Pressurized Liquid Extraction of Macauba Pulp Oil

Caroline Portilho Trentini,[1] Sandra Beserra da Silva,[1] Giovana Menezes Rodrigues,[2] Vitor Augusto dos Santos Garcia,[2] Lúcio Cardozo-Filho[3,4] and Camila da Silva[1,2,3]*

1. Programa de Pós-Graduação em Bioenergia, Universidade Estadual de Maringá (UEM), Maringá, PR, Brasil
2. Programa de Pós-graduação em Engenharia Química, Universidade Estadual de Maringá (UEM), Maringá, PR, Brazil
3. Departamento de Tecnologia, Universidade Estadual de Maringá (UEM), Umuarama, PR, Brazil
4. Centro Universitário Octávio Bastos (UNIFEOB), São João da Boa Vista, SP, Brazil

The aim of this study was to investigate the extraction of oil from macauba pulp using ethanol and isopropanol as pressurized solvents. Experiments were carried out in a semi-continuous extractor system at various temperatures (40, 60, and 80 °C) maintaining the pressure fixed at 10 000 kPa and the solvent flow at 3 mL/min and also using conventional extraction (in a Soxhlet). For both methods assessed, higher yields were obtained with the use of ethanol as the solvent. In the pressurized liquid extraction (PLE), an increase in temperature from 40 to 60 °C provided higher yields at 72 min of extraction, which was not influenced by the extraction carried out at 80 °C. This temperature effect was also observed in the extraction kinetics data. The maximum yields obtained by PLE were 44.78 % and 37.12 % with ethanol and isopropanol, respectively, which represents ~77 % of the yield obtained by conventional extraction. Oleic and palmitic acids are the main fatty acids identified in macauba pulp oil, representing ~88 % of the fatty acids composition, which was not influenced by the extraction method and solvent used. PLE with ethanol provides oils with higher levels of β-carotene. The flavonoid content was higher with the use of isopropanol; however, it was not influenced by the method used.

Keywords: ethanol, isopropanol, fatty acids, β-carotene

INTRODUCTION

Macauba (*Acrocomia aculeata*) is a common species of palm tree found in Brazil, from Pará to São Paulo and Mato Grosso do Sul[1] and it has an average production of 0.25 kg fruit/m^2.[2] Because of its good capacity for adaptation, high productivity, and high oil yield (50 to 70 %) it can be used as feedstock for biofuels,[2–6] to prepare products in the cosmetic and food industries, and it also has technical applications.[7] The oils obtained from the pulp and kernel are commonly used as a tonic in folk medicine due to their anti-inflammatory and antioxidant activities.[8]

Of all the products which can be extracted from macauba, the oils of the kernel and pulp are those with the highest economic value.[9,10] They can be used in the food, cosmetics, and pharmaceutical industries, and for biodiesel production.[9,11]

The macauba fruit consists mostly of pulp,[12,13] which has an oil content of around 18.7 to 32 %,[3,12–15] and contains a high amount of active compounds.[15,17,18] The oil obtained from macauba pulp has high concentrations of β-carotene, flavonoids, and oleic acid,[12,14,16] and lesser contents of phytosterols and tocopherols.[16,17,19]

The method of extraction used to obtain macauba pulp oil (MPO) needs to provide an extract with high quality and yield while retaining the bioactive compounds present in the matrix. Pressurized liquid extraction (PLE) is a technique that combines temperature and pressure with the use of liquid solvents to achieve rapid and efficient extraction,[20] and it is associated with low solvent consumption.[21,13] The literature reports the efficiency of this technique for extracting oil from wheat germ,[24] grape seed,[21] palm,[25] watermelon seed,[26] and safflower;[23] however, PLE does not appear to have been applied to obtain macauba pulp oil.

