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Thermo-physical Properties and Oxidative Stability of the Oil of Babassu Palm (*Attalea vitrivir* Zona, Arecaceae)

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Abstract

Population growth and the concern about its environmental impact have increased the demand for vegetable oils in recent years. Vegetable oils are used in several industry sectors and have been investigated as an alternative energy source to replace traditional fossil fuels. Knowledge of physical and chemical properties is essential to assess the potential use of vegetable oil in the production of biofuels. The present study aimed to determine the oxidative stability of the seed oil of the babassu palm tree, *Attalea vitrivir*, native to the Brazilian cerrado and originated from two different regions, before and after storage for 12 months. The relative density of the oil did not differ between regions and did not change over time. Kinematic viscosity was lower for area 1 and increased with storage in oils from both areas. The induction period in the Rancimat test was lower in area 2 and decreased in the stored oil, regardless of the area, though it remained higher than the minimum required for its use as biofuel. The evaluation of oxidation by the oven method also showed satisfactory results, confirming the high thermo-oxidative stability of the babassu oil rich in lauric acid.

Keywords: biofuel, lauric acid, vegetable oil, Rancimat

1. Introduction

The demand for vegetable oils has increased in the past decades, in part due to population growth (Vasconcelos et al., 2019), boosting the use of these inputs as raw materials in several agro-industrial sectors, such as food, cosmetics, pharmaceutical, paint, and lubricant production (Siddeg & Xia, 2015; Florido et al., 2017; Al Jourdi et al., 2019; Vasconcelos et al., 2019). In addition, vegetable oils have been investigated as an alternative energy source to replace conventional fossil fuels due to the uncertainty regarding the supply of these finite fuels and the environmental problems arising from their use (Moreira et al., 2013; Borugadda et al., 2018; Coutinho et al., 2016; Giuffrè et al., 2016; Guil-Laynez et al., 2019; Singh et al., 2019b). In this sense, the energy produced from biomass, such as biofuel, is more convenient as it is renewable, biodegradable, less toxic, and cheaper (Borugadda et al., 2018; Coutinho et al., 2016; Giuffrè et al., 2016; Biresaw et al., 2017; Guil-Laynez et al., 2019; Singh et al., 2019b; Srivastava & Gaurav, 2019; Chua et al., 2020). The use of vegetable oils in biodiesel production stresses the importance of obtaining data on their properties (Florido et al., 2017).

The fatty acid composition defines the chemical and thermophysical properties of oils (Gomna et al., 2019; Różańska et al., 2019) and is closely related to vegetable oil quality, especially its ability to resist oxidation, which is the main cause of lipid deterioration (Gomna et al., 2019). Oxidative stability is a crucial factor in oil shelf life (Moreira et al., 2013; Forero-Doria et al., 2016). Saturated compounds are less prone to oxidation than unsaturated compounds; thus, the higher the degree of establishment, the more sensitive the oil (Gomna et al., 2019). The stability of some types of vegetable oil results from the antioxidant activity of phenolic compounds, tocopherols, and carotenoids naturally present in them. There are some methods for evaluating the oxidative stability of vegetable oils. Among them, the oven method (*Schaal test*) and the Rancimat stand out, which are inductive methods used to evaluate the induction period, when there is a significant formation (rapid increase) of peroxides and other oxidation products (Gomna et al., 2019; Pereira et al., 2019; Suri et al., 2019).

Physical properties such as density and viscosity can also indicate the oil degradation level (Gomna et al., 2019). Density increases with the establishment, and denser oils have a higher mass per volume (Uzunova et al., 2016; Gomna, 2019; Singh et al., 2019a). Viscosity can be defined as resistance to flow (Marulanda et al., 2008; Gomna et al., 2019), and it plays an important role in oil pumping and fluidity (Pierezan et al., 2015). Viscosity increases with fatty acid chain length in triglycerides and decreases with the establishment and increased temperature (Gomna et al., 2019). Therefore, evaluating how thermo-physical properties and behaviors change over time in storage is essential to examine the quality and industrial use of vegetable oils (Florido et al., 2017).

