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Macauba as Promising Substrate for Crude Oil and Biodiesel Production

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Abstract: The preparation of macauba fruits (*Acrocomia aculeate* (Jacq.) Lodd. ex Martius) both for oil extraction by continuous pressing and biodiesel production were investigated. The fruits and its pulp and kernel were characterized for their resulting oils. Experimental design was carried out to study the transesterification of each oil to obtain the concentrations of ethanol and catalyst that would provide the best biodiesel yield. A higher amount of biodiesel was produced from the best conditions to evaluate some characteristics as specified by ANP (National Agency of Petroleum, Natural Gas and Biofuels). The results showed a variation in the fruits composition and different characteristics presented by the pulp and kernel oil. It was possible to define technological steps to obtain oil from the fruits of macauba and find the optimal conditions to biodiesel production, concentration of catalyst 4.42% and 5% and 13.3:1 and 9:1 ethanol:oil molar ratio to kernel oil and pulp oil, respectively.

Key words: *Acrocomia aculeata*, oil extraction, pulp and kernel oil.

1. Introduction

With the world's increasing demand for energy and unquestionable environmental concerns in using fossil fuel, the search for new sources of energy becomes essential. Adding to this, reduction in the world oil reserves and international conflicts have contributed to an increase in fuel prices. Facing this reality, the use of biodiesel is considered a fine alternative to replace petroleum derived fuels due to its renewable and biodegradable character and its low emission profile. Among several plants capable of producing crude oil, a raw material for biodiesel production, palms are considered the most promising species. Macauba (*Acrocomia aculeata* (Jacq.) Lodd. ex Martius) is an oleaginous palm tree widely distributed in the American continent including Mexico, Antilles, Brazil, Argentina, Uruguay and Paraguay. This palm is native to dry hillsides and open forests from Central America to Southern Brazil [1, 2]. Different from oil palm (*Elaeis guineensis*), which requires a hot humid tropical environment to grow, macauba palm is adapted to different environments, including cooler subtropical and drier semiarid ecosystem [3].

Macauba is highly productive and its fruits can generate more than 5,000 kg of oil per hectare [4], which oil can be used in food, cosmetic, pharmaceutical and biodiesel industries. In addition, a significant amount of a high quality charcoal can be produced from its endocarp. The coconut flavored seed kernels of the fruits are eaten raw. The mesocarp is a food rich in fixed oil, protein and crude fiber [5]. However, macauba palm plantations in Brazil and in many places in the world are still in their primary stage. The macauba palm fruit exploitation occurs mostly by gathering in large natural populations. Its commercial exploitation depends on the development of technology.

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This study aimed at establishing the conditions for the preparation of the raw material fruits of macauba to pulp and kernel oil extraction by continuous pressing and producing biodiesel by ethanolic transesterification, as well as examining the reaction conditions such as the molar ratio (between ethanol and oil) and the catalyst concentration used in the transesterification of the pulp and the kernel macauba oil.

2. Material and Methods

2.1 Fruits Collection and Characteristics

Fruits of macauba were collected in the São Paulo state, Brazil during the harvest season of February 2010.

The average of fruit weight and the quantity of pericarp (shell), mesocarp (pulp), exocarp (kernel shell) and endocarp (kernel) was determined by gravimetry.

2.2 Moisture and Oil Content

The moisture and oil content by direct extraction in a Butt apparatus, using petroleum ether (boiling point 40-60 °C), according to standard procedures [6] was determined.

2.3 Sample Preparation and Press Conditions

The macauba fruits were selected, all types of foreign materials or deteriorated fruits that could affect extraction of the oil being removed.

Shell with pulp together was removed in apropriate apparatus and dried in oven at 100 °C for 2 hours. Kernel was separated from the kernel shell after breaking into hammer mill and by passage through a sieve.

