

## Production and Characterization of Biodiesel from Indigenous Jatropha Plants

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### Abstract

The purpose of this study was to examine the practicability of indigenous jatropha seeds for production of biodiesel and partial replacement of petro diesel. The crude oil was extracted with the help of mechanical expeller and free fatty acids were reduced with acid esterification and biodiesel produced through transesterification process. The total quantity of extracted crude oil from jatropha seeds was 37%, press cake 52% and losses 11%, and yield percentage of biodiesel was around 92% and glycerin 7%. All characteristic parameters of crude oil and biodiesel were found within standards. The sulphur content in crude oil was found extremely lower with 0.0001% and flash point of biodiesel was 170°C against the standards of 0.8% and 130°C respectively. It is concluded that biodiesel produced from indigenous jatropha seeds is a good quality and environmental friendly fuel to be blended with petro diesel in various proportions for internal combustion engine applications.

**Keywords:** Crude Oil, Free Fatty acids, Jatropha Seeds, Transesterification

### INTRODUCTION

Energy is an essential commodity to the quality of our lives. Humans are absolutely dependent on profuse and uninterrupted supply of energy for their existence. It is the most important component in all sectors of contemporary economies. Its growth is directly associated to well-being and prosperity across the globe. Meeting growing energy demand in a safe and environmental friendly manner is a key challenge [1]. There are two main sources of energy namely nonrenewable and renewable. Nonrenewable sources include fossil fuels, such as coal, natural gas and petroleum [2]. Uranium is another nonrenewable source, but it is not a fossil fuel. In order to produce energy from fossil fuels, these are combusted, which results release of carbon monoxide, sulfur dioxide and oxides of nitrogen, which may contribute to acid rain and global warming. Renewable energy sources comprise of solar, wind, geothermal, hydropower and biomass [3]. These resources generate much less pollution as compared to nonrenewables from its collection to production steps. Out of all renewables, biomass is a versatile energy source that can be used for production of heat, power, transport fuels and biomaterials [4]. Biomass energy sources can be categorized as primary or secondary biofuels. The primary biofuels include firewood, wood chips and pellets. Such fuels are directly combusted, usually to supply cooking fuel, heating or electricity production. Secondary biofuels are available in the form of solids (e.g. charcoal), liquids (e.g. ethanol, biodiesel and bio-oil), or gases (e.g. biogas, synthesis gas and hydrogen). These secondary resources can be used for a wider range of applications, including transport and high-temperature industrial processes [5].

Pakistan is an energy deficient country. Energy shortage and the frequent load shedding have created a chaos in every corner of the country [6]. However, the Government is introducing renewables including biodiesel in order to meet the increasing energy demand of the country. Biodiesel is considered as one of the imminent alternate fuel of petroleum diesel. As it contains higher oxygen content (10-12%), sulphur free, and high cetane index (greater than 47) [7]. If 10% biodiesel is utilized in automobile diesel engines in the country, then approximately 56.13 Billion Rupees foreign

exchange would be saved in the country.

Pakistan possesses a high potential for biofuels, which include forest, cattle dung, distillery waste and agricultural biomass. When exploring the agriculture options, the biomasses comprise of rice husk, banana leaves, barley straw, sugarcane, wheat hulls, castor, and jatropha. From all agricultural sources, jatropha is preferable because of its reasonable availability and easy accessibility especially in Sindh province. The bionomical name of Jatropha is Jatropha curcas. It belongs to spurge family. Its seeds are hard in texture and blue in color. Jatropha is a flowering plant with a height of around 6m, cultivated all over the world particularly in the tropical and subtropical regions [8]. Once matured, the plant yields in 9-10 months and 2-3 times in a year [9]. Upon maturing, the Jatropha plant appears with green rounded seeds, which become either light blue or purple shaped hard shells as shown in Figure 1.

Various characteristic properties and chemical composition of jatropha seeds, extracted crude oil and produced biodiesel decides its suitability for the replacement of petro diesel in internal combustion engines and other industrial applications. The characteristic parameters include moisture content, oil content, iodine value, saponification value, free fatty acid and protein. Moisture reacts with catalyst during transesterification process for both soap formation and emulsion [10]. Therefore, it must be reduced or removed from seeds and/or extracted crude oil before biodiesel production. Lower saponification value is preferred for higher yield of biodiesel, because, higher saponification value decreases yield. However, higher saponification values support soap formation in presence of sodium hydroxide as a catalyst [11]. The iodine number measures the double bonds present in biodiesel which determines the degree of unsaturated free fatty acids present in biodiesel. Higher iodine number may lead to deposition in diesel engine injectors [10].

