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Quality attributes of roasted Arabica coffee oil extracted by pressing: composition, antioxidant activity, sun protection factor and other physical and chemical parameters

B.R. Böger[a] ✉, A.L.B. Mori[a], M.C. Viegas[b] and M.T. Benassi[a]

[a]Department of Food Science and Technology, Universidade Estadual de Londrina, Rodovia Celso Garcia Cid (PR 445), Km 380, 86057-970 Londrina, Brazil

[b]Research & Development, Companhia Iguacu de Café Solúvel, Rodovia Mello Peixoto (BR 369), Km 88, 86300-000, Cornelio Procopio, Brazil

✉ Corresponding author: brunaraquel12@gmail.com

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SUMMARY: This research reports a comprehensive characterization of the composition profile and physical and chemical characteristics of roasted Arabica coffee oil obtained by mechanical pressing. The oil presented a peroxide value of 3.21 meq·kg^{-1} and an acid value of 7.3 mg KOH·g^{-1}. A higher proportion of unsaturated fatty acids (58%), predominantly linoleic (L) and palmitic (P) acids, was observed; PLL and PLP were estimated as the main triacylglycerols. The oil was characterized by high contents in diterpenes and tocopherols (3720 and 913 mg·100g^{-1}, respectively), the presence of caffeine and chlorogenic acids, as well as a high sun protection factor (9.7) and ABTS free radical-scavenging capacity (12.5 mg Trolox·mL^{-1}). Among the 35 volatile compounds studied, furfurythiol and pyrazines were the main components of the oil. These properties showed that roasted coffee oil has good potential for use in food and cosmetics.

KEYWORDS: *Coffee Arabica; Diterpenes; Tocopherols; Volatile compounds*

RESUMEN: ***Atributos de calidad del aceite de café Arábica tostado extraído por prensado: composición, actividad antioxidante, factor de protección solar y otros parámetros físicos y químicos.*** Esta investigación reporta una caracterización completa del perfil de composición y características físicas y químicas del aceite de café Arábica tostado obtenido por prensado mecánico. El aceite presentó un índice de peróxido de 3,21 meq·kg^{-1} y un índice de acidez de 7,3 mg de KOH·g^{-1}. Se observó una mayor proporción de ácidos grasos insaturados (58%), ácido linoleico, (L) y palmítico (P); PLL y PLP se estimaron como los principales triacilgliceroles. El aceite se caracterizó por un alto contenido de diterpenos y tocoferoles (3720 y 913 mg·100g^{-1}, respectivamente), la presencia de cafeína y ácidos clorogénicos, así como un alto factor de protección solar (9,7) y capacidad de captación de radicales libres ABTS (12,5 mg de Trolox·mL^{-1}). Entre los 35 compuestos volátiles estudiados, el furfuritiol y las pirazinas fueron los componentes principales del aceite. Estas propiedades mostraron que el aceite de café tostado tiene un buen potencial para su uso en alimentos y cosméticos.

PALABRAS CLAVE: *Café Arabica; Compuestos volátiles; Diterpenos; Tocoferoles*

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1. INTRODUCTION

Coffee is one of the most popular beverages worldwide. In the past 10 years, global coffee production has grown at an average annual rate of around 2.6% from 140.16 million 60-kg bags in 2010/11 to an estimated 168.71 million 60-kg bags in 2019/20. Brazil is the world's second-largest coffee consumer. In addition to being the main producer (57 million in 2019/20), in 2019 Brazil was also the world's largest exporter (37.7 million up to November), and soluble coffee represented around 10% of this total (Ico, 2019).

The mechanical pressing of coffee beans, green (raw) or roasted, is the most common industrial methods for oil extraction in Brazil (Oliveira *et al.,* 2005). Iy is eco-friendly, and does not require the use of any solvents. Roasted coffee oil is a co-product of the soluble coffee industry, and can be obtained by pressing the roasted beans before extraction of the soluble coffee. The roasted coffee oil is applied as a food flavoring, while green coffee oil is used in cosmetic formulations due to its antioxidant, emollient and UV protection properties (Calligaris *et al.,* 2009; Wagemaker *et al.,* 2011; Hurtado-Benavides *et al.,* 2016).

Lipids are among the most abundant coffee components, accounting for 3.2 to 11% of the total green beans and 8.6 to 17% of the roasted coffee. The increase in lipid content with the roasting process is due to losses in CO_2, water vapor and volatile compounds, and the degradation of carbohydrates, amino acids, and chlorogenic acids (Budryn *et al.,* 2012; Dias *et al.,* 2014; Pacetti *et al.,* 2015). Owing to their relatively high thermal stability, lipids protect aromatic compounds from degradation (Wagemaker *et al.,* 2011). The lipid fraction contains the majority of the volatile compounds responsible for the aroma (Calligaris *et al.,* 2009; Wagemaker *et al.,* 2011; Hurtado-Benavides *et al.,* 2016). It also contributes to coffee brew viscosity (Pacetti *et al.,* 2015). Triacylglycerols are the main components of coffee oil (about 75%), which also presents from 15 to 18% of the unsaponifiable matter (UM) (Speer and Kölling-Speer, 2006), composed of hydrocarbons, steroids, and tocopherols (Belitz *et al*., 2009). It has a high proportion of UM compared to other vegetable oils (0.2 - 1.5 %), such as soybean (from 0.6 to 1.2%), olive (from 0.4 to 1.1%), and sunflower (from 0.3 to 1.2 %) (Belitz *et al*., 2009).

