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Effect of different extraction methods on yield and quality of araticum (*Annona crassiflora* Mart.) seed oil

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Abstract

Microwave- and ultrasound-assisted extraction methods can increase the yield and quality of oil extraction. This study investigated their effects on araticum seed oil extraction compared with conventional methods. Oils were extracted using press and Soxhlet techniques, combined with microwave- and ultrasound-assisted methods at different times. Key parameters such as yield, acidity index, peroxide index, iodine index, moisture, saponification index, ether-insoluble impurities, antioxidant activity by DPPH and ABTS methods, and fatty acid profile were evaluated. The highest oil yield (24.22%) was achieved by combining the press with Soxhlet. Based on EC50 values, antioxidant activities ranged from 1.06 ± 0.10 to 5.19 ± 0.39 mg/mL and 2.26 ± 0.33 to 10.43 ± 0.28 μM trolox/g for DPPH and ABTS, respectively. The ultrasound-assisted method showcased superior antioxidant activity. Predominant fatty acids included oleic, linoleic, palmitic, and stearic acids. Extractions by ultrasound-assisted press at 15 and 30 min showed enhanced antioxidant potency and reduced peroxide and acidity indices. The extraction method affected the characteristics of the oil, and changes in the fatty acid profile were observed. Non-heating methods yielded more unsaturated acids but with low extraction yield.

Keywords: antioxidants; araticum; cerrado; seed oil.

Practical Application: Efficient extraction methods improve the quality of araticum oil.

1 INTRODUCTION

Araticum (*Annona crassiflora* Mart.) is an exotic fruit native to the Brazilian Cerrado, which belongs to the Annonaceae family (Santos Oliveira et al., 2022). Their pulp is cone-shaped, light yellow, thick, soft, and sweet (Reis & Schmiele, 2019), while their seeds (70–190 per fruit) are dark brown with obovate-flattened shape, measuring from 10 to 13 mm in width per 20–27 mm in thickness (Arruda et al., 2018).

Several studies have shown the biological properties of the araticum seed as considerable concentrations of phenolic compounds (Arruda et al., 2018), genotoxic action (Ribeiro et al., 2013), antioxidant activity (Prado et al., 2020; Ramos et al., 2023; Roesler et al., 2007), good yield (28.84% m/m), presence of bioactive substances (683.59 mg/kg of phytosterols and 138.90 mg/kg of tocopherols), and 67.76% of unsaturated fatty acids, with a predominance of oleic acid (49.75%) (Luzia & Jorge, 2013). Therefore, the use of seeds as a source of vegetable oil can be relevant and promising.

During the extraction process, vegetable oils may lose their nutritional potential, and it is crucial to assess their efficiency and stability in the face of the entire process (Ferreira et al., 2015; Zhang et al., 2023). Conventional processes for oilseed oil extraction are by the press, solvent, or a combination of both. Press extraction is a mass transfer operation that removes oil from seeds by applying mechanical energy, having its organoleptic properties preserved (Koubaa et al., 2016). However, about 5–6% of the oil remains retained in the press cake and cannot be recovered. In this sense, industrially, the oil extraction process combines press extraction and solvent extraction, reducing residual oil to up to 1% loss (Gupta, 2017).

In solvent extraction, the oils migrate from the seeds to the solvent (hexane, ethyl ether, ethanol, and methanol, among others), as they have a greater affinity with it. Then the solvent is recovered (Chemat et al., 2019; Ferreira et al., 2022). However, in the extraction by this method, thermal degradation of beneficial components occurs as the solvent, time, and temperature directly influence the quality of the extracted oil (Guimarães et al., 2016).

Therefore, other extraction methods are sought to minimize losses in the yield and the final quality of vegetable oils. For example, microwave- and ultrasound-assisted extraction methods can increase extraction yield and maintain oil quality (Ferreira et al., 2022; Thilakarathna et al., 2023). Microwaves can penetrate the solid matrix, generating heat inside the cells,

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causing destruction of the cellular structure of plant tissues, and resulting in the more efficient extraction of intracellular compounds, which, consequently, decreases the extraction time and increases income (Satriana et al., 2019; Taghvaei et al., 2014). On the contrary, ultrasound-assisted extraction facilitates oil extraction by collapsing cell walls and promoting solvent penetration, resulting in higher concentrations of polyphenols and better antioxidant activity when compared with the conventional method (Dias et al., 2019; Ferreira et al., 2022; Pereira et al., 2017).

This study aimed to extract araticum seed oil using conventional methods adjunct with extraction assisted by ultrasound and microwave and verify its effects on the yield and quality of the final product. The importance of this research is to understand the behavior of araticum seed oil, given different extraction technologies, to offer the consumer a quality oil, preserving its nutritional characteristics.

