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Thermal behavior of *Jatropha curcas* oils and their derived fatty acid ethyl esters as potential feedstocks for energy production in Cuba

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Abstract The thermal behavior and oxidative stability of *Jatropha curcas* oils and their derived fatty acid ethyl esters (FAEE) obtained from two different Cuban regions and varieties are evaluated. A decrease in acid value is found for the FAEE compared to the oils from both natural sources. Primary and secondary oxidation products are significantly different among samples. Thermogravimetry and the derivate thermogravimetry were applied to the oils and FAEE samples using air and nitrogen atmospheres. The main differences between oil and FAEE derived from Jatropha oil are the number of decomposition steps that are lower in FAEE samples. Concerning the onset temperature in the mass loss process, no pattern is observed when oils and the derived FAEE are compared. A comparison among biofuels obtained from the different regions shows that the biofuels obtained from one of both plantations are less thermally stable and they also have more oxidation activity. The obtained results can influence the engine performance and exhaust emissions when these biofuels are used as fuels.

Keywords Jatropha · FAEE · Thermal analysis · Oil's decomposition

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Introduction

The study 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.

The potential of biomass helping to meet the world energy demand has been widely recognized [1, 2]. At the time of soaring prices and limited supply of fossil fuels, the use of biomass as a renewable energy source is especially important for most developing countries as their economies are based largely on agricultural and forestry sectors.

Jatropha curcas (JC), native of tropical America, has been later introduced into Africa and Asia and is now cultivated worldwide [3]. 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 [4–8], by preheating of the oil, blending with fossil diesel fuel, or converting them 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 [5].

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 [9], even at low concentration, although there are some species of Jatropha that produce edible oils [10].

Thermal analysis characterizes some physical and chemical properties of substances depending on the temperature at a defined heating rate, or on the time at a constant temperature. These techniques are settled to continuously monitoring of physical or chemical changes of a sample which occur as the temperature of a sample is increased or decreased. Thermogravimetry (TG), Differential thermogravimetry (DTG), and differential scanning calorimetry are the principal thermoanalytical methods [11]. The DTG curve is defined as the derivative of the curve from TG, in which the sample mass loss is proportional to the area under the DTG curve. Using TG, extensive research has been performed on fossil fuels, particularly on combustion kinetics of crude oils [11, 12]. These techniques are used to determine fuel thermal decomposition and combustion characteristics [11, 13, 14]. These techniques also have been used to study oils and fats obtaining a relationship between fatty acid composition and the thermal properties of the fat [15]. TG–DTG curves are used to obtain information on the temperature-controlled combustion of the biofuels.

The oxidative stability evaluated by thermal analysis can be used for evaluating the quality of oils, fats, and biodiesels, and to explain some phenomenon that take place in the engines when these biofuels are used to produce energy. Such property depends on chemical composition, the quality of the raw material, the conditions of refining processes (oils and fats), the transesterification route (biodiesel), and the storage conditions.

The transesterification reaction of oil or fats with short chain alcohols, usually methanol and ethanol, leads to a mixture of corresponding mono-alkyl esters, which are defined as biodiesel. As biodiesel presents the same fatty acids composition of its initial oils or fat, characterized by a considerable amount of unsaturated fatty acids, its oxidative stability is a crucial property, especially during long-time period storage. The thermal stability of the oils is important in defining the operability conditions for lubricants prepared using such vegetable oils [16]. Thermal analysis of biodiesel is reported in [14, 17, 18], and the thermal stability of Jatropha oil is reported using thermal analysis [17, 19].

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 [2, 20, 21]. 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 [22], the biofuels tested in this paper will be named as derived FAEE, as ethanol was used in the chemical synthesis.

The purpose of this study is to evaluate and compare the thermal behavior and oxidative stability of two different species and plantations of *Jatropha curcas* oils and their derived FAEE, obtained by ethanol route.

Experimental procedure

Samples tested

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 (Gt) 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 (SJ) belongs to an African variety. Both samples were tested as oils and as 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 hand operated press. The liquid samples prepared (oils and derived FAEE) were stored in glass vials in nitrogen atmosphere. The biofuels were prepared simultaneously and the samples were tested 1 month after their preparation.