A major advantage of PLE is that the solvents under pressure remain liquid, even above their boiling points at atmospheric pressure, and the higher temperature increases the solubility and diffusion of analytes since it decreases the viscosity and surface tension of solute-solvent, which facilitates the penetration of the solvent into the matrix and diffusion of the components.[21,27] The use of pressure facilitates the process of extraction from samples in which the analyte is retained in the matrix. The pressure forces the solvent to penetrate areas of the matrix that would not normally be reached using a solvent at atmospheric pressure.[28]

In these types of extraction techniques, *n*-hexane is commonly used as the solvent.[29] However, despite its high efficiency in the extraction of vegetable oils, this solvent is considered toxic and harmful at high concentrations.[30] Frequent human exposure to *n*-hexane vapour can cause damage to the nervous system, such as inflammation, as well as irritation of the eyes and respiratory system,[31] and its use is also related to environmental pollution.[32] According to Hanmoungjai et al.,[33] *n*-hexane emitted to the environment during extraction and recovery can react with other pollutants to produce ozone and photochemical oxidants. Thus, to minimize its use, the efficiency of other solvents in the extraction of vegetable oils should be investigated.

* *Author to whom correspondence may be addressed.*
E-mail address: camiladasilva.eq@gmail.com
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Ethanol and isopropanol are less toxic than hexane and thus they have been used in this technique.[34] In addition, they can be used as solvents in the food industry.[35] Unlike fossil fuels, ethanol is a renewable energy source produced by fermentation.[36] Isopropanol leads to a more stable extraction, preventing oxidation of the compounds present in the sample, besides being less flammable and of lower toxicity compared to other solvents.[37] In addition, these solvents are classified with GRAS status.[38]

In this context, and considering the lack of data in the literature regarding oil extraction from macauba pulp, the aim of this study was to evaluate the efficiency of PLE for the obtainment of macauba pulp oil using ethanol and isopropanol as solvents. The results for the extraction under pressurized conditions were compared to those obtained applying the conventional technique, in terms of yield and composition of the extracts.

MATERIALS AND METHODS

Preparation of Raw Material

The fruits of *Aculeata acrocomia* were harvested in the Araripe Plateau (Global Positioning System coordinates 38°0′ and 41°55′ W; 70°10′ and 7°50′ S), Cariri region, Ceara State, Brazil. The fruits were sanitized, separation of the parts was carried out, and the pulp was dried at 60 °C for 8 h in an oven (Marconi, MA035), with the humidity determined as 0.036 ± 0.001 g/g (3.6 ± 0.1 wt%). The dry sample was milled using an electric mill (Marconi, MA 750) and classified using a Tyler sieve (Bertel, ASTM). Particles with an average diameter of 0.5 mm were used in the experiments.

Extraction

Ethanol (Panreac, 0.998 g/g (99.8 wt%) purity) and isopropanol (JT Baker 0.9976 g/g (99.76 wt%) purity) were the solvents used for the oil extraction. Conventional extraction was performed as recommended by the Adolfo Lutz Institute,[39] for 480 min and using ~5 g of pulp. The extraction temperature was kept constant and above the solvent reflux temperature (78 °C and 82 °C for ethanol and isopropanol, respectively).

The extractions under pressurized conditions were carried out at temperatures of 40, 60, and 80 °C, with a constant pressure of 10 000 kPa. The pressure to be applied was determined based on the work of Debien et al.[22] and Conte et al.,[23] who reported that pressures of >10 000 kPa do not influence the oil yield obtained by PLE. Figure 1 shows the laboratory scale unit used in the experiments, which basically consists of a solvent reservoir, high-pressure liquid pump (Acuflow, series III), jacketed extraction vessel with a capacity of 15 cm^3, temperature and pressure indicators, and a pressure control valve (Swagelok).

For each extraction, the extraction vessel was loaded with ~6 g of pulp to form a bed of solids supported by two 200 mesh wire disks at both ends. Before the extraction, the system was kept at the experiment temperature for 30 min for stabilization. The solvent was pumped at a constant flow rate of 3 mL/min and the system was pressurized with the aid of a pressure control valve. The sample was collected in an amber glass vessel, and its mass was determined at time intervals of 3 min (up to 12 min) and 5 min (12–72 min of extraction). This procedure was performed in triplicate for each experimental condition evaluated.

