

PHYSICO-CHEMICAL ANALYSIS OF THE VIABILITY OF MURUMURU FAT FOR BIODIESEL PRODUCTION

Dhyenifer Mayara Guimarães de Oliveira

Universidade Federal Rural da Amazônia
Capanema – Pará
<https://lattes.cnpq.br/9863116291390110>

Débora da Cruz Souza

Universidade Federal Rural da Amazônia
Capanema – Pará
<http://lattes.cnpq.br/9024138185276156>

Marcelo Costa Santos

Universidade Federal Rural da Amazônia
Capanema – Pará
<http://lattes.cnpq.br/8380189608965320>

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Abstract: Society's growing energy demand brings to light its environmental problems in meeting, in a sustainable way, the required development requirements. Murumuru comes in this context as an alternative source of raw material for the production of biofuels. This study analyzes the technical feasibility of fat from the Murumuru oilseed (*Astrocaryum Murumuru*) for biodiesel production through the transesterification reaction. Analyzes were carried out to characterize the fat, including moisture content obtained following method No. 972.20 of the AOAC (1997), ash content using the methodology adapted from ASTM E1755-01 (ASTM, 2003), volatile material content using the methodology of D3175-07 (ASTM, 2013) and fixed carbon which was determined by the ASTM D3172-89 methodology (ASTM, 2013), as well as acidity index with the official AOCS Cd 3d-63 method, saponification index following the official AOCS Cd 3 methodology -25 (1997), Fourier transform infrared spectroscopy (FTIR) analysis and fat extraction yield. Based on the results obtained, the moisture content was determined to be 35.93%. The values of the immediate analyzes (ash content, volatile material and fixed carbon) were satisfactory, recording values of 1.45%, 92.07% and 6.47% respectively. The results of the acidity and saponification analyzes were 2.98% and 260.4% respectively, results consistent with those found in the literature.

Using Infrared spectroscopy analysis, the existence of absorption bands corresponding to axial, symmetrical and asymmetrical stretching or deformation of the C-H bonds of the CH₂ and CH₃ group was observed. It is possible to observe that the fat presented a broad band in the region 3677, this is an aspect of axial deformation of (O-H) groups in hydrogen bonds. The fat yield varied between 54.64% and 53.87, with an average of 53.73%, which indicates a satisfactory percentage.

Keywords: Murumuru; Vegetable fat; Biofuel.

INTRODUCTION

The growing demand of society, as well as its energy needs, brings to light its environmental problems in meeting the required development requirements in a sustainable way. An addition to this demand is the production of biofuels, which is a renewable source of energy produced from natural raw materials (Demirbas, 2011).

The objective of this "escape" valve in the production of biofuels seeks to minimize the disruption of the current global energy matrix, largely composed of non-renewable sources of fossil carbon, such as oil, hard coal and natural gas, where studies envisage the possible scarcity of these resources. As important as the depletion of sources is the role they play in the environment with the emission of greenhouse gases that cause drastic changes (Peres, 2006).

The use of plant biomass as an alternative source of energy is enhanced due to its renewable nature, wide availability, biodegradability and low cost. These factors encourage research and development of new renewable basic inputs, as well as contributing to possible economic and social development. (Suarez, 2007).

Murumuru comes in this context as an alternative source of raw material for the production of biofuels. This medium-sized palm tree with a stunted trunk, commonly known as Murumuruzeiro, reaches around 10 to 15 meters in height and around 17 to 27 centimeters in diameter. The leaves are pinnate and vary in size from 12 to 20 cm, and their stems are covered with long black thorns that make harvesting difficult (Bezerra, 2012).

The core of the fruit is made up of a woody, hard and thin shell, covered by endocarp filaments. The kernel has an almond, conical in shape, and this almond is made up of

firm white masses. On the outer surface, the almonds are gray in color (Lopes, 2007). Yields of 27 to 29 kg of almonds can be obtained per 100 kg of dry kernels, with a moisture content of 12 to 15%, up to 5 - 6%. This type of almond contains around 40% to 42% fat. (Pesce, 1941).

For biofuel production, raw materials can be grouped into three groups (Atabani et al., 2012): Biofuel production can be divided into three different conversion technologies: first generation biofuels; second generation biofuels; third generation biofuels. First-generation biofuels are produced from edible plant materials produced by agriculture, including bioethanol, biodiesel, vegetable oil and biogas. Second-class biofuels are fuels produced from cellulose and other plant fibers present in wood or inedible parts of plants (Mira, 2012).

