b>	8.04 ± 0.17	7.96 ± 0.17	8.00 ± 0.20	8.04 ± 0.17
<b>Taste</b>	8.00 ± 0.20	7.96 ± 0.09	8.04 ± 0.09	8.00 ± 0.14
<b>Aftertaste</b>	8.00 ± 0.14	7.96 ± 0.09	8.04 ± 0.17	8.00 ± 0.14
<b>Acidity</b>	7.86 ± 0.13	7.96 ± 0.17	7.96 ± 0.17	8.04 ± 0.22
<b>Body</b>	7.93 ± 0.12	7.89 ± 0.13	8.00 ± 0.20	8.00 ± 0.14
<b>Balance</b>	7.86 ± 0.24	8.07 ± 0.19	8.00 ± 0.14	8.14 ± 0.20
<b>General</b>	7.89 ± 0.24	8.07 ± 0.19	7.96 ± 0.17	8.07 ± 0.19
<b>Final Score</b>	85.54 ± 0.99	85.89 ± 0.66	86.07 ± 0.28	86.11 ± 0.24

**Table 5.** Sensory analysis scores of coffee samples by type of drying.



**Figure 2.** (A) Tasting parameters with the greatest influence on samples subjected at minimum airflow drying, (B) Tasting parameters with the greatest influence on samples subjected at medium airflow drying, (C) Tasting parameters with the greatest influence on samples dried without airflow, and (D) Tasting parameters with the greatest influence on samples subjected at maximum airflow drying.

## Conclusions

In this study, the influence of the different drying processes and harvest periods on the quality of specialty green bean coffee was evaluated through the characterization of aroma-responsible compounds and their relationship with the tasting attributes of coffee drinks. Four drying types and three harvest times were considered for 36 coffee bean samples. The techniques for extracting and analyzing chemical compounds from coffee were optimized. The concentrations of polyphenols, amino acids and aromatic compounds were quantified and some differed from those reported in the literature, attributing to a series of factors such as geographical origin, environmental factors, agricultural practices, grain species and variety, post-harvest treatments, type of roasting, among others. Slight differences were established in chemical compound content concerning harvest periods, but these differences were not significant. The drying type did not significantly influence the bio compounds concentration determined in specialty coffee. Therefore, both the harvest period and the drying type alone cannot be considered predictive scale variables that explain the sensory differences of specialty coffee. It is suggested to analyze the entire production process of specialty coffee to define other variables that adequately explain these differences in composition for the chemical compounds of coffee. Tasting according to drying type allowed to establish a predictive model, particularly for drying types with minimum and medium airflow, as long as the tasting values are within a range of 8-9.5. Considering that cupping scores for specialty coffee should be high, it is suggested to create an internal cupping scale to obtain a broader cupping range, allowing a more sensitive evaluation of processing variables and even chemical coffee composition.

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## Conflicts of Interest

The author(s) declare that they have no competing interests.

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