1 **Characterization of Jatropha Curcas oils and their derived fatty acid ethyl esters**

2 **obtained from two different plantations in Cuba**

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11

12 **Abstract** The scope of this work is to evaluate some properties of the oils and derived fatty

13 acid ethyl esters (FAEE) from two different Jatropha Curcas species planted in Cuba. The

14 properties that were determined include the acid value, peroxide value, p-anisidine value and

15 fatty acid ethyl esters composition. In order to study the influence of the genus species and

16 geographic conditions on the fuel properties, the oils from Jatropha Curcas planted in two

17 regions of Cuba and their derived FAEE were analyzed and compared. The two plantations

18 were in San José (SJ) and Guantanamo (Gt) representing respectively the western and eastern

19 part of the island.

20 The analyses indicated that the FAEE obtained from Guantanamo has a higher acid value and

21 peroxide value compared with the FAEE from San José. The p-anisidine values did not show

22 a clear trend and the results of gas chromatography-mass spectrometry indicated a similar

23 FAEE composition. The results obtained by gas chromatography are in good agreements with

24 previous reports.

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1. Introduction

The work reported here is part of a 5 year project between Ghent University (Belgium) and the Technical University of Havana (Cuba). Given the limited Cuban natural resources, in terms of oil fields and hydropower, Cuba is dependent on foreign oil for its energy and transportation demands. A promising alternative to imported fossil fuels is the use of renewable sources such as wind or solar power for electricity generation. For the transportation sector the application of renewable fuels from non-edible crops and waste products offers an alternative to diesel and gasoline. A transition to these renewable fuels requires profound knowledge on the biofuels, their characteristics, behavior and effects on storage, durability etc. The project aims to implement a multidisciplinary knowledge cell in order to increase knowledge on the use of renewable fuels and the social awareness of these fuels. The activities include the inventory of possible renewables, the estimation of their potential, and the study of likely candidate renewables. One of the renewables that has been identified as a potential source for transportation fuel is the Jatropha crop.

Jatropha Curcas (physic nut), native of tropical America, has been later introduced into Africa and Asia and is now cultivated worldwide [1]. Jatropha is a genus of approximately 175-200 plants, shrubs and trees, from the family of Euphorbiaceae. It is resistant to drought and produces seeds containing up to 40 % mass of oil. When the seeds are crushed and processed, the resulting oil can be used in a standard diesel engine [2-6] when measures are taken to decrease the viscosity: preheating of the oil, blending with fossil diesel fuel or converting the oil to biodiesel using the esterification reaction. In a comparison between crop efficiencies for biodiesel production, only algae and palm oil yield better results than Jatropha [2].

While important amounts of biodiesel are nowadays produced from edible sources, a challenge for biodiesel production is to use feedstocks that would not compete with human food. In that direction, Jatropha Curcas has been identified among the most promising non-edible oil-bearing seeds for biodiesel production. Jatropha produces mainly a non edible oil due to the phorbol esters that are toxic [7], even at very low concentration. However, there are some species of Jatropha that produce edible oils [8].

Biodiesel fuels are generally classified as fatty acid methyl esters (FAME), which are derived from the alkali-catalyzed transesterification of oils or fats with methanol, although other alcohols can be used [9-11]. Due to the definition of biodiesel given by governmental

62 regulations and standards, that only define biodiesel as FAME which is the result of reaction
63 of fatty acids with methanol [12], the biofuels tested in this paper will be named as derived
64 FAEE as ethanol was used in the chemical synthesis.

65

66 Some physical properties of Jatropha oil are shown in Table 1. The range of values reported
67 for the cetane number (CN) is because this value depends on the Jatropha oil variety and
68 climate conditions, affecting the fatty acid composition [13-15].