Attalea vitrivir, popularly known as babassu, is a palm tree that occurs in the region comprising the state of Bahia and the north of the Minas Gerais State, Brazil (Lorenzi et al., 2010). This species is native to the cerrado (Brazilian savanna) and establishes well in disturbed areas (Neves et al., 2013). *A. vitrivir* produces seeds with a lipid content above 53% (Salvador et al., 2019). The oil is rich in lauric acid (Table 1) and has characteristics that favor its conservation over time (Salvador et al., 2019). The cultivated palm has the potential to produce more than 1000 kg of oil per hectare annually (Guedes et al., 2015) and has been considered for inclusion in biodiesel production programs (Embrapa, 1984; MME, 2014). Previous studies have evaluated the yield and properties of the biodiesel produced from babassu oil (Lima et al., 2007), but there are no reports on the thermo-physical behavior of the crude oil under storage conditions. To ensure the quality and better exploitation of the *A. vitrivir* oil, we determined the oxidative stability of the oil from babassu seeds originated from two different areas by evaluating physical properties and accelerated aging tests resulting from storage.

Table 1. Fatty acid profile of the oil from *A. vitrivir* seeds (Salvador et al. 2019)

Fatty acid profile (%) of babassu oil *		
Caprylic	C8:0	5.77-7.03
Capric	C10:0	6.64-7.94
Lauric	C12:0	44.32-46.57
Myristic	C14:0	11.65-12.38
Palmitic	C16:0	7.23-8.16
Stearic	C18:0	2.92-3.08
Oleic	C18:1	14.8-17.41
Linoleic	C18:2	1.87-2.24

2. Method

2.1 Plant Material and Collection Site

We collected seeds from natural populations of *A. vitrivir* located in the Environmental Protection Area of Rio Pandeiros (EPA-Pandeiros), in the northern region of Minas Gerais State (15°26′10″S-44°40′44″W). The EPA-Pandeiros is within the ecotone between Caatinga and Cerrado biomes, where the dominant vegetation comprises Cerrado *sensu stricto*, gallery forests, and seasonal deciduous forests (Silva et al., 2009). The region has a semi-arid climate, with well-defined dry and rainy seasons, an average annual temperature between 21 and 24 °C, and average annual rainfall ranging from 900 to 1200 mm, with rains concentrated from November to January (INM, 2012).

We sampled two regions: area A has higher vegetation cover with dense babassu groves, where land use includes farming and agriculture activities, whereas area B is a pasture, comprising open grasslands with sparse trees and shrubs and the predominance of herbaceous plants. We collected ripe fruits directly from the bunches in each area and extracted seeds manually.

2.2 Oil Extraction

Seeds were dried in an oven at 105 °C for 24 h. Next, the oil was obtained by the cold extraction method in a mechanic press. The oil obtained was centrifuged for 15 minutes at 3,500 rpm to separate impurities and stored in amber glass bottles in a refrigerator (average temperature of 4 °C). Evaluations were performed before storage (initial condition) and 12 months after storage.

2.3 Determination of Relative Density

Density was measured in a pycnometer following the A.O.C.S. Official method Cc 10a-25 (1990). Oil samples were kept in a pycnometer in a water bath at 25±0.1 °C for 30 min. Then, the pycnometer was weighed. The weight of the empty pycnometer was subtracted to obtain the oil mass. The same process was repeated to measure the water mass. Finally, the relative density was calculated as the ratio between oil and water masses.

2.4 Kinematic Viscosity

Viscosity was determined using a Brookfield viscometer (Model LVDV-III+), equipped with a cylinder suitable for the fluid viscosity (spindle S-63). The viscometer was coupled to a thermostatic bath, and the oil viscosity was measured at 40 °C at 250 rpm. The obtained data were used to calculate the kinematic viscosity (as described in Arrudas et al., 2018, and Damasceno et al., 2018).

2.5 Determination of the Oxidative Stability (Rancimat® Test)

The oxidative stability index was determined following the regulation EN 14112 (EUROPEAN COMMITTEE FOR STANDARDIZATION, 2016), using the Rancimat 873 equipment (Metrohm AG, Herisau Switzerland). Three grams of oil were weighed in a reaction vessel and heated to 110 °C, with an airflow of 20 $L \cdot h^{-1}$. The volatile products released during the oxidation process were collected in a flask containing distilled water. The oxidation process was automatically recorded by measuring the change in water conductivity. The sample induction period (IP) corresponded to the breaking point of the electrical conductivity *vs.* time curves.

2.6 Schaal Oven Test

One hundred grams of oil sample were heated to 65± 2 °C. At the end of each time interval established (0, 1, 2, 3, 4, 10, 24, 48, 72, and 96 h), the samples were evaluated according to the peroxide index (AOCS Official method Cd 8-53, 1993). The induction period (PI) was determined by detecting significant changes in the peroxide index.

2.7 Experimental Design and Statistical Analysis

The experiment was carried out in a completely randomized design. A factorial scheme 2 (oil origin) × 2 (time) was used, with three replications for each treatment. To evaluate the oil resistance degree by the oven method, we also considered the interaction between time intervals in which oil samples were analyzed. Data were submitted to analysis of variance, and means were compared using the Tukey test at a 5% significance level.

