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A N I N T E R N A T I O N A L J O U R N A L

Macauba (*Acrocomia aculeata*) pulp oil quality is negatively affected by drying fruits at 60 ºC

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ABSTRACT

This study aimed to determine if the quality of macauba pulp oil is affected by drying the whole fruits at 60 ºC. Mature fruits were collected at every five days on the ground under 10 palm trees. A mixed batch of 3 kg of whole fruits, with three replications each, was dried in an oven with air circulation for 0, 12, 24, 36 and 48 h at 60 ºC. After every drying time, dried fruits were pulped using an automated device, followed by oil extraction with hexane. Moisture and oil content were determined in the pulp, and the pulp oil quality was analyzed for fatty acid composition, free fatty acids content, peroxide value, molar absorptivity at 232 and 270 nm, refractive index, and total carotene content. The tested temperature was sufficient to decrease moisture to a range suitable for automated pulping, with the best combination and easier pulping being reached after 24 h of drying. In the first 12 h there was an apparent synthesis of unsaturated fatty acids and carotenes. The tested temperature was insufficient to avoid the enhancement of acidity since the beginning and throughout the drying period, nor the degradation of polyunsaturated fatty acids. Thus, it is concluded that drying of fresh fruits of macauba palm at 60 ºC is not appropriate to obtain an overall good quality pulp oil for industrial purposes.

Key words: macauba palm, high oleic, pulping, pulp oil, vegetable oil oxidation, vegetable oil acidity

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INTRODUCTION

Brazilian public politics regarding biofuel production, such as biodiesel[1] and aviation biokerosene[2], and the encouragement for mitigation of climate change by low carbon agriculture[3], among others, have increased the interest in the tropical macauba palm (*Acrocomia aculeata* (Jacq.) Lodd). This species provides a high pulp oil yield, close and even higher than oil palm[4,5], with suitable features for oil-based products besides a large amount of useful coproducts[6,7]. However, the development to obtain high quality macauba pulp oil is still in the beginning.

Macauba fresh fruits present elevated moisture content at ripening. For example, in a study with wild groves in either wet and dry areas, moisture of ripe fruits ranged from 50 to 63%[8]. Fresh fruit pulp also has got a strong stickness, which paired with moisture hinder automated pulping and further mechanical oil extraction by expeller pressing. Practice has shown that automated pulping and pressing is facilitated when moisture is reduced to around 20%.

Currently, fruits are mostly gathered by extractive activity from wild groves, after naturally dropping from the bunch as they mature. Then, the fruits are allowed to dry for months under room conditions in the industry quarters. The fruits processing takes place when a suitable moisture is reached. Such approach results in pulp oil with high acidity, originated from hydrolysis reactions of triglycerides, which may be caused by microbial activity[9] and/or endogenous metabolism[10]. It has been demonstrated that macauba oil with high acidity can be successfully converted to biodiesel and biokerosene in lab conditions[11,12]. However, in Brazil the current industrial processes to convert vegetable oil into biodiesel are mostly based in alkaline-transesterification, which is limited by oil acidity[13]. Therefore, low acid oil is still required by biofuel and other industrial purposes.

The quality of vegetable oils may also be compromised by oxidative reactions during storage and processing, which are influenced by temperature, light irradiation, metals, endogenous and exogenous enzymatic reactions, and their fatty acid composition[14,15]. Macauba pulp oil presents characteristics that possibly imply a good oxidative stability. It is composed predominantly of monounsaturated fatty acids (47.05-81.32% oleic acid), followed by saturated acids (13.42-30.60% palmitic acid), with lower contents of polyunsaturated (1.40-24.71%)[16,17,18]. Rich polyunsaturated lipids oxidize at a faster rate than mono and saturated ones[19]. Also, it has got a significative amount of carotenoids, mostly β-carotene[20,21], that play an important role to prevent the oil oxidation.

For the optimization of obtaining high quality macauba pulp oil is necessary to devise an efficient method of drying the fruits that could prevent acidification and oxidation, as well as to facilitate the mechanical processing of fruits. Drying fruits usually aims to maintain original color and vitamin contents. There is a wide range of temperatures and methods of drying fruits. However, it is usual to find data on fruit drying in the range of 50 to 80 ° C, more usually at 60 °C[22]. There is scarce information on whether accelerated drying processes of oil bearing fruits induce undesirable quality changes on the extracted oil or not, with reports going in both directions[23,24]. Regarding macauba palm it was reported that pulp oil acidity development, during long term storage, may be prevented by pre-treatment that controls lipolytic microorganisms, followed by drying at 60 °C during 15 days[9]. This approach may be expensive and time consuming, especially for large scale processing.