2 MATERIALS AND METHODS

2.1 Materials

Seeds of *Annona crassiflora* Mart. were acquired from a pulp producer in Minas Gerais, in the southeastern region of Brazil, located at 16°41'31.2" S and 43°53'27.7" W. The seed lot was purchased in the 2016 harvest and was previously dried in the sun for 48 h. Then, they were transported in plastic boxes to the Food Engineering sector of the School of Agronomy (Universidade Federal de Goiás, Brazil), where the peel was removed, to obtain the almond, packed in low-density polyethylene bags, wrapped in aluminum foil, to protect from light, and frozen in freezers at -8°C until the time of oil extraction by different methods.

2.2 Oil extraction

The extractions of araticum seed oil evaluated in this study are summarized and identified in Table 1.

Table 1. Identification of the different methods of extracting araticum seed oil.

Treatment	Assisted Method	Extraction	Power (W)	Time (min)
P	–	Press	–	–
S	–	Soxhlet	–	–
UP15	Ultrasound	Press	–	15
UP30	Ultrasound	Press	–	30
US15	Ultrasound	Soxhlet	–	15
US30	Ultrasound	Soxhlet	–	30
MP1	Microwave	Press	100	5
MP2	Microwave	Press	100	10
MP3	Microwave	Press	500	5
MP4	Microwave	Press	500	10
MS1	Microwave	Soxhlet	100	5
MS2	Microwave	Soxhlet	100	10
MS3	Microwave	Soxhlet	500	5
MS4	Microwave	Soxhlet	500	10
OS	–		–	–

2.2.1 Cold extraction (Press)

For mechanical extraction by hydraulic pressing, the MARCON-MPH-15 press was used for 15 tons. Notably, 200 g of araticum almonds were weighed in a stainless-steel cylinder and pressed until the force of 12 tons was marked on the pressure gauge for approximately 40 min. The extracted crude oil was weighed and stored in amber bottles under refrigeration (5°C) in a Consul refrigerator until the moment of chemical analysis.

2.2.2 Soxhlet extraction

About 500 g of the araticum almonds were broken in a Vitalex® OP 30791 low-speed blender for 3 min. A quantity of 150 g of the crushed material was weighed and placed directly in the Soxhlet Tecnal TE-044-5/50 extractor trailer, with 300 mL of n-hexane at 80°C for 6 h. After completing the extraction cycle, the hexane was removed by distillation, under vacuum, using a rotary evaporator MA 120 at 25°C. The crude oil was weighed and stored in amber flasks, at 5°C, in a Consul refrigerator, until the moment of chemical analysis.

2.2.3 Extraction assisted by ultrasound and microwave

In the ultrasound-assisted extraction, 500 g of the almonds were weighed, per treatment, and submerged in distilled water (25°C) in an ultrasonic bath, with a frequency of 40 kHz, in the periods of 15 and 30 min. The almonds were then dried in an oven at 60°C for 48 h, and the cold and Soxhlet extraction proceeded as previously described.

In microwave-assisted extraction, 200 g of almonds were weighed per treatment and heated in a domestic microwave, varying the potency by 10 and 50% and time in 5 and 10 min, as shown in Table 1. Then, cold and Soxhlet extraction was performed, as previously described.

2.2.4 Press with Soxhlet

After performing the cold extraction (see Section 2.2.1), the cake was subjected to extraction by Soxhlet (see Section 2.2.1), and the oils obtained from both extractions were mixed and weighed, forming a single batch.

2.3 Analytical methods

2.3.1 Chemical analysis

The moisture content (ISO 1442) (Mohanta, 2016), acidity index (method Ca 5a-40), peroxide index (ISO 3960—method Cd 8-53) (Mohanta, 2016), saponification index (method Cd 3c-91), iodine index (method Cd 1-25), and impurities insoluble in petroleum ether oil were determined according to the methodology of AOCS (2004). The analyses were performed in triplicate.

2.3.2 Fatty acid profile

The profile was determined following the methodology described by IOC (2015). The extracted araticum seed oils were converted into fatty acid methyl esters (FAMEs) and then analyzed by a Thermo Scientific gas chromatograph TRACE 1310.

A TR-FAME capillary column (30 m × 0.22 mm ID × 0.25 µm) was used for separation purposes. A mobile phase gas (nitrogen) was released through the column at a flow rate of 1.2 mL·min^{-1}. The initial temperature of the column was adjusted to 150°C and increased at the rate of 10°C·min^{-1} to a final temperature of 250°C. The identification of fatty acid compounds was based on the combination of their absolute and relative retention times with those of FAMEs standards. The composition of fatty acids, in percentage, was calculated concerning the total peak areas.

