



Study of Physical and Chemical Properties of Biodiesel from Sorghum Oil

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Abstract

There has been great awareness in the area of the development of biodiesel especially in the developing countries during the recent time. Significant research activities have been performed for its production and new development. Biodiesels can be the fuel of the future as it provides an option of economical, eco-friendly, alternative renewable energy source. In the present investigation sorghum bicolor seed oil and its methyl ester have been chosen to find out their suitability as petro-diesel. Experimental investigation has been made to find out the physico-chemical properties of sorghum oil. Laboratory scale quantities of sorghum oil biodiesel were produced through transesterification reaction using 100 g sorghum oil, 17% methanol (wt% sorghum oil), 1.0% sodium hydroxide catalyst at 60°C in 90 minutes. The experiments were triplicate and average results were evaluated. The obtained biodiesel was characterized as an alternative diesel fuel through series of ASTM and European organization (EN 14214) standard fuel tests. The transesterification process yielded 93.5% sorghum oil biodiesel. The sorghum oil biodiesel had 89.9% reduction of viscosity over its raw vegetable oil at 40°C. Higher specific gravity, pour point and cloud point were obtained and comparison has been carried out to petroleum diesel. Specific gravity values were found to be favorable with results for other vegetable oil biodiesel. Results obtained were found to be within limits set by various International standards for biodiesel.

Keywords: Biodiesel, physical and chemical properties, renewable fuel, transesterification.

Introduction

Currently, biodiesel is becoming popular as an environment friendly fuel. It has been used as an alternative for diesel fuel in the automotive industry, commonly known as No. 2 diesel. The advantage of this biofuel over the conventional diesel fuel includes high cetane numbers, low smoke and particulates, low carbon dioxide and hydrocarbon emissions; it is biodegradable and non toxic¹. Directly or blended edible or non-edible oil can be used in diesel engine but it can create problem in engine because of its high viscosity^{2,3}. Incomplete combustion of edible and non-edible oil produce high smoke thereby causing ring sticking and inefficient oil air mixing effects injector system performance⁴. Biodiesel comprises of monoalkyl esters of long chain fatty acids. It is produced using edible oil, non-edible oil and animal fats by acid or by base catalyzed transesterification with ethanol or methanol⁵. The significant efforts have been made for obtaining biodiesel by transesterification of oil obtained from *Jatropha curcas*⁶, soybean⁷, sunflower^{8,9}, cotton seed¹⁰, rapeseed¹¹, and palm¹². The ASTM-445 specification for viscosity at 40°C of centistokes is generally met by biodiesel and biodiesel blends. The reported viscosity of soy methyl ester is ranging from 3.8 to 4.1 centistokes at 40°C. Glycerin contamination will increase biodiesel viscosity which leads many other problems. Estimates of the surface tension of biodiesel suggest that it may be two to three times as great as that for diesel. The major problem of using biodiesel in diesel engine is higher viscosity and cloud point. High viscosity of edible oil, non edible oil and animal fats tends to cause problem when directly used in diesel engines¹³⁻¹⁶. The fuel droplet size

during injection is affected by the above properties. Higher NO_x emission may be due to larger droplets resulting from both viscosity and surface tension of Biodiesel. Transesterification of oil and fats using short chain alcohols, results in monoesters having viscosity that is closer to petroleum based diesel fuel¹⁷⁻¹⁹. The physical and chemical properties such as viscosity, density, flash point, cloud point, cetane number, and acid value etc. affect the biodiesel engine performance and emission. In turn, these properties are derived from the fatty acid composition and the properties of the individual fatty esters in biodiesel. Increase in chain length and decrease in unsaturation²⁰ of fatty compounds result in increase in cetane number, heating value and viscosity.

Many species of the genus sorghum (*Sorghum bicolor*, family poaceae), are found in tropical and subtropical countries; *Sorghum bicolor* is crop plant; eight species of sorghum are reported to occur in several part of India. The sorghum seed is comprised of 30-50% oil and oil extraction will not affect the food, feed and fodder needs of formers²¹. The fatty acid compositions of sorghum oil consist of palmitic acid, stearic acid, oleic acid, lenoliec acid, and lenolenic acid²¹. Thus, it attract as the starting oil for production of biodiesel fuel. The physical and chemical properties such as free fatty acid, viscosity, density, flash point, cloud point, cetane number, iodine value, saponification value and acid value of sorghum oil were studied and fatty acid composition of sorghum oil was determined and compared with *jatropha* oil.