Coffee oil composition varies with harvesting and post-harvest handling practices, bean origin and genetics (species and varieties), as well as roasting and extraction conditions (Pacetti *et al.,* 2015). The literature reports some data on coffee oil from the Arabica and Robusta species, although the majority of them are related to green coffee - extracted with solvents or more sophisticated methods (such as supercritical extraction). In general, researches focus on specific classes of compounds, such as fatty acids and volatile compounds (Oliveira *et al.,* 2005; Calligaris *et al.,* 2009; Budryn *et al.,* 2012; Getachew and Chun, 2016; Hurtado-Benavides *et al.,* 2016; Raba *et al.,* 2018). Less information is available on UM compounds –such as diterpenes and tocopherols– and on the presence of hydrosoluble components, which could be carried during pressing, such as caffeine and chlorogenic acids (González *et al.,* 2001; Oliveira *et al.,* 2014; Guercia *et al.,* 2016; Bitencourt *et al.,* 2018).

Regarding the physico-chemical characteristics, which are essential for technological use, some studies focus on quality indices such as the peroxide value (Turatti, 2001; Budryn *et al.,* 2012; Getachew and Chun, 2016) and thermal properties (Calligaris *et al.,* 2009; Budryn *et al.,* 2012; Raba *et al.,* 2018), generally correlating these parameters with the fatty acid profile.

Considering the interest and potential use of roasted coffee oil as a food ingredient as well as in the cosmetic area, where green coffee oil is more common nowadays, this study aimed to report a comprehensive characterization of the composition profile and properties of roasted Arabica coffee oil obtained by mechanical pressing.

2. MATERIALS AND METHODS

2.1. Materials

The coffee oil was supplied by Company Iguaçu Soluble Coffee (Cornélio Procópio, Brazil). Commercial dry Arabica coffee beans (4.5 to 5.0% w/w moisture) were medium roasted at 220 °C (air temperature) and 5 mbar for 10 to 12 min. The extraction was carried out at room temperature by cold pressing in an oil expeller SCOTTECH ERT 50 (Scott Tech USA, USA); the coffee reached a maximum of 60 °C during the process. The efficiency of the extraction was around 5 to 6% of oil (w/w). The oil was kept in a freezer at -22 °C until analysis.

2.1.1. Reagents and standards

The HPLC-grade solvents were *tert*-butyl methyl ether (Acros Organics, USA), acetonitrile (Mallinckrodt Baker, USA), and methanol (Merck, Germany). The following reagents and analytical grade materials were also used: potassium hydroxide (Quimex, Brazil), ethanol 98% (JTBaker, Mexico), sulfuric acid 95-97% (Merck, Germany), hydrochloric acid (Quimex, Brazil), sodium hydroxide (Sigma-Aldrich, USA), Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) (Sigma-Aldrich, USA), acetic acid (Merck, Germany), ABTS (2,2-azino-bis-3-ethylbenzothiazoline-6-sulfonic acid) (Sigma-Aldrich, USA), potassium persulfate (Anidrol, Brazil), ethyl acetate (Sigma-Aldrich, USA), sodium thiosulphate (Synth, Brazil),Wijs solution (Anidrol, Brazil), potassium iodide (Synth, Brazil), carbon tetrachloride (Dinâmica, Brazil) and chloroform (Synth, Brazil).The water used to prepare standards and solutions was obtained by a purification system Elga Purelab Option-Q (Veolia Water Technologies, France). Nylon membranes were applied for filtration of solvents (Millipore, USA) and samples (0.22 µm) (Whatman, UK). Standards of 5-caffeoylquinic acid (5-CQA), caffeine, fatty acid methyl esters (FAME Mix C4-C24) and tocopherols (α, β, γ, and δ) (Sigma-Aldrich, USA), and cafestol and kahweol (Axxora, USA) were used. For the volatile profile, the following standards were used: 2-3-dimethylpyrazine, pyrazine, 4-methylthiazole, 2-isobutyl-3-methylpyrazine, 2,3-butanedione, 2,3-pentanedione, acetoin, benzyl alcohol, maltol, furaneol, furfuryl acetate, 3-methylbutanal, 2,5-dimethylpyrazine, pyridine, 2,6-dimethylpyrazine, 4,5-dimethylthiazole, 2-furfurylthiol, 2-acetylpyridine, vanillin, phenylethyl alcohol, 4-ethylguaiacol, 4-vinylguaiacol, cis-isoeugenol, isovaleric acid, methanethiol, dimethyldisulfite, acetic acid, propanoic acid, acetaldehyde, guaiacol, 2,3-diethyl-5-methylpyrazine, furfural, linalool, 2-isobutyl-3-methoxypyrazine and 2-acetyl-3,5-dimethylpyrazine (Sigma Aldrich, USA).

2.2. Physico-chemical analyses

The acid, iodine, and peroxide values were determined according to AOCS (2014). The peroxide value was determined using titrator TitroLine easy (Schott, Germany) with a 0.1 N sodium thiosulphate solution; results were expressed as meq of peroxide·kg^{-1}. The iodine value was determined by the Wijs method using a 0.1 N sodium thiosulphate solution; results were expressed as g of I_2·100 g^{-1}. The saponification value was determined by the fatty acid composition, and was expressed as mg KOH·g^{-1}. All analyses were performed in triplicate.

The moisture and volatile matter were determined in triplicate according to the AOCS (2014) and expressed as a percentage. The oil (5 g) was oven-dried with air circulation TE-394/1 (Tecnal, Brazil) at 130 °C for 2 h.

