Production and Characterization of Biodiesel from Indigenous Castor Seeds

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***Abstract*— The purpose of this study was to examine the practicability of indigenous castor 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 castor seeds was 45.8%, press cake 40% and losses 14.2%, and yield percentage of biodiesel was around 93.5% and glycerin 6.5%. All characteristic parameters of crude oil and biodiesel were found within standards. The sulphur content in crude oil was found extremely lower with 0.001% and flash point of biodiesel was 130^{o}C against the standards of 0.8% and 130^{o}C respectively. In addition, the palmitic, linoleic acid, Oleic and ricinoleic acids found better against literature reported in extracted crude oil and produced biodiesel samples. The palmitic fatty acid was greater; henceforth as a result it produces more yield of biodiesel. While linoleic acid was contained moderate percentage so as to form biodiesel with fewer amounts of reagents and henceforth ensures most economical production of biodiesel. 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.**

***Index Terms*— Crude Oil, Free Fatty acids, Castor Seeds, Transesterification**

I. INTRODUCTION

The energy crisis is increasing continuously worldwide due to enhancement in mechanization, industrialization and declination of fossil fuel resources. The fossil fuels are non-renewable energy sources. At the same time, the unstable prices and environmental concerns of petroleum products are other issues which need to be given attention. Among petroleum fuels, the consumption of diesel is higher than other products. Roughly estimate consumption of diesel and petrol is 70% and 30%, respectively [1]. Therefore, it is a need of today's world to find a better substitute of fossil fuels, mainly, diesel. Presently, biodiesel is being given a full consideration of researchers as an alternative of diesel fuel because it is renewable and reduces the emission of some pollutants to the environment. The properties of biodiesel are also very close to the diesel fuel [2].

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Pakistan is an energy deficient country. Energy shortage and the frequent load shedding have created a utter confusion in every corner of the country [3]. However, the Government of Pakistan is introducing renewable energies including biodiesel in order to meet the increasing energy demand of the country. Biodiesel is considered as one of the impending alternate fuel of petroleum diesel. Biodiesel is defined as the mono-alkyl esters of long chain fatty acids derived from animal fats, vegetable oils or alcohol in presence or absence of catalyst [4]. The commonly used process for biodiesel production is transesterification. In this process, the triglycerides react with an alcohol to produce fatty-acid mono-alkyl ester as a main product and glycerol as a byproduct in presence/absence of acid or basic catalyst. Generally, alkali catalysts are preferred for this type of reactions because they make the reaction faster and the conditions are more moderated [5]. Different feed materials from edible sources such as soybean, palm, rape seed, and sunflower oils have already been studied by various scientists in the world. The use of edible oils as a feedstock for biodiesel production may cause food and feed problems, therefore, in order to avoid such type of shortcomings, the non-edible oils are promising source as a feedstock material for biodiesel production, especially, in developing countries to meet their increasing demand of energy [6-7]. Different non-edible oil sources such as Jatropha curcas, calophyllum inophyllum L, sterculia Foetida L, ceiba pantandra L, pongamia pinnata, nicotiana tabacum L, putranjiva roxburghii, linseed oil, rice bran oil, mahua oil, and castor oil (ricinus communis) might be studied and explored as a feedstock for biodiesel production [8-10]. In this study, castor oil from non-edible oil resources is selected as a feedstock for biodiesel production due to its higher oil content. Castor belongs to the Euphorbiaceae family. It grows in tropical and subtropical areas [11]. The growing period of castor is much shorter (4-5 months) and it is a fast growing, suckering perennial shrub with a size of 12 meters [2]. The oil content in castor seed is around 40-60%, which is rich in triglycerides. It is mainly constituted of triglycerides consisting of three fatty acids and one molecule of glycerol. It is a viscous, non-dry, and pale yellow nonvolatile oil as shown in Figure 1 and it has very low cloud point and pour point making it as a good alternate biofuel in cold climate condition [8-12].

Figure 1: Castor Plant & Seed

Various characteristic properties and chemical composition of castor 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 [4]. 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 [13] Koh MY. 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 [4]. 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 [6]. It is measured as the amount of time taken for a given volume of oil to pass through an orifice of a specified size [4]. 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 [14]. 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 [15]. Lower cetane index containing fuels cause diesel engine knocking and result increased emissions of gaseous and particulate exhaust due to incomplete combustion [14]. 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 [15]. The composition of free fatty acids (FFA) determines the fuel properties of any biodiesel. The castor 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 [16]. Mofijur Higher amount of FFAs cannot be completely converted into biodiesel because it supports the formation of soap [17]. 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 castor 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.