Five kilogram of samples (dried shell with pulp or kernel) were used in each oil process extraction. Pressing was performed using a stainless steel expeller with a capacity to press 40 kg of material per hour (Ecirtec MPE-40). The oils obtained were filtered and the yields determined.

2.4 Oil Analysis

The physicochemical characteristics of the pulp and kernel oils were determined by standard procedures recommended by the AOCS [6] that include free fatty acids, unsaponifiable matter, refractive index, peroxide value, iodine value, saponification value, copper, iron, phosphorus and sulfur.

2.5 Fatty Acid Composition

After extraction, the oils were saponified and the fatty acids converted to methyl esters as described by Hartman and Lago [7]. The fatty acid methyl esters were analysed, using a model Star 3400 Varian Gas Chromatograph, equipped with a flame ionisation detector, a split/splitless injector and a capillary column of melted silica (30 m × 0.25 mm, J & Scientific, USA), containing polyethylene glycol (D B Wax) as the stationary phase. The chromatographic conditions used included: detector temperature of 260 °C, injector temperature of 250 °C, column temperature of 200 °C for 20 min, programmed for 1 ºC per minute up to 220 ºC, hydrogen gas with flow rates of 1.1 mL min^{-1}, make-up gas of nitrogen for 22 mL min^{-1}. The fatty acids were identified by comparing the retention times of the sample peaks with those of methyl ester standards of the fatty acids. The areas of the peaks were calculated and expressed as a percentage of the total area.

2.6 Laboratory Scale Transesterification

Biodiesel by transesterification was produced using anhydrous ethanol (purity 99.5%) and sodium methylate solution (30%) as the catalyst. Two independent variables were evaluated: the ethanol:oil molar ratio (MR) and the catalyst concentration, as related to the amount of oil that had to be transesterified (C). The response was summarized as the ester-rich phase (BY).

The mass of ethanol used in each experiment was calculated according to the mean molecular mass of the oil (Eq. 1).

$$MM_{oil} = 3\left[\sum\left(\%_{f.acids}\right)\left(MM_{f.acids}\right)\right] - MM_g - 3\left(MM_{water}\right) \quad (1)$$

where:

MM_g: molecular mass of the glycerol;

MM_{water}: molecular mass of three water molecules, lost during formation of the triacylglycerols;

$\%_{f.acids}$: quantity of each fatty acid present in the oil molecule;

$MM_{f.acids}$: molecular mass of each fatty acid present in the oil molecule;

MM_{oil}: molecular mass of the oil.

The molecular mass of glycerol is 92 g mol and the molecular mass of three water molecules is 54 g mol.

Initially, 25 g of crude oil were added to the flask, which was heated to 45 °C. In another flask, a pre-specified amount of sodium methylate was added to a predetermined amount of ethanol, and the resulting solution added to the pre-heated oil and stirred for 30 min.

The glycerin phase was recovered immediately after phase separation. The ester-rich phase was analyzed using a Waters 600E liquid chromatograph equipped with two columns in series (one 500 Å and one 100 Å Jordi Gel DVB, both 300 mm × 7.8 mm), refractive index detector, 50 min run time, injector and column temperatures of 40 °C, 0.5 mL/min flow rate and sample volume of 20 μL. Quantification was based on external calibration using triacylglycerol, diacylglycerol, monoacylglycerol and free fatty acid standard solutions and commercial biodiesel. The ester-rich phase samples were prepared in THF (tetrahydrofuran) to a final concentration of 1 mg mL^{-1} [8].

2.7 Reactor Transesterification

The experimental condition corresponding to test 8 for macauba pulp oil and the experimental condition corresponding to test 4 for macauba kernel oil were applied to a stirred batch tank reactor. Two kilogram of each oil, the correspondent quantity of anhydrous ethanol and sodium methylate solution (30%) were used. The chemical reactions were carried out at 45 °C for 30 minutes.