The antioxidant capacity was estimated based on the ABTS free radical scavenging capacity as described by Corso *et al*., (2016). The ABTS^{+} solution was produced by reacting 7 mmol·L^{-1} of a ABTS stock solution with 2.45 mmol·L^{-1} of potassium persulfate solution; the mixture stood in the dark at room temperature for 12-16 h prior to use. The ABTS^{+} solution was diluted with 5 mmol·L^{-1} phosphate buffer (pH 7.4) to an absorbance of 0.70 ± 0.02 at 730 nm. Ethyl acetate (1:12) was used for dilution. After the addition of 10 µL of the sample or standard Trolox in 4 mL of ABTS^{+} solution diluted, 6 min was taken for reaction, and 730 nm readings were performed on a UV-visible Libra S22 spectrophotometer (Biochrom, UK). Quantification was performed using the 5-point analytical curve (in triplicate) with Trolox. The analysis was performed in duplicate, and the results were expressed as mg Trolox·mL^{-1}.

2.3. Physical analysis

The refractive index was determined, in triplicate, in a refractometer RM 40 (Mettler Toledo, USA) at 20 ºC.

The density was determined by an electronic digital densimeter, model DMA-35 (Anton Paar, Austria) using 10 mL of coffee oil; the result was expressed as g·mL^{-1}. The viscosity was evaluated in a Viscometer DV-II (Brookfield, USA) and expressed in mPas. The analyses were performed at 25 ºC in triplicate.

In order to determine the sun protection factor (SPF) in vitro, the oil was dissolved in ethyl alcohol at the concentration of 0.2 µL·mL^{-1}. Three-fold readings were performed in the range of 290 to 320 nm (in 5 nm increments) in a UV-visible spectrophotometer Libra S22. The absorbance was multiplied by the erythemal effect of the radiation at each wavelength (Table 1), and the sum of the values was multiplied by a

correction factor (determined according to two sunscreens with known SPF), as described by Wagemaker *et al.,* (2011) (Eq. 1).

TABLE 1. Normalized product function used for sun protection factor calculation.

Wavelength (nm)	EEx I (normalized)[a]
290	0.0150
295	0.0817
300	0.2874
305	0.3278
310	0.1864
315	0.0839
320	0.0180

[a]EE (λ) = erythemal effect spectrum; I (λ) = solar intensity spectrum.

$$SPF = CF \cdot \sum_{290}^{320} EE(\lambda) \cdot I(\lambda) \cdot Abs(\lambda) \quad \text{(Eq. 1)}$$

Where:
CF = correction factor (= 10);
EE (λ) = erythemal effect spectrum;
I (λ) = solar intensity spectrum;
Abs (λ) = absorbance of the sunscreen product.

2.4. Chemical composition

2.4.1. Fatty acids and triacylglycerols

The hydrolysis and transesterification of the fatty acids were performed according to ISO method 5509, using 2 mol·L^{-1} NaOH in methanol and n-heptane, in triplicate. After separation, the phase containing n-heptane and fatty acid methyl esters was stored in an amber vial at -18 °C until analysis. Methyl esters of fatty acids were analyzed using CG Shimadzu 17A (Kyoto, Japan) equipped with a flame ionization detector and a CP SIL 88 capillary column (100m x 0.25 mm) (Agilent Technologies Inc., USA). The column temperature was programmed as follows: 65 °C (15 min); raised at 10 °C·min^{-1} until 165 °C and held for 2 min; raised at 4 °C·min^{-1} to 185 °C and held for 8 min; raised at 4 °C·min^{-1} to 235 °C and held for 5 min. The detector and injector were maintained at 260 ºC, using 1/100 Split. The gas flow rate was 1.2 mL.min^{-1} for the carrier gas (H_2) and 30 mL·min^{-1} for make-up gas (N_2). Identification of the fatty acids was based on comparison with standards and the results were expressed as relative percentages of the fatty acids identified.

Oil composition in triacylglycerols (TAG) was estimated by software available in the Plataforma Lames (2019) based on the fatty acid profile. This method results in a large number of TAGs, and in order to reduce the number of components, all structural isomers were divided into a set of components with the same number of carbon and double bonds. Each set of isomers was named according to the major TAG and groups with a total TAG content lower than 0.5% (w/w) were not considered, as suggested by Bitencourt *et al.,* (2018).

2.4.2. Diterpenes

Extraction was performed according to Dias *et al.,* (2014), in duplicate. Samples (0.2 g) were saponified with 2.0 mL of 2.5 mol·L^{-1} potassium hydroxide in ethanol (96% v / v) at 80 °C for 1 h. For the extraction of the unsaponifiable matter, 2.0 mL of distilled water and 2.0 mL of *tert*-butyl methylether were added. After stirring and centrifugation at room temperature (3 min at 3000 rpm), the organic phase was collected. The last step was repeated 3 times. Distilled water (2 mL) was added for cleaning, and the organic extract was collected and evaporated to dryness in a water bath (70 °C) and re-suspended in the mobile phase.

The analysis was performed as described by Mori *et al.,* (2016), using UPLC Waters Acquity (Waters, Milford, USA) equipped with an automatic sample injector, solvent quaternary pumping system, column oven, and DAD detector, controlled by the Empower 3 program. Detection was set at 230 nm (cafestol) and 290 nm (kahweol). Kinetex C18 column (150 mm x 4.6 mm, 2.6 µm) (Phenomenex, USA) and volume of injection of 1.4 µL were used. Isocratic elution with water: acetonitrile (45:55 v / v) at a flow rate of 1.2 mL·min^{-1} was performed. The analyses were made in duplicate.

Quantification was performed by external standardization using triplicate 6-point analytical curves ($r \geq 0.999$, $p < 0.001$), with a limit of quantification (LQ) of 3.2 mg·100 g^{-1} and 3.6 mg·100g^{-1} for kahweol and cafestol, respectively. The results were expressed as contents of kahweol and cafestol and as total diterpenes (mg·100 g^{-1}).