In this study, in order to find suitability of the castor oil as feedstock for biodiesel production, the castor oil was converted to biodiesel using transesterification process. Furthermore, the properties of castor oil biodiesel were studied and compared to ASTM standards to find the compatibility of castor biodiesel with diesel fuel. Additionally, the composition of free fatty acids (FFAs) was investigated using gas chromatography-mass spectroscopy (GC-MS).

II. 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.

A. PRODUCTION OF CASTOR BIODIESEL

Initially, castor seeds were purchased from Mirpurkhas and Mithy district of Sindh as 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

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

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.

Figure3: Production of Castor Biodiesel through Transesterification process

The moisture from extracted crude oil was removed by heating the samples at 120°C for 15 minutes. The temperature was brought down up to 50°C through air cooling. The dried crude oil was then mixed with solution of methanol and H_2SO_4 for acid esterification. After that, the mixture was transferred and left in a funnel for 24 hours to separate bottom layer (castor oil) and upper layer unreacted reagents. The sample of crude castor oil was further titrated and brought the level of free fatty acids (FFA) less than 1 percentage. Finally, the biodiesel was produced from castor crude oil using transesterification process as shown in Figure 3. In this process, the pre-treated castor oil and sodium methylate solution was poured into the reactor vessel.

Figure 4: Drying of biodiesel & recovering of methanol through distillation process

The crude oil and sodium methylate solution was stirrer well by mechanical stirrer keeping speed 800 rpm under temperature 50°C 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 completion reaction phase, the collected biodiesel was washed with glacial acetic acid and water to dissolve any organic matters present in it. The moisture of synthesized castor biodiesel was removed by heating at 100°C as shown in Figure 4. At the end, the biodiesel is filtered through a filter paper and collected in a sterilized bottle.

A. 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 castor oil was examined using The *American Oil Chemists' Society* (*AOCS*)standard methods as shown in Table 1

Table 1: Characteristics of Crude Oil, Adopted Methods and Allowable Ranges (11, 20)

Sr. No.	Characteristic Parameters	Methods	Range	Jatropha Crude Oil
1	Oil Content (%)	AOCS (Aa 3-52)	Max: 50	51.12
2	Moisture Content (%)	AOCS (Ae 3-38)	Min: 0.5	1.21
3	Saponification Value (mgKOH/g)	AOCS (Cd 3 – 25)	190 - 209	186.23
4	Iodine Value (gI2/100g)	AOCS (Cd 1- 25)	84.2 - 112	89.35
5	Protein (%)	AOCS (984.13)	< 24	19.4
6	Free Fatty Acid (%)	AOCS (Ca5a-40)	3.38 -38.2	4.02

The chemical composition of FFA of castor 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.

Table 2: The Standards and Methods of Fuel Properties of Biodiesel

S. No.	Fuel Properties	Methods	ASTM Standards of Biodiesel	ASTM Standards of D100
1	Density at 15°C (kg/lit)	ASTM D-1298	0.880	0.840
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	0
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

III. RESULTS AND DISCUSSIONS

A. QUANTITY OF CRUDE OIL EXTRACTED FROM JATROPHA SEEDS

A total of 10 kg castor seed samples were introduced in mechanical expeller for extraction of crude oil. The total quantity of extracted crude oil obtained was 4.58 kg (45.8% or 4.75 liters), press cake 4.0 kg (40%) and the losses were around 1.42 kg (14.2%). The temperature of crude oil at the outlet was in the range of 50-60^{o}C and of press cake was from 55 to 95^{o}C.