The mechanical stirrer operates first at 10 Hz until the oil had reached the ideal temperature. After adding the catalytic solution, the rotation was decreased to 7 Hz and maintained thus until the conversion into biodiesel had occurred. For the final reaction stage, the rotation was again increased to 10 Hz. Adjustment of the rotation took into account the mixing intensity required for adequate blending of the chemical reagents with the oil as evaluated visually, favoring mass transfer during the transesterification reaction and avoiding excessive air incorporation during the process. The mixture was then left to decant for 6 hours to allow for separation of the glycerol and ester-rich phases. Any excess ethanol in the ester-rich phase was evaporated off under reduced pressure at a temperature below 60 °C. So the sample obtained, containing no ethanol, was called the "unpurified biodiesel", and was subjected to purification method that consisted of washing the biodiesel with a 5% solution of hydrochloric acid (37%), heated to 90 °C [9].

The biodiesel were characterized in relation to the some technical specifications for Brazilian biodiesel (Resolution 7 of the National Petroleum, Natural Gas and Bio-fuels, ANP, dated March, 2008), using the following methods: ABNT NBR 7148 for specific gravity at 20 °C, ASTM D 6304 for water content, ASTM D874 for the sulfated ash content, ASTM D4294/5453 for the total sulfur content, ASTM D664 for the acid number, visual aspect, EN 14538 for the calcium, magnesium, sodium and potassium contents, EN 14107 for the phosphorous content and EN 14111 for the iodine number [10].

3. Results and Discussion

3.1 Characteristics of the Fruits

The macauba (*Acrocomia aculeata)* presented average fruit weight of 37.62 g with variation of 17.7% to 27.9% of pericarp (shell), 32.1% to 43.8% of

mesocarp (pulp), 24.1% to 26.5% of exocarp (kernel shell) and 11.1% to 18.7% of endocarp (kernel). Manfio and co-workers [11] in their study determined the average fruit weight that was 32.12 g, with 1.69 g kernel weight.

3.2 Moisture and Oil Content

The moisture content of the pulp ranged from 27.9% to 47.7%, range considered large, requiring the pulp drying in order to preserve and to take the possibility to storage. The oil level of the pulp presented the average of 14.0% to 51.7%, and the oil level of the kernel was 61.1%.

3.3 Press Oil Yields

The result of continuous press yield of dried macauba shell with pulp together was 20% of oil, and the continuous press yield of macauba kernel was 32% of oil. The macauba pulp oil aspect was yellowish liquid at room temperature while the kernel oil had clear appearance and good smell.

3.4 Oil Characteristics

Table 1 shows the physicochemical characteristics of the pulp and kernel oil of macauba fruits from the state of São Paulo respectively. The free fatty acids and peroxide value of the oils indicating that the preparation of raw material and the press processing of pulp and kernel resulted in good quality crude oil. The iodine value of the macauba pulp oil was 74.9 and the iodine value of the macauba kernel oil was 32.7 that is because of the different fatty acid composition. Because of the low level of phosphorus present in the pulp and kernel oils degumming process was not necessary.

3.5 Fatty Acid Composition

Table 2 shows the fatty acid compositions of macauba pulp and kernel oil. In the pulp oil there was a predominance of oleic acid (55.3%), with levels similar to those found in the fruit of other palm trees, palmitic acid (23.6%) being the second most abundant fatty acid. Lauric acid (38.7%) was the predominant fatty acid in the macauba kernel oil, followed by oleic acid (29.1%), in agreement with previously reported macauba species [12]. Lauric oils and their derivatives have many applications both in the food and chemical industries.

The pulp oil showed 60.2% content of monounsaturated fatty acids to those verified in edible oils rich in these fatty acids, such as olive (56%-87%), peanut (15%-47%) and canola oil (54%-75%). In the kernel, 29.2% of monounsaturated fatty acids and a high value of saturated acids (66.4%) were observed. Other palm kernel oils, such as palmiste oil (*Elaeis guineensis* Jacq.), presented lower monounsaturated (12%-19%) and higher saturated (69%-98%) fatty acid values than those verified in the kernel studied here.

