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Characteristics and Composition of Soyabean Oil Seed from India by Alkali-Catalyzed Transesterification and its Potential as Biodiesel Feedstock

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Over consumption of fossil fuels turns the world's pivot in to finding of a new possible substitute fuels. In this category, biodiesel get major attention as an alternative fuels. Production of biodiesel are dependent on various feedstock sources like algae, vegetable oil, food waste, animal fat, fatty acids containing seeds and grease *etc*. In the present study soybean seed oil and its methyl ester have been taken to find out their possibility as petroleum-diesel fuels. Experimental analysis has been made to detect the physico-chemical properties of soybean seed oil. Biodiesel were produced through transesterification reaction from soybean seeds oil. In transesterification process about 90 % yield of biodiesel were produced. The obtained biodiesel was characterized as an alternative diesel fuel through series of ASTM and BIS. Higher specific gravity, flash point, fire point, iodine point, pour point and cloud point were obtained and comparison has been carried out to petroleum diesel. GC-MS analysis of the esters of fatty acids shows that the overall percentage of methyl ester is more than the ethyl ester.

Keywords: Biodiesel, Methyl ester, Fatty acid, Transesterfication, GC-MS.

INTRODUCTION

Biodiesel is a mixture of monoalkyl esters of long chain fatty acids, which are mainly produced from vegetable oils, seeds of fatty acids and animal fats. Biodiesel is produced by the acid or by base catalyzed transesterification reactions of vegetable oils, seeds of fatty acids and animal fats with ethanol or methanol [1]. Without any modification of the engine biodiesel can be used in diesel engines. The remarkable endeavour have been formulate for procure biodiesel by acid-base catalyzed transesterification of oil obtained from jatropha, soyabean, sunflower, food waste, grease, cotton seed, rapeseed, sorghum and palm *etc*. During the acid-base catalyzed transesterification reaction the presence of glycerine increases the viscosity of biodiesel which may leads other problems like higher viscosity. Transesterification of oil and fats using short chain alcohols, results in monoesters having viscosity that is closer to petroleum based diesel fuel. The physico-chemical properties such as viscosity, density, flash point, fire point, cloud point, pour point, iodine value and acid value *etc*. affect the biodiesel engine performance and emission.

There are many advantages of biodiesel *e.g.*, it is eco-friendly, non-flammable and nontoxic, its renewable, safe for traditional diesel engines, good performance of engine, reduces the discharge of exhaust system, perceptible gas, toxic fumes and odour's. The emerge of biodiesel has during the last few years. Generally the production of biodiesel directly dependence upon feedstock and the large per cent of the direct biodiesel production costs, including capital cost and return cost dependence upon the feedstock costs [2]. Soyabean constitute of phytic acid, dietary minerals and B vitamins. Soyabean seed oil comprises of saturated and unsaturated fatty acids. The major unsaturated fatty acids in soyabean seed oil triglycerides are the poly unsaturated α-linolenic acid, linoleic acid and the mono unsaturated oleic acid. Soyabean seed oil also contains the saturated fatty acids *i.e.*, stearic acid and palmitic acid [3]. Oil extraction from soybeans and biodiesel production was performed using conventional and ultrasonication methods [4]. Soyabean oil seed biodiesel can also be prepared by the hydrotalcite was synthesized using the co-precipitation method with Mg/Al molar ratio of 3.0 and calcined at 450 °C (723 K), under Ar flow, for 6 h. The obtained solid was characterized by X-ray powder diffraction and temperature-programmed desorption of CO_2 (CO_2-TPD) [5] but there is no characterization of methyl ester of soyabean seed oil were found. The main objectives of this study are the enhancing the production of methyl ester and their characterization.

EXPERIMENTAL

Collection of soyabean seeds: Soyabean seeds were collected from the local market of India. The seeds were cleaned and dried in the presence of sunlight.

Extraction of oil from soyabean seeds: For the preparation of oil, the seeds should be dried in the presence of sunlight for several hours. During this process cell of the seeds oil containing break down and improve the extraction process. The seeds were finally grinded to the extraction process. In the present study the oil can be extracted from the seeds by two methods *i.e.*, cold percolation method and Soxhlet method.

In cold percolation method [6], 10 g of fine grained powder of seed mixed with 6.6 g of glass power and sodium sulphate into the 500 mL vial. Add the 300 mL of carbon tetrachloride into vial. The solution was kept into rotatory shaker for 24 h. Now filter the solution and the filtrate containing the oil with carbon tetrachloride. Evaporate the carbon tetrachloride at 60 °C. After the evaporation the oil containing vial kept into the desiccator for cooling and weigh the oil.

