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Pyrolysis Oil from the Fruit and Cake of *Jatropha curcas* Produced Using a Low Temperature Conversion (LTC) Process: Analysis of a Pyrolysis Oil-Diesel Blend

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Abstract

Background: the LTC process is a technique that consists of heating solid residues at a temperature of 380˚C - 420˚C in an inert atmosphere and their products are evaluated individually: these products include pyrolysis oil, pyrolytic char, gas and water. The objective of this study was to compare the effects of the use of oils obtained by pyrolysis of *Jatropha curcas* as an additive for diesel in different proportions. Results: a Low Temperature Conversion (LTC) process carried out on samples of *Jatropha curcas* fruit and generated pyrolysis oil, pyrolyic char, gas and aqueous fractions in relative amounts of 23%, 37%, 16% and 14% [w/w] respectively for *Jatropha curcas* fruit and 19%, 47%, 12% and 22% [w/w] respectively for *Jatropha curcas* cake. The oil fractions were analyzed by FTIR, ^{1}H NMR, ^{13}C NMR, GCMS and physicochemical analysis. The pyrolysis oil was added to final concentrations of 2%, 5%, 10% and 20% [w/w] to commercial diesel fuel. The density, viscosity, sulfur content and flash point of the mixtures were determined. Conclusions: the results indicated that the addition of the pyrolysis oil maintained the mixtures within the standards of the diesel directive, National Petroleum Agency (ANP nº 15, of 19/7/2006), with the exception of the viscosity of the mixtures containing 20% pyrolysis oil.

Keywords: *Jatropha curcas*, Low Temperature Conversion, Fuel and Pyrolysis Oil

1. Introduction

The LTC process was developed and refined over many years by Romeiro and colleagues [1-5]. This technique consists of heating the solid residue at a temperature of 380˚C in an inert atmosphere. The LTC products are evaluated individually: these products include pyrolysis oil, pyrolytic char, gas and water. Conversion at a low temperature can be seen as an alternative technology for power generation and can be part of strategy for environmental conservation through the reuse of waste because other waste processing methods such as incineration or landfilling are criticized for causing environmental damage.

The pyrolysis process has been developed and the technique has been modified with respect to residence time in the pyrolysis reactor, [6] heating rate and temperature according to the required analysis, **Table 1** [7,8].

Several parameters of Low Temperature Conversion developed by Bayer and co-works [9-12] can be varied including residence time (1.5 - 2.0 hours), heating race (medium), temperature (380˚C - 400˚C) and products (oil, char, gas and water).

The LTC process involves only thermal decomposition and does not involve the use of solvents or chemical reagents. Other methods to produce alternative fuels are more sophisticated with respect to the instruments required. The LTC process produces pyrolysis oil that is constituted of oxygenated compounds among others. These oxygenated compounds increase the lubricity of the diesel oil like as observed in the case of B2 and B5 when fossil fuels (diesel) and methyl fatty esters (biodiesel) are mixed.

Various biomasses sources can, in principle, be used to produce biodiesel or used as diesel additives [13-21].

Table 1. Pyrolysis methods and their variations.

Pyrolysis technology	Residence time	Heating rate	Temperature (˚C)	Products
Carbonization	days	very low	400	charcoal
Conventional	5 - 30 min	low	600	oil, gas, char
Fast	0.5 - 5 s	very high	650	bio-oil
Flash liquid	<1 s	high	<650	bio-oil
Flash gas	<1 s	high	<650	chemicals, gas
Ultra	<0.5 s	very high	1000	chemicals, gas
Vacuum	2 - 30 s	medium	400	bio-oil
Hydro-pyrolysis	<10 s	high	<500	bio-oil
Metano-pyrolysis	<10 s	high	>700	chemicals

The choice of material to use in a particular region or country depends on various factors; however, the availability of the plant in that region is usually of major importance. Clearly, the components of the plant matrix or extract used must also be compatible with diesel oil and must be suitable for use in diesel engines. The property that best demonstrates this compatibility is the cetane number and additional properties such as density, sulfur content and flash point are important in determining the adequacy of biofuels as alternative fuels.

Recently we described the production of pyrolysis oil from *Castor seeds* via a Low Temperature Conversion (LTC) process and showed that the use of this oil in a pyrolysis oil-diesel blend is a good option [22].