2.3.3 DPPH antioxidant activity and free radical capture ABTS$^{\cdot+}$

To determine antioxidant activity by DPPH, 5 g of oil was solubilized in 25 mL of isopropyl alcohol and then determined by the DPPH method (2.2 diphenyl-1-picrylhydrazyl), as described by Brand-Williams et al. (1995). The results were expressed in concentration (mg/mL), necessary to obtain an antioxidant effect of 50% (EC_{50}).

The sequestering capacity of the 2,2'-azino-bis-3-ethylbenzothiazoline-6-sulfonic acid radical ($ABTS^{+}$) was determined according to the method described by Rufino et al. (2007). The results were expressed as antioxidant activity in µM of Trolox equivalent per gram of sample. Analyses of antioxidant activity were performed in quintuplicate.

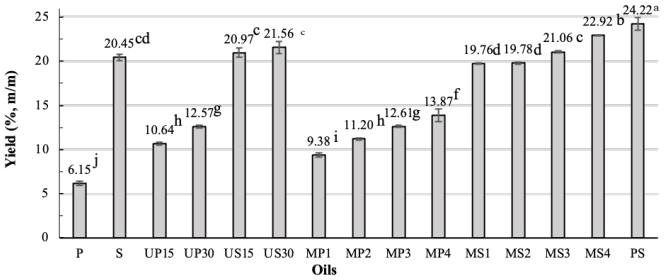
2.4 Statistical analysis

The experimental design was completely randomized (DIC) with 15 treatments. The means were compared by the Tukey test at a significance level of 5%, using the SISVAR software, version 5.0. Principal component analysis (PCA) was performed using the Past software.

3 RESULTS AND DISCUSSION

The effects of different extraction methods on the yield of araticum seed oil are shown in Figure 1. The result indicated a greater yield in the combination of the press and Soxhlet extraction methods (PS—24.22%), followed by the combination of microwaves and Soxhlet (MS4—22.92%). The S, US15, US30, and MS3 methods did not differ statistically, with an average yield of 20.9%. Press extraction (P) was the one with the lowest yield (6.15%), indicating that approximately 18.07% of the oil was retained in the cake, as expected.

The great advantage of press extraction is that the pressed oils maintain their properties preserved (Koubaa et al., 2016; Yang et al., 2021), while in solvent extraction, the beneficial components suffer thermal degradation (Guimarães et al., 2016; Thilakarathna et al., 2023), which is an effect observed in oil quality parameters (Table 2).



Different letters indicate significant differences ($p < 0.05$). P: press; S: Soxhlet; UP15: ultrasound, press, 15 min; UP30: ultrasound, press, 30 min; US15: ultrasound, Soxhlet, 15 min; US30: ultrasound, Soxhlet, 30 min; MP1: microwave, press, 100 W, 5 min; MP2: microwave, press, 100 W, 10 min; MP3: microwave, press, 500 W, 5 min; MP4, microwave, press, 500 W, 10 min; MS1: microwave, Soxhlet, 100 W, 5 min; MS2: microwave, Soxhlet, 100 W, 10 min; MS3: microwave, Soxhlet, 500 W, 5 min; MS4: microwave, Soxhlet, 500 W, 10 min; PS: press combined with Soxhlet.

Figure 1. Yield of araticum seed oil extracted by different methods.

Table 2. Chemical characterization and antioxidant activity of araticum seed oil extracted by different methods.

