



Physicochemical properties of *Syagrus coronata* and *Acrocomia aculeata* oils for biofuel production



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ABSTRACT

This work examines the potential of two species of palm-tree, *Syagrus coronata* (Mart.) Becc. and *Acrocomia aculeata*, for biodiesel and thermo cracked bio-oil production. These species can offer the opportunity to obtain alternative fats and oils from perennial sources, which permits their combination with other crops or cattle production, as well as the recovery of degraded areas. The physico-chemical properties of the oils, biodiesel and bio-oil obtained from the two palms were evaluated. For biodiesel production, the high acid value of the *Acrocomia aculeata* crude vegetable oil required a previous acid esterification step to avoid the formation of soaps and stable emulsions, which was not necessary in the case of *Syagrus coronata* (Mart.) Becc. Concerning their fatty acid composition, SC exhibits mainly short, saturated chains, while AA exhibits long unsaturated chains. The short saturated chains make *Syagrus coronata* (Mart.) Becc. biodiesel less viscous and more stable to oxidation than *Acrocomia aculeata* biodiesel. However, both *Syagrus coronata* (Mart.) Becc. and *Acrocomia aculeata* biodiesels are in the acceptable range for use as biodiesel in diesel engines, showing promise for the economic exploitation of these raw materials, requiring only the use of an antioxidant additive in the case of *Acrocomia aculeata*. The bio-oils obtained from both *Syagrus coronata* (Mart.) Becc. and *Acrocomia aculeata* were not completely deoxygenated, but the physico-chemical properties showed good potential for producing renewable fuel acceptable for use in diesel engines. However, further studies are needed to achieve their complete deoxygenation and improve their properties.

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1. Introduction

The intrinsic concepts of the green chemistry and biorefinery platform comprise the design of chemical products that are less hazardous to the environment and the use of feedstocks derived from renewable resources or from abundant wastes (Sheldon et al., 2007; Santos et al., 2010). From this perspective, biofuels have been proposed as an elegant solution to replace fossil fuels in a sustainable way. Indeed, biodiesel or bio-oil may be obtained from fats and oils (source of triacylglycerides and derivatives) by esterification/transesterification or thermocracking, respectively, and either

used pure or blended with fossil fuel in diesel engines (Suarez et al., 2007).

Several vegetable fats and oils can be used for biodiesel and bio-oil production, and the choice of feedstocks must be based on technical and economic considerations to achieve a sustainable process. Thus, the search for alternative and renewable raw materials for biofuel production is highly strategic, and several studies are available in the literature concerning the use of unconventional vegetable fats and oils to produce biodiesel (Lima et al., 2004; Meneghetti et al., 2006; Doll et al., 2008; Kalbande et al., 2008; Scott et al., 2008; Oliveira et al., 2009; Xue et al., 2009; Liu et al., 2009; Xu and Hanna, 2009; Yang et al., 2009; Sharma et al., 2009; Suarez et al., 2009a,b; Sarin et al., 2010; Silva et al., 2010; Mangas et al., 2012; Shah et al., 2013; Iha et al., 2014).

In this context, palm-trees can offer the opportunity to obtain alternative fats and oils from perennial sources, which permits their combination with other crops or cattle production, as well as the recovery of degraded areas. Thus, palm-trees can be highlighted as

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an alternative oleaginous source that avoids the trilemma of food, energy, and environment (a topic elegantly discussed by Tilman et al. (2009)). To contribute to the knowledge of palm-trees as a fat and oil source, we decided to study the potential of *Syagrus coronata* (Mart.) Becc. (SC) and *Acrocomia aculeate* (AA) to produce biofuels because, as far as we know, information about these species is still scarce (Teixeira da Silva de La Salles et al., 2010; Alves et al., 2010).

Part of the family *Arecaceae*, the species *S. coronata* (SC) is popularly known in Brazil as “licuri” or “ouricuri”. This palm-tree grows mainly in semi-arid regions, and in Brazil, its geographical distribution comprises part of the states of Minas Gerais, Bahia, Sergipe and Alagoas. The SC tree bears fruit throughout the year; however, March, June, and July are the most favorable months for the best production (Noblick, 1986; Crepaldi et al., 2001). These fruits contain nuts that can grow to 2.5 cm in width. This species grows widely and has been used to recover degraded areas of the Brazilian semi-arid region as well as being cultivated alone or with other crops.