The *Jatropha curcas* plant is a Euphorbia that is being introduced in Brazil for the purpose of biodiesel production and income generation for people of small farmers. The use of the cake the co-product produced during the extraction of oil, has so far been limited due to the toxicity of the seed caused by the presence of curcin and diterpene esters [23].

The objective of this study was to compare the effects of the use of oils obtained by pyrolysis of *Jatropha curcas* as an additive for diesel in different proportions. The results of this study show that the pyrolysis oil can be used as an additive and that one of the two raw materials would be better used as an additive.

2. Methods

2.1. Low Temperature Conversion (LTC) Process

Samples of *Jatropha curcas* fruit or cake were subjected to a Low Temperature Conversion process at 380˚C. Each experiment was repeated seven times using 400 g of material each time and the results for the seven replicates were averaged. Each sample was placed in the central region of a cylindrical glass tube, which was then introduced into a reactor coupled to a condensing system. Nitrogen gas was continuously applied, at 500 mL/min, before the start and during the course of the process. After 10 minutes of gas purging, heating was initiated at a rate of 10˚C/min and the temperature was then maintained at 380˚C for 2 hours. After passing through the condenser the condensable gas, pyrolysis oil and water fractions, were collected in a graduated tube and were separated based on density. The pyrolytic char was retained in the middle of the reactor and was collected after cooling. The non-condensed gas was passed through three traps containing, successively, NaOH 10% (w/v), $NaHCO_3$ 10% (w/v) and HCl 10% (w/v) solutions. Parameters such as temperature, time of reaction and nitrogen flow were established prior to this work by Romeiro and co-workers, these values were determined to be the best due to the higher yield of oil, the shorter time required for total conversion of the raw material and the smaller amount of energy used.

2.2. Apparatus Description

The conversions were carried out in a Heraus R/O 100 batch scale instrument (**Figure 1**)—laboratory scale, consisting: 1) oven; 2) dried sample; 3) glass wool; 4) electric resistance; 5) gas N_2 inlet; 6) condenser; 7 and 8) separator funnel; 9) gas washing system [22].

2.3. Spectroscopy Analysis

2.3.1. Infrared Spectroscopy—FTIR

The FTIR spectra (400 to 4000 cm^{-1}) were obtained using a Model 1420 Perkin-Elmer Spectrometer using an

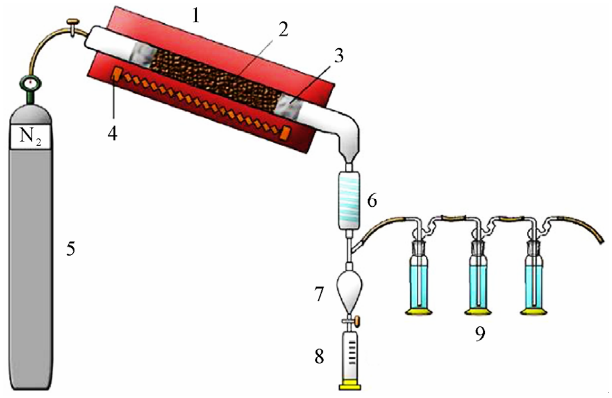

Figure 1. Batch mode equipment of LTC process—laboratory scale.

NaCl window and polystyrene for calibration.

2.3.2. Nuclear Magnetic Resonance—NMR

^{1}H NMR and ^{13}C NMR spectra were recorded using a Varian-Unity plus 300 (300 MHz), in $CDCl_3$ using TMS as the internal standard.

2.3.3. Gas Chromatography Mass Spectrometry Analysis—GCMS

The pyrolysis oil was initially separated chromatographically on a silica gel column using hexane, dichloromethane and methanol as successive mobile phases. Separate fractions were then submitted for GCMS analysis. The conditions used in the GC part of the GCMS set-up were as follows: column: 25 m × 0.2 mm × 0.33 µm, based in the method used for diesel; heating: 5˚C/min from 60˚C to 300˚C and then 300˚C for 20 minutes; He flow rate of 0.6 mL; temperatures: t detector 250˚C, injector 280˚C and interface 300˚C. Samples of 1 - 3 µL were prepared in the reason of 1:100.