In other method mix the 25 g of fine grained powder and 250 mL of *n*-hexane in Soxhlet apparatus. Filter the extract and also extracted the lipid from solvent. Now evaporate the *n*-hexane at 40 °C from the filtrate and store the extracted oil at -2 °C for the degradation.

Conversion of extracted seed oil into methyl ester by transesterification reactions: Pour the known amount of oil into round bottom flask and add the known amount of potassium methoxide (KOH + excess amount of methanol) into the round bottom flask, while continuous stirring of the flask. Maintain the temperature of the reaction mixture at 55 °C for 1 h. After the complete addition of potassium methoxide, some of the sample drawn from the flask to confirm the formation of methyl ester (by titration method and TLC method). After the formation of methyl ester the reaction mixture allow to separate into the phase by standing for overnight in a separating funnel and add 5 mL CH_3COOH to this reaction mixture to neutralize the KOH present in the ester. Glycerol and biodiesel are two phase observed in separating funnel so as to remove the glycerol from the biodiesel. Now wash the biodiesel with water and allow standing for overnight in separating funnel. The dense mixture was carefully removed from the bottom of the funnel. Removed the moisture content from the biodiesel by placing in oven for 1 h.

Characterization of methyl ester: Standard test methods were used for determining the physical and chemical properties of the soyabean seed oil biodiesel such as kinematic viscosity, flash point, fire point pours point, cloudpoint, saponification value, iodine value and acid value. These standard values were compared with ASTM D-6751 [7] are compared with jatropha oil biodiesel [8-12].

Oil content: Oil content is represented in terms of percentage of oil in the dry soyabean seeds powder [13].

$$\% \text{ of Oil} = \frac{W_2 - W_1}{W} \times 100$$

Density: Density of biodiesel was determined by the capillary stopper relative bottle or Pyknometer by using the following formula:

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

where, W_1 = Weight of the empty bottle; W_2 = Weight of the empty bottle + water; W_3 = Weight of the empty bottle + biodiesel; ρH_2O = Density of water at 30 °C.

Specific gravity: Specific gravity of the biodiesel was determined by the specific gravity bottle at 30 °C by using the following formula:

$$\text{Specific gravity} = \frac{A - B}{C - B}$$

where, A = Weight of the specific gravity bottle with biodiesel (g); B = Weight of the empty specific gravity bottle (g); C = Weight of the specific gravity bottle with water (g).

Flash point: Flash point of the biodiesel was determined with the help of Pensky Marten's apparatus. Flash point is the lowest temperature at which the oil gives off enough vapours that ignite for a moment when tiny flame is brought near it.

Cloud point and pour point: This characteristic was determined by cloud point and pour point apparatus. Cloud point is the temperature at which oil becomes cloudy or hazy when oil is cooled at specified rate. Pour point is the temperature at which oil just ceases to flow.

Kinematic viscosity: Kinematic viscosity was determined with the help of Redwood viscometer No. 1 at 40 °C by using the following formula:

$$\text{Kinematic viscosity (cst)} = \frac{0.26t - 171}{t}$$

Saponification value: The saponification value of oil is the number of milligrams of potassium hydroxide required to saponify 1 g of the oil or fat under the specified conditions. The soyabean seed oils are esters of fatty acids and glycerol they react with KOH to form the potassium salts of fatty acids. The saponification value of oil is determined by refluxing a known weight of the sample with a known excess of standard alcoholic KOH solution and determining the alkali consume by titrating the unreacted alkali.

$$\text{Saponification value} = \frac{\text{Normality of KOH used} \times V_2 - V_1 \times \text{eq. wt. of KOH}}{\text{Weight of sample taken (g)}}$$

Acid value: The acid value is number of milligrams of sodium hydroxide which are needed to neutralize the acidity present in 1 g of material.

$$\text{Acid value} = \frac{\text{Volume of 0.1 N KOH used in mL} \times 5.6}{\text{Weight of sample taken}}$$

Iodine value: The iodine value is a measure of the unsaturation of oils. It is expressed in terms of the number of grams iodine absorbed 100 g of sample. Iodine value can be calculated as follows:

$$\text{Iodine value} = \frac{(B - S) \times N \times 12.69}{\text{Weight of the biodiesel (g)}}$$

Analysis of fatty acid methyl esters: Methyl esters of fatty acids were analyzed by GC-MS [14].

