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Influence of refining on the properties of buriti oil (*Mauritia flexuosa* L.f.)

Influência do refino nas propriedades do óleo de buriti (*Mauritia flexuosa* L.f.)

Influencia del refinado en las propiedades del aceite de buritíi (*Mauritia flexuosa* L.f.)

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Darlyson Santos Silva
ORCID: https://orcid.org/0000-0003-3621-3203
Universidade Estadual de Montes Claros, Brazil
E-mail: darlysonsilva90@gmail.com
Sônia Ribeiro Arrudas
ORCID: https://orcid.org/0000-0002-1257-0460
Universidade Estadual de Montes Claros, Brazil
E-mail: sonia.arrudas@unimontes.br
Franciellen Morais-Costa
ORCID: https://orcid.org/0000-0002-4241-9200
Universidade Estadual de Montes Claros, Brazil
E-mail: franmoraisbio@gmail.com
Yule Roberta Ferreira Nunes
ORCID: https://orcid.org/0000-0003-3328-7506
Universidade Estadual de Montes Claros, Brazil
E-mail: yule.rfnunes@gmail.com
Maria Teresa Oliveira Silva Rodrigues
ORCID: https://orcid.org/0000-0003-4322-5501
Universidade Estadual de Montes Claros, Brazil
E-mail: mariateresaealex@gmail.com
Guilherme Araújo Lacerda
ORCID: https://orcid.org/0000-0003-0878-6557
Universidade Estadual de Montes Claros, Brazil
E-mail: guilherme.lacerda@unimontes.br
Victor Soares Rodrigues
ORCID: https://orcid.org/0000-0003-0440-4494
Universidade Estadual de Montes Claros, Brazil
E-mail: victorsrodriguesbio@gmail.com
Paulo Henrique Fidêncio
ORCID: https://orcid.org/0000-0003-3108-9661
Universidade Federal dos Vales do Jequitinhonha e Mucuri, Brazil
E-mail: paulo.fidencio@ufvjm.edu.br

Abstract
Vegetables oils are renewables sources of ready availability with vast commercial employability. The application of these oils, generally, requires refining procedures for the removal of substances that make it unfeasible, such as phosphatides, proteins and free fatty acids. The objective of this study was to evaluate the effect of acid and aqueous degumming on the physical-chemical properties of crude oil of *Mauritia flexuosa* L.f. (buriti), palm tree of the family Arecaceae, very commercially exploited and well adapted to the Cerrado biome. Physical and chemical analysis were performed as acidity index, peroxide index, refraction, viscosity, density, gas chromatography coupled to the mass spectrometer and infrared spectroscopy before and after the refining process. The acidity, density and refraction indices didn´t change significantly during the treatments; however, significant differences were observed for the peroxide and viscosity parameters after the acid degumming process. Chromatographic and infrared spectroscopy results didn´t reveal changes in the constitution of constituent esters. The acid degumming was more efficient for the removal of undesirable substances, with significant variation ($p < 0.05$) for the peroxide index after this process. However, *M. flexuosa* oil presented good stability, keeping indexes acceptable in view of the current resolutions for its use.
Keywords: Refining; Degumming; Vegetable oil.

Resumo
Os óleos vegetais são fontes renováveis de pronta disponibilidade com vasta empregabilidade comercial. A aplicação desses óleos, geralmente, requer procedimentos de refino para a remoção de substâncias que a inviabilizam, como fosfatídeos, proteínas e ácidos graxos livres. O objetivo deste trabalho foi avaliar o efeito da degomagem ácida e aquosa nas propriedades físico-químicas do óleo bruto de *Mauritia flexuosa* L.f. (buriti), palmeira da família Arecaceae, muito explorada comercialmente e bem adaptada ao bioma Cerrado. Foram realizadas análises físicas e

químicas como índice de acidez, índice de peróxido, refração, viscosidade, densidade, cromatografia gasosa acoplada ao espectrômetro de massas e espectroscopia no infravermelho antes e após o processo de refino. Os índices de acidez, densidade e refração não se alteraram significativamente durante os tratamentos; entretanto, diferenças significativas foram observadas para os parâmetros de peróxido e viscosidade após o processo de degomagem ácida. Os resultados de cromatografia e espectroscopia no infravermelho não revelaram alterações na constituição dos ésteres constituintes. A degomagem ácida foi mais eficiente para a remoção de substâncias indesejáveis, com variação significativa ($p < 0,05$) para o índice de peróxido após este processo. No entanto, o óleo de *M. flexuosa* apresentou boa estabilidade, mantendo índices aceitáveis diante das resoluções atuais para seu uso.
Palavras-chave: Refinação; Degomagem; Óleo vegetal.