Oil	Acidity Index (%Oleic acid)	Peroxide Value (mEq/kg)	Iodine Index	Moisture (% w/w)	Saponification Index (mg KOH/g)	Insoluble Impurities in Petroleum Ether (% w/w)	EC_{50} (mg/mL)	ABTS (µM trolox/g)
P	1.64 ± 0.10^{g}	2.67 ± 0.38^{i}	99.49 ± 1.15^{a}	1.89 ± 0.13^{c}	157.99 ± 1.51^{a}	0.79 ± 0.06^{a}	1.06 ± 0.10^{a}	6.21 ± 0.14^{c}
S	3.48 ± 0.11^{cd}	6.40 ± 0.26^{h}	79.37 ± 1.85^{b}	4.58 ± 0.38^{b}	141.75 ± 2.44^{b}	0.22 ± 0.02^{de}	1.77 ± 0.08^{b}	3.10 ± 0.98^{e}
UP15	1.77 ± 0.11^{g}	3.27 ± 0.31^{i}	99.70 ± 1.45^{a}	2.40 ± 0.10^{c}	150.38 ± 1.07^{ab}	0.77 ± 0.15^{a}	1.03 ± 0.07^{a}	8.88 ± 0.16^{b}
UP30	2.26 ± 0.08^{f}	5.07 ± 0.25^{h}	99.63 ± 0.83^{a}	2.23 ± 0.19^{c}	150.20 ± 1.49^{ab}	0.71 ± 0.01^{a}	0.99 ± 0.05^{a}	10.48 ± 0.28^{a}
US15	4.40 ± 0.24^{b}	8.10 ± 0.20^{f}	89.80 ± 6.56^{de}	4.35 ± 0.13^{ab}	136.73 ± 1.45^{c}	0.38 ± 0.13^{c}	1.70 ± 0.15^{b}	3.83 ± 0.31^{d}
US30	4.58 ± 0.11^{b}	9.53 ± 0.35^{e}	81.86 ± 0.27^{b}	4.48 ± 0.56^{b}	133.72 ± 0.67c	0.36 ± 0.04^{cd}	1.40 ± 0.16^{ab}	3.51 ± 0.05^{d}
MP1	2.99 ± 0.08^{e}	6.33 ± 0.12^{dh}	78.86 ± 1.15^{b}	2.10 ± 0.18^{c}	150.13 ± 1.49^{ab}	0.48 ± 0.04^{b}	2.53 ± 0.10^{c}	2.26 ± 0.33^{g}
MP2	3.16 ± 0.14^{de}	8.77 ± 0.23^{f}	94.24 ± 6.35^{ad}	2.21 ± 0.31^{c}	145.63 ± 2.55^{b}	0.46 ± 0.06^{bc}	2.56 ± 0.09^{c}	2.45 ± 0.11^{fg}
MP3	3.39 ± 0.18^{d}	9.83 ± 0.06^{e}	92.30 ± 0.72^{d}	2.21 ± 0.23^{c}	156.55 ± 2.90^{a}	0.45 ± 0.05^{bc}	2.81 ± 0.31^{cd}	3.03 ± 0.20^{de}
MP4	3.64 ± 0.11^{c}	10.17 ± 0.31^{de}	88.30 ± 0.42^{de}	2.67 ± 0.35^{c}	155.62 ± 1.69^{a}	0.66 ± 0.01^{a}	5.19 ± 0.36^{e}	2.44 ± 0.17^{f}
MS1	3.43 ± 0.14^{c}	10.87 ± 0.25^{cd}	71.64 ± 0.59^{f}	4.85 ± 0.09^{a}	139.50 ± 0.96^{c}	0.41 ± 0.02^{bc}	3.11 ± 0.26^{d}	3.36 ± 0.85^{g}
MS2	4.90 ± 0.07^{ba}	11.30 ± 0.17^{bc}	72.03 ± 1.95^{f}	3.01 ± 2.01^{ab}	136.32 ± 1.27^{c}	0.12 ± 0.01^{e}	2.50 ± 0.08^{c}	2.64 ± 0.62^{fg}
MS3	5.04 ± 0.07^{a}	11.63 ± 0.06^{ab}	70.18 ± 5.46^{f}	4.34 ± 0.22^{ab}	125.86 ± 1.53^{d}	0.11 ± 0.02^{e}	3.03 ± 0.30^{d}	1.80 ± 0.41^{g}
MS4	5.11 ± 0.14^{a}	12.07 ± 0.15^{a}	66.75 ± 1.31^{c}	4.31 ± 0.16^{ab}	137.98 ± 2.18^{c}	0.12 ± 0.03^{e}	4.29 ± 0.30^{e}	2.45 ± 0.17^{fg}
PS	3.13 ± 0.04^{de}	6.57 ± 0.06^{h}	86.75 ± 1.31^{e}	3.63 ± 0.32^{b}	147.38 ± 1.13^{ab}	0.37 ± 0.02^{cd}	1.33 ± 0.07^{ab}	3.14 ± 0.57^{e}

Data are expressed as mean ± standard deviation. Different letters in the same column indicate significant differences ($p < 0.05$). P: press; S: Soxhlet; UP15: ultrasound, press, 15 min; UP30: ultrasound, press, 30 min; US15: ultrasound, Soxhlet, 15 min; US30: ultrasound, Soxhlet, 30 min; MP1: microwave, press, 100 W, 5 min; MP2: microwave, press, 100 W, 10 min; MP3: microwave, press, 500 W, 5 min; MP4: microwave, press, 500 W, 10 min; MS1: microwave, Soxhlet, 100 W, 5 min; MS2: microwave, Soxhlet, 100 W, 10 min; MS3: microwave, Soxhlet, 500 W, 5 min; MS4: microwave, Soxhlet, 500 W, 10 min; PS: press combined with Soxhlet.