After the extraction period, the solvent remaining in the samples, obtained by both methods evaluated, was removed at 50 °C until a constant pulp weight was obtained. The yield was calculated as the ratio of the extracted mass to the initial macauba pulp mass.

Analytical Methods

For the determination of the β-carotene content in the extracts, samples were prepared by diluting 20 mg of oil in 10 mL of *n*-hexane (F Maia) and the absorbance of the samples at 450 nm was measured on a UV spectrophotometer (Femto 700 plus). The results were expressed in mg of β-carotene per 100 g of oil, based on the calibration curve for concentrations of 1.0 to 100.0 mg/L of a β-carotene standard (Sigma Aldrich 99.9 % purity), which showed a regression coefficient of 0.998.

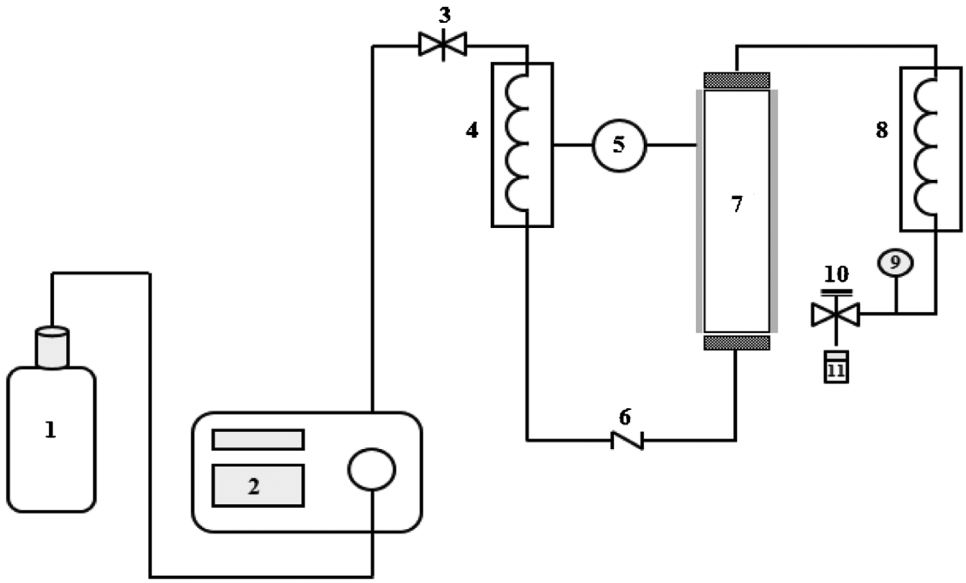

Figure 1. Experimental apparatus used in pressurized liquid extration: (1) solvent reservoir; (2) high pressure liquid pump; (3) needle valve; (4) pre-heating; (5) temperature indicator; (6) check valve; (7) extraction vessel; (8) cooling system; (9) pressure indicator; (10) pressure control valve; (11) sampling.

Table 1. Experimental conditions and oil yields obtained from macauba pulp by PLE and conventional extraction (CE) using ethanol and isopropanol as solvents

Solvent	Extraction method	T (°C)	Time (min)	Yield (%)
Ethanol	PLE	40	62	40.80 ± 1.07^{a}
		60		44.78 ± 0.97^{b}
		80		44.07 ± 1.15^{bA}
	CE	±78	480	57.45 ± 0.61
Isopropanol	PLE	40	62	30.02 ± 1.03^{a}
		60		37.12 ± 0.52^{b}
		80		38.36 ± 0.69^{bB}
	CE	±82	480	49.41 ± 0.45

Means followed by the same lowercase letters (comparison of temperatures for same solvent in PLE) and uppercase letters (comparison of best yields for different solvents in PLE) did not differ statistically ($p > 0.05$).

The total flavonoids content was determined according to the procedure reported by Francis.[40] In this procedure, 1 g of oil was placed in a 50 mL volumetric flask, the volume was completed with ethanol (Anidrol)/HCl (Nuclear) (1.5 mol/L), and the sample was left to stand protected from light for 12 h. The absorbance of the samples was determined at 374 nm using a spectrophotometer (Femto 700plus). The results were expressed in mg of flavonoids per 100 g of oil, using 76.6 as the flavonoids factor.