Resumen
Los aceites vegetales son fuentes renovables fácilmente disponibles con un amplio uso comercial. La aplicación de estos aceites generalmente requiere procedimientos de refinación para eliminar sustancias que la hacen inviable, como fosfátidos, proteínas y ácidos grasos libres. El objetivo de este trabajo fue evaluar el efecto del desgomado ácido y acuoso sobre las propiedades fisicoquímicas del crudo de Mauritia flexuosa L.f. (buriti), palmera de la familia Arecaceae, explotada comercialmente y bien adaptada al bioma Cerrado. Se realizaron análisis físicos y químicos tales como índice de acidez, índice de peróxido, refracción, viscosidad, densidad, cromatografía de gases acoplada a espectrómetro de masas y espectroscopía infrarroja antes y después del proceso de refinación. Los índices de acidez, densidad y refracción no cambiaron significativamente durante los tratamientos; sin embargo, se observaron diferencias significativas para los parámetros de peróxido y viscosidad después del proceso de desgomado ácido. Los resultados de la cromatografía y la espectroscopia infrarroja no revelaron alteraciones en la composición de los ésteres constituyentes. El desgomado ácido fue más eficiente para remover sustancias indeseables, con variación significativa ($p < 0.05$) para el índice de peróxido después de este proceso. Sin embargo, el aceite de M. flexuosa mostró buena estabilidad, manteniendo índices aceptables en vista de las resoluciones vigentes para su uso.
Palabras clave: Refinación; Desgomado; Aceite vegetal.

1. Introduction

Vegetal oils have a great commercial applicability and the attractiveness of this resource is a renewable source of ready availability (Adekunle, 2015). Due to its low cost, vegetal oil is a raw material with numerous advantages, like biodegradability and low toxicity, characterizing its significant employability in the industrial sector (Saurabh et al., 2011). In addition, the global concern with the growing demand for energy and with the finitude and risks of the fossil matrix has driven the most diverse studies on renewable energy sources, such as, for example, the use of oils of vegetable origin for the synthesis of biofuels (Shereena & Thangaraj, 2009; Cruz et al., 2022; Zanini et al., 2022). Other advantages of biofuels include the fact that they are non-toxic and constitute a neutral source of carbon, that is, they generate an amount of carbon dioxide similar to that absorbed by the source of biomass, plants, via photosynthesis (Zore et al., 2021).

For a better utilization of vegetal oils its necessary a refining process, that promotes the remotion of unwanted substances originated from lipidic oxidation processes, such as phospholipids, free fatty acids and proteins, making it more resistant to degradation, especially during storage (Ramalho & Suarez, 2013; Cordeiro, 2020). The phospholipids present in vegetal oils are found in the hydrated form and they can be removed by treatments involving addition of water, or they can be in the not hydrated form, generally phosphatidic acid salts resulting of enzymatic hydrolysis (Gunstone, 2011).

The degumming is the process of removal of substances as hydratable phosphatides (phospholipids), through aqueous degumming, and not hydrated through acid degumming (Morais et al., 2012; Oliveira et al., 2022). This process considerably modifies physical-chemical characteristics of oils, such as levels of acidity, peroxide, iodine, free fatty acids and of proteinaceous and metabolites remains (Luz et al., 2011a). In Brazil chemical refining is the most used, where after degumming is realized a neutralization, which the objective is reduce the free fatty acids, that when reacting with sodium hydroxide produce soap (Aued-Pimentel et al., 2009). Posteriorly the oil wash is realized, that consists in subsequent additions of water at room temperature and warmed alternately, in order to remove soap remains that it can still be present (Prado et al., 2014). The drying consists in removing water remains, that it can be realized in evaporator with constant agitation or by addition of sodium sulphate, separating posteriorly the precipitate (Encarnação, 2008).

Mauritia flexuosa L.f., (buriti) is a palm tree of the Arecaceae family found in the whole Brazilian territory and especially in Amazon and Cerrado biomes (Cândido et al., 2015). *M. Flexuosa* oil is similar in composition of fatty acids to others oleaginousness of great commercial value as olive oil, canola and peanut, in some cases, presents superior indices (Freire et al., 2016). Considering the presence of many oleaginousness plants in the North of Minas Gerais and the importance of *M. flexuosa* to many industries sectors of national scope, this present work objectived evaluate the physical-chemical characteristics of *M. flexuosa* oil before and after refining process.