Acrocomia aculeate (AA) belongs to the *Arecaceae* family, and its more common name in Brazil is macauba. It is a palm-tree species native to tropical regions of the Americas, from southern Mexico and the Caribbean south to Brazil, Paraguay, and northern Argentina. The fruit is a yellowish-green drupe 2.5 to 5 cm in diameter. The inner fruit shell, also called the endocarp, is very tough and difficult to break and usually contains one single, dark brown, nut-like seed 1 to 2 cm in diameter (Hiane et al., 2006; Coimbra and Jorge, 2011). AA grows in a wide region of the Americas and, in Brazil, is largely cultivated for shade for the thermic comfort of cattle in the Savannah region (Suarez et al., 2009a,b).

We evaluated the characteristics and chemical composition of the oils obtained from SC and AA. Furthermore, we produced biofuels from these oils and analyzed their physico-chemical properties.

2. Materials and methods

2.1. Materials

SC fruits were collected from trees that grow in the Maceió/Alagoas ($09^{\circ}36'15''$ South and $35^{\circ}44'30''$ West) and São José da Tapera/Alagoas ($9^{\circ}32'38''$ South, $37^{\circ}23'3''$ West). The isolated nuts were dried at 105°C for 24 h. The oil was extracted by pressing in a hydraulic press (model TE-098 from Tecnal, Piracicaba, São Paulo, Brazil) and used without further purification. Oil of AA was directly obtained in a popular market in Montes Claros/Minas Gerais ($16^{\circ}44'06''$ South and $43^{\circ}51'43''$ West), produced locally, and used as received.

2.2. Biodiesel preparation

The AA methyl esters were synthesized in two steps. First, the AA oil, methanol and sulfuric acid were kept under gentle reflux for 2 h, and then the reaction mixture was washed three times with 5% (wt%) sodium bicarbonate solution. The biodiesel/oil phase was dissolved in hexane and kept over magnesium sulfate for 2 h, and then the mixture was filtered and the volatiles removed under vacuum. In the second step, the previous biodiesel/oil phase was mixed with a methanolic potassium hydroxide solution under magnetic stirring for 2 h at room temperature. The mixture was then allowed to stand, and two phases were obtained: one containing fatty acid methyl esters and the other glycerine, potassium hydroxide and methanol. The excess of methanol in the methyl ester phase was removed by rotary evaporator at 70°C . The methyl ester was then washed once with phosphoric acid (5% v/v) and three times with brine, dissolved in hexane, kept over magnesium sulfate for 2 h, and

filtered; finally, the volatiles were removed under vacuum. The second step was repeated two more times until purity of more than 98% (wt%) methyl esters was detected by HPLC, using the previously described method (Carvalho et al., 2012). The methyl esters were then stored in amber flasks in a freezer.

Because of its lower acidity, the SC oil was transesterified using only the second step of the procedure described for AA methyl ester synthesis.

2.3. Bio-oil preparation

The pyrolysis of SC and AA oils (175 g) was conducted in a three necked flask connected to a condensation system. The oil was heated at 400°C (the temperature was measured by thermocouples throughout the reaction), causing the formation of vapors that left the round flask and were condensed to obtain a two-phase system (an aqueous phase and a bio-oil). The yields of bio-oil were approximately 60%.

2.4. Oil and biofuels characterization

The vegetable oils were analyzed by methods described by the Association of Official Analytical Chemists (A.O.A.C.) (1997). The biodiesel samples were analyzed according to standard methods indicated by ANP (National Agency of Petroleum, Natural Gas and Biofuels) (2012) for this biofuel. The bio-oils were analyzed according to the recommendations of ANP for fossil diesel oil (Lima et al., 2004). The calorific value was evaluated in a Parr 1241 calorimetric bomb using oxygen at a pressure of 3.0 MPa. The fatty acid composition of the oils was determined by gas chromatography (GC) using the official AOCS method Ce 1-62 and Ce 2-26 (A.O.C.S., 1998).