According to Codex Alimentarius (2017), the parameters of quality of vegetable oils recommend that the peroxide index is the maximum 15 mEq of active oxygen/kg of virgin oil and the acidity index is the maximum of 2% AGL of oleic acid for oils unrefined. The peroxide index indicates the beginning of the oxidation of oils and fats, which are the primary lipid oxidation products (Melhaoui et al., 2021; Shahidi & Zhong, 2010; Zhang et al., 2023). The araticum seed oils, extracted by different methods, are below the limit established by Codex (2.67 ± 0.38 to 12 ± 0.15 mEq/kg), indicating efficient extraction quality in both methods used.

The acidity index allows the quantification of acidic substances in the oil, determining the hydrolysis/oxidation suffered by it (Zhang et al., 2023). Therefore, it is essential information for determining the oil's conservation status. The values of the acidity index in the extraction P (1.64% AGL of oleic acid) and UP15 (1.77% AGL of oleic acid) were not different ($p < 0.05$) and are within the determined standards of, at most, 2% AGL (Shahidi & Zhong, 2010), proving the quality of the extracted oil. A lower acidity index indicates less free fatty acids content and higher quality of the oil (Melhaoui et al., 2021; Zhang et al., 2023). The MS4 extraction (5.11% AGL of oleic acid) showed the highest values in the acidity index, which did not differ ($p < 0.05$) from the extraction MS3 (5.04% AGL of oleic acid) and MS2 (4.90% AGL of oleic acid), showing worse quality in the microwave treatment, which was already expected, due to the decomposition of glycerides to be accelerated by the increase in temperature. Therefore, with the increase in power and time of exposure to microwaves, greater heating causes a significant increase ($p < 0.05$) in the acidity index.

According to Gupta (2017), during the processing of crude oil, in the presence of moisture content above 0.5% and elevated temperatures (> 50°C), the triglyceride can be hydrolyzed, releasing free fatty acids, providing an increase in acidity content, and consequently increasing the speed of the oxidation reaction. As the moisture content observed in the treatments varied from 1.89 ± 0.13 to 4.85 ± 0.16%, all araticum seed oils in the tested extractions are susceptible to the acceleration of the self-oxidation process, and it is necessary to carry out some additional refining step, such as filtration.

The iodine index measures the primary oxidation status of unsaturated fatty acids in the oil and is also related to the fatty acid profile (Sotelo-Méndez et al., 2023). Fatty acids containing carbon–carbon double bonds react with iodine. Therefore, the greater the unsaturation number, the higher the iodine index (Gaber et al., 2018). The oils with the highest values of iodine index were found in the extraction by the press (P—99.49) and assisted by ultrasound (UP15—99.7 and UP30—99.63), which did not differ significantly. Comparing press and Soxhlet, it is observed that the iodine index is lower in the Soxhlet extraction, which was expected as it is a hot extraction. It is known that the increase in temperature accelerates the degradation of unsaturated fatty acids.

Among the oils extracted with the help of microwaves, in the extraction by Soxhlet, with greater power and time (MS4), there was a lower value of the iodine index (66.75 ± 1.31), that is, the microwave favored the degradation of the unsaturated fatty acids. As in this study, when researching the effect of different drying temperatures on the oil extracted from physic nut seeds, Cabral (2011) observed that the iodine index of the extracted oil decreased as the drying temperature increased, indicating deterioration of physic nut oil. The results observed for the iodine index corroborate the fatty acid profile of araticum seed oils in Table 3, where the sum of unsaturated fatty acids is observed.

The saponification index is essential to obtain the minimum amount of potassium hydroxide (KOH) necessary to saponify the oil (Martins et al., 2020). The saponification reaction is a reaction in which the chemical bonds of a macromolecule break due to the addition of water, forming smaller molecules. The values found for the saponification index in araticum seed oil were 125.86 ± 1.53 (MS3) to 157.99 ± 1.51 (P) mg KOH/g. All values were lower than those found for coco oil (222–233 mg KOH/g) (Martins et al., 2020), tomato seed oil (172.86 mg KOH/g), orange (181.05 mg KOH/g), passion fruit (174.97 mg KOH/g), guava (189.91 mg KOH/g) (Kobori & Jorge, 2005), and lupine oil (179.91–186.45 mg KOH/g) (Sotelo-Méndez et al., 2023). The low values of the saponification index of araticum seed oils are a possible indication of small amounts of high-molecular-weight fatty acid, as can be seen in Table 3 (fatty acid profile).

