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Experimental evaluation of the transesterification of *Jatropha curcas* oil into biodiesel

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Abstract : In this work the transesterification pathway of *Jatropha curcas* oil for biodiesel production was studied. For the experimental process, *Jatropha* oil was extracted using hexane as a solvent and subsequently refined to be esterified due to the presence of high content of free fatty acids at 50°C and 500 rpm, using 60 % w/w alcohol/oil ratio and 1% w/w H_2SO_4. Finally, the oil was transesterified with different amounts of methanol and NaOH as a catalyst, according to the experimental design. In addition, physicochemical properties for the crude oil and biodiesel such as lipid profile, density, calorific value, kinematic viscosity, cetane number, among others, were determined. Results showed that the highest yield obtained was 95 % at 65°C with methanol/oil molar ratio of 8:1 and a catalyst amount of 0.5 % w/w. Finally, by comparing the property results obtained against data reported in the literature and the ASTM standards for biodiesel, it can be concluded that the biodiesel obtained from *Jatropha curcas* is a viable alternative with potential to reduce the environmental impact and energy dependence caused by fossil fuels.

Keywords : *Jatropha curcas* oil, Biodiesel, Transesterification.

Introduction

Biodiesel is a renewable fuel derived from vegetable oils or fats from animal origin which can be used in whole or in partially to replace the diesel fuel for the ignition engines without requiring a substantial modification thereof[1,2]. Despite biofuels have been fostered as an energy alternative, they present difficulties related to their wide usage due high production costs[3],food competition and monocultures. Therefore, there is a need to evaluate new raw materials that can be grown on land that is not traditionally used for agricultural purposes. In this sense, *Jatropha curcas* has obtained importance due to the high oil content (30 - 40 %) and the potential use that can be given to deforested, eroded and marginal areas[4]for its cultivation. In addition, cultivation of this crop has a positive impact on the soil where it is planted since if it is used as an erosion controller and carbon picker, *Jatropha curcas* crops can be adapted in areas not suitable for food-crops cultivation. Characteristics and benefits of *J. curcas* reflect the need to carry out studies related to biodiesel production process, in order to evaluate and establish the conditions of higher yield, thus presenting an alternative for the biofuels industry.

Materials and Methods

Raw material and sampling

Seeds of *J. curcas*, perennial species of the family *Euforbiaceae*, were obtained from the district of Sincerín - Bolívar (10°8'39''N, 75°16'39''W) located at a distance of 36 km from the city of Cartagena (Colombia). Prior oil extraction, fruits passed through a pulping and peeling processes carried out manually to be dried in an oven at 100 ºC.

Oil extraction and refining

For oil extraction, seeds of *J. curcas* were subjected to a chemical extraction process in a Soxhlet unit using 150 ml of hexane as solvent, for a time of 4 to 6 hours. crudeoil extracted was characterized by methods based on ASTM standards[5], after extraction degumming was carried out, 0.5 %v/v of phosphoric acid (H_3PO_4) was added to oil at 60 °C for 30 minutes. Then, the mixture was cooled at room temperature and washed with hot distilled water to facilitate gums separation by vacuum filtration[6].

Oil esterification and transesterification

Refined oil was subjected to an esterification process using sulfuric acid as catalyst (1 % wt/wt H_2SO_4) and an alcohol/oil ratio of 60 % wt/wt at 50 °C per 1 hour[7], to reduce the FFA concentration in the *J. curcas* oil below 2 %. After the esterification process, oil was subjected to a transesterification process to obtain biodiesel based on a factorial 3^3experimental design, performed varying the alcohol/oil ratio (4: 1, 6: 1 and 8:1) and the amount of catalyst (0.5, 1 and 1.5%), for a total of 9 experiments. Each mixture was stirred at 500 rpm and 65° C for 90 minutes. After this time, the reaction was stopped and mixture was added to aphase separator (1000 mL), where remained at room temperature for 12 hours to ensure separation. The lower phase composed of residual glycerin, methanol and salts was removed, leaving in the funnel the biodiesel phase, which was washed to remove traces of catalyst, glycerol, saponified material and excess alcohol[5]. washed biodiesel was centrifuged and dried at 110 °C for 2 hours; then,it was cooled at room temperature, weighed and stored in amber glass bottles for further analysis[1]. Finally, an ANOVA analysis was performed in order to determine the statistical significance of the effect of parameters in the transesterification stage.

