International Journal of Research in Engineering, Science and Management
Volume-3, Issue-1, January-2020
www.ijresm.com | ISSN (Online): 2581-5792

Production and Optimization of Biodiesel by Transesterification of Chia Seed Oil

Vaibhav V. Mahishi[1], Sourabh Kulkarni[2]
[1]*Student, Department of Mechanical Engineering, Basaveshwar Engineering College, Bagalkot, India*
[2]*Student, Department of Mechanical Engineering, KLE Technological University, Hubballi, India*

***Abstract*: This study investigated production of biodiesel from Chia seed oil. Due to the environmental problems caused by the use of fossil fuels, considerable attention has been given to biodiesel production as an alternative to petro-diesel. Indian refineries import over 70% of their crude oil feedstock which affects the Indian Foreign Reserves. This weakens the value of Indian National Rupee (INR). Biodiesel is an eco-friendly, alternative fossil fuel synthesized from domestic renewable resources. The general method to produce biodiesel is by trans-esterification of oil with methanol in the presence of either base or strong acid catalysts. This work describes the fuel properties of biodiesel, production process and the most important variables that influence the trans-esterification reaction.**

***Keywords*: Biodiesel, Chia seed oil, Transesterification**

1. Introduction

The importance of alternate fuel for internal combustion engine needs no emphasis in the present situation where renewable and low polluting fuels are drawing a lot of attention. Various bio-fuel energy resources explored include biogas, biomass, Vegetable Oils and so on. Of these, vegetable oil plays an important role as an alternative fuel for diesel engine. Oxygenated compounds blended with diesel and vegetable oil plays an important role to achieve stringent emission regulations. According to the National Wasteland Development Board, Ministry of Rural Areas and Employment Board, Government of India, 175 million hectors (mha) of degraded land is available in India (53% of India's total land mass), of which 130 mha is totally degraded land and 45 mha is forest. About 75 mha of wasteland in the country can be used for growing oil seed crops as avenue tress.

The worldwide worry about the protection of environment and the conservation of non-renewable natural resources, has given rise to development of alternate sources of energy as substitute for traditional fossil fuels. Thus, focusing on alternative sources of new and renewable energy such as hydro, biomass, geothermal, hydrogen, wind, solar and nuclear are of prime importance. Technologies for using solar energy, wind energy, hydropower, energy from biomass and geothermal energy are some of the promising ones. Biodiesel is one of the alternatives for fossil fuels.

Currently, more than 95% of biodiesels are produced from oil feedstock like Soya bean oil, Sunflower oil, Niger oil, Rapeseed oil, Palm oil, Sesame oil and Linseed oil due to this there is a considerable imbalance in the human food chain versus fuel. This makes biodiesel economically unfeasible as compared to fossil fuels. To avoid these situations, other oil seeds need to be used for commercial production of biodiesel.

2. Methodology

The preliminary stage involves literature reviews on the previous studies of various biodiesels which are used around the world. In brief, various available biodiesels in India were studied. The second stage involves selection of a suitable oil for the production of biodiesel. After a short survey involving various criteria, we have chosen chia seed oil as the suitable oil for production of biodiesel. The third stage involves determination of Free Fatty Acid (FFA) in the oil, based on the content of FFA in the oil, suitable process is chosen to convert it into biodiesel (single stage/double stage). The fourth stage involves the conversion of chia seed oil to biodiesel, and optimization of chia seed oil biodiesel. The fifth stage involves the study of chemical and physical properties of chia seed oil biodiesel.

A. Production of Biodiesel

1. Measure 1 liter of oil.
2. Transfer this oil into a 3-neck flask provided as part of laboratory set.
3. Place the 3-neck flask on magnetic stirrer.
4. Put the magnetic pellet into the flask.
5. Fix the reflex condenser to the central neck of the 3-neck flask. Connect water pipe line to the condenser and checkup for water circulation from the tap to condenser and outlet.
6. Connect the magnetic stirrer the electric connection.
7. Switch on the magnetic stirrer switch.
8. Set up the heating control to 60° C.
9. Adjust the speed between 600-800rpm to get homogeneous/uniform heating of the oil.
10. Take the glass thermo-well and add some oil into the thermo-well. Insert the thermo-well into the sided neck of 3-neck flask. Stick the thermometer into the thermo-well and note the temperature.
11. Take 300ml methanol per 1 liter of oil in a 500ml

capacity beaker.