The bio-oil composition was determined by gas chromatography (GC-MS) analyzes performed on a Shimadzu GC-17A chromatograph equipped with a mass spectrometer detector (Shimadzu GCMS-QP5050) using a polydimethylsiloxane column (CBPI PONA-M50-042; 50 m, 0.15 mm i.d., and film thickness of $0.42\ \mu\text{m}$), working between 80 and 180°C and heating at a rate of $10^{\circ}\text{C min}^{-1}$. Peaks were identified using a software library (Wiley Library CLASS-5000, 6th edition) with more than 95% similarity.

3. Results and discussion

3.1. Characteristics of SC and AA oils and biofuels

The SC oil content (50%, wt%) is higher than the values reported in the literature (Teixeira da Silva de La Salles et al., 2010), which may be related to different maturation degrees and fruit storage conditions. As described in the experimental section, the AA oil was commercially acquired, and the oil content was not determined experimentally. However, it is reported in the literature that the AA seeds contain up to 50% (wt%) oil (Hiane et al., 2006). These high values of oil content for SC and AA species indicate that processing the nuts to obtain oil would be economic.

Table 1 presents the fatty acid composition of SC and AA oils, which are distinctly different. The SC oil contains a higher amount of saturated fatty acid (90.0%) than the AA oils (30.0%). The 70.0% unsaturated fatty acids present in the AA oil is mainly shared between oleic and linoleic acids.

Table 2 summarizes the physico-chemical properties determined for SC and AA oils and their derived biodiesels. Very few properties of these oils were previously reported in the literature (Noblick, 1986; Crepaldi et al., 2001; Hiane et al., 2006; Teixeira da Silva de La Salles et al., 2010; Coimbra and Jorge, 2011), and the reported values that do exist are different from the results found in this work, most likely because of small variations in the fatty acid composition of the oils.

Table 1

Fatty acid composition of *Syagrus coronata* (Mart.) Becc. (SC) and *Acrocomia aculeata* (AA) oils.

Fatty acid	SC	AA
C8:0 (caprylic)	13.0	–
C10:0 (capric)	8.0	–
C12:0 (lauric)	48.0	–
C14:0 (myristic)	14.0	–
C16:0 (palmitic)	5.0	25.0
C16:1 cis 9 (palmitoleic)	–	6.0
C18:0 (stearic)	2.0	5.0
C18:1 (oleic)	7.0	52.0
C18:2 cis/cis 9,12 (linoleic)	3.0	11.0
C18:3 cis/cis/cis 9,12,15 (linolenic)	–	1.0
Saturated chains (%)	90.0	30.0
Mono-unsaturated chains (%)	7.0	52.0
Poly-unsaturated chains (%)	3.0	18.0

A low acid value was observed for SC ($1.5 \text{ mg KOH g}^{-1}$), but a very high acid value was observed for AA ($112.0 \text{ mg KOH g}^{-1}$). Although this elevated free fatty acid content may be related to the wild origin (different maturation degrees) and storage conditions of the nuts, some species of palms do contain enzymes that accelerate the hydrolysis process of triacylglycerides (Rodrigues and Ramos, 2011).

It is well established in the literature that transesterification of triacylglycerides can be conducted using basic or acid catalysts. It is also well established that basic catalysts are widely preferred because of their lower corrosiveness and higher efficiency, but it is also known that this catalytic system must be avoided for triacylglycerides containing high amounts of free fatty acids due to consumption of the alkaline catalyst and the formation of soap, thus generating emulsions (Alves et al., 2010).

The low acid value observed for the SC oil allows the biodiesel to be obtained in a single step by transesterification. However, two steps were required for AA biodiesel synthesis to obtain methyl esters: first, an acid-catalyzed esterification step, followed by base-catalyzed transesterification, as proposed for acid stocks more than 50 years ago (Keim, 1946). In both cases, a biodiesel containing fatty acid methyl esters with purity higher than 99% was obtained.