Moreover, the fuel properties of biodiesel like lower kinematic viscosity, total acid number, and pour point support higher production and engine efficiencies. It is because the kinematic viscosity is an important property, which is used to determine the efficiency of biodiesel as a fuel. It is directly related to the resistance to the flow of fluids [12]. It is measured as the amount of time taken for a given volume of

oil to pass through an orifice of a specified size [10]. Flash point is measurement of flammability of any fuel. The higher the flash point, the more viable the fuel will be in storage and handling [13]. Cetane index is considered as one of the most important property of fuel affecting the quality of combustion and ignition delay. The lower the cetane index, the higher its ignition delay [14]. Lower cetane index containing fuels cause diesel engine knocking and result increased emissions of gaseous and particulate exhaust due to incomplete combustion [10]. The calorific value is a measurement of heat energy content of any fuel. Generally, higher calorific value is preferred because it releases higher heat; ultimately improving the performance of engine [14]. The composition of free fatty acids (FFA) determines the fuel properties of any biodiesel. The jatropha crude oil contains saturated as well as unsaturated FFAs. The fatty acid with double bond is termed as unsaturated fatty acid, whereas fatty acid containing no double bond is known as saturated fatty acid [15]. Higher amount of FFAs cannot be completely converted into biodiesel because it supports the formation of soap [16]. Therefore, the crude oil which contains higher amount of FFA need further process through acid esterification to lower down its level. Because of higher availability, accessibility, non-edible nature, and lower cost of jatropha seeds, it is selected for production of biodiesel. Its characteristics, properties and composition were studied to examine its viability for partial replacement of petro diesel in internal combustion engines. It is because our country is highly dependent on the import of petroleum products for primary energy needs.

## MATERIALS and METHODS

Materials and methods adopted for this study are categorized into three sections. In first section, the method adopted for production of biodiesel are discussed including the collection of seeds, chemicals used, and technique applied for extraction and pretreatment of crude oil, and production of biodiesel through transesterification process. The characteristic analysis of crude oil are highlighted in section 2, whereas, the fuel properties of produced biodiesel and composition of free fatty acids are discussed in section 3. The chemicals and reagents used for the production of biodiesel were acetic Acid, Carbon Tetrachloride, Wijs Solution, Potassium Iodide Solution, Starch, Sodium Thiosulphide, Di Ethyl Ether, Phenolphthalein indicator, Hydrochloric acid, Sodium Hydroxide, Potassium Hydroxide, Methanol and Sulphuric Acid.

### Production of Biodiesel

Initially, jatropha seeds were purchased from Pipri Model farm Karachi and then these were sorted and washed with distilled water to remove ruminants, clays, and adsorbed pesticides. After that, the seeds were dried for 72 hours for removal of moisture and then crushed for separation of their coats/shells and seed meats. The cleaned crushed seeds were passed through a screen of 10 mm size. Finally, the dried seeds were feed in mechanical screw for extraction of crude oil through compression force as shown in Figure 2. After compression, the crude oil and press cake were separated from each other. The press cake was recycled four times in the hopper to extract maximum oil contents. The crude oil was refined by adding little amount of caustic soda and then filtered and stored in plastic cans at room temperature.

The moisture from extracted crude oil was removed by heating the samples at 100°C for 15 minutes. The tempera-

ture was brought down up to 50°C through air cooling. The dried crude oil was then mixed with solution of methanol and H<sub>2</sub>SO<sub>4</sub> for acid esterification. After that, the mixture was transferred and left in a funnel for 24 hours to separate bottom layer (jatropha oil) and upper layer unreacted reagents. The sample of crude jatropha oil was further titrated and brought the level of free fatty acids (FFA) less than 1 percentage.

Finally, the biodiesel was produced from jatropha crude oil using transesterification process as shown in Figure 3. In this process, the pre-treated jatropha oil and sodium methylate solution was poured into the reactor vessel at a temperature of 50°C with stirring speed of 800 for 30 minutes. During transesterification process, the triglycerides were converted into diglycerides, then to monoglycerides and finally to glycerol and settled at the bottom of funnel and the biodiesel at the top. After that, the collected biodiesel was washed with glacial acetic acid and water to dissolve any organic matters present in it. The moisture of synthesized jatropha biodiesel was removed by heating at 110°C. At the end, the biodiesel is filtered through a filter paper and collected in a sterilized bottle.