Biodiesel characterization

Once the biodiesel was obtained, properties such as density and kinematic/dynamic viscosity were determined according to procedures established on the ASTM D445 and ASTM D1268. In addition, fatty acids profile was obtained using gas chromatography. Cetane (CN) index was determined from the biodiesel using Equation 1, developed by Bamgboye and Hansen (2008)[8], which is recommended as the most accurate for the prediction of this property according to Manzanares (2011)[9]. In Equation 1, $x_2 =$ % Myristic acid, $x_3 =$ % Palmitic acid, $x_4 =$ % Stearic acid, $x_5 =$ % Palmitoleic acid, $x_6 =$ % Oleic acid, $x_7 =$ % Alpha-linoleic acid and $x_8 =$ % Linoleic acid. Finally, the calorific value (HHV) was calculated using Equation 2 reported by Demirbas (2008)[10]and using the ASTM D240 method, where VS is the kinematic viscosity.

$$CN=61.1+0.088x_2+0.133x_3+0.152x_4-0.101x_5-0.039x_6-0.243x_7-0.395x_8 \quad (1)$$

$$HHV=0.4625\ (VS)+39.45 \quad (2)$$

Results and Discussion

Crude oil characterization

Table 1 presents the results of the fatty acids profile of the crude oil. It is observed that high content of unsaturated fatty acids (77.74 %) which improves the oil performance at the moment to be transformed into biodiesel due to the tolerance to lower temperatures, although it decreases the oxidativestability[11].

Table 1. Fatty acids profile of crude oil obtained from *Jatropha curcas*

Fatty acid	Numerical nomenclature	% weight
Myristic	14:0	0.11
Palmitic	16:0	14.26
Palmitoleic	16:1	0.68
Stearic	18:0	7.89
Oleic	18:1	38.01
Linoleic	18:2	38.82
Linolenic	18:3	0.23
Arachidonic	20:0	0.00
Beenic	22:0	0.00
Others	24:0	0.00
Total Monounsaturated		38.69
Total Polyunsaturated		39.05
Total Unsaturated		77.74
Total Saturated		22.56

Table 2 presents the results obtained for some physicochemical properties of crude *J. curcas* oil compared with values reported in the literature. Although the density value of crude oil at 15 °C is below that reported by other authors and for other sources of raw material such as palm oil (0.945 g/ml), soybean oil(0.920 g/ml) and sunflower oil(0.925 g/ml), it does not present a significant difference[12].Likewise, the kinematic viscosity obtained at 40 °C is similar to values reported in the literature for other raw materials (41.93, 35.71 and 36.18 mm^2/s for palm, soy and moringa peregrine oil, respectively)[13]. Calorific value reached does not present significant difference with the values reported by Salaheldeen et al. (2015)[13], Where obtained similar values for crude palm (39.87 MJ/kg) and soybean (39.59 MJ/kg) oil.