2. Methodology

2.1 Study area

The fruits were collected from a natural population in Environmental Protection Area of Pandeiros River - APA Pandeiros, north region of Minas Gerais. The conservation unit embrace municipalities of Januária, Bonito de Minas and Cônego Marinho, presenting different phytophysonomies, like veredas (palm swamps) which has *M. flexuosa* as a characteristic palm species (IEF, 2015).

2.2 Extraction and storagement

The oil used for physical-chemical analysis was extracted from the fruit's mesocarp. Samples were benefited with the cold pressuring method of extraction, with a mechanical press ERT 75 II - Scott Tech. After extraction process, the obtained raw oil was put in a centrifuge for separation of impurities. These processes were executed in the Agricultores Familiares cooperative and Agroextrativistas Grande Sertão LTDA.

2.3 Oil tenor

The oil's tenor was obtained by chemical extraction with solvent through a device of Goldfish type, according to methodology of National Institute of Science and Animal Technology. The extraction process of the oil occurred by continuous reflux of the solvent in samples for 4 hours, with condensation velocity between five to six drops per second. After extraction, the cups, returned to heating chamber at 105° C, for 30 minutes, for complete removal of reagent. The difference between the last cup weight and the empty cup corresponded to the extracted oil's weight. The calculation of yield was obtained by gravimetric relation (Detmann, Souza & Valadares, 2012).

2.4 Physical-chemical analysis

The acidity level was determined by the method that uses as titrant, sodium hydroxide 0.01 mol/L and phenolphthalein as indicator (American Oil Chemists` Society – AOCS, 2004). To refraction index determination an Abbé refractometer was used, adjusted with distilled water at 25 ºC (AOCS, 2004). To density determination a 25 mL pycnometer was used at 25°C, determining posteriorly the reason of mass of the sample in relation to the mass of water (AOCS, 2004). To determination of viscosity a Ford cup viscosimeter was used, using the number three orifice according to the characteristics of the sample. The viscosimeter was leveled, adjusting the levelling feet. The orifice was closed manually and the cup filled with the sample to the highest level, spilling the sample slowly to avoid bubble formation. The excess of sample was removed with a glass plate. The flowability time was measured in seconds for viscosity calculation (ASTM, 2016). Thcamarge peroxide indexes were realized according to the method of AOCS (2004), Where 5 g of oil were dissolved in 25 mL of acetic acid solution – chloroform (3:2 v/v) with addition of 1 mL of saturated solution of potassium iodate. After repose, was added distilled water and 2 mL of 1% amid solution. The titrimetric method was applied, having as titrant solution the sodium thiosulphate at 0.01M (AOCS, 2004).

2.5 Gas chromatography coupled to mass spectrometry (GC-MS)

The samples were analyzed through chromatography in gaseous phase coupled to mass spectrometry, using the device Perkin Elmer Clarus 560-600 MS, with fused silica capillary column DB-5 (30 m; 0,25 mm internal diameter; 0.25 μm film) and helium as carrier gas. The injector temperature was at 260 °C. For the column, the initial temperature was at 100 °C, increasing from 100° to 250 °C in the rate of 10 °C/min, staying in this temperature for 40 min. The detector temperature was at 290 °C and in the system interface GC-MS at 290 °C (Collins, Braga & Bonato, 1997). The mass detector was adjusted to operate with electron impact ionizations (70 eV) and mass scan with interval of 30 to 600 Da. The compounds identification was realized through the comparation of the samples mass spectra to those existing in the device database, with data of literature and with standards injections of samples. For quantitative analysis of the device GC-MS it was calibrated with reference compounds, representatives of the principal classes of compounds present in the samples (Munari, Cavagnino & Cadoppi, 2007).

2.6 Infrared spectrocopy (IR)

Samples were submitted to analysis by infrared spectroscopy in spectrometer with Fourier transform FTIR 640. The spectra were registered in KBr tablets.