### Characterization of Crude Oil and Biodiesel

The characteristic parameters of crude oil namely moisture content, oil content, iodine value, saponification value, free fatty acid and protein of crude jatropha oil was examined using The American Oil Chemists' Society (AOCS) standard methods as shown in Table 1. The chemical composition of FFA of jatropha crude oil and produced biodiesel was analyzed using Gas Chromatography and Mass spectrometry. The methods adopted, American Society for Testing and Materials (ASTM) standards used for investigation of fuel properties of produced biodiesel like density, kinematic viscosity, sulphur, flash point, total acid number, pour point, cetane index, cloud point, water percentage and calorific values as shown in Table 2.

## RESULTS and DISCUSSIONS

### Quantity of Crude Oil Extracted from Jatropha Seeds

A total of 10kg jatropha seed samples were introduced in mechanical expeller for extraction of crude oil. The total quantity of extracted crude oil obtained was 3.7kg (37% or 4 liters), press cake 5.2kg (52%) and the losses were around 1.1kg (11%). The temperature of crude oil at the outlet was in the range of 35-50°C and of press cake was from 40 to 95°C.

### Characterization of Extracted Crude Oil

Table 3 shows the characteristics of extracted crude oil and literature reported values, and the fuel properties of produced biodiesel. The density of extracted crude oil at 15°C was found 0.92 kg/lit, while in the literature reported values for crude oil were in the range of 0.85-0.94 kg/lit, and in produced biodiesel it was found 0.88 kg/lit. The kinematic viscosity of crude oil and literature reported values were 33.45 cSt, and 24.5 to 54.8 cSt respectively. The measured value of kinematic viscosity in produced biodiesel was 4.54 cSt. The flash point and pour point values in crude oil were 216°C and 3°C, whereas, in literature there values were 128°C to 315°C and -2°C to +6°C respectively. The flash point and pour point of biodiesel samples were 170°C and +3 °C respectively. Cetane index in crude oil was found 38, while in

the literature it was in the range of 23 to 41, whereas in produced biodiesel it was 48. In literature the sulphur content of crude oil was in the range of 0.200% to 0.166%, whereas, in examined crude oil samples its value was too low with 0.0001%, whereas, in produced biodiesel its value was around 0.0092%. Free Fatty Acids level in crude oil samples was found 16.9%, whereas, the reported values in literature were from 0.21% to 3.38%. The calorific value of extracted crude oil was 39.85kJ/kg, and in literature there values were found from 33.0kJ/kg to 42.8kJ/kg. The calorific value of produced biodiesel was around 42.15kJ/kg, water content 0.05% of volume, cloud point +3 °C and total acid number 0.77mg KOH/g.

The yield percentage of biodiesel was 92% and glycerin 7.2% and the remaining were losses in the transesterification processes. The kinematic viscosity of jatropha biodiesel as found within the range of ASTM standards of 1.9 to 6.0 cSt. The results were found to be similar biodiesel production through other seeds such as *M. oleifera* (4.83 cSt), *jatropha curcas* (4.723 cSt), *M. stenopetala* (4.58 cSt), canola (4.5281 cSt), palm (4.6889 cSt), and soybean (4.3745) [17, 18]. The flash point of produced jatropha biodiesel was found to be 170°C, which is greater than ASTM standard of 130°C. It was found that jatropha biodiesel fuel was an excellent fuel. The flash point of produced biodiesel was almost double than that of petro diesel. The sulphur content in the produced biodiesel was found less than ASTM allowable limits as well as than petro diesel, which is a good indication of environmentally friendly fuel source. The calorific value of produced biodiesel was found within ASTM standards, but slightly lower than that of petro diesel fuel. Biofuels normally have lower calorific value than diesel fuel due to the oxygen content in biodiesel. However, the presence of higher amount of oxygen in biodiesel leads towards complete combustion of the biodiesel fuel in the diesel engine [19-29].