METHODOLOGY

STUDY AREA

To develop and carry out the research, the proposed procedures were followed. It began with the process of collecting the raw material Murumuru (*Astrocaryum Murumuru*), in the area known as ``*Quarta Travessa*`, located in the interior of Capanema, on the Cuuba branch, precisely located by the coordinates of latitude 1°10'52" and longitude 47°09'08". The analyzes in this study were primarily carried out in the laboratory of the Universidade Federal Rural da Amazônia-UFRA, with the exception of the analysis of the infrared spectrum, which was carried out in the Laboratory of Vibrational Spectroscopy and High Pressures (PPGF/UFPA).

In possession of the Murumuru almonds, pre-treatment was carried out, consisting of breaking the almonds, separating the shells, cutting the almonds, weighing the sample, drying and crushing.

MILLING

It is the initial process of physical characterization, in which the seeds were crushed in order to transform them into paste or bran. This process is essential to facilitate the removal of the oil present in the seeds, making it more accessible for the next step. The comminution process was carried out in a FORTINOX knife mill, willye type, model STAR FT-50.

IMMEDIATE ANALYSIS

Carrying out the immediate analysis process of the Murumuru almond consisted of the following analyses: Moisture Content, Volatile Materials Content, Ash Content and Fixed Carbon.

MOISTURE CONTENT

The calculation involved weighing 5g of sample in petri dishes using a Weblador analytical balance, model BR1700434. The samples were dried at 105° for 24 hours in the Lucadema oven, model 80/85, until reaching a constant mass. After this period, the samples were removed from the oven, cooled in a desiccator and weighed again to determine the percentage of moisture, according to method n° 972.20 of the AOAC (1997).

ASH CONTENT

Determining the ash content is important for evaluating the purity of the material and its influence on the quality of the biodiesel. It was carried out according to a procedure similar to the official ASTM Standard Method Number E1755-01 and calculated according to the equation described in the official methodology. The process began with the identification of porcelain crucibles intended for calcination in an oven at 105°C. After identification, the crucibles were weighed individually on a SHIMADZU ATY224 analytical balance, first without the sample

and then with the addition of 2g (two grams) of sample. The crucibles were sealed and inserted into a SOLIDSTEEL muffle furnace for 12 minutes at 105°C, followed by 30 minutes at 250°C and, finally, 180 minutes at 575°C. After this, cooling occurred in a desiccator with silica gel until it reached room temperature, the ash in the crucibles was weighed and recorded.

VOLATILE MATERIALS CONTENT

The content of volatile materials refers to the fraction of material that vaporizes when heated at high temperatures, this includes volatile organic components present in the seeds. It was carried out according to a procedure similar to the ASTM Standard Method Number and ASTM Method Number D3175-07.

Initially, two porcelain crucibles were selected and calcined at 105°C for 1 hour. They were then weighed without sample and then with 2g of sample in each. The crucibles, together with the sample, were then placed on the muffle door previously heated to 900°C for 3 minutes, and after this period, they were transferred to the interior of the muffle and remained with the door closed for 7 minutes. Subsequently, the crucibles were removed from the muffle and left in the desiccator until they reached room temperature, followed by weighing again.

CARBON CONTENT

The fixed carbon content was determined according to ASTM Standard Method Number D3172-89 “Standard Practice for Proximate Analysis of Coal and Cokel”. According to the standard, it is possible to identify the fixed carbon content, due to the values found in the determination of ash and the content of volatile materials.

MURUMURU FAT EXTRACTION PROCESS BY SOLVENT

Murumuru almonds were subjected to solvent extraction following the Goldfish method and the use of Hexane PA ACS. A Weblador analytical balance, model M214Ai, was used to weigh the material used in the extraction cartridges, as well as to weigh approximately 8g of sample in petri dishes. The cartridges have been prepared to begin the fat elimination process. The procedure was repeated in triplicate, but with 5 samples to increase the amount of fat extracted.

The extraction process firstly covers the preparation of the cartridge with the weighed sample, then the cartridge was placed in a stainless-steel basket and connected to an extractor and then a 190 mL reboiler containing 100 mL of solvent was inserted, submerging the cartridge.

The extractor temperature was adjusted and maintained constant at 120°C for an hour and a half, with a power of 1800 W, carrying out the extraction process. After this period, the system was closed using a PTFE (polytetrafluoroethylene) tap in the extractor to recover the solvent, which could be reused in another batch. The reboiler was removed from the extractor and placed in an oven at 105°C for 20 minutes to evaporate any remaining solvent. Finally, the reboiler was placed in a silica desiccator until it reached room temperature and the extracted fat was weighed and stored in a bottle using a glass funnel. After extraction, the process residue was dried in a Lucadema oven, model 80/85, at 105°C for 24 hours. After drying, the samples were cooled in a desiccator until they reached room temperature.

PHYSICO-CHEMICAL ANALYSIS

Assessment of the quality of fat extracted from Murumuru almonds consisted of physical-chemical characterization using the methods: acidity index and saponification index.