This paper reports the results of a trial devised to determine if macauba fruit pulp oil quality is compromised by drying at 60 ºC until moisture content is reduced to a range that facilitates automated pulping.

MATERIAL AND METHODS

Raw Materials

Ripened fruits of macauba were collected from native palm trees growing in a central Brazil area under typical savanna-like vegetation called Cerrados, in the Campo Grande Municipality, Mato Grosso do Sul State. The minimum average temperature is 19 °C and maximum is 30 °C; the average relative humidity is 68% and annual precipitation is 1400 mm. The ground under the ten macauba palm trees was cleaned, and dropped fruits were collected every five days from January to March, being stored at -18 °C until processing. The fruit collection mimics what is usually done in the actual exploitation of macauba palm. Ten fruits from every sampled palm were randomly selected for fruit mass and size measurements. On average, the transversal external diameter was 35.6 ± 5.3 mm, and the mass of whole fruit was 27.5 ± 6.2 g.

Drying and pulping

Whole unfrozen fruits were grouped randomly into batches of 3 kg each. Three replications were directly pulped, while another three replications were pulped after 12, 24, 36, and 48 hours of drying at 60 ºC. The drier was a convective oven (Marconi, model MA035, Brazil), with air circulation and renovation. Pulping was made with the help of an automated pulper, specifically designed for macauba palm fruits (RM Ltda, Brazil), allowing to process batches of fruits up to 5 kg. The device makes an easy separation and recollection of husk, pulp, and the shell + kernel.

Determination of pulp moisture and lipid content

The moisture content of fresh and dried fruit pulp in each sampling was determined by drying at 105 °C until constant weight[25]. The total content of lipids was determined according to AOAC standard[25], using a Soxhlet apparatus and hexane as solvent.

Physicochemical analysis of pulp oil

Physicochemical analysis were performed using the oil extracted with hexane by refluxing in a Soxhlet apparatus, with further removal of the organic solvent in a rotary evaporator working at 40 °C. The following parameters were analysed: free fatty acid (FFA, expressed as percentage of oleic acid, according to method Ca5a-40[26]; peroxide value (PV), according to method Cd8-53[26]; molar absorptivity at 232 nm (K_{232}) and 270 nm (K_{270}), respectively, to check the eventual formation of any dimers and trimers that could result from oxidation reactions, according to method Ch5-91[26]; refractive index (RI) by refractometry at 20 ºC, according to method Cc7-25[26]; and total carotene content by absorbance at 446 nm (spectrometer Aquamate, v model. 4.), using petroleum ether as organic solvent[27].

In the sequence, the fatty acid profile was determined after the lipids were converted to fatty acid methyl esters (FAME). FAME was obtained by the addition of 2 mL of 7% Boron Fluoride (BF_3) methanolic solution, plus 1 mL of toluene, followed by heating at 100 °C for 45 minutes, and cooling to room temperature with further addition of 5 mL of water, 3 mL of hexane, and 300 mg of sodium sulphate, all mixed by stirring. The top layer was collected and injected into a GC (Agilent 6890N, California, USA) equipped with a flame ionization detector (FID) and a polar capillary column (HP88, 0.25 mm internal diameter, 100 m length and 0.25 mm film thickness), to obtain individual peaks. Injector temperature was 225 °C and detector temperature was 285 °C. The initial column temperature was held at 160 °C for 3 minutes and then increased to 190 °C at 3 °C/minutes for 6 minutes, followed

by an increase to 230 °C at 6 °C/minutes and then held for 12 minutes. Total run time was 37.67 minutes. FAME peaks were identified comparing their relative retention times with individual standards (Supelco C8-C22, USA), spiking the samples with methyl undecanoate. Data were analyzed using Agilent Technologies Chemstation A09.01 Software. The relative percentage of a given fatty acid was calculated comparing its individual peak with the total peak area of all fatty acids in the oil sample.

Experimental Design

An entirely randomized experimental design was applied, encompassing five drying times as the independent variable, with three replications of the drying time. Analytical procedures were done in triplicate for every sample. The results were submitted to analysis of variance and regression, using GraphPad InStat software.