The composition of Free Fatty Acid (FFA) of extracted crude oil and produced biodiesel are tabulated in Table 4. The palmitic acid in extracted crude oil and produced biodiesel samples were 12.7 and 22.1 %. Whereas, the literature reported values of extracted crude oil and produced biodiesel samples was in the range of 11.3 - 16.9% and 12.9-31.4 respectively. The linoleic acid in extracted crude oil was 37.5% and in literature its range was 19.0 - 41.0%, while in produced biodiesel its value was 32.3% and literature reported values were 31.9% to 40.4 %. Similarly, the oleic acid in extracted crude oil was 36.5% and in literature its range was 34.3 to 45.8%, while in produced biodiesel its value was 36.1% and literature reported values were 37.1% to 45.8%. Likewise, stearic acid in extracted crude oil was 13.4% and in literature its range was 3.7 to 9.8%, while in produced biodiesel its value was 7.9% and literature reported values were 5.4% to 7.6%. The value of lauric acid and palmitoleic acid in produced biodiesel were found 0.1% and 1.4%, whereas, in literature reported values were in the range of 0.5 to 1.4% and 0.6% to 1.1% respectively [20-31].

## CONCLUSION

The characteristic parameters of crude oil was examined using AOCS standard methods and the chemical composition of FFA of jatropha crude oil and produced biodiesel was analyzed using Gas Chromatography and Mass spectrometry as per ASTM standards. Initially, the crude oil was extracted from jatropha seeds with the help of mechanical expeller. The total quantity of extracted crude oil obtained was 37%,

press cake 52% and the losses during extraction were around 11%. Then, the fatty acids present in the crude oil were reduced using acid esterification process. Finally, the biodiesel was produced through transesterification process and its characteristics and composition were investigated. The results revealed that all characteristic parameters and chemical composition of extracted crude oil and produced biodiesel were found permissible limits and literature reported values except sulphur and flash point. The sulphur content of crude oil was found 0.0001% against literature reported values of 0.200% to 0.166%, which was highly less than ASTM standards. Moreover, the flash point of produced biodiesel was found to be 170°C, which is greater than ASTM standard of 130°C. The flash point of produced biodiesel was almost double than that of petro diesel. It indicates a best quality and environmental friendly fuel. The yield percentage of biodiesel was 92% and glycerin 7.2% and the remaining were losses in the transesterification processes. In addition, the values of lauric acid of produced biodiesel were found 0.1% against literature reported value of 0.5% to 1.4% and palmitoleic acid 1.4% against 0.6% to 1.1%. The lauric acid value was lower and palmitoleic acid was more than literature reported values, which indicates better quality fuel due to presence of polyunsaturated fatty acids. It is concluded from the study that biodiesel produced from indigenous jatropha seeds is a best alternate to petro diesel fuel and can be successfully blended and used in internal combustion engines.

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**Table 1.** Characteristics of Crude Oil, Adopted Methods and Allowable Ranges

Sr. No.	Characteristic Parameters	Methods	Range	Jatropha Crude Oil
1	Oil Content (%)	AOCS (Aa 3-52)	Max: 45	45.0
2	Moisture Content (%)	AOCS (Ae 3-38)	Min: 0.5	0.19
3	Saponification Value (mgKOH/g)	AOCS (Cd 3 – 25)	190 - 209	198.37
4	Iodine Value (gI <sub>2</sub> /100g)	AOCS (Cd 1- 25)	84.2 - 112	108.61
5	Protein (%)	AOCS (984.13)	< 24	24.60
6	Free Fatty Acid (%)	AOCS (Ca5a-40)	3.38 -38.2	16.09

**Table 2.** Methods and Standards of Fuel Properties of Biodiesel

S r . No.	Fuel Properties	Methods	ASTM Standards of Biodiesel	ASTM Standards D100
1	Density at 15°C (kg/lit)	ASTM D-1298	0.880	0.8401
2	Kinematic Viscosity at 40°C (cST)	ASTM D-445	1.9 – 6.0	3.06
3	Sulphur (% by wt.)	ASTM D-4294	0.05 max	0.735
4	Flash point (°C)	ASTM D-93	≥ 130 min	74
5	Total Acid Number (mg KOH/g)	ASTM D-664	0.80 max	0.249
6	Pour point (°C)	ASTM D-97	-15 to +5°C	o
7	Cetane index	ASTM D-976	47 min	52
8	Cloud point (°C)	ASTM D-2500	-3 to -12°C	+10
9	Water (% by vol.)	ASTM D-85	0.05	0.05
10	Calorific value (MJ/kg)	ASTM D240-14	37.5 to 42.80	44.2