Food antioxidants can be defined as any compounds that inhibit oxidative processes that deteriorate the quality of dietary lipids (Benzie & Strain, 1996). The EC_{50} values of araticum seed oils, analyzed by the DPPH method, were between 1.06 ± 0.10 and 5.19 ± 0.39 mg/mL, and their capacity by the ABTS method varied from 2.26 ± 0.33 to 10.43 ± 0.28 μM trolox/g. Using the ABTS method, extraction by ultrasound-assisted pressing, in 30 min (UP30), was the one with the highest antioxidant capacity (8.88 ± 0.16 μM trolox/g), followed by UP15 (10.48 ± 0.28 μM trolox/g). Ultrasound-assisted extractions obtained oil with greater antioxidant capacity due to cavitation effects, such as ultrasound waves that cross the medium of the mass, causing compression and shearing of molecules, breaking part of the structure by the abrupt increase and decrease in pressure, and generating tiny bubbles, which collapse in the pressure cycle and produce turbulent flow conditions (Goula, 2013).

When observing the oil quality analyses, it is noted that the oil extracted by the press was the one that presented the best quality, despite the small yield. The UP15-assisted ultrasound and rush method also showed good quality, although the yield is still low. The methods assisted by ultrasound and those extracted by Soxhlet showed good yield. However, the quality of the oils was lower as the heat accelerated the oxidation process.

Fatty acids participate in the formation of sensory attributes, add caloric and nutritional value to foods, and are precursors of essential metabolites in the human body. The determination of the fatty acid profile (Table 3) of the araticum seed oil is valuable information on its nutritional value and possible deterioration by oxidation.

The most essential fatty acids found in araticum seeds were oleic > linoleic > palmitic > stearic, which accounted for about 97% of the total fatty acids, ranging from 41.66 to 48.34% oleic acid, 34.98 to 36.14% linoleic acid, 8.54 to 15.09% palmitic acid, and 4.80 to 5.16% stearic acid. This pattern of fatty acids is

Table 3. Fatty acid profile of araticum oil extracted by different methods.