To determine the fatty acids composition, a gas chromatograph (Thermo-Finnigan) coupled to a mass spectrophotometer was used. The samples were derivatized with BF3-methanol (Sigma-Aldrich) following the method of AOAC Ce 2–66[41] and analyzed using the following chromatographic conditions: initial temperature of the column 120 °C, held for 5 min, increasing to 180 °C at a rate of 15 °C/min and to 240 °C at a rate of 5 °C/min, held 5 min. An Agilent HP-5MS capillary column (30 m × 0.250 mm × 0.25 μm) was used, with the injection of 0.4 μL in split mode (1:10) and helium was used as the carrier gas, at a flow of 1 mL/min. The identification of the components present in the samples was performed with Xcalibur® software (Thermo Electron) and quantified using methyl heptadecanoate (Sigma-Aldrich, >0.99 g/g (99 wt%) purity) as the internal standard.

Analysis of Data

All assays and analysis were performed in duplicate and the data collected were subjected to ANOVA using Excel® 2010 software and the Tukey test (with a 95 % confidence interval), to evaluate differences between the results.

RESULTS AND DISCUSSIONS

Extract Yield

Table 1 shows the experimental conditions and extract yields for the conventional extraction and using solvents under pressurized conditions (PLE). It is clear from Table 1 that ethanol is the most effective solvent for extracting oil from macauba pulp, providing greater yields when compared with isopropanol, in both methods evaluated. Similar results have been reported by Dunford and Zhang[24] for the extraction of oil from wheat germ performed at 105 °C, with 10 300 kPa and 30 min of extraction time, obtaining yields of ~23 % and ~17 % using ethanol and isopropanol, respectively. Debien et al.[22] studied the pressurized extraction of oil from Brazilian ginseng roots and also reported higher yields with ethanol (2.2 %) when compared to the use of isopropanol (0.70 %). This is due to the higher polarity of ethanol, since the polarity of the solvent directly influences the extraction efficiency.[42]

The solvents ethanol and isopropanol have relatively high affinity for polar solutes, such as phospholipids, polyphenols, pigments, soluble sugars, waxes, and some proteins,[24,43,44] resulting in relatively high yields. Oliveira et al.[35] and Baümler et al.[44] also reported higher yields for oil extraction from white rice and wheat germ, respectively, using polar solvents such as ethanol.

In relation to the results obtained for the PLE, analysis of the results in Table 1 indicates that increasing the temperature from 40 to 60 °C favoured the extraction process, obtaining yields of 40.80 and 44.78 % for ethanol and 30.02 and 37.12 % for isopropanol, respectively. However, the yield was not affected by increasing the temperature to 80 °C. These results indicate that the macauba pulp oil has high solubility at low temperatures, 40 and 60 °C, in the solvents evaluated.

Other studies indicate that the extraction yield is affected by the temperature at which the extraction is carried out. Colivet et al.[26] performed the extraction of watermelon seed oil using pressurized ethanol at 10 340 kPa and obtained yields of 25.8 and 28.7 % at 40 and 60 °C, respectively. For the extraction of oat oil using ethanol and isopropanol as solvents at 6 895 kPa, Moreau et al.[43] reported