Table 1 Physicochemical characteristics of the pulp and kernel oil of macauba, *Acrocomia aculeata* (Jacq.) Lodd. from the state of São Paulo, Brazil.

Characteristic	Macauba pulp oil	Macauba kernel oil
Free fatty acids (%)	2.10	2.14
Peroxide value (meq O_2 kg^{-1})	4.89	4.82
Iodine value (g Iodine per 100 g)	74.90	32.70
Saponification value (mg KOH g^{-1})	196.77	232.96
Unsaponifiable matter (%)	0.85	0.51
Refractive index 40 °C	1.464	1.455
Copper (mg kg^{-1})	0.02	n.d.*
Iron (mg kg^{-1})	7.11	0.38
Phosphorus (mg kg^{-1})	14.60	0.61
Sulfur (mg kg^{-1})	7.7	7.1

n.d.* = not detected.

3.6 Laboratory Scale Transesterification

Table 3 presents the real values corresponding to the experimental design. The ethyl ester-rich phase (EE) mass obtained in the different trials varied from 23.85% to 98.39% to macauba pulp oil and from not detected to 98.77% to macauba kernel oil (Table 4). From the experimental design, the optimal conditions corresponded to an ethanol:oil molar ratio of 9:1, which corresponds to an excess of 6 moles of alcohol and a catalyst concentration of 5% to macauba pulp oil transesterification. The optimal conditions corresponded to an ethanol:oil molar ratio of 13.3:1 and a catalyst concentration of 4.42% to macauba kernel oil transesterification.

Table 4 shows the fitting of the coefficients to the experimental data. The results demonstrated that all the coefficients were considered to be statistically significant at a 90% confidence level to macauba pulp oil transesterification. The positive sign of both coefficients indicated that the mass of ester-rich phase showed a tendency to increase as the molar ratio and catalyst concentration increased.

The variance analysis for both oils were carried out and analyzing the values about pulp oil transesterification, the coefficient of determination (R^2) was 0.62, meaning that the generated model could explain 62% of the variation of the experimental data with the remainder attributed to random errors. The value of F calculated for the regression was greater than the tabulated F value, indicating that the model can be used for predictions. The low value of F calculated for the F table indicates that the model showed no lack of fit.

As the model generated for the production of EE from macauba pulp oil was predictive, showed no lack of fit and had an appropriate value of R^2, we can construct the response surface, as shown in Fig. 1.

Table 2 Fatty acid composition (%) of the pulp and kernel oil of the macaúba, *Acrocomia aculeate* (Jacq.) Lodd. from the state of São Paulo, Brazil.

Fatty acid (%)	Macauba pulp oil	Macauba kernel oil
C 6:0 Caproic	n.d.*	0.3
C 8:0 Capryllic	n.d.*	3.9
C 10:0 Capric	n.d.*	3.4
C 12:0 Lauric	n.d.*	38.7
C 14:0 Myristic	0.1	8.9
C 16:0 Palmitic	23.6	8.2
C 16:1 Palmitoleic	4.7	0.1
C 18:0 Stearic	2.1	2.8
C 18:1 Oleic	55.3	29.1
C 18:2 Linoleic	12.1	4.4
C 20:0 Araquidic	0.2	0.2
C 18:3 Linolenic	0.7	n.d.
C 20:2 13,16 Docosadienoic	0.5	n.d.
Σ Saturated fatty acids	26.2	66.4
ΣMonounsaturated fatty acids	60.2	29.2
Σ Polyunsaturated fatty acids	13.4	4.4

n.d. * = not detected.

Table 3 Real values for the molar ratio and catalyst concentration in the experimental design for the transesterification reaction.