Table 2. Physicochemical properties of crude oil obtained from *J. curcas*

Property	Authors	Lafarge et al. (2012)[14]	Ong et al. (2013)[6]	Mofijur et al. (2012)[15]	Piloto et al. (2011)[16]	Achten et al. (2008)[17]
Density, g/mL	0.8765	0.9140	0.9150	0.918	-	-
Kinematic viscosity, mm^2/s	34.28	33.89	-	35.40	34.60	-
HHV, MJ/kg	39.14	-	38.96	-	39.63	
Saponification index, mg KOH/g	228.69	-	-	-	-	102.9 - 209
Acidity index, mg KOH/g	6.38	11.84	-	-	-	3.71

The saponification index is a parameter directly proportional to the average molecular mass of the fatty acids present in the oil. Therefore, by comparing the value obtained with the ranges found in the literature of 102.9 – 209 mg KOH/g[17]and 182 - 199 mg KOH/g[18], it is observed that it is slightly superior, which has no relevance in the biodiesel production process but in the biodiesel quality, since as the average length of the fatty acid chains increases, it might be obtained a biodiesel with a higher cetane index correlated with minors emissions of NO[19]. On the other hand, the acid index is slightly different to the reported by Achten et al. (2008)[17]and Lafargue et al. (2012)[14]. This value indicates that the oil obtained does not present a high amount of FFA, and therefore, it will have a lower tendency towards the formation of soaps during transesterification[20]. However, prior to carry out this step, the oil was subjected to an esterification reaction to reduce the free fatty acid content.

Oil transesterification and statistical analysis

Figure 1 show the yields obtained in the transesterification step of *J. curcas* oil using different molar ratios of methanol/oil and different percentages by mass of sodium hydroxide as catalyst. From the results, it can be said that yield tends to increase at lower catalyst concentrations and higher alcohol/oil molar ratios, so the highest yield (95 %) was obtained for a catalyst percentage of 0.5 % and an alcohol/oil molar ratio of 8:1, which is higher than stoichiometric quantity leading to favor the reaction towards the products[21]. This value is comparable to the 98 % reported by Qian et al. (2010)[22]and the 96.75% reported by Ong et al. (2013)[6].Likewise, by comparing this value with the yield achieved in the transesterification of other types of oils, it can be observed that the trend is similar (97.5 and 97.72 % for *Sterculia foetida* and *Ceiba pentadra* oil, respectively)[6].