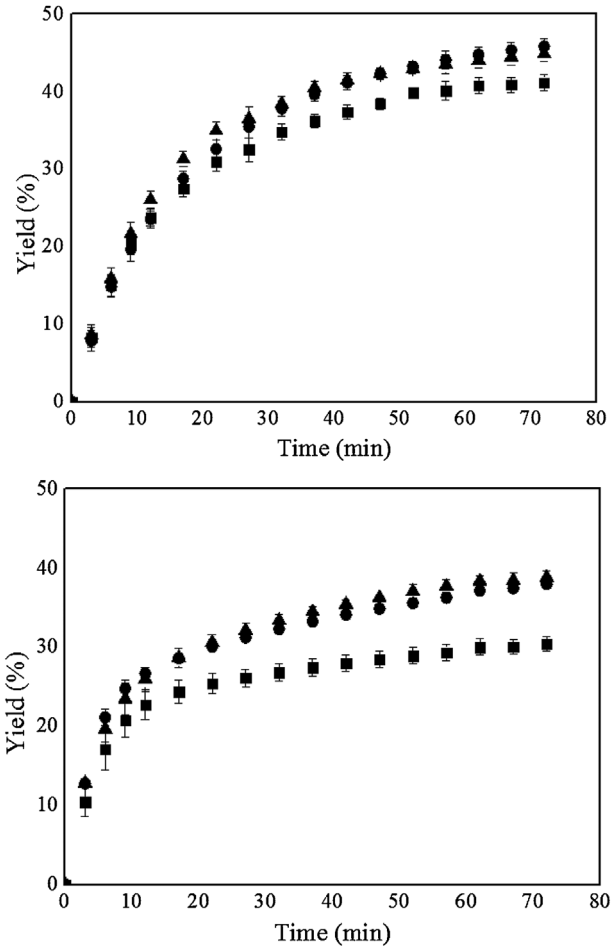

Figure 2. Kinetics of oil extraction from macauba pulp at ■ 40 °C, ● 60 °C, and ▲ 80 °C with: (a) ethanol and (b) isopropanol.

Table 2. Comparison of the results obtained in this study with those reported in the literature

Method/solvent	Experimental conditions	Yield (%)	Reference
PLE/ethanol	60 °C; 62 min; 100 bar	44.78	This study
PLE/isopropanol		37.12	
SFE/carbon dioxide	55 °C, 180 min; 150 bar	14.49	Nascimento et al.[5]
CE/n-hexane	80 °C; 6 h	30.09	
CE/ petroleum ether	40–60 °C; 8 h	16.50	Hiane et al.[12]
CE/ethanol	78 °C; 8 h	~47.00	Lescano et al.[13]
LPE/isopropanol	40 °C; 4 h	27.43	Trentini et al.[16]

PLE: pressurized liquid extraction; CE: conventional extraction (Soxhlet); SFE: supercritical fluid extraction; LPE: low pressure extraction.

an increase in the yield when the temperature was increased from 40 to 100 °C. However, the increase in the yield was relatively small (between 10 and 17 %) considering the wide range of the temperatures applied, indicating that the extraction can be conducted at temperatures below 100 °C.

At higher temperatures the solubility of the compounds of the vegetable matrix in the solvent increases due to the lower viscosity and density of the solvent[21,45] and higher vapour pressure of the solutes,[45,46] which increase the diffusion coefficient[47,48] and the spontaneity of the extraction process.[47,49] Moreover, the application of elevated temperatures softens the tissue of the solid matrix, facilitating the elution of the oil bound in the solvent.[50]

The best yield obtained by PLE represents ~76.7 % and ~77.6 % of those of the Soxhlet method for ethanol and isopropanol, respectively, in shorter time extraction.

The kinetics data for the PLE carried out with ethanol and isopropanol are shown in Figure 2. It is clear from this figure that the extraction occurs in two steps. The beginning of the extraction process is dominated by a washing mechanism (up to ~20 min) which is followed by a diffusion step until the equilibrium yield was obtained (at 62 min for both solvents).

The results in Figure 2a show that the temperature, in the higher range, had little influence on the initial extraction rate for ethanol, but did affect the yield at equilibrium when the temperature increased from 40 to 60 °C. In the extraction with isopropanol, as shown in Figure 2b, the temperature had a more pronounced effect on the yield compared to ethanol, when the temperature increased from 40 to 60 °C. For both solvents, it can be observed that the use of a temperature of 80 °C had no effect on the extraction kinetics and equilibrium yield compared to the results obtained at 60 °C.