Fatty acids	Oils														
	P	S	UP15	UP30	US15	US30	MP1	MP2	MP3	MP4	MS1	MS2	MS3	MS4	PS
C < 12	n.d.	0,02^{a}	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,01^{a}	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
12:0 (Lauric)	0.01^{a}	0.01^{a}	0.01^{a}	0.01^{a}	0.01^{a}	0.01^{a}	0.02^{a}	0.02^{a}	0.01^{a}	0.01^{a}	0.01^{a}	0.01^{a}	0.01^{a}	0.01^{a}	0.01^{a}
14:0 (Myristic)	0.10^{a}	0.09^{a}	0.10^{a}	0.09^{a}	0.10^{a}	0.10^{a}	0.14^{a}	0.14^{a}	0.12^{a}	0.11^{a}	0.09^{a}	0.09^{a}	0.09^{a}	0.08^{a}	0.09^{a}
16:0 (Palmitic)	8.66^{e}	15.06^{a}	8.72^{e}	8.72^{e}	11.31^{b}	9.86^{d}	8.57^{e}	8.54^{fg}	8.56^{f}	8.45^{g}	15.04^{a}	14.98^{a}	14.97^{a}	15.09^{a}	11.20^{c}
16:1 (Palmitoleic)	0.22^{c}	0.93^{a}	0.22^{c}	0.22^{c}	0.50^{b}	0.35^{bc}	0.28^{c}	0.27^{c}	0.24^{c}	0.24^{c}	0.93^{a}	0.92^{a}	0.92^{a}	0.94^{a}	0.50^{b}
17:0 (Margaric)	0.06^{a}	0.06^{a}	0.07^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}	0.06^{a}
17:1 (Heptadecanoic)	0.03	0.05^{a}	0.03^{a}	0.03^{a}	0.04^{a}	0.04^{a}	0.04^{a}	0.03^{a}	0.04^{a}	0.03^{a}	0.05^{a}	0.05^{a}	0.05^{a}	0.05^{a}	0.04^{a}
18:0 (Stearic)	5.16^{a}	4.80^{c}	5.18^{a}	5.19^{a}	5.05^{b}	5.07^{ab}	4.82^{c}	4.82^{c}	4.99^{b}	5.02^{b}	4.80^{c}	4.82^{c}	4.82^{c}	4.82^{c}	5.03^{b}
18:1 (Oleic)	47.81^{a}	41.90^{c}	47.81^{a}	48.10^{a}	45.01^{b}	46.36^{b}	48.31^{a}	48.34^{a}	47.74^{a}	47.60^{a}	41.66^{c}	41.88^{c}	41.72^{c}	41.61^{c}	45.21^{b}
18:2 (Linoleic)	35.64^{a}	34.98^{b}	35.54^{ab}	35.25^{ab}	35.63^{a}	35.86^{a}	35.48^{ab}	35.49^{ab}	35.94^{ab}	36.14^{ab}	35.23^{ab}	35.03^{b}	35.20^{ab}	35.19^{ab}	35.61^{a}
18:3 (Linolenic)	0.97^{bc}	1.07^{a}	0.97^{bc}	0.94^{c}	1.05^{a}	0.99^{b}	1.00^{b}	1.00^{b}	0.98^{b}	0.99^{b}	1.08^{a}	1.07^{a}	1.08^{a}	1.08^{a}	1.01^{b}
20:0 (Arachnoid)	0.70^{a}	0.58^{c}	0.70^{ab}	0.71^{a}	0.66^{b}	0.68^{ab}	0.66^{b}	0.67^{ab}	0.68^{ab}	0.69^{ab}	0.58^{c}	0.59^{c}	0.59^{c}	0.59^{c}	0.65^{b}
20:1 (Gadoleic)	0.29^{a}	0.20^{c}	0.29^{a}	0.30^{a}	0.26^{b}	0.28^{ab}	0.27^{ab}	0.27^{ab}	0.28^{ab}	0.29^{a}	0.20^{c}	0.20^{c}	0.20^{c}	0.20^{c}	0.25^{ab}
20:0 (Behenic)	0.22^{a}	0.16^{c}	0.21^{a}	0.16^{c}	0.21^{a}	0.16^{c}	0.22^{a}	0.16^{c}	0.19^{bc}	0.15^{c}	0.21^{a}	0.19^{b}	0.22^{a}	0.21^{a}	0.21^{a}
22:1 (Ketoleic)	0.02^{a}	0.01^{a}	0.01^{a}	0.02^{a}	0.02^{a}	0.01^{a}	0.01^{a}	0.01^{a}	0.02^{a}	0.02^{a}	0.02^{a}	0.02^{a}	0.02^{a}	0.02^{a}	0.01^{a}
24:0 (Lignoceric)	0.12^{a}	0.11^{a}	0.12^{a}	0.11^{a}	0.12^{a}	0.11^{a}	0.13^{a}	0.11^{a}	0.12^{a}	0.10^{a}	0.12^{a}	0.12^{a}	0.13^{a}	0.12^{a}	0.12^{a}
Σ saturated fatty acids	15.03^{c}	20.87^{a}	15.11^{c}	15.05^{c}	17.52^{b}	16.05^{bc}	14.62^{c}	14.52^{c}	14.73^{c}	14.59^{c}	20.91^{a}	20.86^{a}	20.89^{a}	20.98^{a}	17.37^{b}
Σ unsaturated fatty acids	84.98^{ab}	79.14^{d}	84.87^{a}	84.86^{ab}	82.51^{c}	83.89^{bc}	85.39^{ab}	85.41^{ab}	85.24^{ab}	85.31^{ab}	79.17^{d}	79.17^{d}	79.19^{d}	79.09^{d}	82.63^{b}
Σ monounsaturated fatty acids	48.35^{a}	43.08^{c}	48.35^{a}	48.65^{a}	45.81^{a}	47.03^{ab}	48.90^{a}	48.91^{a}	48.30^{a}	48.16^{a}	42.84^{c}	43.05^{c}	42.89^{c}	42.80^{c}	46.00^{b}
Σ polyunsaturated fatty acids	36.16^{a}	36.06^{a}	36.52^{a}	36.21^{a}	36.70^{a}	36.86^{a}	36.49^{a}	36.50^{a}	36.94^{a}	37.15^{a}	36.33^{a}	36.12^{a}	36.30^{a}	36.29^{a}	36.63^{a}

Data are expressed with average. Different letters in the same column indicate significant differences ($p < 0.05$). P: press; S: Soxhlet; UP15: ultrasound, press, 15 min; UP30: ultrasound, press, 30 min; US15: ultrasound, Soxhlet, 15 min; US30: ultrasound, Soxhlet, 30 min; MP1: microwave, press, 100 W, 5 min; MP2: microwave, press, 100 W, 10 min; MP3: microwave, press, 500 W, 5 min; MP4: microwave, press, 500 W, 10 min; MS1: microwave, Soxhlet, 100 W, 5 min; MS2: microwave, Soxhlet, 100 W, 10 min; MS3: microwave, Soxhlet, 500 W, 5 min; MS4: microwave, Soxhlet, 500 W, 10 min; PS: press combined with Soxhlet.

similar to that described in the literature for peanut oil (*Prunus dulcis*), whose main fatty acids are oleic acid (35–69%), linoleic acid (12–43%), and palmitic acid (8–14%) (FAO/WHO, 2001; Lin et al., 2016).