3. Results

The Oil density from area A differed statisticaly with storage, where a soil density from area B did not differ between treatments and was the same as oil density from area A after 12 months (Figure 1A). The kinematic viscosity of the oil from area A increased with storage (from an average of 29.91 at initial condition to 32.01 mm²/s). The oil from area B showed a higher initial viscosity, which reached 37.59 mm²/s after 12 months (Figure 1B).

The induction period for the oil from area A was 29.17 h and 25.46 h after 12 months (Figure 1C). The oil from area B showed the same trend when submitted to the Rancimat test, from 22.70 h to 17.64 h after storage (Figure 1C).

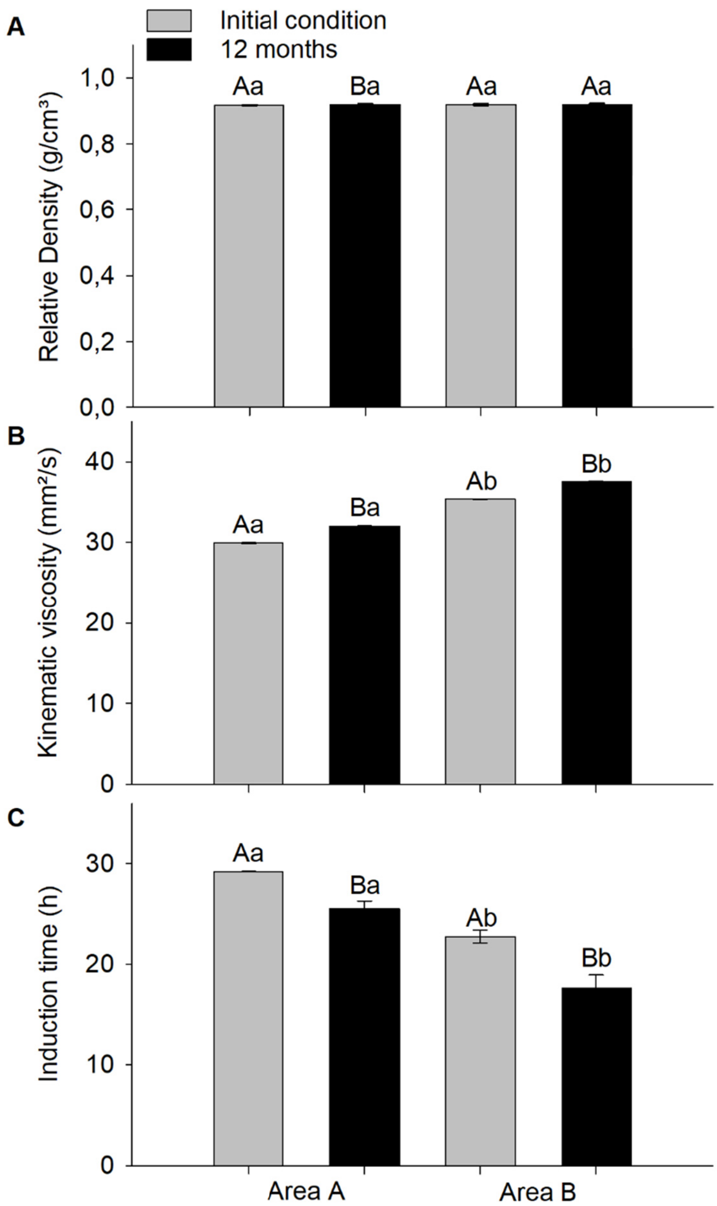

Figure 1. Relative density (A), kinematic viscosity (B), and IP by the Rancimat test (C) of the oil of *Attalea vitrivir* from two regions, before and after storage. Uppercase letters indicate differences between treatments in the same area, and lowercase letters indicate differences between areas in the same treatment ($P < 0.05$) by the Tukey test

Before storage, the peroxide index of the oil from area A was 5.95 meq·1000 g^{-1} (Figure 2). Subjecting the oil to heating reduced the amount of peroxide radicals identified; the value had already changed after four hours (4.61 meq·1000 g^{-1}), and this could be the induction period considered by the oven method (Figure 2). The peroxide index observed for the oil from area B before storage was 11.25 meq·1000 g^{-1}, higher than that from area A under the same condition (Figure 2). The peroxide index decreased with heating, and after ten hours of experiment, it statistically differed from the initial condition, with an average of 10.43 meq·1000 g^{-1} (Figure 2).The peroxide index increased with storage for the babassu oils from both areas (A: 4.71 and B: 10.66 meq·1000 g^{-1}; Figure 2). It also increased with oil heating and differed from the beginning of the experiment when the oil remained in the oven at 65 °C for 24 h (oil from area A: 5.33 meq·1000 g^{-1}) and 48 h (oil from area B: 11.25 meq·1000 g^{-1}). These were the induction periods considered for the stored oil (Figure 2).