A comparison of the best results obtained in this study using the PLE technique with those reported by other authors (including other extraction methods) can be seen in Table 2. It is clear from this table that with the use of polar solvents, in this study and Lescano et al.,[13] higher yields can be obtained in comparison with *n*-hexane,[5,14] which is probably due to the higher percentage of polar lipids in macauba pulp. Trentini et al.[16] obtained lower yields using isopropanol due to the lower temperature applied and because the process was conducted at low pressure. Applying an extraction procedure using supercritical carbon dioxide, Nascimento et al.[5] obtained low yields due to the low solubility of the triglycerides in the solvent.

Extract Characterization

To evaluate the effect of the extraction method and solvent on the extract characteristics, the samples obtained by PLE and conventional extraction were analyzed in terms of the β-carotene and flavonoids contents as well as the fatty acids composition, and the results are shown in Tables 3 and 4. For this analysis, the samples obtained applying the PLE conditions which gave the best oil yields (60 °C) were selected.

It can be seen from the data presented in Table 3 that the fatty acids composition was not affected by the solvent and extraction method used ($p > 0.05$). Oleic and palmitic acids are the main fatty acids identified, in agreement with results reported in the literature for oils obtained from pressing[13,14,51,52] and conventional extraction in a Soxhlet.[1,13,53] On average, the major fatty acids in the macauba pulp oil (oleic and palmitic) represent ~88 % of the oil composition.

The macauba pulp oil contained lower proportions of polyunsaturated fatty acids, such as linoleic (~5 %) and linolenic acid (~2 %), and saturated fatty acids, such as stearic acid (~2 %). Linoleic and linolenic acids, omega-6 (ω-6) and omega-3 (ω-3) are considered essential in the diet and a deficiency can result in

Table 3. Fatty acids composition in macauba pulp oil obtained by PLE and conventional extraction (CE) using ethanol and isopropanol as solvents

Extraction method		PLE		CE	
Solvent		Ethanol	Isopropanol	Ethanol	Isopropanol
Fatty acid[1]	C16:0	25.96 ± 0.28^{aA}	26.39 ± 0.79^{aA}	27.14 ± 2.25^{aA}	26.54 ± 0.18^{aA}
	C16:1	2.80 ± 0.21^{aA}	2.62 ± 0.51^{aA}	2.49 ± 0.12^{aA}	2.25 ± 0.20^{aA}
	C18:0	1.30 ± 0.08^{aA}	1.35 ± 0.12^{aA}	1.51 ± 0.35^{aA}	1.54 ± 0.28^{aA}
	C18:1	62.20 ± 0.33^{aA}	62.69 ± 0.21^{aA}	60.42 ± 1.82^{aA}	61.53 ± 0.71^{aA}
	C18:2	5.28 ± 0.01^{aA}	5.05 ± 1.32^{aA}	5.65 ± 0.58^{aA}	5.39 ± 0.05^{aA}
	C18:3	2.38 ± 0.19^{aA}	2.20 ± 0.11^{aA}	2.48 ± 0.16^{aA}	2.74 ± 0.38^{aA}
	SFA[2]	27.26	27.74	28.65	28.08
	MUFA[3]	65.00	65.31	62.91	63.78
	PUFA[4]	7.66	7.25	8.13	8.13
	PUFA/SFA	0.28	0.26	0.28	0.28

[1]Results in g/100 g of oil; [2]SFA - saturated fatty acids; [3]MUFA - monounsaturated fatty acids; [4]PUFA - polyunsaturated fatty acids. Means followed by different lowercase letters (comparison of methods for same solvent) and uppercase letters (comparison of solvents for same method) did not differ statistically ($p < 0.05$).