2.4. Blend Analysis: Pyrolysis Oil-Diesel

The pyrolysis oil from the LTC process was obtained as described above and the diesel was acquired from a local filling station. Analyses of the mixtures, which contained 2%, 5%, 10% or 20%. pyrolysis oil in commercial diesel, [referred to as PD2, PD5, PD10 and PD20, respectively] was carried out at the Fuel Laboratory of the School of Chemistry of UFRJ (LABCOM—Rio de Janeiro, Brazil). This analysis followed the standards of resolution ANP nº 15, of 19/7/2006. The following quantities were measured: total sulfur content (ASTM D 4294), density at 15˚C and 20˚C (ASTM D 4052), flash point (ASTM D 56) and viscosity at 40˚C (ASTM D 445).

2.4.1. Sulfur Content (ASTM D-4294)

The amount of sulfur the sample was determined according to ASTM standard D 4294/02, using the Sulfur Meter Tanak Scientific device, model RX-3505. The samples were exposed to emitted x-rays and the results were compared with previously prepared calibration standards. The sulfur concentration in the sample was then calculated from the calibration curve generated in the assay (repetitively, $r = 0.02894$ ($X \pm 0.1691$) and reproducibility, $R = 0.1215$ ($X \pm 0.05555$). X is the sulfur concentration in percentage mass.

2.4.2. Density at 20˚C (ASTM D 4052) and Density at 15˚C (ASTM D 4052)

The samples were brought into thermal equilibrium in a thermal bath kept at 15˚C or 20˚C according to ASTM standard D-4052. The densities were determined at each of these temperatures. An Anton Paar, electronic aerometer, model DMA 35N, was used to determine the density at each temperature. This experiment was performed in triplicate for each sample.

2.4.3. Flash Point (ASTM D 6450)

For the determination of the flash point of the samples ASTM standard D 6450 was used. A Grabner Instruments miniflash FLPH was used, this instrument can be used to analyze the liquid products of oils that are used in the range of 40˚C - 360˚C. The samples were assayed separately in a system that contained the sample in an airtight bronze cup, which was used for the constant agitation of the sample. An ignition source was directed at the cup containing the sample at regular intervals during the agitation process; this was continued until a specific amount of vapor was formed and caught fire as a result of contact with the ignition source. At the time of ignition, the system detected and recorded the imposed tax of heating and the tax of agitation, as well as the flash point of the sample. (accuracy, $r = \pm$ 1.9˚C and reproducibility, $R = \pm$ 3.1˚C)

2.4.4. Viscosity at 40˚C (ASTM D 445)

The viscosity of each sample was determined using an Ubbelohde-certified Herzog model HVB-438 viscometer in a thermal bath, in accordance with ASTM standard D-445. The time required for the oil to drain out of the viscometer was determined and the viscosity was calculated using the equation $\eta = t$. C, where t = the draining time in seconds and C = a constant inherent to the dimensions of the specific viscometer. Each experiment was performed in triplicate.

3. Results and Discussion

The LTC of the *Jatropha curcas* fruit and cake was carried out under a constant nitrogen flow at 380˚C, as indicated in the experimental section. This process resulted in the production of four fractions: pyrolysis oil [23%], an aqueous fraction [12%], pyrolytic char [41%] and gas [24%] for *Jatropha curcas* fruit and pyrolysis oil [19%], an aqueous fraction [12%], pyrolytic char [47%] and gas [22%] for *Jatropha curcas* cake.

3.1. Spectroscopy Analyses of the Crude Oil Fraction

Significant absorptions in the FTIR spectra of the crude pyrolysis oil, were found at 3418 cm^{-1} (axial deformation of OH); 2920 cm^{-1} and 2854 cm^{-1} (axial deformation of C-C aliphatic); 1700 cm^{-1} (carbonyl group) and 1463 cm^{-1} (angular deformation of CH_3 and CH_2) for *Jatropha curcas* fruit and 3407 cm^{-1} (axial deformation of OH); 2923 cm^{-1} and 2873 cm^{-1} (axial deformation of C-C aliphatic); 1737 cm^{-1} (carbonyl group) and 1466 cm^{-1} (angular deformation of CH_3 and CH_2) for *Jatropha curcas* cake. The absorptions observed by FTIR show that the mixture contained hydrocarbons and carbonyl compounds in both cases and that there were probably hydrogen bonds are present.