RESULTS AND DISCUSSION

Drying effects upon macauba pulp moisture, lipid content, and pulping

The pooled macauba fruits showed pulp with an initial moisture content averaging 390 g kg^{-1}, which was reduced to 370, 220, 180, and 130 g kg^{-1} after drying at 60 °C during 12, 24, 36, and 48 hours, respectively, following a significant ($P<0.001$) linear pattern of moisture decrease (Fig. 1). As water was removed from the pulp, there was a parallel concentration in oil amount, whose content raised from 68 to 184 g kg^{-1} (Fig. 1). As expected, both trends were significantly correlated (Pearson Correlation 0.94, $P<0.001$). Only 5% of the water content was lost in the first 12 h of drying, suggesting a period of adjustment for the heated water inside the fruit to move from the inner parts through the lignocellulosic epicarp and out to the environment. Such long period under a range of temperature and moisture that might enhance some metabolic activities, may lead to either synthesis or degradation of fruit compounds.

The pulp moisture dropped after 24 h to a value close to that commonly practiced to macauba fruit processing of drying, around 20%. With such moisture content, automated pulping was easily performed, being necessary only a single step operation of 2.5 minutes to pulp a batch of 3 kg fruits. When fruits had a moisture content higher than 220 g kg^{-1}, at least three steps were needed for a complete pulping, with interruptions to disperse the jammed pulp in the machine, taking more than 10 minutes each batch.

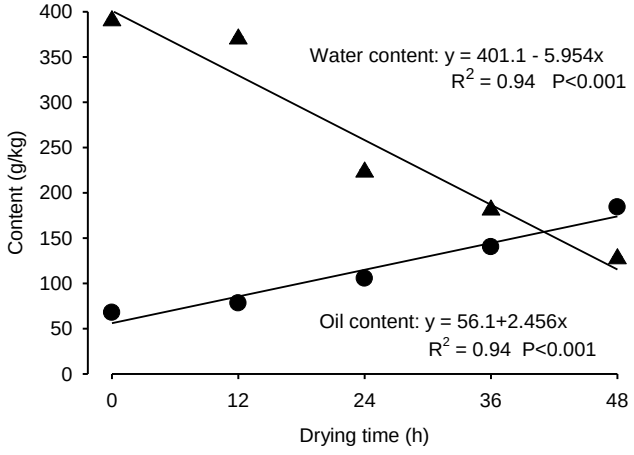

Figure 1. Moisture and oil content of macauba pulp fruit along drying at 60 °C.

Effects of drying on the composition of macauba pulp oil fatty acid composition

Monounsaturated fatty acids were predominant in the extracted macauba pulp oil (Table 1), with oleic acid encompassing 70% of the total composition. Palmitic acid was predominant within the saturated fatty acid fraction, accounting for 17% of the total. Polyunsaturated fatty acids corresponded to only 2.6% of the total. Overall, there were amounts higher in oleic acid, almost similar to those of palmitic acid, and lower in linoleic acid than reported in other studies[9,18,21], although within the range determined for a large number of wild genotypes[17]. Oleic and palmitic acids contents are the traits related to the profile of fatty acids which most contributed to the total divergence and differentiation of macauba genotypes[28].

Table 1: Fatty acid profile of macauba pulp oil over fruit drying at 60 ºC.

Fatty acids	Drying time (h)				
	0	12	24	36	48
Lauric acid ($C_{12:0}$)	0.22±0,01	0.14±0.00	0.15±0.00	0.14±0.00	0.14±0.00
Myristic acid ($C_{14:0}$)	0.61±0.02	0.42±0.001	0.49±0.01	0.50±0.00	0.50±0.00
Palmitic acid ($C_{16:0}$)	17.00±0.03	17.00±0.37	16.50±0.08	16.00±0.08	16.16±0.09
Palmitoleic acid ($C_{16:1}$)	1.26±0.01	1.14±0.01	1.14±0.03	0.95±0.01	0.95±0.02
Margaric acid ($C_{17:0}$)	0.07±0.01	0.06±0.00	0.06±0.00	0.07±0.01	0.06±0.01
Stearic acid ($C_{18:0}$)	2.21±0.03	2.45±0.02	2.54±0.02	2.67±0.02	2.66±0.03
Oleic acid ($C_{18:1}$)	70.00±0.03	70.03±0.37	71.44±0.24	73.36±0.08	73.38±0.13
Linoleic acid ($C_{18:2}$)	2.36±0.05	3.18±0.34	1.98±0.14	0.81±0.05	0.75±0.02
Linolenic acid ($C_{18:3}$)	0.22±0.01	0.30±0.02	0.23±0.01	0.20±0.00	0.20±0.00
Arachidic acid ($C_{20:0}$)	1.25±0.01	1.23±0.04	1.10±0.02	0.94±0.02	0.92±0.01
Cis-11-eicosenoic acid ($C_{20:1}$)	0.21±0.00	0.26±0.02	0.20±0.01	0.19±0.01	0.18±0.01
Behenic acid ($C_{22:0}$)	0.07±0.00	0.11±0.01	0.07±0.00	0.05±0.01	0.05±0.00
Unidentified	3.02±0.02	3.01±0.54	2.90±0.03	2.73±0.02	2.73±0.02
Saturated fatty acids	19.22	18.96	18.37	17.7	17.83
Monounsaturated fatty acids	71.47	71.43	72.78	74.5	74.51
Polyunsaturated fatty acids	2.58	3.48	2.21	1.01	0.95