Table 4. Flavonoids and β-carotene contents obtained by PLE and conventional extraction (CE) using ethanol and isopropanol as solvents

Extraction method	PLE		CE	
Solvent	Ethanol	Isopropanol	Ethanol	Isopropanol
β-carotene (mg/100 g)	232.44 ± 1.53^{aA}	219.19 ± 3.12^{aB}	212.00 ± 1.45^{bA}	205.02 ± 2.60^{bB}
Flavonoids (mg/100 g)	14.33 ± 0.41^{aA}	16.44 ± 0.51^{aB}	14.22 ± 0.55^{aA}	16.10 ± 0.78^{aB}

Means followed by same lowercase letters (comparison of methods for same solvent) and uppercase letters (comparison of solvents for same method) did not differ statistically ($p < 0.05$)

adverse clinical symptoms such as scaly skin rashes, neurological abnormalities, and poor growth.[13] The amount of ω-6 and ω-3 available in macauba pulp oil can assist in food enrichment and enhance the benefits to human health. According to Coimbra and Jorge,[1] the presence of unsaturated fatty acids has led to the oil attracting interest from a nutritional point of view, as these are precursors of eicosanoids, which play an important role in human health.

However, a relatively higher concentration of unsaturated fatty acids (MUFA and PUFA) and a lower concentration of saturated fatty acids was observed in the lipid composition. The demand for foods with a higher content of unsaturated fatty acids has increased, mainly due to a trend toward a healthy lifestyle.[54] The lower concentration of PUFAs in Macauba pulp oil leads to greater stability and less susceptibility to lipid peroxidation.[55]

Differences in relation to the fatty acids composition of macauba oil pulp reported in the literature may be associated with the maturation stage of the fruits, the storage conditions, the drying temperature, and the extraction method used.[6,56,57]

It can be observed in Table 4 that the extracts obtained from the process conducted under pressurized conditions showed higher levels of β-carotene. This result suggests that the degradation of β-carotene may occur during conventional extraction, since the solvent remains under reflux for a long period of time.[34] Also, in this method the extraction temperatures are higher compared with PLE (78 and 82 °C for ethanol and isopropanol, respectively). Results obtained by Mustapa et al.[46] suggest that β-carotene degradation could occur at 80 °C. Cardenas-Toro et al.[27] analyzed the β-carotene content in the oil obtained from palm fibre using ethanol as the solvent, and observed that the extraction with Soxhlet provided lower concentrations of β-carotene compared with PLE. Sanagi et al.[20] evaluated the efficiency of PLE based on the β-carotene content of a residue oil obtained from pressed palm fibre and reported higher values for this technique compared to Soxhlet extraction.

The solubility of β-carotene is influenced by the polarity of the solvent and interactions with other oil constituents and according to Yunus et al.[58] solvents with higher polarity provide a higher yield of this compound. Thus, the use of ethanol increases the extraction of β-carotene for both techniques assessed. Yunus et al.[58] observed an increase in β-carotene extraction from palm oil using methanol compared with isopropanol. Similar results were obtained by Castro-Puyana et al.[59] using ethanol as the solvent in PLE compared with less polar solvents.

It can be observed in Table 3 that the extractions conducted with isopropanol led to higher concentrations of flavonoids (mg per 100 g) in the macauba pulp oil (16.10 to 16.44) compared to the use of ethanol (14.22 to 14.33), and this was not influenced by the extraction method ($p > 0.05$). Liu et al.[60] reported that solvents with a higher polarity have a lower capacity for extracting flavonoids, which may explain the lower concentrations of flavonoids in the oil obtained using ethanol (polarity of 5.2), which has a higher polarity than isopropanol (polarity of 3.9). For the low-pressure extraction of macauba pulp oil with isopropanol, Trentini et al.[16] obtained an extract with a flavonoids content of 14.78 mg/100 g at 40 °C with 4 h of extraction.

CONCLUSIONS

In this study, oil extraction from macauba pulp using pressurized liquid extraction (PLE) was investigated. It was observed that the use of ethanol as a solvent led to higher yields compared with isopropanol. The temperature, in the range of 40 to 60 °C, influenced the yield obtained by PLE, and this technique can provide ~77 % of the yield obtained with the conventional extraction method (Soxhlet). It was found that ~88 % of macauba pulp oil corresponds to oleic and palmitic acids. The use of ethanol provides extracts with a greater β-carotene content and higher levels of these compounds were observed with the use of PLE. The flavonoids content was higher with the use of isopropanol as a solvent, which was not influenced by the technique used.

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