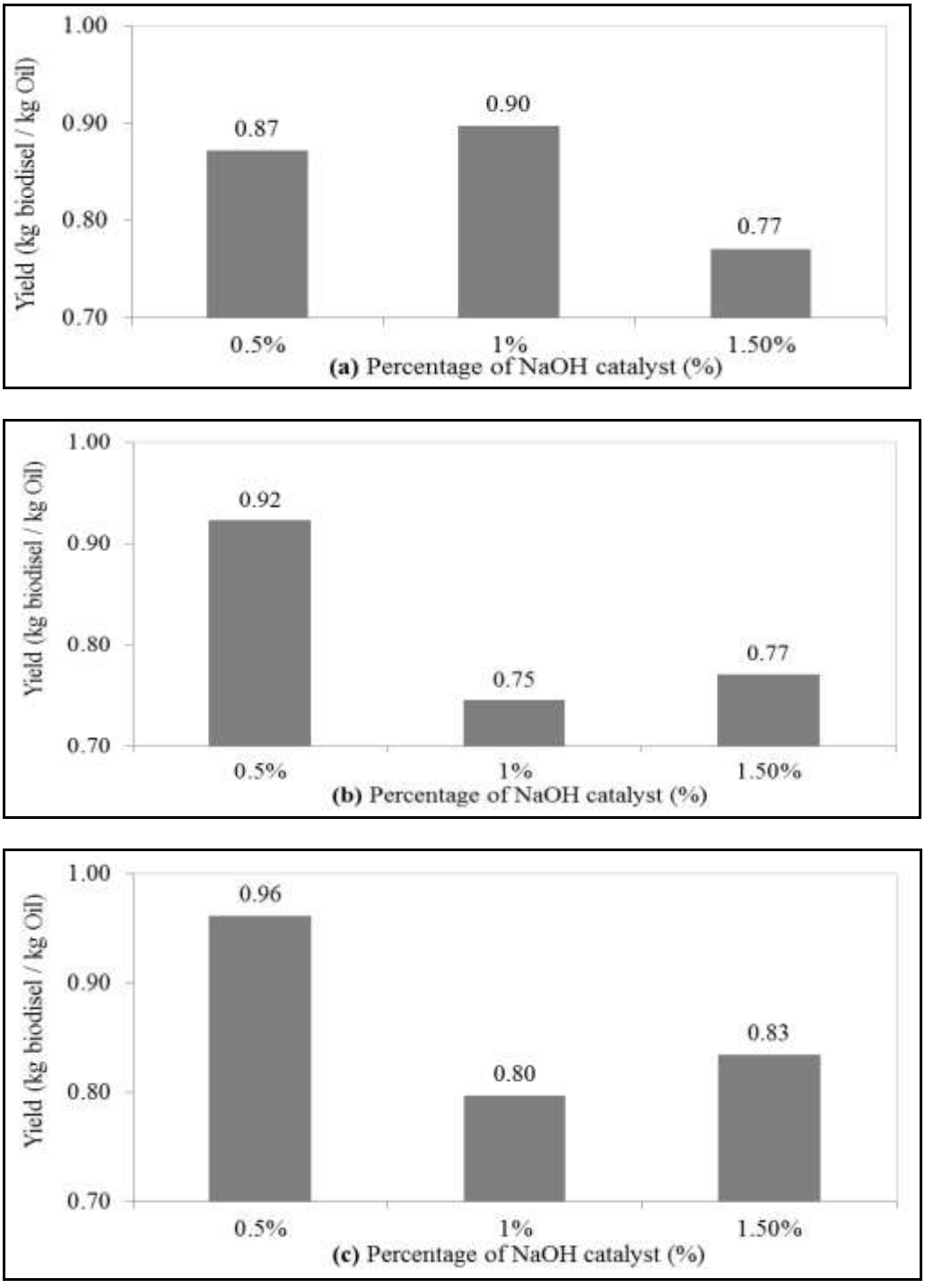

Figure 1.Transesterification yield for methanol/oil molar ratio of a) 4:1, b) 6:1 y c) 8:1

Based on the experimental data collected, the ANOVA analysis was performed for the transesterification reaction with a 95 % confidence level. Figure 2 presents the Pareto diagram obtained, which allows visualizing in order of importance the estimated effects of each variable on yield, identified so that the variable that most affects the transesterification process under the conditions evaluated, is the amount of NaOH catalyst.

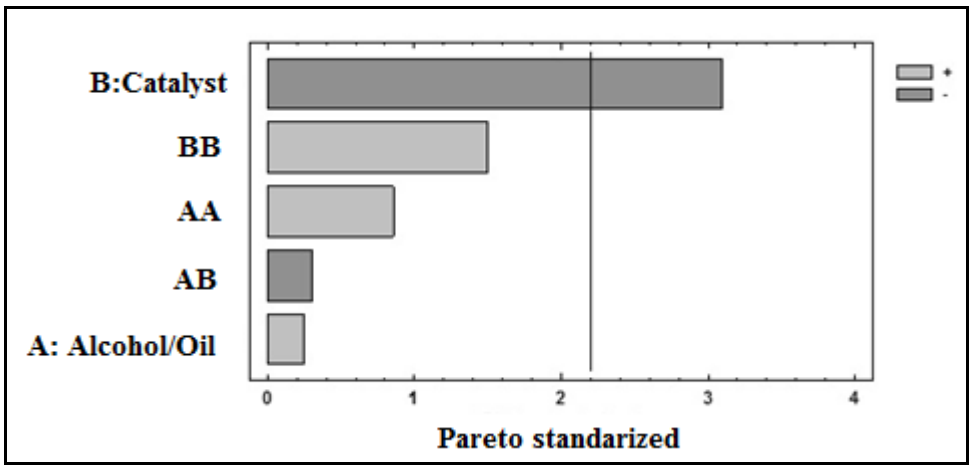

Figure 2. Standardized Pareto diagram for transesterification yield

Biodiesel characterization

The biodiesel characterization shows the presence of 13 different fatty acids, among which linoleic and oleic acid are in highest concentration. Table 3 presents the fatty acid profile obtained respect to others reported in literature [23, 7.]