B. CHARACTERIZATION AND ANALYSIS OF EXTRACTED CRUDE OIL AND BIODIESEL

The moisture content found through analysis was 1.21%, which was greater the reported range. Moisture content is an impurity which must be reduced or removed before biodiesel production through heating crude oil. Further, moisture reacts with catalyst during transesterification process for both soap formation and emulsion [14]. The Saponification value (mgKOH/g) was found 186.23 which was well within the vrange. Strictly speaking, lower saponification value is preferred for higher yield of biodiesel. Higher saponification value decreases biodiesel yield percentage; however, on the contrary it will support soap formation in presence of sodium hydroxide as a catalyst [13]. The iodine value was found 89.35 (gI_2/100g), which was good and within the range. The iodine number measures the double bonds present in biodiesel which determines the degree of unsaturation of biodiesel. Further, a higher iodine number may lead to deposit formation in diesel engine injectors [13, 18]. Protein was found to be 19.4%, which was lower than the range. The FFA was found 4.02 which were within the range.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 15ºC was found 0.963 kg/lit, while in the literature reported values for crude oil were in the range of 0.856-0.967 kg/lit, and in produced biodiesel it was found 0.924 kg/lit. The kinematic viscosity is an essential property of fuel which is remained very high in jatropha and castor oils [11]. The kinematic viscosity of crude oil and literature reported values were 196 cSt, and 6.6 to 248.8 cSt respectively. The measured value of kinematic viscosity in produced biodiesel was 7.5 cSt. The flash point and pour point values in crude oil were 138ºC and -15ºC, whereas, in literature there values were 178ºC to 325ºC and -14ºC to -18ºC respectively. The flash point and pour point of biodiesel samples were 130ºC and -12 ºC respectively. Cetane index in crude oil was found 40, while in the literature it was in the range of 23 to 40, whereas in produced biodiesel it was 45. In literature the sulphur content of crude oil was in the range of 0.2% to 0.166%, whereas, in examined crude oil samples its value was too low with 0.038%, whereas, in produced biodiesel its value was around 0.001%. Free Fatty Acids level in crude oil samples was found 4.22%, whereas, the reported values in literature were from 0.5% to 5.0%. The calorific value of extracted crude oil was 35.18kJ/kg, and in literature there values were found from 29.6kJ/kg to 38.65kJ/kg. The calorific value of produced biodiesel was around 42.5kJ/kg, water content 0.05% of volume, cloud point -10 ºC and total acid number 0.96mg KOH/g.

The yield percentage of biodiesel was 93.5% and glycerin 6.5% and the remaining were losses in the transesterification processes.

Table 3: Fuel Properties of Castor Crude Oil [8, 11, 19, 20, 21, 24]

Sr. No.	Properties	Extracted castor Crude Oil	Crude Oil Cited in Literature	Produced Castor Biodiesel
01	**Density at 15°C (kg/lit)**	0.963	0.856 – 0.967	0.924
02	**Kinematic Viscosity at 40°C (cSt)**	196.0	6.6 – 248.8	7.5
03	**Flash Point (°C)**	138	178 – 325	130
04	**Pour Point (°C)**	-15	-14 to -18	-12
05	**Cetane Index**	40	23 - 40	45
06	**Sulphur Content (%)**	0.038	0.2 - 0.166	0.001
07	**Free Fatty Acid, FFA (%)**	4.22	0.5 – 5.0	--
08	**Calorific Value (kJ/kg)**	35.18	29.6 - 38.65	42.5
09	**Water Content (% by vol.)**	--	--	0.05
10	**Could Point (°C)**	--	--	-10
11	**Total Acid Number (mg KOH/g)**	--	--	0.96

The higher rate of conversion is attributed to the reduced amounts of FFAs present in these materials. 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 [6]. The kinematic viscosity of castor biodiesel as found little bit higher than the range of ASTM standards of 1.9 to 6.0 cSt. This may be endorsed to the presence of hydroxyl groups in castor biodiesel [11]. The kinematic viscosity can be reduced by using blending with petro diesel [19-21]. The 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 [14]. The flash point of produced castor biodiesel was found to be 130^{o}C, which is equal to ASTM standard of 130^{o}C but less than petro diesel which indicates castor biodiesel fuel is an excellent biofuel.. The higher value of flash point of biodiesel in comparison to diesel fuel suggests that the flammability hazard of biodiesel is much lesser than that of diesel fuel [22].The sulphur content in the produced biodiesel was found less than ASTM allowable limits as well as than petro diesel, which is a good sign of environmentally friendly fuel source. 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 [15].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 more 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 [10-23]. Table 4 shows the composition of FFA, which are to be used to determine the fuel properties of any biodiesel. Oil from castor seeds contained high percentage of ricinoleic acid with substantial amounts of palmitic, linoleic, Oleic, stearic and Dihydroxy stearic acid. The composition of Free Fatty Acid (FFA) of extracted crude oil and produced biodiesel are tabulated in Table 4. The palmitic fatty acid is the most common fatty acid found in animals, plants and microorganisms. Its percentage is greater; henceforth as a result it produces more yield of biodiesel. [24].The palmitic acid in extracted crude oil and produced biodiesel samples were 3.39 and 0.65 %. Whereas, the literature reported values of extracted crude oil and produced biodiesel samples was in the range of 0.7-2.0 and 0.41-2.0 respectively. The linoleic acid has a lower boiling and melting point.[10,15]. Linoleic acid is a polyunsaturated omega-6 fatty acid. It is a colourless becomes liquid at room temperature [24-26]. The linoleic acid in extracted crude oil was 2.62 and in literature its range was 0.30 – 5.5%, while in produced biodiesel its value was 4.55% and literature reported values were 4.4 – 5.0%. The Oleic acid occurs naturally in various animal and vegetable fats and oils.