Degradation of the oils and the derived FAEE is very important to understand some changes or phenomenon that can be presented in these biofuels concerning their thermal behavior or the further engines performance when these biofuels are used. Therefore, the thermal analysis is in this research complemented with the degradation analysis of the tested biofuels.

Degradation parameters

Acid value

The acid value (*AV*) 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, it is possible to calculate the amount of acids in oil. The acid value is expressed as mg KOH g^{-1} of oil [23–25].

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 L^{-1} in ethanol. The neutralization solution was NaOH 0.01 mol L^{-1}. The determination of the acid value is based on [25].

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, and 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 (*PV*). 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 [26]. Each experiment was repeated three times. The determination of the peroxide value (*PV*) is based on [26].

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 biodiesel. The secondary oxidation products are reacted with *p*-anisidine, resulting in the production of a colored compound which is assessed spectrophotometrically.

The experiments were set using isooctane as an optically clear dissolution and a solution of 0.25 g of *p*-anisidine in 100 mL glacial acetic acid. The solution was kept refrigerated (4 °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 [27] using a pair of identical quartz cells. Each experiment was repeated three times. The determination of the *p-AV* is based on [27].

Characterization of thermal degradation processes

In this research, the thermal degradation characteristics of *Jatropha curcas* were studied using a thermogravimetric analyzer: Mettler Toledo: TGA-SDTA 851E. Thermogravimetric analysis provides a measurement of weight loss of the sample as a function of temperature and time.

The apparatus consists of a microbalance within a furnace, allowing the weight of the sample to be continuously monitored while the temperature is controlled. Samples can be run either in a dynamic (temperature ramp) or in an isothermal mode. Once these data are collected, analysis of the raw numbers can lead to the kinetic rate parameters (order of reaction, activation energy, and the pre-exponential factor).

In this study, experiments were carried out in two different atmospheres using an inert gas (N_2) with flow rate of 100 mL min^{-1} and using an oxidative flow (O_2) at 100 mL min^{-1}. Samples taken were 120–200 mg for each run. Dynamic trials were performed at a constant heating rate of 5 °C min^{-1} with a temperature programming of: 25–400 at 5 °C min^{-1} and 400 °C during 5 min. Prior to each experiment, the mass and temperature modules of the analyzer were calibrated to obtain reliable and reproducible mass and temperature data. Mass losses that correspond to temperature change were continuously recorded with a data acquisition system working in coordination with the furnace and control unit of the analyzer. Variation of sample mass with respect to temperature change (TG data) and the first derivative (DTG data) were continuously collected.

Results and discussion

Degradation analysis

The results of the acid value determination for the samples are shown in Table 1. A decrease in acid value is found for the FAEE samples compared to the oils from both sources, related to a decrease of the FFA present in FAEE compared

Table 1 Acid, peroxide, and *p*-anisidine values for the different samples

Sample	AV/KOH g^{-1}	PV/meq. O_2 kg^{-1}	p-AV
JC oil SJ	0.7	5.9	0.6
JC oil Gt	1.7	20.2	1.6
FAEE SJ	0.5	5.3	1.5
FAEE Gt	1.0	14.7	1.2

to the original oil. The comparison between Jatropha oil samples shows that the *AV* of Guantanamo oil is much higher compared to the *AV* of the oil from San José.

The difference in *AV* for FAEE samples is related to the differences found in their oil sources. The *AV* obtained for biodiesel is typically less than 1 mg KOH g^{-1} of oil [1, 23]. Berchmans [28] 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 and 38 mg KOH g^{-1} [29]. The wide range found in literature is because these values depend on the biofuels' degradation stage and on the natural source.

An increase in *AV* can also be observed with the increase in fuel's storage time due to the natural biodegradability of the oil or the derived FAEE. The degraded fuel may contain higher volatility components that accelerate decomposition [28]. The more volatile the degradation products, the lower the onset temperature will be for the combustion reactions, influencing the combustion process in an engine. In this case, the fuels' storage time was similar for all samples, so we can conclude that 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 also shown in Table 1. The *PV* is reported in milliequivalents of oxygen per kilogram of oil (meq. O_2 kg^{-1}). Significant differences can be observed for the samples from different species and regions. The *PV* can also be increased during the time the oil or the ethyl esters are stored. The obtained *PV* for Jatropha oils samples is higher than the reported values by Sharma (4.26 ± 0.12) [16].