69 Some important properties for biofuels and issues encountered with them, are discussed in the
70 following.

71 • The Fatty Acid (FA) content in these kinds of biofuels has an important influence on
72 their properties [16]. There are reports of the use of the FA composition of biodiesel
73 obtained by Gas Chromatography (GC) for predicting the viscosities of biodiesel [9].
74 One of the contributing factors to the variation in engine performance can be the
75 viscosity of the fuel. This is because the atomization process, which is the initial stage
76 of the combustion process in a diesel engine, is significantly affected by the fuel's
77 viscosity [9]. The viscosity of oil or its derivative (m)ethyl ester is influenced by its
78 fatty acids composition[16]. It is also possible to estimate the vapor pressure and the
79 normal boiling point of biodiesel fuels from their FAME or FAEE composition [17].
80 Jatropha oil is composed of the following fatty acids: myristic (14:0), palmitic (16:0),
81 stearic (18:0), oleic (18:1) and linoleic (18:2) [2]. Depending on the origin of the crop,
82 the average fractions of oleic and linoleic acids found in Jatropha oil are 50 and 30 %
83 respectively. Samples from Indonesia contain fractions of 40 % and 37 % of oleic and
84 linoleic acids respectively. In Brazil, the reported composition for these two FA are 30
85 and 50 % respectively, indicating the predominance of the most unsaturated fatty acid
86 and the higher susceptibility to oxidation of the oil [18, 19].
87 The FA composition of Jatropha Curcas planted in Sancti Spiritus (located in the
88 central part of Cuba) using chemical and mechanical extraction of the oil from the
89 seed is reported in [10]. The authors found fractions of 35.2 % of oleic acid, 39 % of
90 linoleic acid, but also 17 % of palmitic acid in Jatropha Curcas' oil.
91 Jatropha-derived biodiesel has the same FA relative composition compared to its
92 initial oils or fats. A study of the derived fatty acid composition of four different
93 Jatropha species from Mexico is reported in [7]. They found variations in the oleic
94 acid fractional composition between 40-49 % within different species, depending on

95 the sample procedure and between 35-44 % for linoleic acid. Another report from
96 Mexico gave similar proportions of oleic and linoleic acids (40 %) [20, 21].

- 97 The oxidative stability of a fuel is also a crucial property, especially for long-term
  98 storage [18]. Oxidative degradation also affects some properties of the biodiesel:
  99 kinematic viscosity, cetane number, and acid value (*AV*) of the fuel [22, 23]. The
  100 German biodiesel standard DIN V 51606 requires esters to have an acid number below
  101 0.5 $mg(KOH) \cdot g^{-1}$ and a iodine value less than 115 $g(iodine) \cdot (100g)^{-1}$, , among many
  102 other specifications [22].

- 103 The cetane number is one of the most commonly used indicators of diesel fuel quality.
  104 It is dependent on the composition of the fuel and has a strong impact on the engine's
  105 performance, noise level, and exhaust emissions [16, 24].

- 106 CO emissions have been reported to be influenced by the saturation of the fatty acids.
  107 CO emissions decrease as the saturation level is increased. The effect of biodiesel
  108 acidity and oxidation on CO emissions is also reported, with CO emissions increasing
  109 as the acid value is increased [11].

- 110 Engine durability testing with biodiesel has shown that biodiesel may be subject to
  111 fuel filter plugging/clogging problems caused by sediments and gums. This is due to
  112 the fuel chemically changing to produce these compounds, which represents a fuel
  113 stability problem [25].

- 114 Acidity, characterized by a high acid value, can lead to a lower than usual heating
  115 value, affecting the engine performance [11]. The acidity is the result of oxidation
  116 processes, which explains the lower heating value. A way to estimate the primary and
  117 secondary oxidation products presented in derived (m)ethyl esters or oil is evaluating
  118 the peroxide value (*PV*) and p-anisidine value (*p-AV*).

119

120 It is clear from the above that the determination of the fatty acid composition and the levels of
121 oxidation products in oils and their derived esters is very important to predict and understand
122 the engine behavior when these biofuels are used, especially when comparisons are to be
123 made with diesel fuel or with biofuels with similar physical properties [16, 24].

124

125 The scope of this work is to evaluate the acid value, peroxide value, p-anisidine value and
126 fatty acid composition of the oils and derived FAEE from two different Jatropha Curcas
127 species planted in Cuba and to compare the obtained results in order to study the influence of

128   the genus species and geographic conditions on fuel properties that could affect the behavior

129   of a diesel engine when these biofuels are to be used as diesel replacement.

130

131   **2. Samples and Analysis Methods**

132

133   *2.1 Samples*

134   The samples tested came from two different sources: one is in the western part of the Cuban

135   island: San José de las Lajas (23° N, 82°10´ W), located 30 km south-east from Havana city,

136   with tropical wet climate conditions, where the annual precipitation rate is normally higher

137   than 1500 mm. The second plantation is settled in the Farm Paraguay, located in the

138   Guantanamo province (20°10´ N, 75°15´ W) that is settled more than 800 km from Havana

139   and is 70 km from Santiago de Cuba. The soil characteristics in Guantanamo are mainly dry

140   and saline with a half deserted or totally deserted soil trend. The Jatropha planted in

141   Guantanamo belongs to a Cuban genetic variety and the Jatropha planted in San José belongs

142   to an African variety. Both samples were tested as oils and as their derived FAEE. Both

143   Jatropha Curcas plantations have the potential to provide Jatropha oil or biodiesel for the local

144   needs. All the seeds were selected from manually-collected fruits. The fruits were harvested in

145   April 2010 in young plantations (3 years old for Guantanamo and 1.5 years old for San José).

146   The seeds were dried exposed to solar radiation and manually dehusked because these are the

147   usual methods used by the farmers in both places.