12. Weigh a required NaOH based on FFA% determined earlier for chia seed oil (i.e. for 0 FFA 3.5 grams of NaOH) and add to methanol. Stir well and this mixture is called “Methoxide” mixture.
13. When the temperature reaches 63ºC, add the Methoxide mixture slowly to the hot oil into the 3-Neck flak through the loading opening neck and maintain the speed at 600 rpm. Close the opening with a stopper.
14. Maintain the temperature at 60-63ºC (Total reaction time is 2 hours). (Boiling point of methanol is 64.07ºC).
15. Run the process for 30 minutes and observe the color of mixture turns from turbid orange to transparent chilly red (In the reaction vessel).
16. After the first 30 minutes drain sample and allow it to settle. Two distinct layers will be obtained indicating the chemical reaction is proceeding in right direction. The bottom layer is glycerin.
17. Run the process for another 1½ hour.
18. After 1½ hour, drain one more sample and observe for glycerin separation. Then switch off the power and remove the reflux condenser.
19. Transfer the mixture into a separating funnel and allow it to settle for two hours.
20. After two hours, glycerin will settle at the bottom and bio-diesel will suspend as the top layer.
21. Drain the glycerin layer from the separating funnel from the bottom, carefully, and store it.
22. Allow the biodiesel layer to settle for another ½ hour and observe if any further glycerin content settles. If glycerin layer is seen, then drain the same.

B. Washing of Biodiesel

1. Transfer the methanol after the methanol recovery into the plastic “Washing Funnel” specially assembled for this purpose.
2. Spray 300ml of warm water slowly into biodiesel without any agitation (Water 40oC).
3. Allow to settle for 15 minutes. A bottom layer of soap water will gradually start to form.
4. Drain the bottom layer (soap water) carefully.
5. Repeat the above procedure (300ml water) for five times and shake vigorously and allow it to settle for 1 hour and drain the soap water.
6. Continue to wash with the warm water till the biodiesel reaches the 7 pH.

C. Drying of Biodiesel

1. Transfer the washed biodiesel from the "Washing Funnel" to the 1liter beaker. Add the magnetic pallet and adjust the rotation to suitable speed.
2. Heat the biodiesel to a temperature of 100oC (Moisture evaporates).
3. Allow the biodiesel to cool gradually.
4. Measure the finished biodiesel. Record the quantity.
5. Store it in a clean, dry container.

D. FFA Calculation

FFA Content:

$$= \frac{28.2 \times (Normality\ of\ NaOH) \times (Titration\ value)}{(Weight\ of\ the\ oil)}$$

$$= \frac{28.2 \times 0.1 \times 4}{10} = 1.12 \text{ amount of FFA}$$

The amount of FFA obtained is 1.12.

Since the value of FFA obtained is less than 4, we concluded that it is single stage base catalyzed.

3. Results and Discussions

Table 1
Properties of Chia Seed Oil

S. No.	Properties	Chia seed Oil
1	Saponification Value, mg of KOH/g	193.01
2	Specific Gravity, at 30 ^{O}C	0.92
3	Density, kg/m^3	923
4	Flash point, ^{0}C	240
5	Acid Value, mg of KOH/g	2.05

A. Effect of methanol to oil ratio

The methanol to oil ratio is one of the most important variables affecting the ester yields. Fig. 1. Shows the effect of methanol oil ratio on yield of biodiesel at different catalytic concentrations. It was observed that the yield of biodiesel increased with increase in methanol to oil ratio. This result is comparable to the reported values. At higher oil ratio, more triglyceride would have reacted. However, an excess of methanol interferes with separation of glycerin because there is an increase in solubility. When glycerin remains in solution, it helps drive the equilibrium back to the reverse direction,

Table 1
Comparison of Properties of Chia Seed Methyl Ester and Neat Diesel

S. No.	Properties	Chia seed Methyl Ester	Neat Diesel	Standards Protocol (IS:1448)
1	Specific Gravity, at 37 ^{O}C	0.87	0.83	IS:1448 (P:32)
2	Density, kg/m^3 at 37 ^{O}C	870	830	IS:1448 (P:32)
3	Kinematic Viscosity, at37 ^{O}C	5.1	3.05	IS:1448 (P:25)
4	Saponification Value, mg of KOH/g	174.86	_	IS:1448 (P:55)
5	Iodine Value, gm/1000 gm of oil	96.12	_	
6	Cetane Number	55.88		IS:1448(P:09)
7	Flash point, ^{O}C	164	56	IS:1448 (P:66)
8	Fire point, ^{O}C	167	65	IS:1448 (P:66)
9	Gross calorific value (kJ/kg)	38,952	44,000	IS:1448 (P:06)

lowering the yield of esters. A methanol to oil ratio 3.5:10 and above resulted in 3 layers in the separating funnel viz. bottom layer of glycerin, middle layer of esters and top layer of unreacted methanol. The percentage increase in Biodiesel yield decreased with increase in molar ratio. The methanol to oil ratio of 3:10 gave the maximum yield of 96%.