The results of the *p*-anisidine determination are also shown in Table 1. Again, important differences between the samples from different species and 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 Guantanamo (Cuban variety) according to the fact that all the biofuels tested were prepared simultaneously.

Thermal analysis

The TG–DTG data of Jatropha oil from Guantanamo using air and nitrogen at a constant heating rate are plotted against furnace temperature in Figs. 1 and 2, respectively. Significant differences between both thermal profiles are observed. The thermal decomposition processes using air start earlier due to the air atmosphere enhancing the thermal decomposition processes presented in the fuel. The process of decomposition involves the absorption of oxygen as well as the liberation of volatile compounds. The same number of decomposition steps are observed in both atmospheres but settled at higher temperatures when nitrogen is used due to a reduction of the reactivity in inert atmosphere.

The maximum peak of the DTG curve shown in Fig. 1 is attributed to the decomposition of the unsaturated molecules and lighter fractions, as oleic and linoleic acid and the other two peaks correspond to the decomposition of the saturated molecules and the heavier components, such as palmitic and stearic acids, and also include the carbonization process of residual products.

As is observed in Fig. 2, there are also three decomposition steps for Jatropha oil Gt using nitrogen, but some variations in the relative peak areas are observed.

The results exposed above can also be observed in Table 2, next to other experimental TG data of all the samples in both atmospheres. The most important parameters of the TG analysis are the initial decomposition temperature, the maximum temperature of conversion, and the final decomposition temperature.

The TG–DTG curves for Jatropha oil SJ using different atmospheres are shown in Figs. 3 and 4. Only one decomposition step is observed in both atmospheres. Less decomposition steps are present compared to Jatropha oil Gt due to more active oxidation processes present in Jatropha oil Gt as was shown in Table 1. Therefore, a

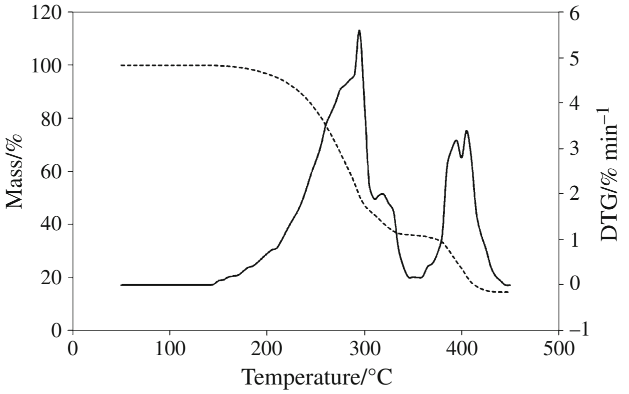

Fig. 1 TG–DTG curves for *Jatropha curcas* oil Gt in air (*dashed line* TG, *solid line* DTG)

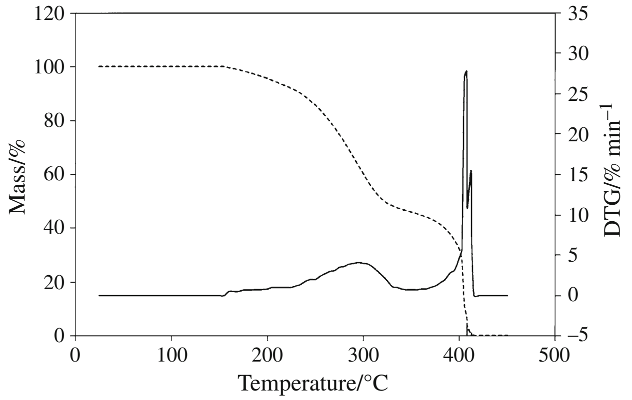

Fig. 2 TG–DTG curves for *Jatropha curcas* oil Gt in nitrogen (*dashed line* TG, *solid line* DTG)

relationship between degradation parameters and the thermal profiles is observed. Degradation of the oil may bring amounts of higher volatility components and therefore creates important variations in the thermal profile (more peaks). Oxidative degradation can affect some properties of the fuel such as kinematic viscosity, cetane number, and acid value. The oxidation degradability is clearly different comparing the Jatropha oil produced in both regions as is observed in Table 1 and in the TG–DTG curves obtained, that can be influenced by the difference in soil characteristics, climate conditions and the different genetic Jatropha varieties tested.