148   The seeds were stored in plastic bags and transported to the laboratory for oil extraction and

149   FAEE synthesis. The oil was squeezed out from the kernels with a handoperated press. The

150   liquid samples prepared (oils and derived FAEE) were stored in glass vials in nitrogen

151   atmosphere. They were transported by air to Ghent University, Belgium. The biofuels were

152   prepared simultaneously and the samples were tested one month after their preparation.

153   As reference, Cuban standard diesel fuel was also tested.

154

155   *2.2 Acid Value*

156   The acid value gives an idea of the amount of free fatty acids (FFA) contained in the oil, as a

157   result of the hydrolysis of the ester bond between the fatty acid and the glycerol molecule.

158   The presence of FFA in the oil is undesired as it can have a corrosive effect on some parts of

159   the engine and can also give undesired saponification reactions. The amount of FFA is

160   detected by titration with NaOH or KOH. By knowing the exact amount of base required to

161   neutralize all the acids [26-28], it is possible to calculate the amount of acids in an oil. The

162   acid value is expressed as *mg (KOH)·g^{-1}* of oil. To obtain the acid value data, each experiment

163   was repeated three times.

164   For the acid value determination a solvent mixture 1:1 by volume with ethanol at 99.9 % and

165   diethylether was used. The indicator used in the titration was a phenolphthalein 10 *g·L^{-1}* in

166   ethanol. The neutralization solution was NaOH 0.01 *mol·L^{-1}*. The determination of the acid

167   value is based on [28].

168

2.3 Peroxide Value

170   The content of peroxides (primary oxidation products) is correlated with the degree of

171   oxidation of oils or fats. Hydroperoxides are unstable and will form secondary oxidation

172   products (aldehydes, ketones, alcohols, epoxides). The esters constituting the biodiesel are

173   subject to oxidation through contact with the oxygen in the air. When this process occurs at

174   ambient temperatures, the initial hydroperoxides are formed by the addition of oxygen to a

175   carbon atom adjacent to a carbon-carbon double bond. The extent of this level of oxidation

176   can be characterized by the peroxide value. As oxidation proceeds, the peroxides may split

177   and form aldehydes and short chain acids.

178   For titration, a solvent mixture of 3:2 of glacial acetic acid and chloroform, KI saturated

179   solution, sodium thiosulphate and a diluted starch solution were used [29]. Each experiment

180   was repeated three times. The determination of the peroxide value (*PV*) is based on [29].

181

2.4 p-Anisidine Value

183   The *p*-anisidine value (*p-AV*) is a measure for secondary oxidation products that can be

184   present in oil or its derived FAEE. The secondary oxidation products are reacted with p-

185   anisidine, resulting in the production of a coloured compound which is assessed

186   spectrophotometrically.

187   The experiments were set using iso-octane as an optically clear dissolution and a dissolution

188   of 0.25 *g* of *p*-anisidine in 100 *mL* glacial acetic acid. The solution was kept refrigerated (4°*C*)

189   in the glacial state. Test tubes with Teflon lined screw caps were used. The

190   Spectrophotometer UV-Visible Varian Cary 50 Probe was suitable for observation at 350 *nm*

191   [30] using a pair of identical quartz cells. Each experiment was repeated three times.

192   The determination of the *p-AV* is based on [30].

193

2.5 GC-MS

The composition of the FAEE was analyzed using a Gas Chromatograph Carlo Erba 8065 equipped with a flame ionization detector and a HT8 capillary column. The GC oven was kept 2 *min* at 70 °C, heated at 30 °C·min^{-1} and kept 30 *min* at 260 °C. The samples analyses by GC were carried out by injecting 1 *µL* of the sample solution at 260 °C, split 1:40. The formed ethyl esters were identified by Mass Spectrometry (MS) and by comparison of their retention times with the spectrums catalog of the standard methyl esters of fatty acids. The MS device was a Fisons MD 800, Detention EI+, and Ionization Energy 70 *eV*.