Table 4 :. Composition of Free Fatty Acid of Extracted castor Crude Oil and Produced Castor Biodiesel [10, 11, 15, 24, 25, 26]

S. No	Name of Fatty acid	Extracted Crude Oil (%)	Produced Biodiesel (%)	Crude Oil (%) Literature Cited	Biodiesel (%) Literature Cited
1	Palmitic acid	3.39	0.65	0.7 – 2.0	0.41-2.0
2	Linoleic acid	2.62	4.55	0.30 – 5.5	4.4 – 5.0
3	Oleic acid	9.61	4.41	2.7 – 7.0	2.57- 7.0
4	Stearic acid	1.51	1.38	0.9 – 7.4	0.13- 0.7
5	Dihydroxy stearic acid	--	0.45	--	--
6	Ricinoleic acid	81.56	87.89	80-90	86-89

It is an odourless and a colourless oil.. The oleic acid in extracted crude oil was 9.61 and in literature its range was 2.7- 7.0 . While in produced biodiesel its value was 4.41 and literature reported values were 2.57- 7.0. It has a moderate percentage so as to form biodiesel with fewer amounts of reagents and henceforth ensures most economical production of biodiesel. Likewise stearic acid is a saturated fatty acid with an 18-carbon chain and has the IUPAC name octadecanoic acid. The stearic acid in extracted crude oil was 1.51% and in literature its range was 0.9 – 7.4, while in produced biodiesel its value was 1.38% and literature reported values were 0.13- 0.7. Its value was little bit greater than the range. Since its characteristic is waxy in biodiesel that's why, kinematic viscosity slightly higher, its percentage is reasonable so that in turn falls within ASTM standard, whilst the cetane becomes higher than petro diesel. Another very important acid namely ricinoleic acid was found in extracted crude oil 81.56% while in produced biodiesel was found and 87.89%, whereas, in literature reported values of were in the range of 80-90 for crude oil and 86-89 for biodiesel respectively. The ricinoleic acid value was reasonable value. Primarily, due to existence of this acid in the castor oil, the oil is used for coatings, plastics, inks and cosmetics but nowadays biodiesel is made from this oil successfully. [11-26].

IV. CONCLUSION

The characteristic parameters of crude oil was examined using *AOCS* standard methods and the chemical composition of FFA of castor crude oil and produced biodiesel was analyzed using Gas Chromatography and Mass spectrometry as per ASTM standards. Initially, the crude oil was extracted from castor seeds with the help of mechanical expeller. The total quantity of extracted crude oil obtained was 45.8%, press cake 40% and the losses during extraction were around 1.42%.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 under permissible limits and literature reported values except sulphur , flash point and calorific value . The sulphur content of crude oil was found 0.038% against literature reported values of 0.2% to 0.166%, which was extremely less than ASTM standards. Moreover, the flash point of produced biodiesel was found to be 138^{o}C, which is greater than ASTM standard of 130^{o}C. The flash point of produced biodiesel was more than that of petro diesel. Due to high flash point of castor oil has convinced advantages over petro diesel i.e. greater safety during handling, storage and transportation It indicates a best quality and environmental friendly fuel. The calorific values were found reasonable. The yield percentage of biodiesel was 93.5% and glycerin and 6.5% the remaining were losses in the transesterification processes.

In addition, the palmitic, linoleic acid, Oleic and ricinoleic acids found better against literature reported in extracted crude oil and produced biodiesel samples. The palmitic fatty acid was greater percentage; henceforth as a result it produces more yield of biodiesel. While linoleic acid was contained moderate percentage so as to form biodiesel with fewer amounts of reagents and henceforth ensures most economical production of biodiesel. It is concluded from the study that biodiesel produced from indigenous castor seeds is a best alternate to petro diesel fuel and can be successfully blended and used in internal combustion engines.

V. REFERENCES

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