The thermal profiles of the FAEE derived from *Jatropha curcas* oil Gt are shown in Figs. 5 and 6. The main difference between oil and the FAEE is concerning the

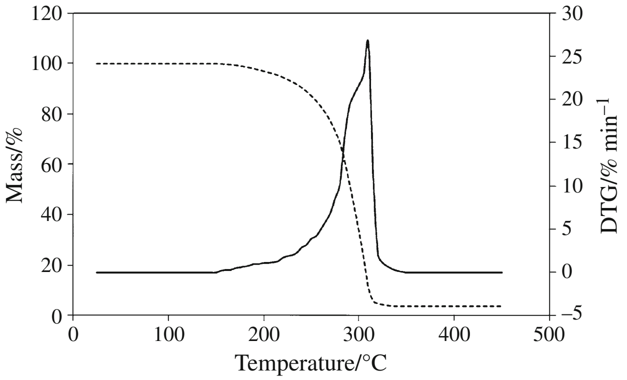

Fig. 3 TG–DTG curves for *Jatropha curcas* oil SJ in air (*dashed line* TG, *solid line* DTG)

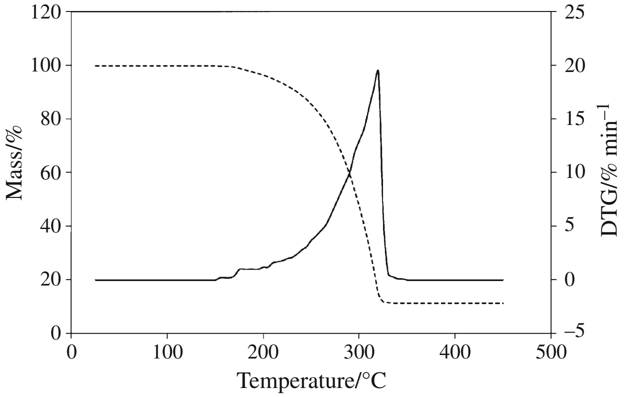

Fig. 4 TG–DTG curves for *Jatropha curcas* oil SJ in nitrogen (*dashed line* TG, *solid line* DTG)

Table 2 Main TG data of the samples in different atmospheres

Sample	Atmosphere	Step	Temperature range/°C	Peak temperature/°C	Offset temperature/°C	Δmass/%
JC oil Gt	Air	First	140–345	295	445	63.9
		Second	355–400	395		10.4
		Third	400–445	405		11.1
JC oil Gt	N_2	First	215–340	290	440	43.5
		Second	360–408	406		41.8
		Third	408–415	408		14.5
JC oil SJ	Air	First	175–335	310	335	96.1
JC oil SJ	N_2	First	175–330	320	335	87.9
FAEE Gt	Air	First	140–350	300	450	89.9
		Second	370–440	400		10.9
FAEE Gt	N_2	First	205–345	295	450	56.9
		Second	370–420	405		41.3
FAEE SJ	Air	First	210–320	300	440	81.0
		Second	390–410	400		19.0
FAEE SJ	N_2	First	240–380	365	440	54.2
		Second	395–420	400		39.1

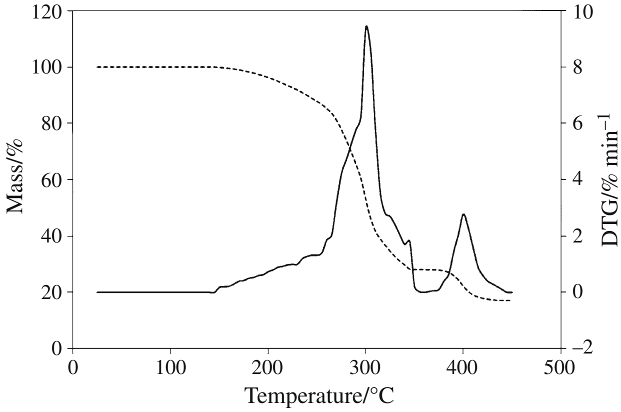

Fig. 5 TG–DTG curves for FAEE Gt in air (*dashed line* TG, *solid line* DTG)