Table 2 also summarizes the physico-chemical properties of biodiesel from SC and AA. To the best of our knowledge, the reports in the literature of using SC and AA to produce biodiesel are very scarce (Teixeira da Silva de La Salles et al., 2010; Alves et al., 2010), and thus, it is difficult to compare the results presented here with previous data. As can be observed, the values of the properties studied here are distinctly different between the two species, which may easily be related to the diverse fatty acid composition exhibited for the SC and AA oils. First, SC biodiesel presents lower viscosity, density and cold filter plugging point when than AA biodiesel. Despite the higher unsaturated chain content of AA, the higher content of fatty acids with C8 to C14 in SC may readily explain this behavior (O'Brien et al., 2000). In addition, the lower oxidation

Table 3

Calculated cetane number (CN) for *Syagrus coronata* (Mart.) Becc. (SC) and *Acrocomia aculeata* (AA) oils and their biodiesel.

FAME	CN ^a	SC	AA
		Composition (mass%)	Composition (mass%)
Caprylate	33.6	13.0	–
Capriate	47.5	8.0	–
Laurate	61.5	48.0	–
Myristate	70.6	14.0	–
Palmitate	81.1	5.0	25.0
Palmitoleate	51.0	–	6.0
Stearate	88.9	2.0	5.0
Oleate	62.3	7.0	52.0
Linoleate	42.1	3.0	11.0
Linoleneate	22.7	–	1.0
Calculated CN	56.3		62.7

^a The CN for each FAME was calculated as the average of values reviewed by Murphy and co-workers (Murphy et al., 2004).

stability presented by AA biodiesel may be due to the high degree of unsaturation in its composition, as previously established in the literature (Souza et al., 2012). Finally, the lower chain length, which indicates higher oxygen content, explains the lower heat value observed for SC.

The investigated properties of SC biodiesel match the standard values accepted in Brazil for biodiesel (Rodrigues and Ramos, 2011). It is worth mentioning that the cold properties of SC biodiesel, together with its high oxidation stability and low viscosity and density, make this crop an excellent option even in low temperature regions. In addition, AA biodiesel is not specified because of its low oxidation stability, which may be easily adjusted using antioxidant agents.

Because of the challenges in experimentally determining the cetane number (CN) of biodiesel, certain authors have recently proposed empirical equations to calculate CN based on the FAME content and their individual CN (Bamgbose and Hansen, 2008; Tong et al., 2011). For instance, Tong and co-workers have proposed Eq. (1), where CN is the predicted CN of biodiesel, CN_i is the CN of each pure FAME, and w_i is the weight percentage of each FAME in biodiesel.

$$CN = 1.068 \sum_{i=0}^n CN_i \times w_i - 6.747 \quad (1)$$

The CN for SC and AA FAME were calculating using Eq. (1), and the results are presented in **Table 3**. As shown in **Table 3**, SC FAME has lower CN than AA FAME, which was expected because it consists mainly of smaller fatty acids. However, both FAME match the current ANP specifications for CN in diesel/biodiesel blends (min. 45).

Usually, the thermocracking of fats and oils leads to a complex mixture of more than 500 hydrocarbons and oxygenated

Table 2

Physico-chemical properties of *Syagrus coronata* (Mart.) Becc. (SC) and *Acrocomia aculeata* (AA) oils and their biodiesel.

Analysis	SC		AA		Standard method ^a
	Oil	Methylic biodiesel	Oil	Methylic biodiesel	
Kinematic viscosity ($\text{mm}^2 \text{s}^{-1}$)	27.6	2.9	41.9	5.6	ASTM D445
Acidity (mg KOH g^{-1} oil)	1.5	2.1	112.0	>0.5	AOCS CD 3d-63
Density at 20°C (g m^{-3})	922	871	919	883	NBR 7148
Cold filter plugging point ($^\circ\text{C}$)	5	-10	10	-2	ASTM 6371
Carbon residue (%)	0.20	0.02	2.70	0.05	ASTM D189
Heat of combustion (MJ kg^{-1})	38.6	37.0	40.3	43.5	ASTM D240
Oxidative stability (h)	10.7	9	0.07	0.17	EM 14112
Copper corrosion	–	1A	–	1A	ASTM D130

^a Methods in accordance with specifications by the Brazilian National Petroleum Agency (ANP (National Agency of Petroleum, Natural Gas and Biofuels), 2012).