Variables	Codified levels				
	-1.41	-1.0	0	1.0	1.41
Molar ratio	3.0	4.7	9.0	13.3	15.0
Catalyst conc. (%)	1.00	1.58	3.00	4.42	5.00

Table 4 Average yield of ester-rich phase in the transesterification on a laboratory scale, based on a 2^2 factorial experimental design, with four axial and factorial points and three central points.

Test	Variables		Ester-rich phase (%)	
	Molar ratio	Catalyst conc. (%)	Pulp	Kernel
1	4.7 (-1)	1.58 (-1)	35.60	7.10
2	4.7 (-1)	4.42 (+1)	73.83	94.78
3	13.3 (+1)	1.58 (-1)	31.95	20.37
4	13.3 (+1)	4.42 (+1)	97.51	98.77
5	3.0 (-1.41)	3.00 (0)	48.35	93.47
6	15.0 (+1.41)	3.00 (0)	95.37	97.73
7	9.0 (0)	1.00 (-1.41)	23.85	n.d.*
8	9.0 (0)	5.00 (+1.41)	98.39	98.34
9	9.0 (0)	3.00 (0)	75.60	87.30
10	9.0 (0)	3.00 (0)	96.86	92.11
11	9.0 (0)	3.00 (0)	95.81	91.77

n.d * = not detected.

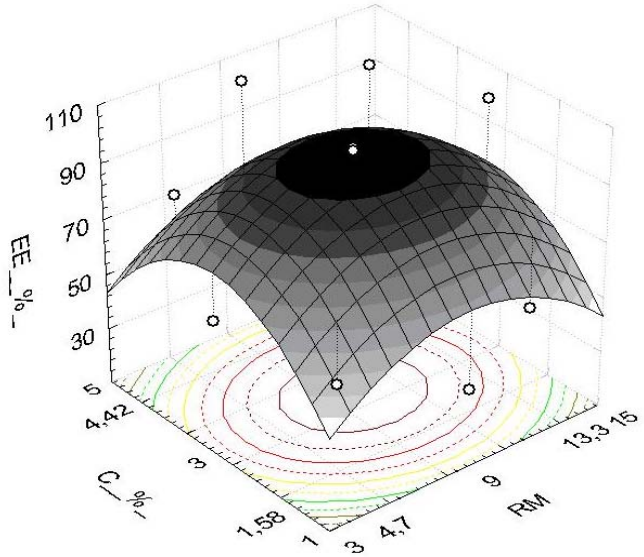

Fig. 1 Response surface showing the effects of catalyst concentration (C%) and molar ratio (RM) on the production of biodiesel (EE%) from macauba pulp oil.

One of the central point was removed because it had a value very different from the others, resulting in a high coefficient of variation. A duplicate of the center point shows that the variation was 0.77% (CV) indicating that the experiments showed good accuracy and repeatability.

In the case of kernel oil transesterification, analyzing the values obtained in Table 5, shows that the coefficient of determination (R^2) was 0.96, meaning that the generated model could explain 96% of the variation of the experimental data and the remainder attributed to random errors. The value of F calculated for the regression was greater than the tabulated F value, indicating that the model can be used for predictions. The high value of F calculated for the F table indicates that the model presented lack of fit, so the surface does not appear to respond.

The enzymatic microwave assisted biodiesel synthesis from macauba (*Acrocomia aculeata*) oil and ethanol using Novozyme 435 and Lipozyme IM was studied by Nogueira et al. [13] using statistically designed experiments. The investigated variables were reaction temperature, time and enzyme loading. It was observed a significant effect of the reaction time in

Table 5 Biodiesel parameters for ethyl esters and ANP specification.