It is observed that araticum seed oils are mainly composed of unsaturated fatty acids, about 80% for all extractions, being a source to be explored as the moderate consumption of food sources of unsaturated fatty acids is related to a decrease in circulating cholesterol levels and, consequently, a lower risk for the onset of cardiovascular diseases. Oleic acid was the one with the highest concentrations. Therefore, araticum seed oil can be used as a food additive as this fatty acid participates in the metabolism, playing a fundamental role in the synthesis of hormones, or even by industries in the manufacture of soaps and cosmetics.

It is observed that the main difference in the fatty acid profile, in the different extraction methods, is in the proportion of oleic (C18: 2) and palmitic (C16: 0) fatty acids. Probably, with heating, in the extraction of Soxhlet, there was a transformation of oleic fatty acid into palmitic acid, by the β-cleavage reaction, which promotes the transformation of unsaturated fatty acids, breaking the molecules into pairs (Gupta, 2017).

Figure 2 presents the analysis of the main components, graphically, for the interaction of the methods given the chemical analyses carried out. There is a division into two sectors (PC1—59.38% and PC2—20.28%), explaining 79.66% of the data variation between the different methods, making it possible to divide them into four quadrants. Observing the layout of the

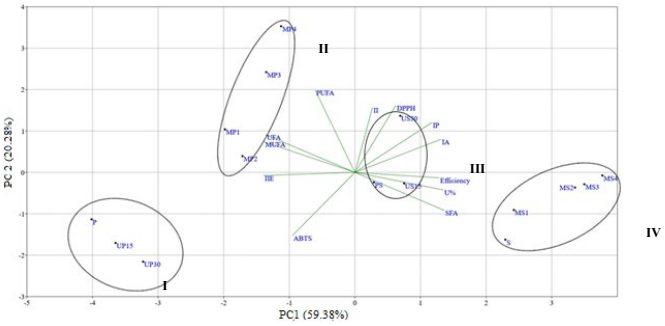


Figure 2. Analysis of the main components of the oil extracted by different methods of the parameters: PUFA: polyunsaturated fatty acids, MUFA: monounsaturated fatty acids; SFA: saturated fatty acids; UFA: unsaturated fatty acids; II: iodine index; IA: acidity index; IP: peroxide index; U%: humidity; IIE: impurities insoluble in ether; antioxidant activity by the DPPH and ABTS method; P: press; S: Soxhlet; UP15: ultrasound, press, 15 min; UP30: ultrasound, press, 30 min; US15: ultrasound, Soxhlet, 15 min; US30: ultrasound, Soxhlet, 30 min; MP1: microwave, press, 100 W, 5 min; MP2: microwave, press, 100 W, 10 min; MP3: microwave, press, 500 W, 5 min; MP4, microwave, press, 500 W, 10 min; MS1: microwave, Soxhlet, 100 W, 5 min; MS2: microwave, Soxhlet, 100 W, 10 min; MS3: microwave, Soxhlet, 500 W, 5 min; MS4: microwave, Soxhlet, 500 W, 10 min; PS: press combined with Soxhlet.

quadrants, the extractions by pressing (quadrants I and II) have greater antioxidant power, lower peroxide, and acidity index, with low yield. Microwave-assisted extractions have a higher proportion of unsaturated fatty acids; the Soxhlet extractions (quadrants III and IV) have higher efficiency, higher peroxide

and acidity index, less antioxidant activity, and a higher proportion of saturated fatty acids. Looking at quadrant III, the combination of extraction by press and Soxhlet (PS) and Soxhlet assisted by ultrasound (US15 and US30) has greater antioxidant activity, lower levels of acidity and peroxide, and higher iodine, compared with quadrant IV.

Although the ultrasound-assisted method has a lower yield in oil extraction, it proved to be the best technique for better quality oil, with lower peroxide levels and acidity and greater antioxidant power, which is the most recommended for use in the food industry.

4 CONCLUSION

Oleic acid (41.66–48.34%), linoleic acid (34.98–36.16%), palmitic acid (8.54–15.09%), and stearic acid (4.80–5.16%) were the predominant fatty acids in araticum seed oil. The extraction yield (24.22%) was higher in combining the press method with Soxhlet (PS).

The different extraction methods have shown to have a great influence on obtaining the oil, with changes in the fatty acid profile being observed, in which the methods that have not been heated have a higher proportion of unsaturated, however, with low extraction yield. The extractions by press assisted by ultrasound, in 15 and 30 min, exhibited greater antioxidant power of the oils, presenting lower peroxide levels and acidity.

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