Table 4

Physico-chemical properties of thermocracked bio-oils obtained from *Syagrus coronata* (Mart.) Becc. (SC) and *Acrocomia aculeata* (AA) oils.

Analysis	SC	AA	Standard method ^a
Kinematic viscosity ($\text{mm}^2 \text{s}^{-1}$)	5.1	4.0	ASTM D445
Flash point (°C)	25	35	ASTM D93
Acidity (mg KOH g ⁻¹ oil)	180.9	133.6	AOCS CD 3d-63
Density at 20 °C (g m ⁻³)	876	848	NBR 7148
Cold filter plugging point (°C)	5	2	ASTM D6371
Cetane number	39.8	44.5	ASTM D4737
Carbon residue (%)	0.19	0.40	ASTM D189
Heat of combustion (MJ kg ⁻¹)	38.6	38.7	ASTM D240
Automatic distillation (°C)			
10%	217.4	148.3	ASTM
50%	290.9	279.4	D613
90%	346.1	349.9	
Copper corrosion	1A	1A	ASTM D130

^a Methods in accordance with specifications by ANP (National Agency of Petroleum, Natural Gas and Biofuels) (2012).

compounds, mainly aliphatic olefins and paraffins and carboxylic acids, with short and long chains (Lima et al., 2004; Santos et al., 2010; Teixeira da Silva de La Salles et al., 2010; Mangas et al., 2012; Shah et al., 2013). According to the GC-MS analysis, the thermocracking of both SC and AA oils leads to a similar and complex mixture of more than 500 hydrocarbons and oxygenated compounds, in good agreement with previous literature reports. The physico-chemical properties for the mixtures are presented in Table 4. It is important to note that high acid values were observed, in agreement with the GC-MS results, strongly indicating that the secondary cracking did not completely deoxygenate the carboxylic acids formed in the primary cracking. However, the secondary cracking may be easily improved if the reaction is assisted by Lewis acid catalysts (Quirino et al., 2009) or is performed under hydrogen atmosphere in the presence of platinum group metal catalysts (Gusmão et al., 1989). The acid value of SC bio-oil is slightly higher than the acid value of AA, which may be responsible for its higher density and viscosity. However, the values observed for the physico-chemical properties show a high potential for these bio-oils to obtain a mixture suitable for use as renewable diesel after improving the secondary cracking using the methods cited above. The CN calculated for SC is lower than the CN calculated for AA. This behavior is most likely a result of the lower fatty acid carbonic chains in SC compared to AA. It is important to note that the CN calculated for SC bio-oil does not match the ANP specifications for CN in diesel/biodiesel blends (min. 45), and the value calculated for AA bio-oil is slightly below the limit.

4. Conclusions

In summary, we have studied two different palm tree species as oleaginous sources for biofuel production. Note that these two species grow in the semi-arid lands of Brazil and in a large region of South and Central America; thus, they may be an elegant solution to produce biofuels while aiding in the preservation of native vegetation.

The high acid value of the AA crude vegetable oil required the use of an initial acid esterification step to avoid the formation of soaps and stable emulsions, which was not necessary in the case of SC. Regarding their fatty acid composition, the two oils are remarkably different: while SC consists mainly of short saturated chains, AA consists of long unsaturated chains. As expected, these differences in their fatty acid compositions lead to different physico-chemical properties. For instance, the short and saturated chains make SC biodiesel less viscous and more stable to oxidation than AA biodiesel. However, both SC and AA biodiesel are in

an acceptable range for use as biodiesel in diesel engines, showing promise for the economic exploitation of these raw materials, requiring only the use of an antioxidant additive in the case of AA.

The thermocracked bio-oils obtained from both SC and AA were not completely deoxygenated, but their physico-chemical properties showed good potential for producing renewable fuel acceptable for use in diesel engines. However, further studies are needed to perform their complete deoxygenation and improve their properties.

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