2.7 Degumming

For the acid degumming process, a sample of 200 g of raw oil was heated to 90° C and acidified with 0.30 mL of phosphoric acid. After 10 minutes of agitation, was added 9.8 mL of sodium hydroxide at 5%. The oil was centrifuged at 3000 rpm for 20 minutes, according to adapted methodology (Penedo & Coelho, 1997). Posteriorly, the oil was poured in a separate hopper for process of washing with water at room temperature and at 90 °C alternately every 30 minutes. After testing the pH of the last washing, the sample was liberated for dehumidification, with addition of 4 g of anhydrous sodium sulfate, maintained for 15 minutes e posterior discard of precipitate. In the aqueous degumming, 200 g of raw oil were heated at 80 °C, adding 8 ml of distilled water with posterior agitation for 30 minutes. The subsequent processes of neutralization with sodium hydroxide at 5%, washings with water in alternative temperatures and dehumidification with anhydrous sodium sulfate were identical to the utilized in the acid degumming.

2.8 Statiscal analysis

For the physical-chemical analysis was utilized the One-way ANOVA test and for percentage of majoritarian fatty acids, Two-way ANOVA and Turkey test for multiple comparation between the averages with 5% of probability ($P<0.05$). The software used was GraphPad Prism 6.01.

3. Results and Discussion

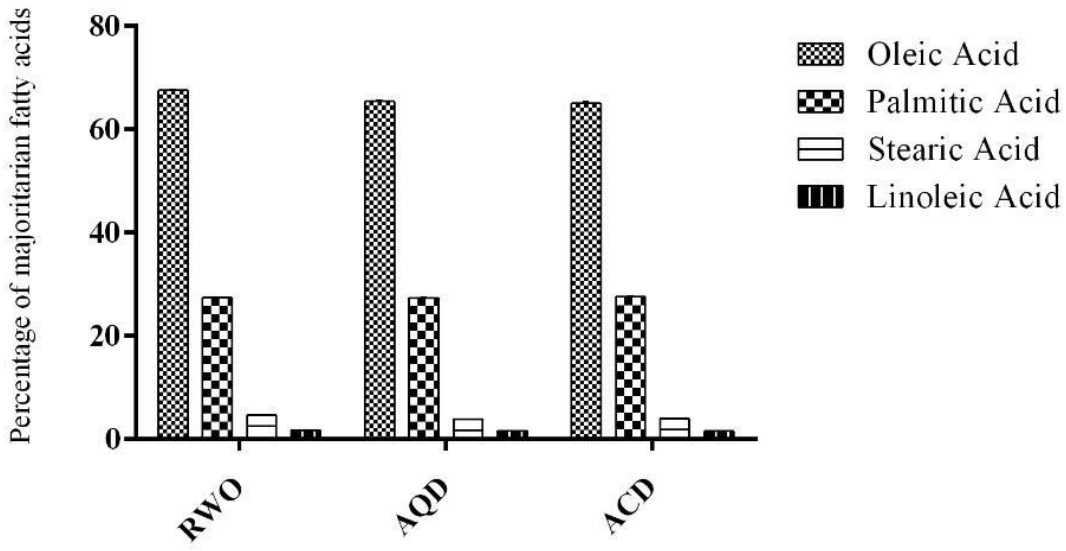
3.1 Oil tenor

M. flexuosa presented an oil tenor of 42% in dry mass of the mesocarp. This elevated rate is present in other oleaginousness of the Arecaceae family such as “babaçu” (*Orbignya phalerata* Mart.) between 62% and 66% and in “urucuri” (*Attalea phalerata* Mart.), with 60% of oil tenor (Stachiw et al., 2016).

3.2 Caracterization of *Mauritia flexuosa* oil

The acid and aqueous degumming did not lead to alterations in composition of the fatty acids of the *M. flexuosa* oil (Figure 1). There was no significative difference between the treatments for majoritarian oleic, palmitic, stearic and linoleic fatty acids (Table 1).

Figure 1 - Percentage of majoritarian fatty acids presents in buriti oil before (OBR) and after the refining treatments (DAQ e DAC).



Source: Authors.

Table 1 - Average proportion of majoritarian fatty acids of buriti oil before and after refining processes.