Table 3. Fatty acids profile of biodiesel obtained from *J. curcas*oil

Fatty acids, % wt/wt	Authors	Sanjidet al. (2013)[23]	Berchmans and Hirata(2008)[7]
Capric	0.092	0.000	0.000
Myristic	0.012	0.100	0.100
Palmitoleic	0.827	0.600	1.100
Palmitic	14.404	14.100	14.960
Heptadecanoic	0.099	0.000	0.000
Linoleic	40.620	31.900	47.430
Stearic	7.461	7.600	3.850
Oleic	36.012	45.100	32.490

Table 4 presents the results for the main physicochemical properties of biodiesel compared to international standards.The calculated density for the biodiesel produced during the experimental part is within the range of values reported by the ASTM D6571 standard. This property is important because it guarantees optimum energy content for the biofuel, preventing it from solidifying at temperatures around 25 °C [24]. The value reached for the kinematic viscosity at 40 °C, is slightly higher to those reported by Mofijur et al. (2012)[15], Ong et al. (2013)[6] and Kartika et al. (2013)[25], possibly due to a residual content of glycerin or the presence of gums due to insufficient washing. By comparing this value to the range established by ASTM D6571, it can be said that a high viscosity favors the use of biodiesel as biofuel due to assist a complete combustion and reduce the smoke emissions in the injection system of fuel. The value obtained for the cetane index does not present significant differences with the values reported by Ong et al. (2013)[6], Becker and Makkar (2008)[26] and Ying and Mohd (2011)[27]. A value higher than the one established by ASTM D6571 indicates that the biodiesel obtained can contribute to improve the combustion characteristics in relation to petroleum diesel, since the greater this value favors the reduction of ignition delay.

Table 4. Physicochemical properties of biodiesel obtained from *J. curcas*oil

Property	Authors	ASTM D6571	ASTMD240
Density, g/cm^3	0.8864	0.8800	-
Kinematic viscosity, mm^2/s	6.2	1.9 – 6.0	-
Cetane Index	52.79	Min 47.00	-
HHV, MJ/kg	42.31	-	Min 39.00
Acidity index, mg KOH/g	0.96	Max 0.50	-

Regarding the calorific value, this result is positive for the biodiesel because of as higher this value isthe motor performance is more favored[28]. Finally, the acidity index is higher than the reported by Qian et al. (2010)[22] (0.48 mg de KOH/kg) and Mofijur et al. (2012)[15] (0.4 mg de KOH/kg), possibly due to the presence of a high content of polyunsaturated fatty acids. This high value of acidity is a disadvantage of biodiesel as biofuel because it favors the corrosion in the system and the formation of deposits[28].

Conclusions

In this work, the effect of alcohol/oil molar ratio and the amount of catalyst on biodiesel yield for the transesterification of *J. curcas* oil was studied. Characterization of *J. curcas* oil obtained allowed to find that the lipid profile as well as the values for density (0.8765 g/mL), kinematic viscosity (34.28 mm^2/s), calorific value (39.14 MJ/kg) and acidity index (6.38 mg KOH/kg)favor their use as raw material for the biodiesel production. On the other hand, it was found that the transesterification reaction yield is favored at high alcohol/oil molar ratios and low concentrations of catalyst, being the catalyst concentration the most influential variable. In this sense, the highest yield (95 %) was reached using a catalyst amount of 0.5 % wt/wt NaOH and a methanol/oil molar ratio of 8:1.For biodiesel produced, values of density, kinetic viscosity, cetane index and calorific value were in accordance with the ASTM standard for the use of biodiesel as biofuel, however, the acidity index was higher than the value allowed, which can be adjusted by the use of additives or improving the esterification and washing stages.

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