RESULTS AND DISCUSSION

Physico-chemical analysis: The physico-chemical results of biodiesel sample are given in Table-1.

The oil content of the soyabean is 15.85 % which is less than the jetropha oil non-edible seed. The oil content indicated that the soyabean seeds are suitable as an edible vegetable oil feedstock in oleochemical industries (biodiesel, fatty acids, *etc.*). The density of the soyabean biodiesel is 0.8534 is less

TABLE-1
PHYSICO-CHEMICAL DATA OF SOYABEAN BIODIESEL AND STANDARD ASTM D6751

Properties	Soyabean biodiesel	ASTM D6751
Oil content	15.84 %	–
Density (at 30 °C)	0.8534	0.875-0.90
Specific gravity (at 30 °C)	0.8497	0.88
Kinematic viscosity (at 40 °C)	8.2	1.9-6.0
Acid number	1.3621	< 0.8
Saponification value	3.6235	–
Iodine value	48.7296	–
Flash point	169 °C	100-170 °C
Cloud point	-1 °C	-15 to 16
Pour point	-4 °C	-3 to 12

than the jetropha biodiesel 0.930 g/mL. Density of oil is decreases with increases molecular weight, increases with unsaturation. Specific gravity of the soyabean biodiesel is 0.8497 which is likely to be similar as jetropha biodiesel 0.94. Kinematic viscosity of the soyabean biodiesel is much more than the other biodiesel. High viscosity of the oil seed are not fit for the directly use as an engine fuel. Iodine value of soyabean biodiesel is 48.7296 which is less than ASTM D6751 and jetropha. Lower iodine value indicates that the less unsaturation of fats and oil. Saponification value of the studied soyabean biodiesel were 3.6 which is less than the jetropha oil, indicated that normal triglycerides are not useful in production of oil industries (likely shampoo, soap). The acid value of soyabean biodiesel is 1.3621 which is higher than the ASTM D6751, indicates the quality of the fatty acid present in biodiesel. The higher value of the fatty acid present in oil affected the esterification process and to reduces the values. Flash point and Pour point of the biodiesel is likely similar to ASTM D6751.

GC-MS analysis of the methyl ester: On the analysis of biodiesel by GC-MS around 90 % of methyl ester formed and the analysis of spectra different constituents of methyl ester and their derivatives are produced (Fig. 1). During transesteri-