ACIDITY LEVEL

The acidity index is a measure of the amount of free fatty acids present in Murumuru oil. Its determination is extremely important, as high levels of acidity can directly interfere with the quality of biodiesel. It was carried out according to the official AOCS Cd 3d-63 method (AOCS, 1999) and calculated according to the equation described in the standard.

The procedure began with weighing 1g of sample (Murumuru fat) in a 250mL Erlenmeyer flask. Then, 25mL of a 1:1 solution of isopropyl alcohol and 25mL of toluene were added. After sample dilution, 2 drops of phenolphthalein were added as a turning point indicator. The sample was titrated with a liquid KOH solution (0.2 N) using a volumetric buret until the color turned pink. And then note the volume of KOH used in the titration.

SAPONIFICATION INDEX

The saponification index is the process of converting the esters present in Murumuru oil into soap and glycerol. It was carried out following the official methodology AOCS Cd 3-25 (1997), applicable to all oils and fats.

Initially, 1g of sample was weighed in a 250mL Erlenmeyer flask. Then 25mL of 4% KOH alcoholic solution / (M/V) was added using a volumetric pipette. The Erlenmeyer flask was then connected to the device's condenser and the internal contents were gently brought to a boil under reflux. After 1 (one) hour of process, the heating plate was turned off and after cooling the Erlenmeyer flask and condenser.

OBTAINING INFRARED SPECTRA

The qualitative chemical characterization of Murumuru almond fat was carried out using Infrared Spectroscopy (FTIR).

The infrared spectroscopy was carried out at the Vibrational Spectroscopy and High-Pressure Laboratory (PPGF/UFPA). Fourier transform infrared spectroscopy (FTIR) analysis was applied to determine which functional groups were present in Murumuru (*Astrocaryum Murumuru*) fat. The spectra were obtained with an FTIR spectrometer, and were measured in an ambient environment using ATR (BRUKER, model VERTEX 70 V). The spectral resolution used was 4 cm⁻¹, the scanning range was 4000 to 400 cm⁻¹ and the Scan number was 32 Scan/s. the spectrum graph was generated using the ORIGIN 2016 software.

RESULTS

IMMEDIATE ANALYZES

The following table shows the average percentage content of moisture, ash, volatile material and fixed carbon of murumuru almonds (*Astrocaryum murumuru*)

Parameters	Average (%)
Moisture	35,93
Ashes	1,45
Volatile materials	92,07
Fixed carbon	6,47

Table 1: Results of immediate analysis of murumuru almond

Source: Authors, 2023.

The average moisture content was 35.93%, higher than the value of 10.55% reported by Teixeira (2010) for the Murumuru almond and lower than the value of 56.42% found by Siqueira (2014) for the tucumã pulp. It was also lower than the 33.66% obtained by Lima (2021) for buriti mesocarp.

The difference in values can be explained by variation in biomass, degradation methodology, desiccation time and planting and soil conditions. Moisture is a critical parameter, directly linked to fruit stability.

The average ash content was 1.45%, similar to the value of 1.37% found by Teixeira (2010) for Murumuru almonds. This value is considered low and satisfactory, as values above 7% can harm the combustion process.

The volatile material content was 92.07%, significantly higher than the values found in other studies for different biomasses. This difference may be related to the carbonization temperature and the chemical composition of the biomass. The presence of volatile materials is important as it affects ignition and reactivity in the combustion process.

The average fixed carbon content was 6.47%, considerably lower than the values reported by Magriotis (2018) of 19.3% for Murumuru cake. This can be justified by the high content of volatile materials in the sample. An increase in fixed carbon will lead to increased reactivity in the combustion process, that is, the high level of fixed carbon would lead to slower burning, which implies a greater resistance time within the equipment (Spanish, 2015).

YIELD OF THE FAT EXTRACTION PROCESS

The table presents the values referring to the results of the yield per cartridge after the extraction and desiccation process, according to the methodology of the Adolfo Lutz Institute (2008).

	Sample mass (g)	Sample mass (g) after extraction	Mass yield (%)
Cartridge 1	7,78	6,68	20,03
Cartridge 2	7,93	7,10	14,87
Cartridge 3	7,90	6,63	22,72
Average	7,87	6,80	19,20
Standard deviation	0,06	0,21	3,26

Table 2: Residue yield per cartridge

Source: Authors, 2023.

The average mass yield was 19.24%, with cartridge 3 presenting the highest yield at 22.78%. Comparing with previous studies, Lima (2021) obtained yields of 50.66% to 54.15% for buriti pulp, while Silva (2011) obtained yields of 65.22% to 94.02% for the same biomass. The results of this work are close to those of Lima (2021) and differ from those of Silva (2011), possibly due to the removal method, type of biomass and amount of mass used. The processing of almonds to produce fat had a good yield, considering the small amount of mass used.

