Characterization of Jatropha Curcas oils and their derived fatty acid ethyl esters obtained from two different plantations in Cuba

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Abstract The scope of this work is to evaluate some properties of the oils and derived fatty acid ethyl esters (FAEE) from two different Jatropha Curcas species planted in Cuba. The properties that were determined include the acid value, peroxide value, p-anisidine value and fatty acid ethyl esters composition. In order to study the influence of the genus species and geographic conditions on the fuel properties, the oils from Jatropha Curcas planted in two regions of Cuba and their derived FAEE were analyzed and compared. The two plantations were in San José (SJ) and Guantanamo (Gt) representing respectively the western and eastern part of the island.

The analyses indicated that the FAEE obtained from Guantanamo has a higher acid value and peroxide value compared with the FAEE from San José. The p-anisidine values did not show a clear trend and the results of gas chromatography-mass spectrometry indicated a similar FAEE composition. The results obtained by gas chromatography are in good agreements with previous reports.

Keywords: Jatropha Curcas, FAEE, acid value, peroxide value, anisidine.

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

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

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

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

- The Fatty Acid (FA) content in these kinds of biofuels has an important influence on their properties [16]. There are reports of the use of the FA composition of biodiesel obtained by Gas Chromatography (GC) for predicting the viscosities of biodiesel [9]. One of the contributing factors to the variation in engine performance can be the viscosity of the fuel. This is because the atomization process, which is the initial stage of the combustion process in a diesel engine, is significantly affected by the fuel's viscosity [9]. The viscosity of oil or its derivative (m)ethyl ester is influenced by its fatty acids composition[16]. It is also possible to estimate the vapor pressure and the normal boiling point of biodiesel fuels from their FAME or FAEE composition [17]. Jatropha oil is composed of the following fatty acids: myristic (14:0), palmitic (16:0), stearic (18:0), oleic (18:1) and linoleic (18:2) [2]. Depending on the origin of the crop, the average fractions of oleic and linoleic acids found in Jatropha oil are 50 and 30 % respectively. Samples from Indonesia contain fractions of 40 % and 37 % of oleic and linoleic acids respectively. In Brazil, the reported composition for these two FA are 30 and 50 % respectively, indicating the predominance of the most unsaturated fatty acid and the higher susceptibility to oxidation of the oil [18, 19].

The FA composition of Jatropha Curcas planted in Sancti Spiritus (located in the central part of Cuba) using chemical and mechanical extraction of the oil from the seed is reported in [10]. The authors found fractions of 35.2 % of oleic acid, 39 % of linoleic acid, but also 17 % of palmitic acid in Jatropha Curcas' oil.

Jatropha-derived biodiesel has the same FA relative composition compared to its initial oils or fats. A study of the derived fatty acid composition of four different Jatropha species from Mexico is reported in [7]. They found variations in the oleic acid fractional composition between 40-49 % within different species, depending on

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

- The oxidative stability of a fuel is also a crucial property, especially for long-term storage [18]. Oxidative degradation also affects some properties of the biodiesel: kinematic viscosity, cetane number, and acid value (AV) of the fuel [22, 23]. The German biodiesel standard DIN V 51606 requires esters to have an acid number below $0.5 \text{ mg(KOH)} \cdot \text{g}^{-1}$ and a iodine value less than $115 \text{ g(iodine)} \cdot (100\text{g})^{-1}$, , among many other specifications [22].
- The cetane number is one of the most commonly used indicators of diesel fuel quality. It is dependent on the composition of the fuel and has a strong impact on the engine's performance, noise level, and exhaust emissions [16, 24].
- CO emissions have been reported to be influenced by the saturation of the fatty acids. CO emissions decrease as the saturation level is increased. The effect of biodiesel acidity and oxidation on CO emissions is also reported, with CO emissions increasing as the acid value is increased [11].
- Engine durability testing with biodiesel has shown that biodiesel may be subject to fuel filter plugging/clogging problems caused by sediments and gums. This is due to the fuel chemically changing to produce these compounds, which represents a fuel stability problem [25].
- Acidity, characterized by a high acid value, can lead to a lower than usual heating value, affecting the engine performance [11]. The acidity is the result of oxidation processes, which explains the lower heating value. A way to estimate the primary and secondary oxidation products presented in derived (m)ethyl esters or oil is evaluating the peroxide value (PV) and p-anisidine value (p-AV).

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

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

the genus species and geographic conditions on fuel properties that could affect the behavior of a diesel engine when these biofuels are to be used as diesel replacement.

2. Samples and Analysis Methods

2.1 Samples

The samples tested came from two different sources: one is in the western part of the Cuban island: San José de las Lajas (23° N, 82°10' W), located 30 km south-east from Havana city, with tropical wet climate conditions, where the annual precipitation rate is normally higher than 1500 mm. The second plantation is settled in the Farm Paraguay, located in the Guantanamo province (20°10' N, 75°15' W) that is settled more than 800 km from Havana and is 70 km from Santiago de Cuba. The soil characteristics in Guantanamo are mainly dry and saline with a half deserted or totally deserted soil trend. The *Jatropha* planted in Guantanamo belongs to a Cuban genetic variety and the *Jatropha* planted in San José belongs to an African variety. Both samples were tested as oils and as their derived FAEE. Both *Jatropha Curcas* plantations have the potential to provide *Jatropha* oil or biodiesel for the local needs. All the seeds were selected from manually-collected fruits. The fruits were harvested in April 2010 in young plantations (3 years old for Guantanamo and 1.5 years old for San José). The seeds were dried exposed to solar radiation and manually dehusked because these are the usual methods used by the farmers in both places.