mean± standard deviation

The fatty acid composition of macauba palm oil showed some changes along the 48 h drying period (Table 1). Oleic acid content increased by around 5%, while the palmitic acid content decreased in the same proportion. These changes suggest that there were ongoing metabolic activities during the drying, with the biosynthesis of monounsaturated fatty acids. It seems that palmitic acid was converted into oleic acid by lipid chain elongation reaction in macauba pulp fruit under the drying conditions, following the normal pathway of lipid synthesis. Lipids biosynthesis begins with a source of carbon, Acetil CoA, which is converted to palmitato by a sequence of enzymes activity, is further elongated and desaturated to build up unsaturated fatty acids[29]. On the other hand, changes were also observed in polyunsaturated fraction, which increased in the first 12 h of drying, but was strongly reduced afterwards, remaining only 68% of its initial content after 48 h of drying (Table 1), probably because of oxidative reactions on the double bonds of those fatty acids. A similar pattern of decrease in polyunsaturated fatty acid content was also reported when macauba fruits were dried at the same temperature for 15 days[9].

Effects of drying on the macauba pulp oil quality

The overall quality of a given vegetable oil is expressed in terms of its acidity (FFA, as oleic acid) and oxidative indexes such as K_{232}, K_{270}, PV, RI and minor

components like pigments, among others. All these were measured in this study, at every sampling time along the drying period.

A superior limit for vegetable crude oil of 5.0% FFA is established by international[30] as well as by Brazilian regulations[31], although the industry looks for oils with lower than 3% FFA to make feasible the further processing. In this study, the initial FFA of the extracted oil was 4.8%, as oleic acid, that jumped to 5.3% during the first 12 h of drying, with a faster release of fatty acids afterwards, reaching 30.2% after 48 h. The best fit of the data is a first order equation (Fig. 2A), and its pattern indicates that the temperature of 60 ^{o}C was not suitable to prevent hydrolitic reactions upon tryacylglicerides of the oil. It is usually assumed that macauba oil has got high acidity, although reports in the literature show variable results, with values as low as 0.8%[18] and as high as 65%[32]. Acidity development in the macauba pulp oil seems to be dependent on how the fruits are collected, stored[33,34], and samples are prepared for analytical procedures[35,36]. It is commonly accepted that fruits should be collected from the bunch and processed as soon as possible to render low acidification, but there is not enough support in the literature for such a claim. Acidity of macauba pulp oil lower than 1% was reported in fruits kept on the ground during seven days after abscision[33], while a 5.6% acidity was registered in oil obtained from fruits collected directly from the bunch[34].

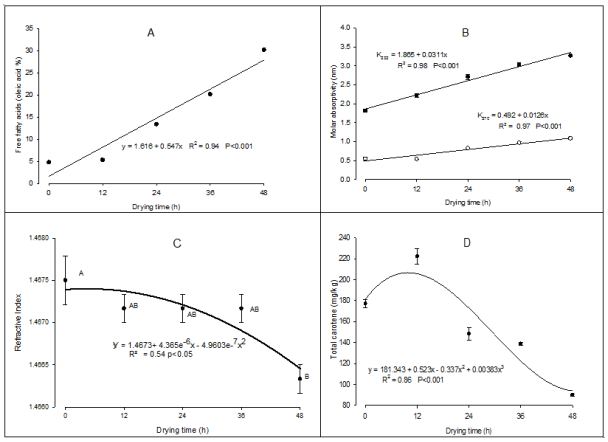

Figure 2. Physicochemical characteristics of macauba pulp oil along the fruit drying at 60 ºC. (bars indicate standard deviation and different letters means significative difference at P<0.01 by Tukey test).