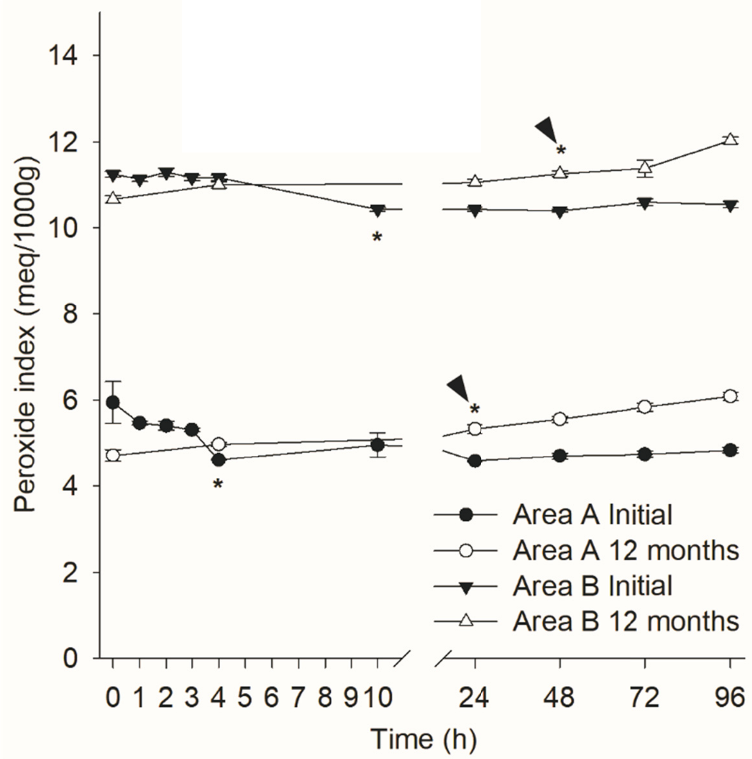

Figure 2. Peroxide index of the oil of *Attalea vitrivir* oil from the two areas, before (closed symbols) and after storage (open symbols). Asterisks (*) indicate a statistical difference in relation to the beginning of the experiment ($P < 0.05$) by the Tukey test. Arrows indicate the values for the oil stored for 12 months

4. Discussion

The oil from *Attalea vitrivir* seeds has good thermo-oxidative stability and desirable physicochemical characteristics for use in several industrial sectors. Oils rich in lauric acid are important due to their chemical properties and oxidative resistance (Martini et al., 2018). They are broadly used in the food industry and the production of soap and detergents. They also stand out as a potential raw material for producing biofuels (Radice et al., 2014; Silva et al., 2015; Damasceno et al., 2018).

Density and viscosity influence the atomization and combustion of fuels and represent important parameters to assess the suitability of using a vegetable oil. Therefore, these properties can determine the best performance of a fuel in diesel engines (Borugadda et al., 2018; Coutinho et al., 2016, Giuffrè et al., 2016; Guil-Laynez et al., 2019). The density of the oil from *A. vitrivir* is within the range reported in the literature for other babassu species (Singh et al., 2019a; de Oliveira et al., 2020; da Silva et al., 2020b) and is similar to those from other palm seeds (Moreira et al., 2013; Singh et al., 2019a). The values of this parameter did not change during storage due to the stability of the babassu oil, conferred by a high content of saturated fatty acids.