2.4.3. Tocopherols

The tocopherol profile was determined based on the AOCS Ce 8-89 methodology (AOCS, 2014). The oil was directly solubilized in hexane (1% w/v). A Lab Alliance LC305 HPLC (Scientific Systems, Inc., USA) with Radpump III pump and LC 305 fluorescence detector and a LiChrospher Si 60 column (125 mm x 4 mm, 5 µm) (Merck, Germany). Fluorescence excitation was set at 325 nm and emission at 480 nm. Isocratic elution was performed with hexane: ethyl acetate: glacial acetic acid (98: 1.3: 0.7% v / v / v), at a flow rate of 1.5 mL·min^{-1} and an injection volume of 250 µL.

Quantification was performed by external standardization using triplicate 6-point analytical curves for each compound (α, β, γ, and δ-tocopherol), with LQ of 0.1 mg·100 g^{-1}. The results were expressed as individual tocopherols and as total tocopherol (mg·100 g^{-1}).

2.4.4. Caffeine and chlorogenic acids

Extraction was performed as described by Carvalho *et al.*, (1990), in triplicate. Coffee oil (2 g), water (200 mL), and MgO (5 g) were boiled for 45 min. After cooling and filtration, 4 mL of a sulfuric acid solution (1:9 acid:water) and 20 mL of chloroform were added to the mixture in a separatory funnel. After stirring, the chloroform layer was transferred to another funnel; the step was repeated five times. A potassium hydroxide solution 1% (5 mL) was then added to the extract, and after stirring and phase separation, the extract was filtered and diluted with chloroform.

A chromatographic analysis was performed according to Corso *et al.*, (2016), using a Shimadzu HPLC (Kyoto, Japan) with two pumps (LC-10 AD), a Rheodyne injection valve with 20 µL loop, a UV/visible detector (SPD-10 A), CBM-101 interface and Program CLASS-CR10, version 1.2. A Spherisorb ODS1 column (250 × 4.6 mm, 5 µm) (Waters, Ireland) was used, and detection was set at 272 nm (caffeine) and 320 nm (chlorogenic acids). A gradient of 5% acetic acid (A) and acetonitrile (B) solution was used as follows: 0-10 min: 5% B; 10-25 min: 13% B; 25-35 min: 5% B, flow rate 0.5 mL min^{-1}. The injections were made in duplicate.

The quantification was performed by external standardization using duplicate 6-point analytical curves ($r \geq 0.999$, $p < 0.001$). The sum of the compounds was detected at 320 nm, using the 5-CQA as standard, and applied to estimate the total chlorogenic acid content (Corso *et al.*, 2016).

2.4.5. Volatile compounds

The analysis was performed by solid-phase micro-extraction followed by quantification in an Agilent 6890 N CG equipped with Agilent 5973 mass spectrometry detector and MSD Chemstation software (Agilent Technologies Inc., USA). Sample preparation and chromatographic conditions were applied according to Kalschne *et al.*, (2018).

The oil was weighed (1.0 g) in a 20 mL vial (Agilent, California, USA) immediately sealed with a silicone septum and kept in a water bath (70 ºC). After 10 min, the septum was punctured, and a DVB/CAR/PDMS fiber (Sigma Aldrich, USA) was exposed to the headspace for 30 min. After injection, the compounds were heat-desorbed from the fiber (desorption time 10 min) and transferred to an Innowax column (60 m x 0.32 mm x 0.25 µm) (Agilent, California, USA). Helium was used as carrier gas at 1.3 mL·min^{-1} flow rate and the injector temperature was 250 °C. The heating profile started at 40 °C, held 5 min, raised to 60 °C at 4 °C·min^{-1}, held at 60 °C for 5 min and up to 250 °C at 8 °C·min^{-1}, held for 3 min. The mass spectrometer operated at 280 °C interface temperature, ion source temperature of 230 °C, quadrupole temperature of 150 °C, scanning in a range of *m/z* of 35-400 amu.

The standards (1 mL) were placed in vials (20 mL), and injected into the GC-MS using the same extraction technique applied to volatile compounds. Quantification was performed by external standardization using duplicate 6-point analytical curves. Sensory attributes related to each volatile compound, based on those described in literature (Akiyama *et al.*, 2007, Belitz *et al.*, 2009. Dulsat-Serra *et al.*, 2016, Toledo *et al.*, 2016, and Kalschne *et al.*, 2018), were also reported (Table 5).

3. RESULTS AND DISCUSSION

Peroxide, acid, iodine and saponification values can be correlated with the stability and quality of oils. They indicate the oxidation degree, stability status, degree of unsaturation, and the relative amount of low fatty acids and high molecular weight (AOCS, 2014).

Coffee oil showed a peroxide value of 3.208 meq·kg^{-1} (Table 2). This was higher than that described by Sanches (2016) for roasted Arabica oil stored at a different time and under temperature conditions up to 2.38 meq·kg^{-1}, and Turatti (2001), up to 2.4 meq·kg^{-1}. However, it was still lower than the maximum value (15 meq·kg^{-1}) recommended for cold-pressed oil by the Brazilian regulation (Anvisa, 2005).

TABLE 2. Physico-chemical characterization of roasted Arabica coffee oil.

Parameters	Oil
Peroxide value (meq·kg^{-1})[a]	3.208 ± 0.001
Acid value (mg KOH·g^{-1})[b]	7.3 ± 0.2
Iodine value (g I_2 100 g^{-1})[b]	113.5 ± 0.3
Saponification value (mg KOH·g^{-1})[b]	195.26 ± 0.08
Moisture and volatile matter (%)[b]	0.85± 0.05
Refractive Index[b]	1.4798 ± 0.0000
Density (g·mL^{-1})[b]	0.938± 0.002
Viscosity (mPas a 25ºC)[b]	228.7 ± 0.5
ABTS (mg Trolox·mL^{-1})[a]	12.5 ± 0.1
Sun protection factor[b]	9.7 ± 1.2

[a]Means of duplicate ± standard deviation.
[b]Means of triplicate ± standard deviation.