The actual agents of tryacilglycerides hydrolysis in macauba fruits are not yet properly known. Lipases produced by microorganisms that contaminate the fruits have been suggested to be responsible for the development of acidity during oil storage[9]. Lipase activity was detected in macauba fruit without isolation of endogenous or exogenous sources up to forty days, and it was, also, correlated to the decay index[10]. On the other hand, a study on lipases in macauba pulp carried out in our laboratory (data not published), employing the methodology described by Iaderoza and Baldini[37], indicated the occurrence of endogenous lipolytic activity in fruits lying on the ground up to 30 days. The peak of activity was within a 40 to 70 °C temperature range. Therefore, acidification could have been further favored by drying temperature.

Besides the acidification, polyunsaturated lipid oxidation also seems to have taken place on the macauba pulp oil during the drying, as indicated by significant (P<0.001) increases measured in the K_{232} and K_{270} values (Fig. 2B). Increases of K_{232} values are related to the formation of primary compounds of polyunsaturated lipid degradation, indicating the beginning of the oxidative process, while increase of K_{270} results from further stages, are related to stable substances like conjugated trienes[38].

Changes in the polyunsaturated fatty acids are shown by the decrease in the amount of linoleic acid over drying process (Table 1). The slope of the best fit of the data measured along the drying period suggests distinct states of oxidative evolution into the analyzed samples. There were increases in K_{232} since the beginning of drying, measured at 12 h, while K_{270} value was significantly altered after 12 h of drying.

Although it is important to know the possible changes that could affect macauba pulp oil quality during the drying process, these measured variations in K_{232} and K_{270} cannot be assumed as constrain to determine its usage or not in the oleochemical industry, because there is not yet a regulation for this oil source. Further investigations should address this issue and contribute to set legal ranges for quality indexes of macauba pulp oil.

On the other hand, the monounsaturated lipid fraction of the macauba pulp oil does not seem to be affected by oxidation, since there were no peroxides measured as PV, neither there were increases in RI over the whole drying period (Fig. 2C),which is related to oxidative reactions upon lipids. The initial macauba pulp oil RI was 1.4675 (Fig. 2C), higher than that reported in other works, such as 1.466[16], 1.464[39], and 1.443[40]. These variations are probably caused by sampling of different wild groves of macauba palm, Mato Grosso do Sul State (Southwest), São Paulo State (Southeast) and Paraíba State (Northeast) of Brazil, respectively to the citations. During the first 36 h of drying, RI decreased slightly, but it decreased significantly ($P<0.05$) by the next evaluation (Fig. 2C). Together with no detection of peroxides, RI results suggest that the bulk of macauba pulp oil shows some extent of oxidative stability under those drying conditions, probably confered by the well known stability of oleic acid[19], which accounts fo a high proportion of macauba pulp oil. This oil has got a crosslinked structure, due to its diglycerides content[33], which, added to its degree of monounsaturation, confers the high thermal stability observed in this study.

The oxidative stability may also be helped by the carotene content of the macauba pulp oil, which averaged 179.88 mg kg^{-1} in fresh fruits (Fig. 2D). There was a significant ($P<0.05$) increment during the first 12 hours of drying, to 221.31 mg kg^{-1}, suggesting a possible synthesis of carotenes, as observed for the fruits of *Lycium barbarum* dried at 55 ºC[41]. In the sequence of drying, carotene content dropped to a final content of 89.11 mg kg^{-1} after 48 h of drying. Such a pattern is usually observed when processing temperatures are higher than the environmental conditions, in the presence of oxygen[42].

Overall, results suggest that drying macauba fruits at 60 ºC is not a suitable procedure to obtain a final oil with a quality acceptable for most industrial purposes. The reduction of moisture to a water content that facilitates pulping, around 20%, was reached after 24 h of drying (Fig. 1), a period of time long enough to allow some important degradation processes to happen, despite the occurrence of synthesis of interesting components as well. The main constraint was the development of acidity. Actually, the acidity of the fresh fruits was at the limit for a suitable vegetable oil, and it was enhanced by the drying process, increasing more than twofold after 24 h (Fig. 2A). Perhaps this temperature of drying would be suitable for fresh fruits with lower acidity, resulting in dried fruits that would be easily pulped before acidity reaches the standard industrial limit. Furthermore, oxidation reactions in the macauba pulp oil were observed to a small extent during the first 24 h of drying, which can be an effect of its fatty acid composition (Table 1) and carotene content (Fig. 2D). Thus, it would be worthy to carry out further studies with higher temperatures of drying, searching for a combination of temperature and time that would provide a reduction of moisture to around 20%, without allowing lipids degradation reactions.

CONCLUSIONS

Accelerated drying of fresh fruits of macauba palm at 60 °C is not suitable to obtain an overall good quality pulp oil for industrial purposes, because it allows the development of undesirable acidity, although keeping its intrinsic oxidative stability.

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