3. Results and Discussions

3.1 Acid, peroxide and p-anisidine values

The results of the acid value determination for the samples are shown in Table 2. The values reported in the table represent the mean values of three experiments per sample. The uncertainties given here and in the following are absolute values and were determined from error analysis following the methods described [28].

A decrease in acid values is found for the FAEE compared to the oils for both sources, which is related to the decrease of the FFA present in the original oil. The comparison between the Jatropha oil samples shows that the *AV* of Guantanamo oil is much higher compared to the *AV* of the oil from San José. The *AV* for the reference diesel fuel was negligible due to the non presence of FFA in the fuel.

The difference in *AV* for the FAEE samples is related to the differences found in their source oils. The *AV* obtained for biodiesel is typically less than 1 *mg (KOH)·g^{-1}* of oil [26, 31]. Berchmans [20] established 2 *mg (KOH)·g^{-1}* as the amount that assures a FFA concentration less than 1 % but he found 30 *mg (KOH)·g^{-1}* for pure Jatropha oil. The range of *AV* that can be obtained for Jatropha Curcas oil varies widely, with values reported between 1-38 *mg (KOH)·g^{-1}* [32]. The wide range found in literature is because these values depend on the biofuels degradation stage and on the natural source.

Hamasaki et al. [33] reported engine tests on biodiesels with *AV* variations between 0.3-0.9, with similar engine thermal efficiencies compared to diesel fuel. On the contrary, the exhaust gas emissions of a diesel engine are claimed to be influenced by differences in fuel *AV* of 0.9 *(KOH)·g^{-1}* [11, 33].

The observed increase in *AV* for samples from Guantanamo must be related to a slightly higher degradation process of the oils, through oxidation or hydrolysis of triglycerides.

227   The results of the peroxide value determination of the samples are shown in Table 3. The *PV*

228   is reported in milliequivalents of oxygen per kilogram of oil ($meq.O_2 \cdot kg^{-1}$). Significant

229   differences can be observed for the samples from different regions. The uncertainty levels are

230   similar but the obtained uncertainty value for diesel fuel is of the same level of the peroxide

231   value itself. This is because the amount of secondary oxidation products found in diesel fuel

232   are of the same order as the detection limits of the applied method.

233   The peroxide value can increase during the time the oil or the ethyl esters are stored, but also

234   if the preparation of the biofuels includes distillation of the final product or not. In this case

235   the biofuels were prepared simultaneously and without distillation. The samples were tested

236   one month after their preparation. The obtained *PV* for the Jatropha oil samples are higher

237   than the reported values by Sharma (4.26 ± 0.12) [21], which can be influenced by the storage

238   time.

239   It has been reported that the oxidation level of the FA has an impact on the cetane number

240   since peroxide compounds have been proposed as cetane improvers [34, 35]. The presence of

241   peroxides, as a consequence of the oxidation process, may result in lower THC emissions, as

242   also reported in [11].

243

244   The results of the p-anisidine determination are shown in Table 4. Again, important

245   differences between the samples from different regions are observed. The secondary oxidation

246   products are also higher for the Guantanamo Jatropha oil and its derived FAEE. This result

247   indicates more secondary oxidation processes for the fuels obtained from the Guantanamo

248   plantations according to the fact that all the biofuels tested were prepared simultaneously.

249

250   **3.2 Composition of the oils and derived FAEE**

251   The results of the GC-MS applied to the FAEE samples are shown in Fig.1. The FAEE peaks

252   correspond to ethyl palmitate (16:0), ethyl linoleate (18:2), ethyl oleate (18:1) and ethyl

253   stearate (18:0). The chromatogram A represents the result for FAEE from Guantanamo while

254   chromatogram B represents that obtained for the FAEE from San José. The chromatogram C

255   represents the peaks distribution obtained for a FAEE standard injected into the system for

256   comparison with the sample peaks for qualitative and quantitative analysis of the biodiesels.

257   The GC identification of peaks was possible by applying the GC method to standard mixtures

258   of FAEE and FAME. The retention times of the peaks for the standard mixtures used are

259   shown in detail in Fig.2.

No significant differences concerning the GC results between the biodiesel samples were found, as is observed in Fig.1. Thus, it can be expected that possible differences in engine emissions between the two biodiesels will not be due to differences in FAEE composition. CO emissions have been found to decrease as the saturation level is increased [36] but we found no differences in saturation level among the biofuels tested. An increase in saturation level can also generate increases in the soluble organic fraction due to the lower volatility of saturated esters. Chang [37] observed an inverse relationship between esters saturation and NO_x emissions. The higher the ester saturation, the more stable compounds and therefore less peroxides and a lower cetane number should be expected, leading to higher maximum combustion temperatures in the combustion chamber and higher NOx in the exhaust gases.