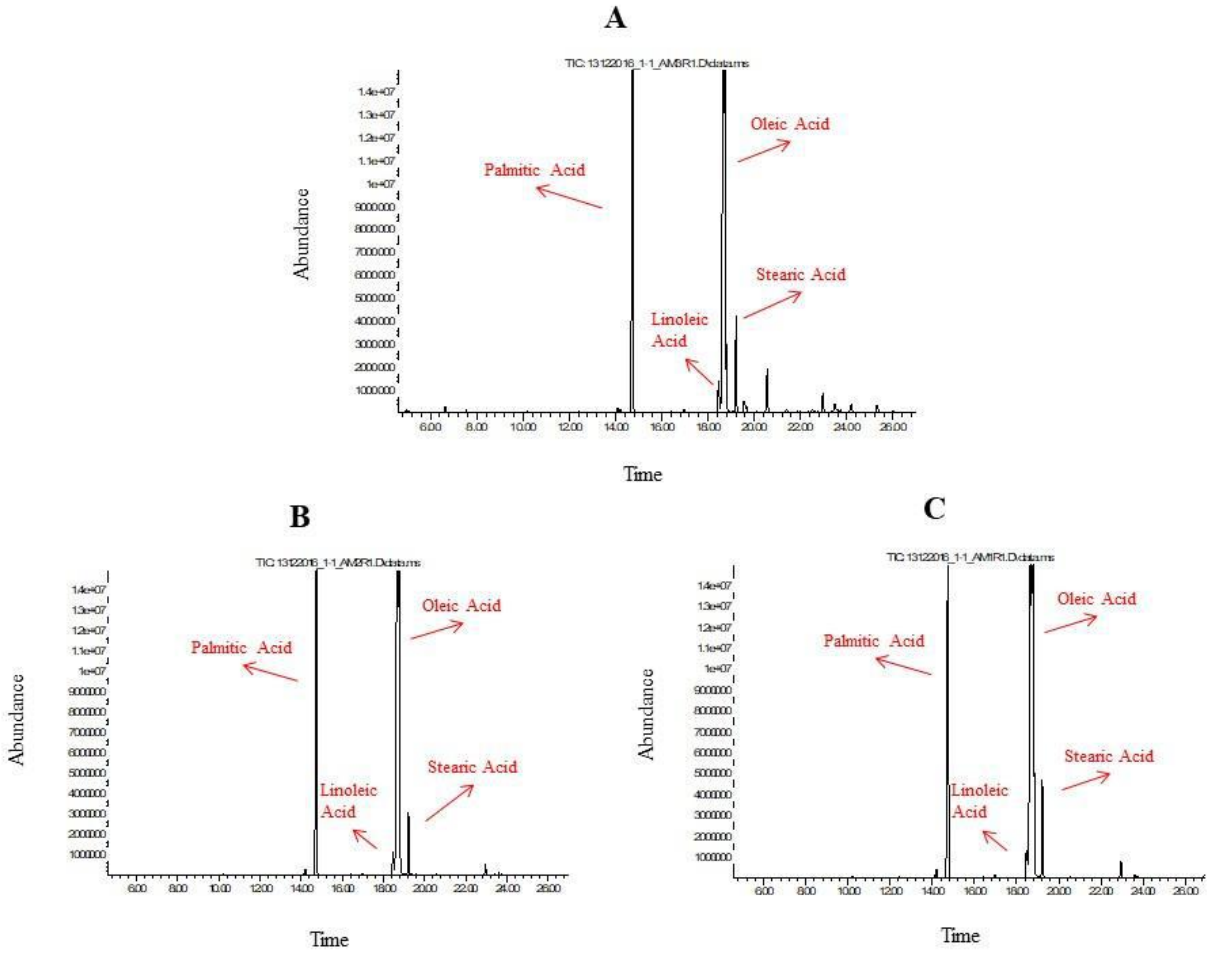
Compounds	Relative area rate (%)			Time of retention (min.)		
	RWO*	ACD*	AQD*	RWO	ACD	AQD
Oleic Acid	67.43	64.72	65.64	18.67	18.66	18.67
Palmitic Acid	27.55	27.77	27.19	14.75	14.76	14.74
Stearic Acid	4.66	4.15	3.86	19.26	19.21	19.20
Linoleic Acid	1.69	1.56	1.63	18.46	18.46	18.45

* Raw Oil (RWO), Acid Degumming (ACD) and Aqueous Degumming (AQD). Source: Authors.

This constancy observed in the oil's constituent fatty acids even after refining is a relevant characteristic for its utilization (Oliveira et al., 2013). The averages of relative areas obtained in chromatographic analysis are in accordance with the values cited by Freire et al. (2016) for oleic and palmitic acids, but differs for stearic and linoleic acids.

The chromatograms obtained in this study showed similar peaks for samples before and after the refining processes (Figure 2).

Figure 2 - Chromatograms of buriti oil and its majoritarian acids. (A) raw oil; (B) after aqueous degumming; (C) after acid degumming.