In order to compare the ^{1}H NMR data of the comercial diesel and the crude pyrolysis croud oil, we considered the "Total aromatic" (δ 6.3 - 9.3 ppm) and "Total aliphatic" (δ 0.5 - 4.5 ppm) regions in the ^{1}H NMR spectra. The high number of aliphatic hydrogens in both spectra demonstrated the presence of sp^3 carbons. The most important difference was the presence of olefinic compounds in the pyrolysis oil from *Jatropha curcas* fruit and cake.

The oils were then chromatographically separated using, hexane, dichloromethane and methanol as mobile phases. The fraction obtained with hexane (the apolar fraction) was analyzed by GCMS (retention times and *m/z* peaks) and 44 hydrocarbons were observed: 14 alkanes from C_9 to C_{30}, 22 alkenes from C_9 to C_{20}, 5 alkyl-aromatic compound and 3 alkyl-aliphatic componds, **Table 2**. In the polar fractions, which were eluted with dichloromethane and methanol, **Table 3**, it was observed 8 acids (66%), 1 ester, 1 aldehyde and 1 ketone.

In order to try to elucidate how these changes occured at 380˚C we compared the composition of the *Jatropha curcas* sample with the compounds obtained in the chromatographic analysis. The composition of the sample before was as follows:

- 37% triglycerides, the identity of which depended on the fatty acid structure and distribution: palmitic—C16:0; palmitoleic—C16:1; estearic—C18:0; oleic—C18:1; linoleic—C18:2 and eicosanoic—C20:0 acids; and

Table 2. Main compounds detected in the hexane fraction by GCMS of the *Jatropha curcas* fruit and cake.

Compounds	RT	Quality	*m/z*
1-nonene	3.075	90	126
nonane	3.194	93	128
1-decene	4.773	95	140
decane	4.950	97	142
(Z)-2-decene	5.054	94	140
(E)-2-decene	5.231	93	140
butylbenzene	6.196	97	134
1-undecene	7.025	97	154
undecane	7.233	97	156
(Z)-2-undecene	7.350	94	154
(E)-2-undecene	7.549	92	154
pentylbenzene	8.667	95	148
isobutyltoluene	8.875	90	148
1-dodecene	9.583	95	196
dodecane	9.808	97	170
(Z)-2-dodecene	9.926	96	168
(E)-2-dodecene	10.149	93	168
hexylbenzene	11.353	93	162
1-tridecene	12.225	96	224
tridecane	12.460	93	196
(Z)-2-tridecene	12.559	93	196
heptylbenzene	14.057	90	176
1-tetradecene	14.825	96	252
tetradecane	15.042	98	198
(Z)-2-tetradecene	15.156	91	198
undecylcyclopentane	16.233	94	224
1-pentadecene	17.342	97	210
pentadecane	17.558	98	212
n-pentadecylcyclohexane	18.767	91	294
cyclohexadecane	19.400	94	224
(Z)-8-hexadecene	19.542	93	224
1-hexadecene	19.733	96	210
hexadecane	19.925	96	240
(Z)-2-hexadecene	20.017	94	224
8-heptadecene	21.667	96	238
1-heptadecene	22.028	96	238
heptadecane	22.200	96	240
1-octadecene	24.191	92	252
octadecane	24.333	96	254
nonadecene	26.381	92	268
eicosane	28.344	93	282
heneicosane	30.224	94	296
docosane	32.019	93	310
tricosane	33.790	92	324

Table 3. GC/MS analysis of the pyrolysis oil, polar fraction, obtained by LTC process.

Compounds	RT (min)	Quality	***m/z*** (other fragments)
Tridecane	16.84	81	184 (43, 57, 71, 141)
Heptilbenzene	18.06	91	176 (43, 77, 91, 105)
Tetradecane	18.60	87	198 (43, 57, 71, 85, 99)
Heptadecane	22.76	96	240 (57, 71, 85, 99, 141)
Methyl tridecanoate	25.36	94	228 (74, 87, 129, 143)
Tetradecanoic acid	26.64	86	228 (60, 73, 129, 185)
9-octadecenoic acid	28.26	83	282 (55, 60, 73, 129)

- 63% ash, protein, fiber and lignin.