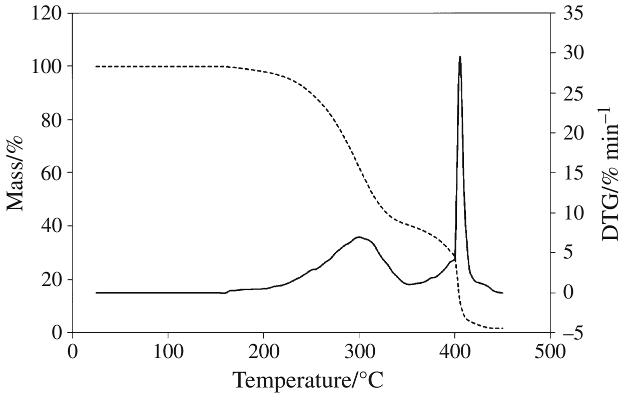

Fig. 6 TG–DTG curves for FAEE Gt in nitrogen (*dashed line* TG, *solid line* DTG)

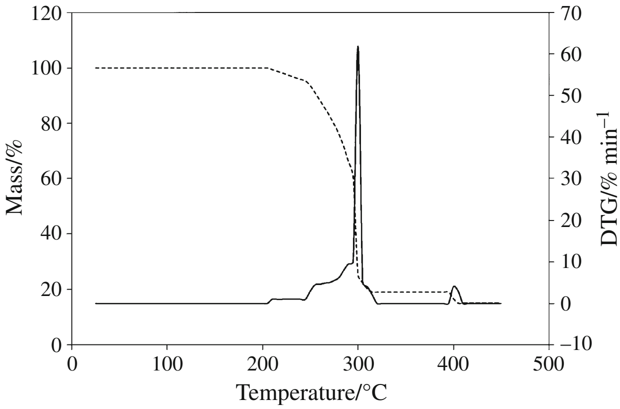

Fig. 7 TG–DTG curves for FAEE SJ in air (*dashed line* TG, *solid line* DTG)

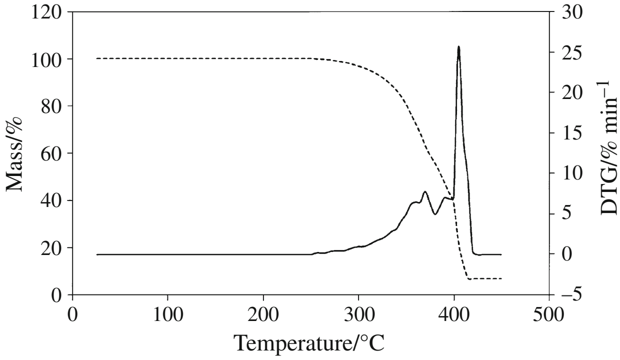

Fig. 8 TG–DTG curves for FAEE SJ in nitrogen (*dashed line* TG, *solid line* DTG)

number of decomposition steps that are lower in FAEE from Gt in both atmospheres. Both decomposition steps observed in FAEE from Gt are due to events of thermal decomposition assigned to the volatilization and combustion of the components present in the derived ethyl esters. The first one corresponds to the unsaturated ethyl esters and the second corresponds to the saturated ethyl esters.

The thermal profiles of the FAEE derived from *Jatropha curcas* oil SJ are shown in Figs. 7 and 8. Compared to the thermal decomposition profile of the Jatropha oil SJ, a more active decomposition process for FAEE (two decomposition steps) is observed. This can be explained due to the physical chemical differences between the derived FAEE and the oil source. Both FAEE samples have two decomposition steps assigned to the volatilization and combustion of the chemical components present in the derived ethyl esters.