An acid value of 7.3 mg KOH·g^{-1} was observed (Table 2), which was lower than that described by Turatti (2001) for roasted coffee oil (8.95 mg KOH g^{-1}) and by Amin *et al.,* (2019) for pumpkin seed oils (from 11.5 to 13.5 mg NaOH·g^{-1}). These values were higher than those defined for cold-pressed oils (maximum 4.0 mg KOH·g^{-1}) (Anvisa, 2005). However, no specific regulation can be found for oils that undergo a previous heat treatment such as roasted coffee oil. Furthermore, the literature describes that the roasting process can release acidic compounds, increasing acidity values (Sanches, 2016).

The coffee oil presented an iodine value of 113.50 and a saponification value of 195.26 mg KOH·g^{-1} (Table 2). The data were in the range of those reported by Sanches (2016) for roasted Arabica coffee oil: from 92.17 to 114.10 g I_2·100g^{-1} and from 192.98 to 233.44 mg KOH·g^{-1} for iodine and saponification values, respectively. Values in a similar range were reported by Amin *et al.*, (2019) for pumpkin seed oils: iodine value from 106.6 to 113.2 g I_2·100g^{-1} and saponification value from 115.7 to 236.0 mg KOH·g^{-1}.

The moisture and volatile matter of 0.85% (Table 2) were attributed to press extraction, since solvent-extracted oils do not contain water. Sanches (2016) reported lower moisture contents (up to 0.2%) for roasted coffee oil, although he pointed out that industrial limits varied between 0.30 and 2.00%.

The refractive index can be used as a physical parameter of oil quality. It increases with increasing fatty acid chain length and degree of unsaturation (AOCS, 2014). The coffee oil presented a refractive index of 1.4798 (Table 2), similar to that described by Amin *et al.*, (2019) for pumpkin seed oils (1.5).

Density and viscosity are important parameters for oil processing, since they are determinant for the correct design of the pumping, sedimentation, and filtration steps (Bonnet *et al.,* 2011). The coffee oil had a density of 0.938 g·mL^{-1} and a viscosity of 228.7 mPas at 25 °C (Table 2). Oliveira *et al.,* (2014), evaluating pressed green Arabica coffee oil, reported similar values for density and lower viscosity (from 95 to 127.9 mPas). Roasted coffee oil is denser than several other vegetable oils. Stanciu (2019) reported density values from 0.84 to 0.93 g·mL^{-1} for soybean, corn, sunflower, grape seed, and olive oils, among others.

Sun protection factor (SPF) and antioxidant capacity are important parameters to evaluate the potential of the oil as an ingredient in food and cosmetics. SPF indicates the relationship between the time of exposure to the sun without generating erythema (redness to the skin) with the use of the product compared to unprotected skin. Consequently, the higher the SPF, the longer the time the skin will be protected against UVB radiation (Wagemaker *et al.,* 2011). For the roasted coffee oil, a SPF of 9.7 and ABTS free radical-scavenging capacity of 12.5 mg Trolox mL^{-1} (Table 2) were observed. No data was found regarding the antioxidant capacity of coffee oil extracted by pressing. Wagemaker *et al.,* (2011) described a SPF of 1.50 for green Arabica coffee oil, traditionally used in cosmetics. Kaur and Saraf (2010) reported a wide range of SPF for several herbal oils used in cosmetics, from 0.248 (rose oil) to 7.549 (olive oil); besides olive oil, the highest values were found for coconut (7.119), peppermint (6.668), tulsi (6.571) and lemon grass (6.282) oils but they presented lower SPF than roasted coffee oil. Therefore, the efficient protection afforded by roasted coffee oil indicates its potential for use in cosmetic products.

Roasted coffee oil presented 57.5% of unsaturated fatty acids (Figure 1-a), indicating susceptibility to lipid oxidation, which highlights the importance of studying chemical parameters related to stability (Table 2). The literature described a wide range for the proportion of saturated, monounsaturated and polyunsaturated fatty acids in coffee oil from 29.45 to 47.3%, 42.72 to 59.17% and from 4.30 to 17.81%, respectively (Calligaris *et al.,* 2009; Getachew and Chun, 2016; Hurtado-Benavides *et al.,* 2016). These differences may be due to the coffee species used, as well as to the extraction method applied.

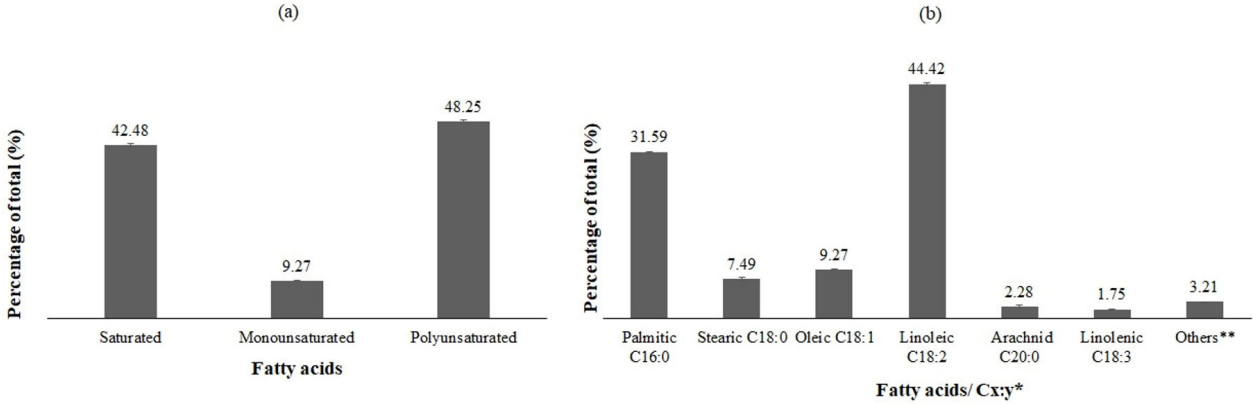
Regarding the fatty acid profile, the high proportion of polyunsaturated linoleic acid (44.42%) in roasted coffee oil (Figure 1) can be important for the health benefit of the compound ingestion, as it is an essential fatty acid (Spector, 1999). The presence (9.27%, Figure 1) of monounsaturated oleic acid –an omega-9 fatty acid – is also interesting because of its effect in reducing LDL cholesterol oxidation and as a precursor to the production of most other polyunsaturated fatty acids and hormones (Watkins and German, 2008). On the hand, palmitic acid, the main saturated fatty acid found in the roasted coffee oil (31.59%, Figure 1), which can increase low-density blood cholesterol levels, is interesting for extended use in several skin product formulations such as soaps and shaving creams and, along with linoleic, stearic and oleic fatty acids, is described as an excellent cosmetic material (Dangarembizi *et al*., 2015).