When molecules are heated to high enough temperatures, the bonds break and radicals are formed. The normal energy power of the C-C bond is about 90 kcal/mol, and thermal excitation of molecules at a temperature of 450˚C - 650˚C is required to break C-C bonds. For example, this is the temperature region in which thermal cracking of oil occurs. However, some compounds that have exceptionally weak bonds break down to form radicals at lower temperatures and these compounds can be used to initiate radical reactions at temperatures from 50˚C to 150˚C. Thus, comparing the results obtained at 380˚C, by LTC, the GCMS of the hexane fraction after conversion it is clear that at this high temperature, the triglycerides, protein, fiber and lignin may undergo dehydratation (–H_2O), decarboxylation (–CO_2), decarbonylation (–CO), deamination (–NH_3), radical fragmentations (R. or Ar.), recombination reactions and rearrangement reactions, thereby generating different hydrocarbons. In the polar fraction, eluted with dichloromethane and methanol we can imagine the same thermal mechanism accuring.

The *Jatropha curcas* cake obtained results similar to those for the *Jatropha curcas* fruit, this result is unsureprising because the composition is the same. The only difference between the fruit and the cake is that the oil has been extracted from the cake.

The pyrolysis oil obtained in this work was miscible with commercial diesel in all ratios. Based on the large number of articles concerning additives for diesel [20] and in order to investigate the quality of the pyrolysis oil obtained using LTC we blended pyrolysis oil and diesel in different ratios in a manner similar to that previously described by Romeiro for *Castor seeds* [22].

3.2. Characteristics of the Pyrolysis Oil-Diesel (PD) Blend Versus Diesel

The results of the analysis are listed in **Table 4**. All measurements were made using mixtures of diesel as a reference. From the data presented it can be deduced that pyrolysis oil mixed with commercial diesel at concentrations up to 10% pyrolysis oil does not cause significant alterations in the specifications of the commercial diesel. With the exception of the viscosity of the 20% pyrolysis oil mixture, the standards of resolution ANP were met.

Theoil from pyrolysis usually dark, has a strong smell and contains a large amount of particulate matter. These characteristics along with others characteristics such as viscosity, can be improved by mixing pyrolysis oil with

Table 4. Results obtained for the mixtures of the pyrolysis oil and commercial diesel.

Analysis	Sulfur content	Density at 15˚C	Density at 20˚C	Viscosity kinematics	Flash point
Unit	% m/m	g/cm^3	g/cm^3	Mm^2/s	˚C
Specification for diesel			0.820 0.865	2.0 - 5.0	Min. 38
Diesel (reference)	0.042	0.839	0.836	3.59	84.0
*PD2	0.038	0.8468	0.8435	3.45	86.0
*PD5	0.036	0.8483	0.8450	3.78	87.0
*PD10	0.069	0.8502	0.8469	4.14	87.0
*PD20	0.061	0.8528	0.8495	6.23	91.0
**PD2	0.036	0.8446	0.8413	3.74	88.0
**PD5	0.040	0.8465	0.8432	4.04	87.0
**PD10	0.043	0.8503	0.8470	4.54	87.0
**PD20	0.113	0.8579	0.8546	6.48	83.0

PD: pyrolysis oil-diesel blend; *Pyrolysis oil from *Jatropha curcas* fruit; **Pyrolysis oil from *Jatropha curcas* cake.

other fuels. The experiments performed here show that mixtures containing up to 10% pyrolysis oil in diesel can be made without considerably changing in the properties of the fuel.

Pyrolysis oil can be used as a fuel for stationary engines and can be mixed with other fuels, such as diesel or alcohol, to improve your application.

4. Conclusions

Jatropha curcas fruit and cake have been found to be a useful renewable sources of pyrolysis oil. *Jatropha curcas cake* is a byproduct of the production of biodiesel and is not currently being used, the cake is correntely a waste product. It was noted that the results for the materials used were very similar. Binary mixtures of diesel and pyrolysis oil containing up to 10% pyrolysis oil (PD10) were very effective. Binary mixtures containing between 10% and 20% (PD20) were not very effective because the viscosity and sulfur content of these mixtures were above the standards of the ANP resolution. This could be a problem in diesel engines as the fuel lines can get hot and decomposition of the fuel at high temperatures could plug the injectors. Therefore the oil obtained by the LTC needs further study to improve the characteristics of the oil. It is important to stress that no organic solvents, no reagents and very simple assemblies were used in the LTC process. Thus the LTC processing of *Jatropha curcas* is a very promising, environmentally friendly and potentially commercially viable process for producing second-generation fuel.

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