Material and Methods

Transesterification Reaction: The reaction of triglyceride (oil/fats) with an alcohol in the presence of acidic or alkaline catalyst is the process of transesterification and it requires the reaction temperature to be below the boiling point of alcohol used, and reaction time should not be less than 30 minutes or more than 2 hours to form mono alkyl ester that is biodiesel and glycerol. It is a widely employed procedure to reduce the high viscosity of triglycerides. The transesterification reaction is shown in scheme 1.

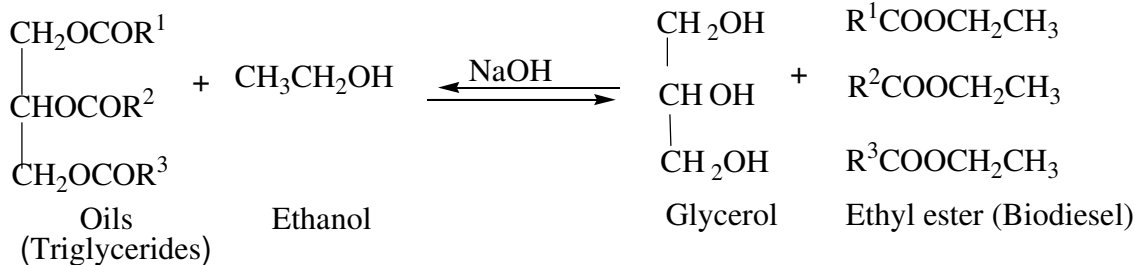
Chemical and Equipments: The sorghum oil used in the study reported herein is produced locally in India. The sorghum oil was purchased from local market Moga, Punjab, India. The methanol used (99% pure) is of analytical grade with boiling point of 78°C; while the NaOH used were of analytical grade and purchased from Aldrich Chemical Co. Ltd. Sodium sulfate, Potassium dichromate, Hydrochloric acid, Potassium iodide, starch, sodium thiosulphate, Phenolphthalein used were also an analytical grade product of Merck Co Ltd. The magnetic stirrer with hotplate, three necks round bottom flask, measuring cylinder, beaker, separating funnel, burette, density bottle, funnels, pet-bottle thermometers and measuring flask were used.

Experimental Procedure for Production of Biodiesel: The alkali catalyzed transesterification of sorghum oil was carried out in a laboratory scale setup. A 250 ml three necked round bottom flask was used as a reactor. Two necks were equipped with a condenser and thermometer, while the other neck used as an inlet for the reactants. The mixture was heated in the reactor and stirred well by magnetic stirrer with hotplate at the same speed for all experiment. In a typical experiment a known amount of sorghum oil is charged to a round bottom flask. A known amount of catalyst sodium hydroxide based on weight percent of oil is mixed in excess mole percent of methanol. The mixture of sodium hydroxide in methanol is added to the sorghum oil in the round bottom flask, while stirring the material of the flask. Required temperature is maintained by controlling the electrical heating till the reaction is completed. After complete addition of methanol sodium hydroxide solution,

samples are drawn at regular interval (10-20 minutes) to confirm the formation of methyl ester. The formation of methyl ester is checked by using thin layer chromatography (TLC) technique. After the completion of methyl ester formation, a known amount of sulfuric acid in methanol is added to the methyl ester to neutralize the sodium hydroxide present in the ester. The excess methanol present in the methyl ester is recovered by distillation with electrical heating and constant stirring. The amount of sorghum methyl ester is analyzed for acid value and the refined with sodium hydroxide solution to remove the free fatty acid (FFA). The transesterification reaction temperature was maintained at 60°C for 120 minutes keeping the molar ratio of oil to methanol at 5:1 and sodium hydroxide concentration 0.5(g) weight percentage of oil and percentage of excess methanol used is 200%. The refined sample was further cooled and centrifuged to remove residual soap. The pH level of the organic layer is measured and neutralized separately. The washed sample was further fried. Under optimal condition the yield of sorghum methyl ester from sorghum oil is about 92%. The reaction parameter such as methanol/oil molar ratio, percentage of excess alcohol, reaction time and temperature, concentration of catalyst were optimized for the production of sorghum methyl ester. Various fuel properties of sorghum oils and sorghum methyl esters were determined experimentally to ascertain their suitability as diesel fuel.