The fatty acid profile (Figure 1- b) was similar to that reported in other studies (Turatti, 2001; Oliveira *et al.,* 2005; Calligaris *et al.,* 2009; Raba *et al.,* 2018), since linoleic (L) and palmitic (P) are the main fatty acids, followed by oleic (O) and stearic (S). Some authors have reported higher palmitic acid contents, followed by linoleic acid (Rocha *et al.,* 2013; Hurtado-Benavides *et al.,* 2016). Cornelio-Santiago *et al.,* (2017) and Bitencourt *et al.,* (2018) described a predominance of linoleic, palmitic, oleic, and stearic acids in green coffee oil obtained by supercritical extraction.

Triacylglycerols are the main components of roasted coffee oil. It was estimated that the main TAGs in the roasted coffee oil were PLL (18.7%), PLP (13.3%), LLL (8.8%), PLO (7.8%), SLP (6.3 %) and OLL (5.5%) (Table 3).

González *et al.,* (2001) reported higher contents in PLL (20.1 to 31.5%) and PLP (15.8 to 28.9%) for Soxhlet-extracted roasted Arabica coffee oil. For green coffee oil, Cornelio-Santiago *et al.,* (2017) reported SLP (12.9%), PLL (12.3%), and PLP (11.6%) as the main TAGs, while Bitencourt *et al.,* (2018) highlighted the high PLP (22.9%) and PLL (22.6%) contents.

The profile of fatty acid and triacylglycerols observed for the studied roasted coffee oil (Figure 1 and Table 3) was similar to that described in the literature for oils obtained by different extraction methods and green coffee oil, showing the potential use of pressed roasted coffee oil.



Means of triplicate; error bars: standard deviation

*Cx:y where Cx = number of carbons and y = number of double bonds.

**Others: Myristic, Margaric, n-Heneicosanoic, Eicosadienoic, Behenic, Timnodonic, Adrenic and Clupanodonic acids presented in contents up to 1%.

FIGURE 1. Fatty acids of roasted Arabica coffee oil. (a) Percentage of saturated, monounsaturated and polyunsaturated fatty acids. (b) Fatty acid profile.

TABLE 3. Hypothetical triacylglycerol profile of roasted Arabica coffee oil.

Triacylglycerols[a]	Cx:y[b]	Percentage of total (%)[c]
PPP	48:0	3.16 ± 0.13
SPP	50:0	2.25 ± 0.04
POP	50:1	2.78 ± 0.06
PLP	50:2	13.30 ± 0.29
SOP	52:1	1.32 ± 0.03
SLP	52:2	6.31 ± 0.03
PLO	52:3	7.80 ± 0.06
PLL	52:4	18.69 ± 0.19
PLnL	52:5	1.46 ± 0.06
PLA	54:2	1.88 ± 0.03
SLO	54:3	1.85 ± 0.06
SLL	54:4	4.44 ± 0.11
OLO	54:4	1.14 ± 0.04
OLL	54:5	5.48 ± 0.13
LLL	54:6	8.75 ± 0.21
LLnL	54:7	1.03 ± 0.04
ALL	56:4	1.32 ± 0.05
Others[d]		8.25 ± 1.08

[a]Fatty acids: Arachnid (A), Adrenic (Ad), Behenic (Be), Clupanodonic (Cp), Linoleic (L), Linolenic (Ln), Oleic (O), Palmitic (P), Stearic (S), Timnodonic (Tm).[b]Cx: y where Cx = number of carbons and y = number of double bonds. [d]PLnP (50: 3), SPS (52: 0), PAP (52: 0), POO (52: 2), SLS (54: 2), PTmL (54: 7), BeLP (56:2), ALO (56: 3), PAdL (56: 6), PCpL (56: 7) presented in contents up to 1%.[c]Means of triplicate ± standard deviation.

Diterpenes, the major components of UM, correspond to 86 to 88% of the UM for Arabica coffee (Pacetti *et al*., 2015), and their contents remained stable during the roasting process (Dias *et al*., 2014). Kahweol and cafestol are the main diterpenes in coffee and produced only by plants of the *Coffea* genus (Dias *et al*., 2014). They are of interest due to their anticarcinogenic, antioxidant, anti-inflammatory, and hepatoprotective activities, and also to their skin hydration and sun protection effects (Kim *et al.,* 2009; Muriel and Arauz, 2010) although cafestol is also related to an increase in serum cholesterol levels (Speer and Kölling-Speer, 2006). The coffee oil presented a total diterpenes content of 3720 mg·100g^{-1}, with 1980 and 1740 mg 100 g^{-1} of kahweol and cafestol, respectively (Table 4). Oliveira *et al.,* (2014) and Bitencourt *et al.,* (2018) reported higher efficiency of supercritical extraction of green coffee oil diterpenes compared to pressing. It was also observed that the kahweol content (Table 4) was comparable to that reported by Bitencourt *et al*., (2018) (up to 1500 mg·100 g^{-1}) for green coffee oil using supercritical extraction.