The seeds were stored in plastic bags and transported to the laboratory for oil extraction and FAEE synthesis. The oil was squeezed out from the kernels with a handoperated press. The liquid samples prepared (oils and derived FAEE) were stored in glass vials in nitrogen atmosphere. They were transported by air to Ghent University, Belgium. The biofuels were prepared simultaneously and the samples were tested one month after their preparation.

As reference, Cuban standard diesel fuel was also tested.

2.2 Acid Value

The acid value gives an idea of the amount of free fatty acids (FFA) contained in the oil, as a result of the hydrolysis of the ester bond between the fatty acid and the glycerol molecule. The presence of FFA in the oil is undesired as it can have a corrosive effect on some parts of the engine and can also give undesired saponification reactions. The amount of FFA is detected by titration with NaOH or KOH. By knowing the exact amount of base required to neutralize all the acids [26-28], it is possible to calculate the amount of acids in an oil. The

acid value is expressed as $mg (KOH) \cdot g^{-1}$ of oil. To obtain the acid value data, each experiment was repeated three times.

For the acid value determination a solvent mixture 1:1 by volume with ethanol at 99.9 % and diethylether was used. The indicator used in the titration was a phenolphthalein $10 g \cdot L^{-1}$ in ethanol. The neutralization solution was NaOH $0.01 mol \cdot L^{-1}$. The determination of the acid value is based on [28].

2.3 Peroxide Value

The content of peroxides (primary oxidation products) is correlated with the degree of oxidation of oils or fats. Hydroperoxides are unstable and will form secondary oxidation products (aldehydes, ketones, alcohols, epoxides). The esters constituting the biodiesel are subject to oxidation through contact with the oxygen in the air. When this process occurs at ambient temperatures, the initial hydroperoxides are formed by the addition of oxygen to a carbon atom adjacent to a carbon-carbon double bond. The extent of this level of oxidation can be characterized by the peroxide value. As oxidation proceeds, the peroxides may split and form aldehydes and short chain acids.

For titration, a solvent mixture of 3:2 of glacial acetic acid and chloroform, KI saturated solution, sodium thiosulphate and a diluted starch solution were used [29]. Each experiment was repeated three times. The determination of the peroxide value (PV) is based on [29].

2.4 *p*-Anisidine Value

The *p*-anisidine value (*p*-AV) is a measure for secondary oxidation products that can be present in oil or its derived FAEE. The secondary oxidation products are reacted with *p*-anisidine, resulting in the production of a coloured compound which is assessed spectrophotometrically.

The experiments were set using iso-octane as an optically clear dissolution and a dissolution of 0.25 g of *p*-anisidine in 100 mL glacial acetic acid. The solution was kept refrigerated ($4^{\circ}C$) in the glacial state. Test tubes with Teflon lined screw caps were used. The Spectrophotometer UV-Visible Varian Cary 50 Probe was suitable for observation at 350 nm [30] using a pair of identical quartz cells. Each experiment was repeated three times.

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

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.

The results of the peroxide value determination of the samples are shown in Table 3. The *PV* is reported in milliequivalents of oxygen per kilogram of oil ($meq.O_2 \cdot kg^{-1}$). Significant differences can be observed for the samples from different regions. The uncertainty levels are similar but the obtained uncertainty value for diesel fuel is of the same level of the peroxide value itself. This is because the amount of secondary oxidation products found in diesel fuel are of the same order as the detection limits of the applied method.

The peroxide value can increase during the time the oil or the ethyl esters are stored, but also if the preparation of the biofuels includes distillation of the final product or not. In this case the biofuels were prepared simultaneously and without distillation. The samples were tested one month after their preparation. The obtained *PV* for the *Jatropha* oil samples are higher than the reported values by Sharma (4.26 ± 0.12) [21], which can be influenced by the storage time.

It has been reported that the oxidation level of the FA has an impact on the cetane number since peroxide compounds have been proposed as cetane improvers [34, 35]. The presence of peroxides, as a consequence of the oxidation process, may result in lower THC emissions, as also reported in [11].

The results of the p-anisidine determination are shown in Table 4. Again, important differences between the samples from different regions are observed. The secondary oxidation products are also higher for the Guantanamo *Jatropha* oil and its derived FAEE. This result indicates more secondary oxidation processes for the fuels obtained from the Guantanamo plantations according to the fact that all the biofuels tested were prepared simultaneously.

3.2 Composition of the oils and derived FAEE

The results of the GC-MS applied to the FAEE samples are shown in Fig.1. The FAEE peaks correspond to ethyl palmitate (16:0), ethyl linoleate (18:2), ethyl oleate (18:1) and ethyl stearate (18:0). The chromatogram A represents the result for FAEE from Guantanamo while chromatogram B represents that obtained for the FAEE from San José. The chromatogram C represents the peaks distribution obtained for a FAEE standard injected into the system for comparison with the sample peaks for qualitative and quantitative analysis of the biodiesels. The GC identification of peaks was possible by applying the GC method to standard mixtures of FAEE and FAME. The retention times of the peaks for the standard mixtures used are 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 NO_x 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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401 **Figure and Table captions**

402

403 Table 1. Physical properties of Jatropha Curcas oil

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405 Table 2. Acid values for the different samples

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407 Table 3. Peroxide values for the different samples

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409 Table 4. P-anisidine values for the different samples

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

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413 Fig.1 Gas Chromatograms for the samples A: Guantanamo FAEE, B: San José FAEE, C:
414 FAEE standard

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416 Fig.2 Chromatograms obtained for FAEE and FAME standard mixtures

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Table 1. Physical properties of Jatropha Curcas oil

Physical properties	Value
density	901 $g \cdot m^{-3}$
melting point	5 °C
kinematic viscosity (at 40°C)	34.6·10 ⁻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	<i>meq.O₂·kg⁻¹</i>	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