The viscosity of the babassu oil was similar to that observed for other commercial oils, such as soybean (Moreira et al., 2013; Singh et al., 2019b) and sunflower (Bertrand & Hoang, 2003). Saturated oils tend to be more viscous, but the viscosity of the oil analyzed in the present study was similar to that of the soybean oil, rich in oleic acid, the main raw material for biodiesel production in Brazil (Ogunkunle & Ahmed, 2019; Singh et al., 2019b). Crude (unrefined) oils naturally represent a condition that favors higher viscosity due to more intense interactions between fatty acid molecules, which is the main obstacle preventing the direct use of vegetable oils in traditional diesel engines (Ogunkunle & Ahmed, 2019; Borugadda et al., 2018; Coutinho et al., 2016; Giuffrè et al., 2016; Guil-Laynez et al., 2019; Singh et al., 2019a). The transesterification process is the most common method used to reduce the viscosity of these products (Giuffrè et al., 2016; Singh et al., 2019b; Chua et al., 2020). Transesterification is also the most common technique used in biodiesel production (Ogunkunle and Ahmed, 2019; Singh et al., 2019a), in which triacylglycerides react with a short-chain alcohol in the presence of a catalyst to produce [alkyl] esters of fatty acids (biodiesel) and glycerol as a by-product of the reaction (Singh et al., 2019a). Viscosity values differed between the two sampled areas, which may be related to genetic and environmental factors (Salvador et al., 2019). The viscosity of babassu oil increased after storage in both areas, probably resulting from the degradation of the oil (Singh et al., 2019a), as evidenced by the analyses carried out.

Oxidation is the main cause of oil degradation, and it can change the physical and chemical properties of the oil (Gomna et al., 2019). Vegetable oils are oxidized in contact with the oxygen in the air during processing and storage and through autoxidation, but temperature, time, and humidity can also affect them. The oxidation process happens as a chain mechanism involving initiation, propagation, and termination steps (Gomna et al., 2019).

The Rancimat test is broadly used in the food industry to assess the oxidative stability of edible oils and predict their shelf life (Vidrih et al., 2010).This test has also been adopted as one of the quality parameters of biofuels. The induction period determined for the babassu oil was higher than that for unrefined oils from other palm trees (Moreira et al., 2013, Vásquez-Ocmín et al., 2010). It is worth noticing that the oxidative stability index of the oil analyzed is higher than those exhibited by vegetable biodiesel (Guil-Laynez et al., 2019) and is within the standard established by EN 14112 of at least 3 h (Singh et al., 2019a), and ANP (2014), between 3 and 6 h. We also observed the area effect on oxidative stability, and storage time affected oil resistance, though the induction period remained relatively high.

Before storage, the peroxide index of the oil was lower for area A than B. Despite that, both values are within the recommended limit for human consumption (ANVISA, 2005). Contrary to what was expected, the peroxide index decreased with oil heating, regardless of the area, making it difficult to accurately determine the induction period of the samples by the oven test. The significant drop in the peroxide index after the first hours of the experiment could have resulted from the oil quality, which may not have developed a considerable amount of oxidation products in relation to the initial condition, or which were produced at a slower rate than the conversion of peroxide and hydroperoxide radicals into unidentified stable products. Gomna et al. (2019) point to the rapid decomposition of peroxide radicals as a crucial factor; thus, this parameter is not the best option for investigating the decomposition status of an oil at a high temperature. Besides, the evaluation of the peroxide index of stored babassu oil could validate such an idea. Both areas showed lower indices than those observed before storage, and heating increased the amount of peroxides in the oil, whose induction period occurred at least 24 hours after the beginning of the experiment.

The oil quality oil is closely related to its properties, especially its ability to resist oxidation, i.e., to remain stable for as long as possible. The fatty acid composition of vegetable oils defines their thermo-physical behavior. Saturated (and short-chain) oils are less prone to oxidation (Gomna, 2019), and promising raw materials for the production of biodiesel (Chua et al., 2020) and aviation biokerosene (Damasceno et al., 2018; da Silva et al., 2020a). Lima et al. (2007) produced good quality babassu biodiesel, with encouraging perspectives regarding its insertion in the Brazilian production chain. Damasceno et al. (2018), and da Silva et al. (2020a) investigated the suitability of the oils from macaw palm (*Acrocomia aculeata*) and African oil palm (*Elaeis guineenses*) seeds to produce biokerosene and were optimistic about the possibility of producing this fuel from oil with similar characteristics to the babassu oil studied here.

Our results showed that the oil of *A. vitrivir* seeds has thermo-physical properties that make it suitable for use in energy production, and storage does not significantly compromise the oil quality. Seed origin affected oil properties; thus, further studies are needed to determine the edaphic-climatic effect and biological variables on mother plants to contribute to establishing appropriate management and collection practices and genetic improvement.

5. Conclusions

Despite the influence of the collection region on oil characteristics, the values obtained for oil parameters analyzed from the two areas were in accordance with the requirements for its use in biofuel production. In addition, storage did not significantly compromise the oil quality. *A. vitrivir* seed oil has high thermo-oxidative stability and desirable physical-chemical characteristics for use in several industrial sectors.The results found here showed the potential of the babassu and emphasized the importance of studies with native species, besides serving as a basis for future studies.

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