TABLE 4. Unsaponifiable matter and hydrosoluble compounds of roasted Arabica coffee oil.

	Compounds	Content (mg·100g^{-1})
Diterpenes[a]	Kahweol	1980± 50
	Cafestol	1740 ± 60
	Total	3720
Tocopherols[b]	α	30.350 ± 0.250
	β	881.123 ± 17.080
	δ	2.226 ± 0.004
	Total	913
Hydrosoluble[a]	Caffeine	350± 10
	Chlorogenic acids	10.71 ± 0.03

[a]Means of duplicate of extraction ± standard deviation.
[b]Means of triplicate ± standard deviation.

In the UM, the presence of tocopherols also stands out, both for vitamin activity and antioxidant effect, which contributes to the stabilization of cell membranes which protect other bioactive compounds. The main component was β-tocopherol (97% of the total), followed by α and δ isomers; γ-tocopherol was absent (below the LD of 0.07 mg·100g^{-1}) (Table 4). Thus, a high total tocopherol content of 913 mg·100g^{-1}was observed, corresponding to 271 mg of vitamin E (expressed as α-tocopherol)·100g^{-1} or 298 IU of vitamin E·100g^{-1}.

In the literature, no consensus is found on the tocopherol profile of roasted Arabica coffee oil. González *et al.,* (2001) reported the β-isomer (from 9.4 to 16.1 mg·100g^{-1}) as the major one, followed by γ and α-tocopherol (5.9 to 9.5 and 2.1 to 3.4 mg·100g^{-1}) and the absence of δ-tocopherol. Ribeiro (2015) reported a higher γ-tocopherol content (182 mg·100g^{-1}), followed by β, δ, and α isomers (94, 25, and 1 mg·100g^{-1}, respectively). It should be noted that, besides the difference in the isomer profile, those authors reported lower total tocopherol contents than those obtained in this study, probably due to the high temperature used in Soxhlet extraction. For pressed green coffee oil, contents of 13.3 and 34.7 mg·100g^{-1} of α and β tocopherol, respectively, were reported (Bitencourt *et al.,* 2018). Therefore, the efficient extraction of the UM compounds of the roasted coffee by pressing can stand out.

As previously discussed, as roasted coffee was pressed, it was also possible to extract some hydrosoluble compounds of known antioxidant effects such as caffeine and chlorogenic acids. The coffee oil presented 350 mg·100g^{-1} of caffeine and 10.71 mg·100g^{-1} of total chlorogenic acids (Table 4). The higher caffeine extraction was attributed to its lower molecular weight (194.194 g·mol^{-1}), and higher water solubility (22 g·L^{-1}) (Pubchem, 2018) compared to chlorogenic acids.

Similar caffeine contents, from 320 to 340 mg·100g^{-1}, were reported by Sanches (2016) for roasted Arabica coffee oil obtained by pressing. Oliveira *et al.,* (2014) highlighted a higher efficiency of the supercritical extraction process with caffeine contents from 260 to 1650 mg· 100g^{-1} in green Arabica coffee oil.

No data on chlorogenic acid content in roasted coffee oil were found. For green coffee oil, Bitencourt *et al.,* (2018) reported 8.8 mg GAE·100g^{-1} using pressing extraction, and Oliveira *et al.,* (2014) reported a wider range of values (0 to 262 mg GAE·100g^{-1}) depending on supercritical extraction conditions. It should be considered, however, that the Folin-Ciocalteu estimation is not specific for phenolic compounds, and the response may also be due to other reducing compounds.

The presence of these bioactive compounds (Table 4) may also be associated with the antioxidant capacity and SPF characteristics observed for roasted coffee oil (Table 2).

Thirty-five volatile compounds of different classes (carboxylic acids, ketones, furans, thiols, pyrazines, phenols, pyridines, aldehydes, terpenes, alcohols, sulfur compounds, and thiazoles) were quantified in the oil (Table 5), several of them being described as typical of roasted coffee aroma (Akiyama *et al.,* 2007; López-Galilea *et al.,* 2006).

For roasted Arabica coffee oil, Getachew and Chun (2016) described the presence of aldehydes, ketones, furans, pyrroles, pyrazines, pyridines, and phenolic compounds (24 volatile compounds), and Hurtado-Benavides *et al.,* (2016) reported a greater number of compounds (41 volatiles), mainly furans and pyrazines, in products obtained by supercritical extraction. In pressed oil, Oliveira *et al.,* (2005) identified 32 volatile compounds, including hydrocarbons, pyrazines, furans, and ketones.

The volatile compounds found in higher contents belong to carboxylic acids, ketones, furans, and thiol classes. We highlight the acetic acid, with a negative impact on the aroma profile, and maltol, 2-furfurylthiol, furfuryl acetate, and 2,6-dimethylpyrazine, which presented potential positive aroma characteristics. The pyrazine class contained the highest number of volatile compounds, and the lowest number of compounds was observed in the classes of terpenes, alcohols, sulfur compounds, and thiazoles (Table 5). The volatile compounds in the roasted coffee oil can be formed by the thermal degradation of carbohydrates, amino acids, ascorbic acid, lipids, esters, and the auto-oxidation of aldehydes and ketones during the roasting process (Buffo and Cardelli-Freire, 2004).

