Extraction and Chemical Conversion of *Jatropha* seed oil into Biodiesel

Hareesh Chandra. P, Suraj Goyal, and Solomon Raju A.J

***Abstract*—** The seeds of *Jatropha curcas* was collected from Mohali (Punjab) and utilized to determine seed characterization. The oil was extracted using Methanol (70°C) by Soxhlet apparatus. The physicochemical properties of oil were evaluated. The result showed that the seeds consist of 22.15 dry% w/w oil, moisture and volatilities 6.1%v/w and lipid content 15%. The physic-chemical properties showed acid value at 30.67 and saponification value at 195 mg/g.

***Keywords*—** *Jatropha curcas*, physiochemical properties, seed characterization

I. Introduction

BIODIESEL helps in saving the environment in many ways. It produces lower emissions and is more energy efficient when compared to other forms of energy. Biodiesel helps in the reduction of greenhouse emissions, biodegradation and pollution. It also helps in reducing the demand for first generation biofuels which are very likely to cause over-reliance on other forms of energy.

Jatropha curcus is a woody shrub or small tree. It is native to Latin America and related very closely to the Castor plant, Ricinus communis. The plant flourishes well in different climatic zones in the tropical world. It requires moderate rainfall and soil fertility. Its leaves are not edible to animals and are used for fencing in crop fields/house premises. It does not require large doses of fertilizer. Once planted, it produces seed crop for more than 50 years. A plant of about 7 years of age produces about 2-5 Kg seed per year with 30-35% oil content. Estimates of *Jatropha* seed yield vary widely, due to lack of research data many applications of *Jatropha* are still unknown. A hectare of *Jatropha* has been claimed to produce 1,892 litres of fuel [4]. However, as it has not yet been domesticated or improved by plant breeders, yields are variable. *Jatropha* can also be intercropped with other cash crops such as coffee, sugar, fruits and vegetables.

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It can be used for halting and reversing land degradation and are fast growing when compared to many tree-borne oilseeds [3].

It has physical and chemical properties that make it highly suitable for processing into biodiesel. *Jatropha* can also be used directly in suitable diesel engines, lamps and cooking stoves and its by-products have potential value, such as seed cake for fertilizer, animal feed (non-toxic varieties) or biogas, fruit shells, seed husks for biogas and combustion. As *Jatropha* seeds are storable, their processing can be delayed, which makes its production restricted to remote areas. *Jatropha* has attracted many investors especially from the private sector. Plant breeders are also trying to make efficient varieties of *J. curcus* strains for improved and stable oil yields.

II. Materials And Methods

J. curcas seeds was collected from the Mohali, India. Damaged seeds were discarded and healthy and clean seeds were selected. The seeds were dried for some time before use. All other chemicals and solvents used were of analytical grade. Official and tentative methods of ASTM (American Society for Testing Materials) were followed for the determination of physicochemical characteristics of seed oil [6].

A. Moisture and Volatilities

About 10g of seeds were accurately weighed in a Petri dish and kept in a hot-air oven maintained at 110°C for 4 hrs after cooling in a desiccators, the loss in weight was recorded in each case. The procedure was repeated till constant weight was obtained.

B. Lipid Content

100 mg of sample was taken in a test tube and added with 10ml 0.2M ice cold $HClO_4$. Mixture was thoroughly vortexed with the help of pestle mortar for 15min keeping intermittently on ice bath and the contents were centrifuged and supernatant was decanted carefully. Procedure was repeated three times with further aliquots of $HCLO_4$. After discarding supernatant, 10ml of chloroform and methanol mixture was added to pellet,

vortexed and allowed to stand for 5 minutes. Sample was centrifuged and supernatant was collected. To this 2ml volume of distilled water was added and the Solution was shaken gently to mix thoroughly and centrifuged to separate phases. Lower organic phase containing lipids were collected in a pre-weighed Petri plate (w1). Chloroform: methanol was allowed to evaporate. After complete evaporation, Petri-plate was reweighed and lipid was estimated (w2). Difference between reweighed and pre-weighed Petri-plate gave final weight of Total lipids in *Jatropha* samples, which was calculated in terms of percentage.

C. Solvent extraction using different catalysts

The hard and dark coloured (Black) fruits were selected and picked; the kernels were then removed from the fruits and weighted. The kernels were then crushed using a pestle and mortar, wrapped in the filter paper to make a themble. The oil was then extracted using chemical solvent extraction method at 70°C using a Soxhlet unit for around 5-7 cycles [2]. The process took 10 days to complete. The crude *Jatropha* oil was checked with 0.5, 1, 2 and 3% KOH as a catalyst and also with 0.5% NaoH. The oil extracted was then made to undergo trans-esterification with the help of methanol and agitated using a magnetic stirrer followed by the separation of biodiesel to give a less viscous pure biodiesel.

D. Viscosity [7]

A viscometer was taken and the flow of water was measured along with that of the sample biodiesel using a stop watch

*η1 (viscosity of biodiesel) = (ρ1 (density of b/d)*t1 (flow time of b/d)*η2 (viscosity of water))/(t2 (flow time of water)*

E. Ash Content [9]

2ml of sample was taken in the glass crucible and kept in a desiccator to avoid moisture from entering and then weighed. They were then transferred to a muffle furnance for 1 hour at 775°C and left for cooling. The left over residue was weighed. After this, drop-wise addition of concentrated sulphuric acid was done to mix the left-over ash. It was then heated at a much lower temperature using a hot plate. After the sulphur fumes died down the weight of the sulphated ash was weighed and calculated.