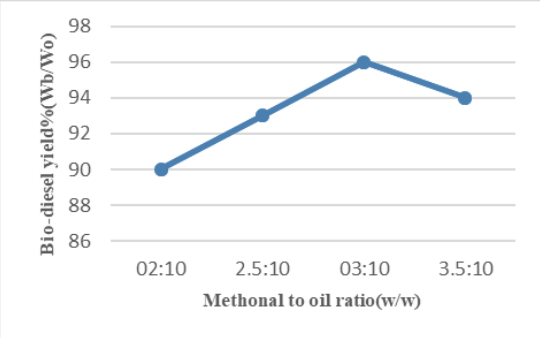

Fig. 1. Effect of molar ratio (methanol to oil) on biodiesel yield at different NaOH concentration keeping other process parameters constant

B. Effect of catalyst concentration

Transesterification of chia seed oil was carried out with Sodium Hydroxide (NaOH) as a catalyst at a concentration 0.4-1.4% in steps of 0.4%. Fig. 2. shows the yield of biodiesel vs NaOH concentration at different methanol-oil ratio. The lower catalytic concentration viz. 0.4-0.8% of NaOH was insignificant to catalyze the reaction to completion. It was found that the biodiesel yields increased as the amount of catalyst concentration increased from 0.8% to 1.2% for the entire methanol-oil ratios understudy. With the increase in concentration of catalyst viz. beyond 1.2% NaOH, there was decrease in the yield of biodiesel. The decrease in the yield of biodiesel may be due to formation of emulsion, increasing viscosity and leading to the gelation. The NaOH concentration of 0.8% gave the maximum yield of 96%.

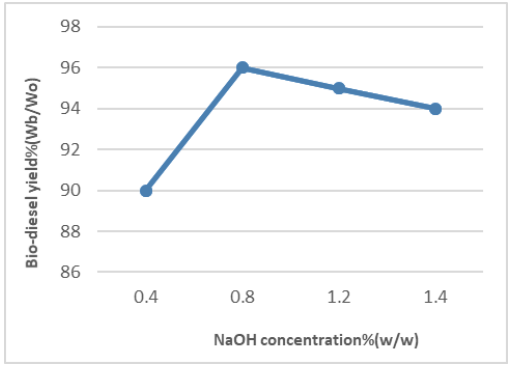

Fig. 2. Effect of NaOH concentration on biodiesel yield for different NaOH concentration keeping other process parameters constant

C. Effect of reaction temperature

Catalytic transesterification of chia seed oil is normally investigated close to the boiling point of methanol. Experiments were carried out at different temperatures such as 50, 60 and 70°C with 0.8% NaOH as a catalyst and a reaction time of 120min. From Fig. 3. It was observed that temperature had positive effect on transesterification of chia seed oil for 50, 60 and 70°C while for 45°C no significant effect was seen. The biodiesel yield follows an increasing trend with increase in reaction temperature up to 60±1°C, this may be attributed to the fact that higher reaction temperature helps in faster settlement of glycerol. The biodiesel yield decreases after a reaction temperature of 60±1°C. The reason may be due to saponification of the triglycerides before the completion of transesterification.

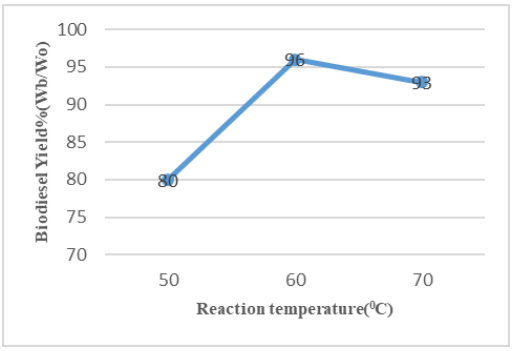

Fig. 3. Effect of reaction temperature on biodiesel yield for different temperature keeping other process parameters constant

4. Conclusion

The objective of this study was to characterize Chia seed oil, Chia seed oil biodiesel, and properties and how the properties changed when the oil is treated with varied parameters and also compare it with neat diesel oil. Chia seed oil could be trans esterified. A two-stage transesterification process has been studied which comprised of alkali transesterification and post treatment. The alkali catalyzed transesterification with 7gm of NaOH at 60±1 ^{0}C for 120 min at 03:10 methanol to oil ratio(w/w) this combination giving optimum reaction conditions for alkali transesterification of chia seed oil, followed by thrice gentle washing of the Bio-diesel with distilled water at 60^{0}C. The kinematic viscosity and specific gravity of the chia seed oil has reduced to a great extent by the transesterification process and calorific value has slightly increased. The biodiesel obtained by means of transesterification, given there is no un-reacted oil and glycerol, is suitable for use in diesel engines.

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