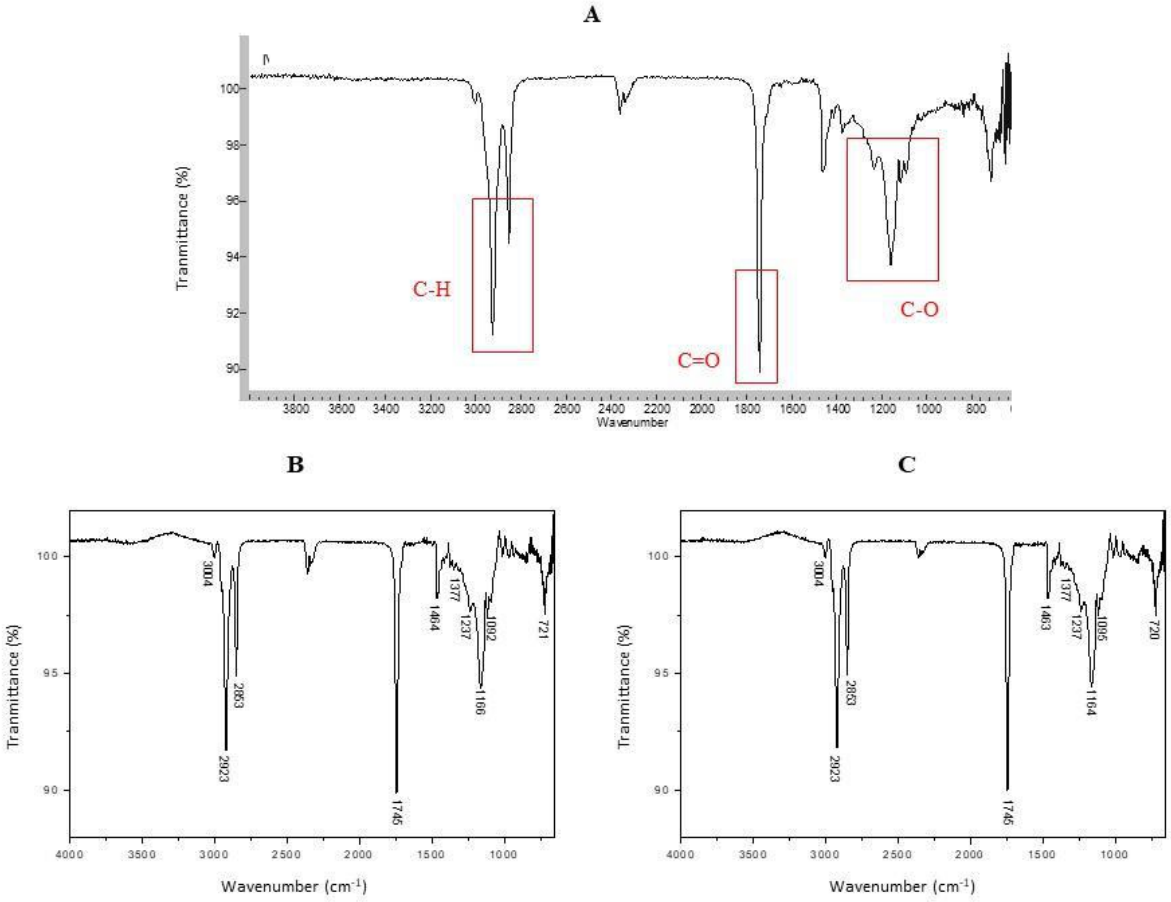


Source: Authors.

Luz et al. (2011b) furthermore was constated a stability of the *M. flexuosa* oil in its study comparing the chromatograms of the treatments of thermal cracking and thermal cracking with solid catalyzer for obtainment of biodiesel with similar characteristics to mineral diesel. Herewith, is possible to infer that the refining processes do not interfere in the basic constitution of the *M. flexuosa* oil.

The infrared spectra (Figure 3-A, B, C) presented absorption bands around 2920 and 2850 cm-1 attributed to C-H sp3 stretching (groups CH, CH2 and CH3) common in many classes of aliphatic compounds such as fatty esters (Silverstein et al., 2006).

Figure 3 - Collection of spectra in infrared of buriti oil during the treatments with evidence of the main functional groups of fatty esters. (A) raw oil infrared; (B) after ACD and (C) after AQD.



Source: Authors.