F. Acid Value [8]

Two gram of the pure oil was weighed accurately by transfer method into a 250 mL conical flask. Neutral ethanol (20 mL) was added by means of a pipette and the flask heated on a steam bath for 3 minutes. Then the flask was cooled and the contents titrated with 0.1N alcoholic potassium hydroxide solution using phenolphthalein as an indicator. A blank titration was also conducted side by side [10].

G. Cloud point, Pour point and Flash point [5]

The cloud point of petroleum products and biodiesel fuels is an index of the lowest temperature of their utility for certain applications. This method determines the temperature of the test specimen at which crystals have formed sufficiently to be observed as a cloud with a resolution of 0.1°C. The pour point of a liquid is the lowest temperature at which it will flow under the prescribed conditions. It is an indication of the lowest temperature at which oil is readily pumpable. Flash point of a volatile liquid is the lowest temperature at which it can vaporize to form an ignition mixture in the air. Measuring a liquids flash point requires an ignition source.

H. Saponification value

Two gram of oil was weighed accurately by transfer method into a 250 mL round bottom flask. Freshly prepared 0.5 N alcoholic potassium hydroxide solution (25 mL) was added to the sample by means of pipette and the mixture gently refluxed on a water bath using an air-condenser for one hour. Then the flask was cooled, the condenser tip washed with little distilled water and the contents were titrated with 0.5N hydrochloric acid solution using phenolphthalein as indicator. A blank titration was carried out simultaneously.

SAP value (mg/g) = (BL1-EP1)*TF*C1*K1/SIZE

EP1: Titration volume (ml)

BL1: Blank level (25.029ml)

TF : Reagent (HCl) factor (1.006)

Cl : Concentration conversion coefficient (28.05mg/ml) (Potassium Hydroxide in Eq: 56.11*5)

Kl : Unit conversion coefficient (1)

Size: Sample Size (g)

III. Results and Discussion

Although sulphuric acid gives maximum yield, it has a high acid number which is harmful to the engine performance. Alternatively, certain potassium hydroxide and sodium hydroxide give a good yield (Table II). Since sodium hydroxide is cheaper and can be used in a very less amount it is most commercially viable catalyst to be used for trans-esterification. Viscosity describes a fluids internal resistance to flow, all fluids (except super fluids) have some resistance to stress and therefore are viscous, but a fluid which has no resistance to shear stress is known as an ideal fluid. Hence, biodiesel 0.5%KoH shows the lowest viscosity when compared with other catalysts containing biodiesel (Table III). The ash content observed for biodiesel was less than 0.1% which meets the ASTM requirements and makes it more eco-friendly as compared to other sources. This also helps in determining the metal additives if any present in the oil.

The test samples for cloud point and pour point meet the ATSM standards and have been found to be much better than the biodiesel obtained from other sources (Table IV). The characteristics of *Jatropha* seeds such as weight, oil content, ash content, moisture and volatilities were calculated (Table

I). According to the ASTM standards the flash point of biodiesel should lie above 130 and in most cases the biodiesel obtained does not meet the requirement (Table V). The saponification value calculated was 195mg/g for *Jatropha* biodiesel. The saponification value of Malaysian *J. curcas* seed oil (208.50 mg/g) was higher compared to the Nigerian *J. curcas* seed oil (198.85 mg/g), while it is less for African *Jatropha* reported in the study [1]. The acid value for the sample was found to be (30.67). The lipid content of the sample was found to be 15%. *Jatropha curcus* oil is having a good potential for oleochemical application such as surface coating and low pour point biodiesel, there is a need of the hour to have more research on *J.curcas* seed oil as it has a great potential to be the next source for biodiesel and also to encourage it as a commercial future industrial oilseeds crop. Further studies targeting the complete testing of *Jatropha* biodiesel and its activity in automotive engines are planned for future studies.

TABLE I
CHARACTERISTICS OF *JATROPHA CURCAS* SEEDS

Analytical Parameter	**Values**
Weight of 100 seeds	52.32g
Volume of 100 seeds	72mL
Oil content (%v/w)	22.15
Colour	Dull brownish black
Ash content (%w/w)	2.45
Odour	Disagreeable
Taste	Bitter
Moisture and volatilities (%w/w)	6.1

TABLE II
BIODIESEL PRODUCTION USING DIFFERENT CATALYST

LIPID+ METHANOL ⟶ BIODIESEL + GLYCEROL
(Presence of Catalyst)

Volume (ml)	***Jatropha* oil (ml)**	**Catalyst (g)**	**Methanol (ml)**	**Biodiesel (ml)**
20	15.5	0.1 (KOH)	4.4	14
20	15.5	0.1 (NaOH)	4.4	14
20	9.6	6 (H_2SO_4)	4.4	16.8

TABLE III.
VISCOSITY OF ALL SAMPLES

Catalyst	**Density of sample (g/cm^3)**	**Flow time of oil (s)**	**Viscosity of biodiesel**
0.5% KOH	.76	97	2.399
0.5%NaoH	.812	107	2.828

TABLE IV
CLOUD POINT AND POUR POINT

S. No	**Catalyst**	**Cloud point°C**	**Pour point°C**
1	0.5% KOH	2	0
2	1%KOH	0	-3
3	2%KOH	-3	-7
4	3%KOH	-2	-6
5	0.5%NaOH	-1	-4

Table V
Flashpoint [5]

S. No	**Catalyst**	**Flashpoint° C**
1	0.5%KOH	102
2	1%KOH	112
3	2%KOH	120
4	3%KOH	130
5	0.5%NaOH	120

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