**Table 3.** Fuel Properties of Jatropha Crude Oil [19-32]

Sr. No.	Properties	Extracted Crude Oil	Crude Oil Cited in Literature	Produced Biodiesel
01	Density at 15°C (kg/lit)	0.92	0.85 – 0.94	0.88
02	Kinematic Viscosity at 40°C (cSt)	33.45	24.5 - 54.8	4.54
03	Flash Point (°C)	216	128 – 315	170
04	Pour Point (°C)	3	-2 to +6	+3
05	Cetane Index	38	23 - 41	48
06	Sulphur Content (%)	0.0001	0.2000 - 0.1660	0.0092
07	Free Fatty Acid, FFA (%)	16.90	0.21 - 3.38	--
08	Calorific Value (kJ/kg)	39.85	33.00 - 42.80	42.15
09	Water Content (% by vol.)	--	--	0.05
10	Cloud Point (°C)	--	--	+3
11	Total Acid Number (mg KOH/g)	--	--	0.77

**Table 4.** Composition of Free Fatty Acid in Extracted Crude Oil and Produced Biodiesel [19-32]

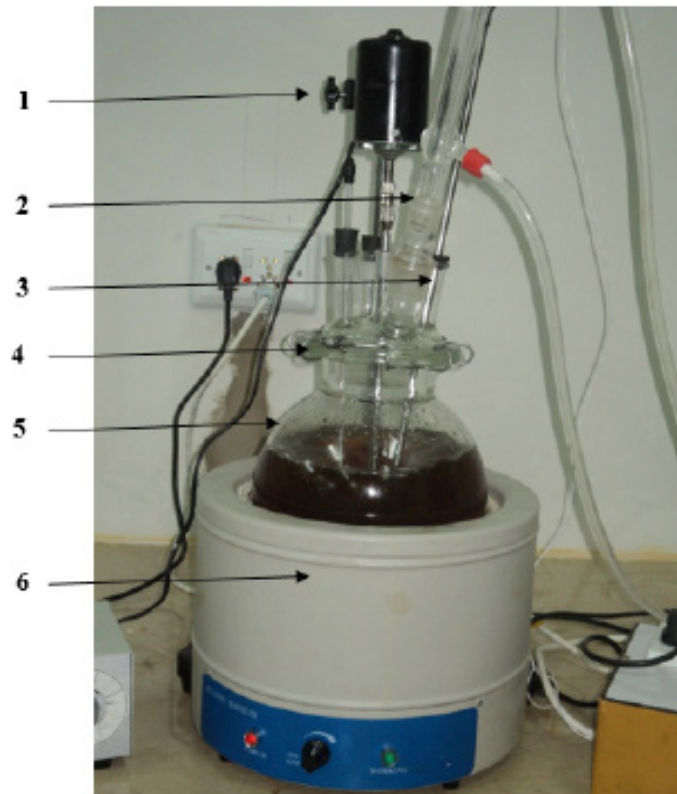
Sr. No.	Name of Fatty acid	Extracted Crude Oil (%)	Produced Biodiesel (%)	Crude Oil (%) Literature Cited	Biodiesel (%) Literature Cited
1	Palmitic acid	12.7	22.1	11.3 - 16.9	12.9-31.4
2	Linoleic acid	37.5	32.3	19.0 - 41.0	31.9 - 40.4
3	Oleic acid	36.5	36.1	34.3 - 45.8	37.1- 45.8
4	Stearic acid	13.4	7.9	3.7- 9.8	5.4- 7.6
5	Lauric acid	--	0.1	--	0.5-1.4
6	Palmitoleic acid	--	1.4	--	0.6 - 1.1



Figure 1. Jatropha Plant, Seed and Kernels



Figure 2. Extraction of Crude Oil through Mechanical Screw Oil Expeller



- |                              |                            |
|------------------------------|----------------------------|
| <b>1. Mechanical Stirrer</b> | <b>2. Reflux Condenser</b> |
| <b>3. Thermocouple</b>       | <b>4. Five hole lid</b>    |
| <b>5. Round bottom flask</b> | <b>6. Heating mantle</b>   |

Figure 3. Production of Biodiesel through Transesterification Process