The absorption bands around 1745 cm-1 represents the C=O stretching of carbonyl, common in fatty acids esters. The absorption around 1460 cm-1 corresponds to the angular deformation band in the plan, symmetrical (CH2) and asymmetrical (CH3); the band around 1377 cm-1 is attributed to the angular deformation in the plan, symmetrical (CH3). The bands around 1237, 1160, 1090 cm-1 are attributed to ester C-O stretching. Is worth mentioning that, the absence of large band (type "bell") of O-H stretching, between 3400 – 2500 cm-1 which indicates the absence of free fatty acids.

Similar values were found by Albuquerquer, Guedes, Alcantra and Moreira (2003); Pardauil et al. (2011) when analyzing the infrared spectra of *M. flexuosa* in comparation to babaçu oils, andiroba and copaiba, being the absorption bands around 2922 and 2854 cm-1 for C-H stretching, the band around 1744 cm-1 for carbonyl C=O and bands around 1040 to 1290 cm-1 attributed to the functional group ester C-O.

3.3 Acid and aqueous degummings

The acid level wasn´t present significative difference although a reduction of its percentage of RWO (3.14%) for ACD (2.84%) and for AQD (2.77%) (Table 2).

Table 2 - Averages of physical-chemical analysis of buriti oil before and after refining processes.

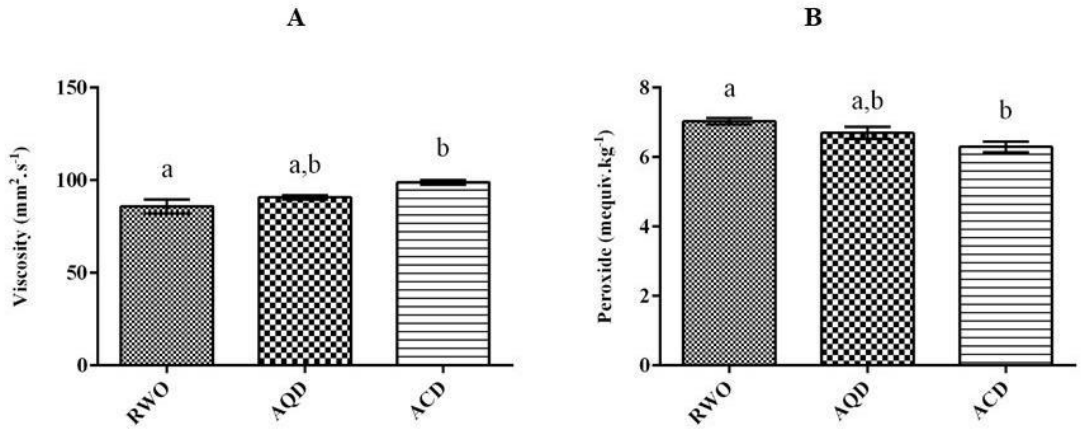
Physical-Chemical Analysis	**RWO** Average±standard deviation*	**ACD** Average±standard deviation*	**AQD** Average±standard deviation*
Acidity Index (%)	3.14 ± 0.35^{a}	2.84 ± 0.08^{a}	2.77 ± 0.04^{a}
Peroxide Index (mequiv.kg^{-1})	7.03 ± 0.1^{a}	6.29 ± 0.26^{b}	6.69 ± 0.30^{ab}
Viscosity (mm².s^{-1})	85.87 ± 6.3^{a}	98.90 ± 2.24^{b}	90.76 ± 2.10^{ab}
Density (mg.mL^{-1})	46.14 ± 0.00^{a}	46.24 ± 0.00^{a}	46.26 ± 0.00^{a}
Refraction (25°C)	1.46 ± 0.00^{a}	1.46 ± 0.00^{a}	1.46 ± 0.00^{a}

*Means followed by the same letter in the columns are not significantly different at a significance level of 0.05 of probability.
Source: Authors.

This small reduction can be related to the degumming process, since as, this value is related to purity conditions of the oils' constituent lipids. Elevated acidity levels indicate a higher lipids decomposition and a higher consume of sodium hydroxide for pigment neutralization (Aquino et al., 2012).

Other two parameters that weren´t presents variation during the treatments were the relative density and refraction index. According to Silva, Carvalho, Conrado, Fook and Leite (2012), the density of an oil is a key-factor, since, in smaller values present a higher oil fluidity increasing its applicability. In turn the refraction index, is specific for each oil and can suffer alterations by thermal and oxidative processes (Jung, Park & Yoon, 2016). In this study, the refining processes didn´t modify the oil constitution to the point of influencing these two indices that remained unaltered. There was significative variation ($p < 0.05$) of the peroxide index after the acid degumming process (Figure 4-B).