Carboxylic acids account for a high proportion of roasted coffee's volatile fraction (Kalschne*et al.,* 2018). Volatile acids present characteristic odors, and acetic acid is present in high contents in the roasted coffee oil (1287.63 ng·g^{-1}) (Table 5), is related to vinegar odor (Belitz *et al.,* 2009).

Ketones are also abundant in roasted coffee (Toledo *et al.,* 2016), presenting aromas such as fruit, butter, mushroom, mold, caramel, and tea (López-Galilea *et al.,* 2006; Akiyama *et al.,* 2007). Maltol, present in higher contents (835.94 ng·g^{-1}) (Table 5), presents a caramel odor (Belitz *et al.,* 2009).

Furans are described as the main chemical class found in Arabica coffee, followed by pyrazines, pyridines, and pyrroles (Toledo *et al.,* 2016). They can give an aroma of roasted malt, sweet, grass, fruits, burnt, burnt sugar, and others (López-Galilea *et al.,* 2006; Akiyama *et al.,* 2007, Nascimento *et al.,* 2007; Belitz *et al.,* 2009). The furan found in higher contents in the roasted coffee oil was furfuryl acetate (539.88 ng·g^{-1}) (Table 5), which has a floral and fruity odor (Nascimento *et al.,* 2007).

Although presented in lower contents, thiols and pyrazines have a significant impact on the characteristic aroma and flavor of coffee brews. Thiols are related to aromas of roasted, fresh coffee, roasted meat, and nuts, among others (Dulsat-Serra *et al.,* 2016). A high content in 2-furfurylthiol in the oil (727.16 ng·g^{-1}) (Table 5) was observed, which is a key aromatic compound in roasted coffee products (Toledo *et al.,* 2016, Nascimento *et al.,* 2007; Belitz *et al.,* 2009). Pyrazines are described as presenting aromas of nut, earth, roasted and grass (Czerny and Grosch, 2000; Akiyama *et al.,* 2007; Toledo *et al.,* 2016).

TABLE 5. Profile of volatile compounds of roasted Arabica coffee oil.

Sensory Group[a]	Volatile Compound / Class	Compound content (ng·g^{-1})[b]	Class content (ng·g^{-1})
	Carboxylic acids		
Fermented	Isovaleric acid	68.82	
Chemist / Pungent	Acetic acid	1287.63	1422.01
Vegetable / Herbaceous	Propanoic acid	65.56	
	Ketones		
Sweet, Burned	2,3-Butanedione	1.58	
Sweet, Burned	2,3-Pentanedione	0.12	869.74
Sweet, Burned	Acetoin	32.10	
Sweet, Burned	Maltol	835.94	
	Furans		
Floral	Furfuryl acetate	539.88	
Vegetable	Furfural	199.90	741.87
Sweet, Burned	Furaneol	2.09	
	Thiols		
Burned (Coffee)	2-Furfurylthiol (Fufurylmercaptane)	727.16	727.17
Putrid	Methanethiol	0.01	
	Pyrazines		
Seasoning	2,3-Diethyl-5-methylpyrazine	1.05	
Nuts	2,5-Dimethylpyrazine	114.29	
Nuts	2,3-Dimethylpyrazine	39.59	
-	2-Isobutyl-3-methylpyrazine	0.27	560.29
-	2-Isobutyl-3-methoxypyrazine	0.31	
-	2-Acetyl-3,5-dimethylpyrazine	6.00	
Burned (Coffee)	2,6-Dimethylpyrazine	361.41	
Moldy / Earth	Pyrazine	37.37	
	Phenols		
Chemical	Guaiacol	113.54	
Sweet / Vanilla	Vanillin	3.31	
Smoked	4-Ethylguaiacol	89.14	491.95
Smoked	4-Vinylguaiacol	285.15	
Smoked	Cis-isoeugenol	0.81	
	Pyridines		
Burned (Coffee)	Pyridine	386.92	388.84
Burned (Coffee)	2-Acetylpyridine	1.92	
	Aldehydes		
Pungent	Acetaldehyde (ethanal)	87.88	113.83
Frutal	3-Methylbutanal	25.95	
	Terpenes		
Vegetable	Linalool	4.02	4.02
	Alcohols		
Sweet, Burned	Benzyl alcohol	1.52	3.71
Smoked	Phenylethyl alcohol	2.19	
	Sulfur compounds		
Putrid	Dimethyldisulfite	3.27	3.27
	Thiazoles		
Burned (Coffee)	4,5-Dimethylthiazole	0.48	1.11
Moldy / Earth	4-Methylthiazole	0.63	

[a]Sensory attributes related to each component are cited based on those described by Akiyama *et al.*,(2007), Belitz *et al*., (2009), Dulsat-Serra *et al*., (2016), Toledo *et al*., (2016), and Kalschne *et al*., (2018). [b]Means of duplicate.

The main pyrazine identified in the roasted coffee oil was 2,6-dimethylpyrazine (361.41 $ng \cdot g^{-1}$), which presents a characteristic aroma of burnt coffee and roasted cocoa/nut (Nascimento *et al.,* 2007).

4. CONCLUSIONS

Roasted coffee oil proved to be a high quality product due to its low peroxide and acid values, significant contents in tocopherols and diterpenes, in addition to the presence of caffeine and chlorogenic acids, resulting in high antioxidant capacity. The roasted oil presented a high sun protection factor effect (compared to green coffee oil and others herbal oils), and the profile of fatty acids and triacylglycerols was similar to that described in the literature for green coffee oil. In the complex profile of volatiles, thirty-five compounds of different classes were identified, with pyrazines and furfurylthiol as the predominant ones. These properties show that roasted coffee oil has good potential for use in food and cosmetics.

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