Parameter	Macauba pulp biodiesel	Macauba kernel biodiesel	ANP limit
Aspect	Limpid	Limpid	Limpid
Density at 20 °C (kg m^3)	0.8745	0.8737	850-900
Ash (%)	0.01	0.01	0.02
Sulphur (mg kg^{-1})	2.91	< 0.1	50
Sodium + Potassium (mg kg^{-1})	< 0.3	2.18	5
Calcium + Magnesium (mg kg^{-1})	< 0.1	0.91	5
Phosphorus (mg kg^{-1})	< 0.1	< 0.1	10
Copper corrosion, 3 h at 50 °C	1	1	1
Acid value (mg KOH g^{-1})	0.52	0.28	0.50
Iodine value (g per 100 g)	78.69	26.13	Report
Water content (ppm)	160	240	500

Table 6 Regression coefficients associated with the codified mathematical models for biodiesel transesterification of macauba pulp oil.

	Macauba pulp oil		
Regression coefficient	Effect	Standard deviation	p
b_0	109.5237	0.5000	0.0029
b_1	1.3565	0.5007	0.2251
b_{11}	-7.0535	0.6640	0.0598
b_2	0.8550	0.5007	0.3373
b_{22}	-5.0416	0.6640	0.0834
b_{12}	0.0000	0.7071	1.0000
	Macauba kernel oil		
Regression coefficient	Effect	Standard deviation	p
b_0	92.0412	0.1700	0.0012
b_1	5.8340	0.1702	0.0186
b_{11}	-4.9769	0.2257	0.0289
b_2	76.4122	0.1702	0.0014
b_{22}	-51.6848	0.2257	0.0028
b_{12}	-4.6400	0.2404	0.0329

Values marked in italics are statistically significant coefficients at a level of 90% to pulp oil and 95% to kernel oil;
b: regression coefficient;
$_1$: linear effect of the molar ratio;
$_2$: linear effect of the catalyst concentration;
$_{11}$: quadratic effect of the molar ratio;
$_{22}$: quadratic effect of the catalyst concentration;
$_{12}$: interaction effect between molar ratio and concentration.

reducing the catalytic activity which interpreted in terms enzyme deactivation due to microwave exposure. The enzyme loading also played an important role, however the effect of temperature was minor appearing only in the effect of variable interactions. The result comparison between biocatalyst activity in absence and presence of microwave showed that the activity is increased about one order of magnitude due to microwave.

3.7 Biodiesel Characterization

The biodiesel samples from macauba pulp and from kernel oil presented a clean aspect and Table 6 shows their characterizations. The density of biodiesel is strongly linked to its molecular structure, such that an increase in size of the carbon chain of

the alkyl ester increases its density. The density decreased at higher oil unsaturation levels, and another factor that influenced the density is the presence of impurities. The results showed that the specific gravities of the samples were very close, indicating that this characteristic influenced the molecular structure of the oil from which the biodiesel was derived. The values obtained were within the ANP specifications.

The water content of the samples was below the limit stipulated by the Brazilian legislation. The presence of water is not desirable, since it promotes hydrolysis of the biodiesel resulting in the production of free fatty acids, and also because it allows for microbial proliferation. The acid value of biodiesel from macauba pulp oil was above the ANP specifications. The presence of water in the sample can cause hydrolysis of the esters to alcohol and fatty acids, the ethyl esters being oxidized to fatty acids, thereby increasing the biodiesel acid number. The complete removal of water after the washing of biodiesel is very important to avoid high acid value.

4. Conclusions

The macauba (*Acrocomia aculeata*) fruit pulp and kernel are rich sources of lipids containing. The continuous press yield of dried macauba shell with pulp together was 20% of oil, and the continuous press yield of macauba kernel was 32% of oil. The results showed a variation in the fruits composition and different characteristics presented by the pulp oil, with higher content of mono and unsaturated fatty acids about 73%, while kernel oil contained about 66% saturated fatty acids. The kernel macauba oil belongs to lauric group. It was possible to define technological steps to obtain oil from the fruits of macauba by continuous pressing and find the optimal conditions to biodiesel production by transesterification, with concentration of catalyst 4.42% and 5% and 13.3:1 and 9:1 ethanol:oil molar ratio to kernel oil and pulp oil, respectively.

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