Results and Discussion

Composition of Fatty Acid: Sorghum grains oil contained high proportion of Linoleic acid with considerable amount of oleic acid palmitic acid, stearic acid, lenolenic acid and myristic acid as compared to Jatropha oil from literature in table 1. The ability of biodiesel to meet ASTM D 6751 standard criteria is dependent on the fatty acid composition^{22, 23}. The petro-diesel contained carbon chain length of 8 to 10 carbon atom are made from hydrocarbon while sorghum oil contained free fatty acid comprising 16 to 18 carbon atom²⁰. Cetane number is increased with increasing carbon atom that indicates the fuel quality for diesel engine²⁴. Branched chains and double bonds improve low temperature flow properties²³.



Scheme-1
 Transesterification of ethyl ester (biodiesel) production

Table-1
Fatty acid composition of sorghum oil as compared with Jatropha oil

Fatty acid	Structure	Formula	Sorghum oil	Jatropha oil
Myristic acid	C 14: 0	C ₁₄ H ₂₀ O ₂	0-0.1	Nd
Palmitic acid	C16 : 0	C ₁₆ H ₃₂ O ₂	10-14	4.2
Stearic acid	C18 : 0	C ₁₈ H ₃₆ O ₂	03- 06	6.9
Oleic acid	C18 : 1	C ₁₈ H ₃₄ O ₂	03-47	43.1
Linoleic acid	C18 : 2	C ₁₈ H ₃₂ O ₂	40-55	34.3
Lenolenic acid	C18 : 3	C ₁₈ H ₃₀ O ₂	0-0.1	Nd

Physical and Chemical Properties of Sorghum oil: The physical and chemical properties of crude sorghum oil were investigated for the production of biodiesel for use in diesel engine. The chemical and physical properties that were studied are following: kinematic viscosity, flash point, density, specific gravity, saponification value, iodine value and acid value. Fatty acid composition and physical and chemical properties of the sorghum oils were also determined and compared with jatropha oil given in table 2.

Table-2
Physical and chemical properties of sorghum oil and compared with jatropha oil

Properties	Sorghum oil	Jatropha oil
Kinematic viscosity	31.4	40.4
Density	0.9099	0.92
Specific gravity	0.9153	0.94
Flash point	225	167
Saponification value	181-191	195
Iodine value	108-122	101.7
Acid value	0.434	5.31

Characterization of Sorghum Methyl ester (Biodiesel): Standard test methods were used for determining physical and chemical properties of sorghum oil biodiesel. These standard values were calculated and compared with USA (ASTM D6751), Germany (DIN 51606), Indian (BIS) and European organization (EN 14214). The physical and chemical properties such as kinematic viscosity, flash point, pours point, cloud point, saponification value, iodine value, and acid value are given in table 3. The important physical and chemical experimental value of sorghum oil biodiesel such as kinematic viscosity, density, saponification value, iodine value, acid value and flash point of sorghum oil biodiesel were calculated by following equations.

Acid value: The number of NaOH required to neutralizing the free fatty acid present in 1.0 gm of the sample²⁵.

$$\text{Acid value} = \frac{5.61 \times T}{W}$$

Where; T = Volume in ml of 0.5N NaOH required for titration in ml. W = Weight in gm of sample taken.

Saponification value: A known quantity of oil is refluxed with an excess amount of alcoholic KOH. After saponification, the

remaining NaOH is estimated by titrating it against a standard acid²⁵.

$$\text{Saponification value} = \frac{28.05 \times (T_2 - T_1)}{W}$$

Where; T₂ = Volume in ml of 0.5N acid required for the blank.
 T₁ = Volume in ml of 0.5N acid required for the sample. W = Weight in g of the sample taken.