The average fat yield from Murumuru almond was 53.73%, which is significantly higher than the yield rates obtained in other studies. This result is considered satisfactory, especially considering the small amount of hexane used in the removal process. According to Pesce (2009), Murumuru fat industrially obtained by pressing has a yield of 35%, while solvent extraction easily exceeds 40%. França (2006), Altman (1958) and Balick (1979) present similar results (41.2%, 38% and 40% respectively). Therefore, the results of this study present a satisfactory yield, highlighting the high energy value and high percentage of yield.

PHYSICOCHEMICAL ANALYSIS

The following table presents the data obtained in the physicochemical analyzes for the characterization of murumuru fat (*Astrocaryum murumuru*).

Parameters	Average
Acid value (mg KOH/g)	2,98
Saponification index (mg KOH/g)	260,4

Table 3: Result of physicochemical analyzes of murumuru fat

Source: Authors, 2023.

Murumuru fat had an acidity value of 2.98 mg KOH/ g⁻¹. This value was lower than that found by Almeida (2018) and higher than the value obtained by Lopes (2007). The difference in results can be attributed to the fat elimination process, the time between sample collection and processing, temperature, atmospheric air and light. It is worth mentioning that the acidity index is important to determine the quality of the fat, in this case, the value is close to that of other literature and meets the parameter established by RDC n° 270 of ANVISA (BRASIL, 2005).

The saponification index obtained was 260.4 mg KOH/g, a result close to the values found in the literature. This way, presenting a satisfactory result and within the framework of ANVISA resolution RDC n° 270 (BRASIL, 2005).

INFRARED SPECTRUM ANALYSIS

The spectra obtained by infrared spectroscopy for the fat studied are shown in Figure 1.

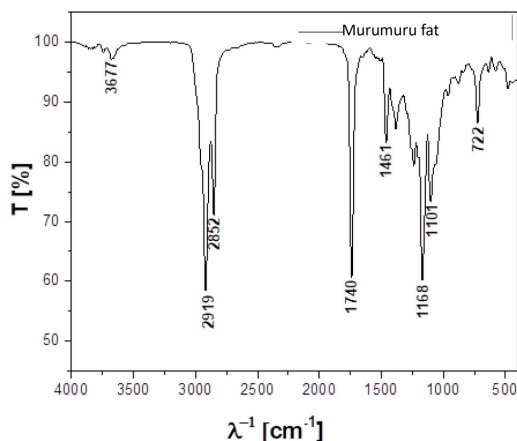


Figure 1: Infrared absorption spectroscopy for murumuru fat.

Source: Authors, 2023.

When observing the spectra obtained by infrared spectroscopy for Murumuru almond fat, it appears that in the region from 2919 to 2852 there is the existence of absorption bands corresponding to stretching or axial deformation of the C–H bond, both for –CH₂ and –CH₃. The absorption bands in 1740–1736 depict the C=O stretching, characteristic of esters. It is possible to observe that fat presented a broad band between 3677–3440, this is an aspect of axial deformation of groups (O–H) in hydrogen bonds, in a way that suggests the presence of water.

CONCLUSION

After analyzing the results, we can state that the global purpose envisaged in this study was successfully achieved. The evaluation of the data suggests that Murumuru fat can be used for the production of biodiesel, since its physicochemical properties demonstrate that this oilseed is a viable technical choice as a raw material for this purpose.

- The first stage of this project involved the collection of oleaginous biomasses, Murumuru (*Astrocaryum Murumuru*), as a raw material for the production of biodiesel. The collection was carried out

in accordance with the best accessible practice, ensuring the obtaining of adequate material, with sample quality and representativeness for the subsequent process.

- Biomass pre-treatment, including selection and cleaning of Murumuru almonds, was moderate to eliminate impurities and ensure that the raw material was ready for fat removal.
- Carrying out immediate analyzes of Murumuru almonds, with the exception of moisture, provided essential information, indicating their quality and viability for biodiesel production.
- The moisture content obtained was higher than literature values, possibly due to fruit storage, temperature

conditions, sample exposure time and soil characteristics.

- The acidity index obtained meets ANP standards, while the saponification index presents values compatible with the literature.
- Analysis of the infrared spectrum reveals absorptions corresponding to stretching and axial deformations, both symmetric and asymmetric, reflecting significant variations in the distances between the nuclei.
- The fat yield was satisfactory, demonstrating effectiveness in its production, considering the high natural oiliness of the almonds.
- It can be concluded that Murumuru fat has viable potential for fuel production.

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