The fractional composition for the FAEE samples is between 19-20 % for ethyl palmitate (16:0), 47-50 % of ethyl linoleate (18:2), 25.5-27 % of ethyl oleate (18:1) and 4.5-5 % of ethyl stearate (18:0). These results are close to those reported by other authors. A comparison of the GC results obtained in this research with other reports is presented in Table 5.

The results obtained for ethyl palmitate and ethyl stearate are similar to those reported by Martin [10]. The results for ethyl oleate and ethyl linoleate are near the reported values by Berchmans [20], but also near those reported by Martin [10]. It is interesting to point out that the Jatropha Curcas analyzed by Martin [10] comes also from Cuba, obtained from a plantation in the central part of the island. The relative composition obtained for ethyl linoleate is higher than that obtained for ethyl oleate. This result obtained for the samples from San José and Guantanamo was also observed for the Jatropha planted in Sancti Spiritus (21° 59´N, 79°14´ W) by Martin, that is the opposite of some reports presented in Table 5.

These results show that both varieties of Jatropha Curcas planted in different parts of the Cuban island do not cause significant differences concerning the FAEE composition obtained by trans-esterification.

4. Conclusions

The Jatropha oil obtained from Guantanamo and the derived FAEE have higher acid, peroxide and p-anisidine values compared to the oil and derived FAEE from the Jatropha planted in San José, representing more active oxidation processes in the first one. The increase in acid value and peroxide value found in Guantanamo samples may result in lower HC emissions and a higher cetane number. This will be checked in future work through engine testing.

The composition of the FAEE obtained from Jatropha (two species) planted in both regions is quite similar. The FAEE results are in good agreement with the reported values found in literature.

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Table 1. Physical properties of Jatropha Curcas oil

Physical properties	Value
density	901 $g \cdot m^{-3}$
melting point	$5\,^{\circ}C$
kinematic viscosity (at 40°C)	$34.6 \cdot 10^{-6} \, m^2 \cdot s^{-1}$
cetane number	41-51
iodine number	103
heat of combustion	39.63 $MJ \cdot kg^{-1}$

Table 2. Acid Values for the different samples

Sample	$mg\ (KOH){\cdot}g^{-1}$	Standard Deviation	Uncertainty
Jatropha oil- SJ	0.7	0.05	0.03
Jatropha oil-Gt	1.7	0.09	0.28
FAEE-SJ	0.5	0.05	0.23
FAEE-Gt	1.0	0.07	0.23
Diesel fuel	0.1	0.03	0.03

Table 3. Peroxide Values for the different samples

Sample	$meq.O_2 \cdot kg^{-1}$	Standard Deviation	Uncertainty
Jatropha oil- SJ	5.9	0.19	1.4
Jatropha oil-Gt	20.2	1.47	2.5
FAEE-SJ	5.3	0.88	1.15
FAEE-Gt	14.7	1.65	2.05
Diesel fuel	0.8	0.20	0.6

Table 4. P-anisidine Values for the different samples

Sample	p-AV	Standard Deviation	Uncertainty
Jatropha oil- SJ	0.6	0.05	0.01
Jatropha oil-Gt	1.6	0.04	0.01
FAEE-SJ	1.5	0.04	0.01
FAEE-Gt	1.2	0.05	0.01
Diesel fuel	0.4	0.05	0.01

Table 5. Comparison between ethyl ester composition in the samples and reports

(m)ethyl esters (%)	Present samples	[10]	[7]	[20]	[21]	[32]	[36]	[37]
16:0	19-20	16.8	10-13	15	12.1	15.6	14-15	14-15
18:0	4.5-5	6.7	2.4-2.8	3.8	16.8	6.7	7-7.4	3-10
18:1	25.5-27	35.2	45-49	32.5	13.0	42.6	34-45	34-46
18:2	48-50	39.0	34-44	47.4	49.7	33.9	31-43	29-44

Ethylester

100-

%

0-

4.131
7.142
10.082
12.753
C14:0 15.173
C16:0 17.373
C18:0 19.393
21.244
22.964
24.594

TR

Methylester

100-

%

0-

C16:0 16.938

C14:0 14.645

C18:0 19.017

3.581
6.332
9.333
10.754
12.114
13.405
C15:0 15.806
18.757
19.897
20.838
20.928
21.828
22.698
23.529
24.349
26.280

5.000   7.500   10.000   12.500   15.000   17.500   20.000   22.500   25.000