Iodine value: The most important analytical determination of oil is the measurement of its unsaturation. The generally accepted parameter for expressing the degree of carbon to carbon unsaturation of oil or their derivatives is iodine value. Iodine value is defined as g of iodine absorbed by 100g of oil. It is useful parameter in studying oxidative rancidity of triacylglycerols since, higher the unsaturation, greater is the possibility of rancidity²⁵.

$$\text{Iodine value} = \frac{12.7 (B - S)N}{\text{Weight of the sample (g)}}$$

Where; B = Volume in ml of standard sodium thiosulphate solution required for the blank. S = Volume in ml of standard sodium thiosulphate solution required for the sample. N = Normality of the standard sodium thiosulphate solution, W = Weight in g of the material taken for the test.

Kinematic viscosity: Kinematic viscosity of sample was measured with the help of Red wood Viscometer No.1. Time of gravity flow of fixed value (50 ml) of sample was measured. The experiment was performed at 38/40°C. Kinematic viscosity was calculated using the following formula.

$$\begin{aligned} V_k &= 0.24 t - 50/t \text{ for } t > 100 \\ V_k &= 0.26t - 179/t \text{ for } 34 < t < 100 \end{aligned}$$

Where: V_k = Kinematic viscosity, cS, t = time of flow.

Specific gravity: Specific gravity was measured using the standard method (Indian standard is (1964). Capillary stopper relative specific gravity bottle (pycnometer bottle) of 50 ml capacity was used to determine specific gravity of biodiesel.

$$\text{Specific gravity at } 30^\circ\text{C}/30^\circ\text{C} = \frac{A - B}{B - C}$$

Where: A= Weight in gm of the specific gravity bottle with sample at 30°C, B= Weight in gm of empty specific gravity bottle, C= Weight in gm of specific gravity bottle with distilwater at 30°C.

Density: Density was measured using the standard method (BIS, 1972), capillary stopper relative density bottle of 50 ml capacity were used to determine density of biodiesel. Density was calculated using the following equation.

$$\text{Density} = \frac{W_3 - W_1}{W_2 - W_1} \times \rho_{H_2O}$$

Where; W1 = Weight of empty bottle, W2 =Weight of empty bottle with water, W3 = Weight of empty bottle with sample, ρ_{H_2O} = Density of water.

Flash Point: Flash point of a fuel is defined as the temperature at which it will ignite when exposed to flame or spark. The flash point of sample was determined by Pensky Martens Flash Point apparatus.

Conclusion

This study revealed that biodiesel could be produced successfully term the sorghum oil by alkali- catalyzed transesterification. The effects of different parameters such as reaction time, temperature, catalyst concentration and reactant ratio on the biodiesel yield were analyzed. The good combination of the parameters were found as 5:1 molar ratio of oil to ethanol, 0.5% NaOH catalyst, 60°C reaction temperature and 90 minutes of reaction time. The viscosity of sorghum oil reduces substantially after transesterification and is comparable to petro-diesel and the physical and chemical properties of biodiesel produced conform to available standards.

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Table-3
Comparison of fuel properties of Sorghum biodiesel and Petro-diesel

Properties	Sorghum biodiesel	Petro-diesel	Biodiesel	Biodiesel
		ASTM D0975	ASTM D6751	EN 14214
Density at 30°C (kg/m ³ or g/gL)	0.871	0.876	0.875- 0.90	0.86-0.90
Viscosity at 40°C	3.237	1.9-4.1	1.9-6.0	3.5-5.0
Specific gravity (kg/m ³ or gm/mL)	0.875	0.850	0.88
Flash point °C	155	60-80	100-170	>120
Pour point	06	-35 to -15	-15 to 16
Cloud point	08	-15 to 5	-3 to 12
Acid value (mg NaOH/g)	0.31	0.35	<0.8	<0.5
Iodine value	101
Saponification value (mg NaOH/g)	165
Ash content (%)	Nil	0.01	<0.02	<0.02
Water content (%)	Nil	0.02	0.03	<0.05

ASTM= American Society for Testing and Materials, EN= European Organization

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