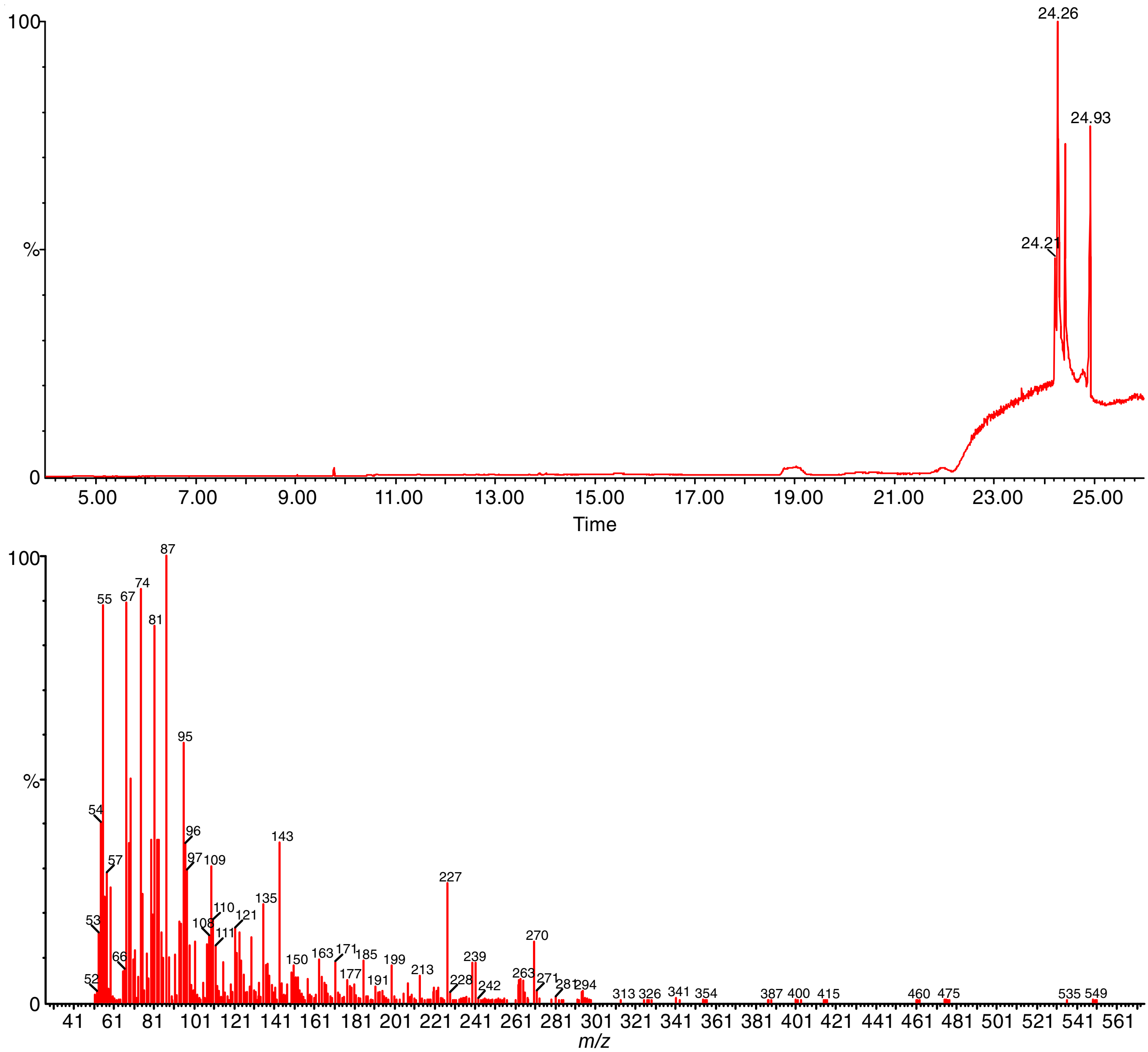

Fig. 1. GC-MS of analysis of soyabean biodiesel

fication process ethyl ester was comparatively less than that of formation of methyl ester (Table-2).

TABLE-2

Compound name	m.w.	m.f.
10,13-Dimethyl tetradecanoic acid	270	$C_{17}H_{34}O_2$
Methyl 5,12-octadecadienoate	294	$C_{19}H_{34}O_2$
17-Octadecynoic acid, methyl ester	294	$C_{19}H_{34}O_2$
Methyl 5,13-docosadienoate	350	$C_{23}H_{42}O_2$
6,11-Eicosadienoic acid, methyl ester	322	$C_{21}H_{38}O_2$
12-Tridecynoic acid, methyl ester	224	$C_{14}H_{24}O_2$
Cyclopropanebutanoic acid, 2-[[2-[[2-[(2	374	$C_{25}H_{42}O_2$
Methyl 12,13-tetradecadienoate	238	$C_{15}H_{26}O_2$
10-Undecynoic acid, methyl ester	196	$C_{12}H_{20}O_2$
3-Cyclopentylpropionic acid, dodec-9-yny	306	$C_{20}H_{34}O_2$
Cyclopentaneundecanoic acid, methyl ester	268	$C_{17}H_{32}O_2$
13-Tetradecynoic acid, methyl ester	238	$C_{15}H_{26}O_2$
10-Undecynoic acid, methyl ester	196	$C_{12}H_{20}O_2$
14,17-Octadecadienoic acid, methyl ester	294	$C_{19}H_{34}O_2$
Methyl 11-cyclohexylundecanoate	282	$C_{18}H_{34}O_2$
8,11,14-Eicosatrienoic acid, methyl este	320	$C_{21}H_{36}O_2$
Octadecanoic acid, 9,10-dihydroxy-, meth	522	$C_{23}H_{36}O_6F_6$
Hexadecanoic acid, methyl ester	270	$C_{17}H_{34}O_2$
13,16-Octadecadiynoic acid, methyl ester	290	$C_{19}H_{30}O_2$
13,16-Octadecadienoic acid, methyl ester	294	$C_{19}H_{34}O_2$

Conclusion

This study shows that the biodiesel could be produced successfully from the soyabean seeds by alkali-catalyzed transesterification process. The soyabean seeds are the good source and potential of renewable biodiesel. GC-MS study confirms the formation of methyl ester. But the production cost is affected on the formation of soyabean biodiesel. The physical and chemical properties of the biodiesel produced confirm to available standards. In this viscosity, acid number of the biodiesel is comparatively higher than the standards. Furthermore work may be carried out to characterize the soyabean biodiesel to reduce the viscosity, acid number and also create a gentle relationship between ester formation and the other important parameters such as viscosity, lubricity and stability for the future fuels.

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