A comparison of the onset temperatures for FAEE samples shows that the onset temperature of Jatropha oil Gt in air atmosphere is lower than the SJ sample due to a higher degradation activity in the Gt oil source, and the derived fuel. That was also shown and remarked in Table 1.

The thermal behavior observed for *Jatropha curcas* oil samples from Guantanamo shows three mass loss steps, which is in accordance with a similar report on Brazilian crops [17]. There are reports [18, 30] observing three steps in the thermal profile of oils and less steps for its derived FAME as is observed in this article for *Jatropha curcas* FAEE from Gt, but not in the SJ samples.

In the case of the thermal behavior of *Jatropha curcas* FAEE, the two mass loss steps observed are in good agreement with a study reported for *Jatropha curcas* from Brazil [17]. In this study, we found the same result for Jatropha oil Gt but only one step for Jatropha oil SJ due to a clear difference in the oxidation stability of these biofuels that is evidence of the strong relationship between the degradation parameters evaluated in this research with the thermogravimetric behavior of the fuels tested.

Concerning the onset temperature, the differences found in the thermal behavior comparing the oil and the FAEE show a lower onset temperature for the FAEE from Gt compared to the oil, but an opposite behavior for the SJ samples. This could be due to the presence of water traces remaining in the raw seeds, later eliminated in the FAEE chemical synthesis. As consequence, secondary hydrolysis has probably occurred to the oil from SJ, yielding to an unusual decrease in the onset temperature and oil thermal stability compared to its FAEE.

A comparison among biofuels obtained from different regions (species) shows that the biofuels obtained from the Guantanamo plantation are less thermally stable (corresponding to the African variety) and they also have more oxidation activity. A relationship between the thermal stability, volatility characteristics and the ignition quality of a fuel has been reported [13, 31]. Thus, the obtained results indicate that the engine performance could be influenced when these biofuels will be used in engines.

The thermal analysis is very useful in the case of oils that will be used for energy production in diesel engines, due to the fact that the vegetable oils should be warmed up before their use into the engine. The temperature increase of the oil should assure adequate levels of oil viscosity, but without the degradation of the oil. In this study, the stability of the Jatropha oil samples as a function of temperature was clearly obtained from their thermal profiles. Supported by the results shown in Table 1 and the thermal profiles, it is possible to conclude that the JC oil from Gt is more active and less stable concerning thermal stability and oxidation processes and therefore its quality is lower compared to the oil obtained from the other source. The onset temperature value mentioned here was selected taking into account all the results obtained. Fixing this warning value (140 °C) as the maximum allowed preheating temperature we can assure that thermal degradation will not occurs if a preheating system for the oils is made before their use in diesel engines. In the case of the oil, a heating above 140 °C will degrade the oil, starting the thermal conversion. Differences in molecular structures influence the physical and chemical processes occurring during the atomization, vaporization and combustion of the fuel after it is injected into the combustion chamber. These differences lead to distinctive patterns in fuel energy release rates and pollutant formation from various molecules [32]. The change in the molecular structure influences the cetane number [13], and the cetane number influences the ignition delay [33, 34]. Concerning the derived FAEE the onset temperature has no real importance because there is no pre-heating due to the FAEE viscosities are closer to the neat diesel fuel. As is observed in Table 1, the lower onset temperature (140 °C) obtained for Jatropha oils suggest the maximum allowed pre-heating temperature. The change in composition above 140 °C can influence the physical chemical fuel characteristics and the engines performance.

In further work, these biofuels will be evaluated in engine bench tests and the relationship between TG and the fuels' combustion parameters, such as ignition delay, will be checked.

Conclusions

The *Jatropha curcas* oil and FAEE oxidation stability obtained from different regions of Cuba and different genetic varieties showed significant differences. The degradation activity for the oil and the FAEE obtained from Guantanamo plantations is higher than for the San José. Jatropha oil and their derived FAEE from different plantations were thermally stable until 140 °C. The volatility of the FAEE, indicated by the onset decomposition temperature and the number of decomposition steps, showed that a different combustion process should be expected compared to their oils. Concerning the onset temperature, no pattern is observed when oils and the derived FAEE are compared. The number of thermal decomposition steps in *Jatropha curcas* fuels was not influenced by the applied atmosphere.

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