Figure 4 - Significative difference for viscosity and peroxide values after acid degumming treatment. (A) Variation analysis for viscosity value. (B) Variation analysis for peroxide value. Means followed by the same letter are not significantly different according at a significance level of 0.05 of probability.



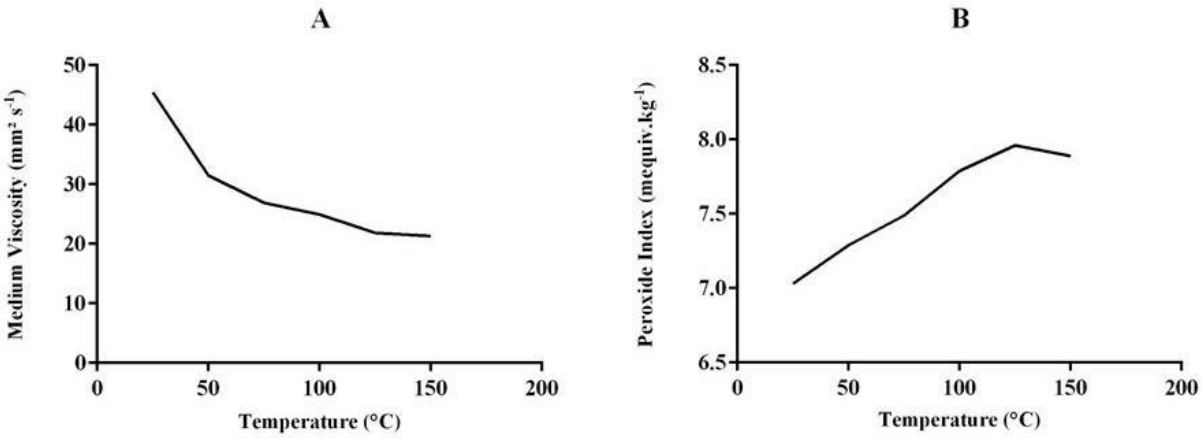
Source: Authors.

The obtained values for this index (Table 2) assemble under the 15 meq/kg limit established by the Resolution RDC nº 270, September 22th of 2005 ANVISA (Brasil, 2005) for vegetal oils. The viscosity also significatively variated showing an elevation in its values after the treatments AQD and ACD (Figure 4-A).

3.4 Effects of temperature in the values of peroxide and viscosity

The peroxide and viscosity values are physical-chemical properties susceptible to significative variations in function of temperature. In oils storage at room temperature, the viscosity tends to increase with the elevation of peroxide index (Ribeiro et al., 2010*)*. Such peroxides, in turn, are related to the deterioration of fats and oils by hydrolysis and oxidation (Mitrea et al., 2022). However, with temperature variation, the relation of viscosity with peroxide index is different (Figure 5-A).

Figure 5 - (A) Reduction of drainage time of buriti oil in function of heating. (B) Elevation of peroxide value of buriti oil in function of temperature increase.



Source: Authors.

According to Canciam (2010) the viscosity is connected to the size of the constituent carbon-chains of an oil and also to the amount of unsaturation of these molecules which have its forces reduced with temperature increase, promoting the decline of this index. Due to the process of auto-oxidation the peroxide index tends to get higher proportionally to the temperature increase (Figure 5-B). However, there is a small decline in this index, at a given temperature, related to the oxygen consume and the transformation of peroxides into hydroperoxides. This step consists in the finishing of the auto-oxidation process, where the absence of free radicals to reacts with oxygen promotes the end of the process (Manhães, 2014).

4. Conclusion

The degumming processes reduced the acidity and peroxide values of raw *M. flexuosa* oil. There was a significative reduction for the peroxide index after acid degumming and an increase of viscosity in the same process. The degummed oil assembles inside the acceptable parameters for its commercial utilization even though after refining processes. The stable characteristic of *M. flexuosa* oil was observed before degumming treatments involving addition of chemical substances and temperature increase necessary for the treatments. The results after the physical-chemical analysis, chromatographic and spectrometric in infrared confirmed this stability.

Research of this nature should be extended to other palm trees native to the cerrado, such as those of the genus *Acrocomia* and *Attalea*, covering not only the mesocarp as a source of raw material for the production of oils, but also the almond. In this way, it is possible to add value to plant species and encourage the income of communities and traditional peoples who depend on the use and exploitation of palm trees as a source of income and survival.

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