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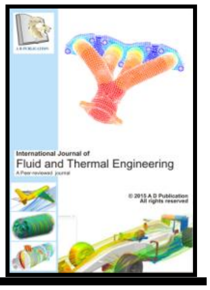

Design and development of small scale biodiesel production unit

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A B S T R A C T : As biodiesel is produced from renewable resources and it has environmental benefits, it's become more fascinated nowadays. In the present work non-edible oils which is available in plenty in India, which possess high calorific value and is under-utilized namely Leptadenia Reticulata (LR) has been identified for the production of biodiesel. Sodium Methoxide (CH3ONa) was used as a catalyst to assess its technical feasibility and process conditions for transesterification of the oils. A dual step process has been used for biodiesel preparation from high FFA containing materials. The first step of the process is to reduce the FFA content in the oil by esterification with methanol catalyzed by acidic catalyst, followed by the transesterification process, in which the triglyceride present in pre-treated oil is converted into methyl esters in presence of a solid base catalyst. Sequencing of process has been identified to produce biodiesel from LR oil.

Keywords: Biodiesel, trans esterification, leptadenia reticulate (LR), free fatty acid

1. Introduction

Increasing energy demand will pose challenges to security of supply as resources are scattered around the globe. Biofuels help to enhance and safeguard energy security by reducing the world's reliance on fossil energy sources. Bio-diesel is a clean burning alternative fuel for diesel engines which produced from domestic, renewable resources such as vegetable oils or animal fat. Biodiesel is domestically produced, non-toxic and renewable fuel source. Bio-diesel can be used, in any concentration with diesel fuel, in existing diesel engines with no or little modification. First generation biodiesel is prepared by using edible oil such as Sunflower oil, Palm, Cotton seed oil, Soybean oil, Almond oil etc.; however, usage of edible oil to produce bio-diesel is not suitable for India because it ultimately adversely influenced food security of the nation. Second generation biodiesel is produced by using non edible oil such as Jatropha oil, Mahua oil, Neem oil, Jojoba oil, Waste cooking oil etc. Since these non-edible oils have the great potential make biodiesel, they are successfully utilized to produce biodiesel.

This paper presents the details of development of a biodiesel from Leptadenia reticulata oil, a non-edible vegetable oil. Leptadenia reticulata belongs to a genus of a family Apocynaceae and subfamily Asclepiadoideae. Its Common names are Jivanti, Dodi, Jivantica, Radarudi (Gujarati), Hiranvel (Marathi), Bhadjivai (Bengali), Hiriyahalle (Kannada). Leptadenia reticulata is found in India, Sri Lanka, Myanmar and Mauritius. In India, it grows in rain fed parts of Gujarat, Punjab and Uttar Pradesh. The flowering season of this plant is from June to August. The flowers are small and greenish yellow in color. The seeds of this plant contain 40-43% oil. Raw Leptadenia reticulata oil contains nearly 16-18% of free fatty acid (FFA).

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Biodiesel can be produced by various conventional methods such as, dilution, transesterification, pyrolysis, hydrodynamic cavitation and micro-emulsions. Among all the methods, transesterification is the regularly used method to reduce mainly the FFA, viscosity, density, flash point, etc. In this process, the oil is reacted in the presence of alkali or acidic catalyst with an alcohol to give corresponding alkyl ester of Fatty Acid mixture. Usually, Alkali catalyzed transesterification process is used because Alkali catalyzed is extremely fast compared to acid catalyzed for transesterification.

Charpe and Rathod[1] produced biodiesel from waste sunflower frying oil by using enzymatic trans esterification process in which methanol used as an alcohol in the presence of P. fluorescens enzyme catalyst. Due to low cost the researchers used waste sunflower frying oil as raw material and P. fluorescens as catalyst because of its higher conversation rate. They got highest 63.84 % yield at 45^0C temperature and 3:1 molar ratio of alcohol to oil. Kumar and Babu[2] first removed moisture content from Mahua oil by heating and then reduced the fatty acid concentration of the oil by acid esterification process using H_2SO_4 as catalyst. Then, they followed the normal transesterification process using methanol in the presence of NaOH as a catalyst and attained a maximum yield of 85%. Sharma and Singh[5] produced biodiesel from mixture of two non-edible oils, which is karanja and mahua, in 1:1 volume ratio. Due to The higher FFA content of the above said two straight vegetable oils, they are forced to use two step reactions. The first step was acid esterification, which used H_2SO_4 as acidic catalyst, to reduce the FFA content within the limit. The second step was alkaline transesterification, which used KOH as alkali catalyst, to convert pre-treated oil to methyl esters of Fatty Acid mixture or biodiesel. In both reactions methanol was used as alcohol because it was less costly and the reaction was also faster. They got maximum yield of 94 % at 50^0C reaction temperature and 8:1 molar ratio of alcohol to oil.

2. Seed for Biodiesel

Leptadenia reticulate (LR) oil was purchased from local oil mill of Naswadi Taluka of Chhota Udaipur district (Gujarat state, India). The free fatty acid content, Acid value, water content, flash point, pour point, viscosity, density, water contain, calorific value of the oil were tested & find out in fuel testing laboratory according to Indian Standard (IS-15607) and these values are shown in Table 1. Apart from LR oil, Methanol(99.9%), Sulfuric acid, sodium methoxide, Brine solution were used during the biodiesel production in present work.

Table 1. Leptadenia reticulata oil properties

Sr. No	property	unit	value
1	Acidic value	-	34.8
2	Calorific value	kcal	6500
3	density	gm/cm^3	0.89
4	Flash point	^{0}C	245
5	Free fatty acid contain	%	17.5
6	Pour point	^{0}C	5
7	viscosity	cps	39.9
8	Water contain	%	0.22

3. Procedure adopted for Biodiesel production

FFA value of oil plays a vital role in the transesterification process. If the free fatty acid content of the oil is greater than 2%, it is necessary to reduce FFA value of oil by pre-treatment (acid esterification) before base is transesterification carried out, because the major problems arise due to high value of free fatty acid

(FFA) such as (1) more amount of catalyst required which leaded to higher cost. (2) Soap is formed during washing process which reduces the quality of finished product. (3) Formation of water as by-product that lead to retard the main reaction. (4) The FFA is not completely converted into Biodiesel, which consequently reducing the yield and increase the acid value of biodiesel.

3.1 Preheating of raw LR oil

Preheating process is done to remove moisture content from raw oil. In this process 200ml of LR oil is taken in a necked round bottomed flask for heating at 110^0 C for 20 minute as shown in figure 1.

3.2 Esterification reaction process

The etherification was carried out to reduce FFA content of LR oil within limit. 100 ml LR oil, 45 ml methanol and 1% w/w sulphuric acid were taken in necked round bottle flask equipped with water – cooled condenser and a magnetic stirrer. The whole reaction mixture was vigorously stirred at 600 rpm for 2 hr under reflux condition at 60^0 C temperatures. After the reaction, the reaction mixture was poured into a separation funnel and was allowed to settle for 8 hr. After the separation process, two layers was separated out, upper layer was esterified oil while bottom layer known as residues which contain excess methanol with water. Then the upper layer was collected and washed with brine solution for 2-3 times.

3.3 Transesterification reaction process

In this process the acidic esterified LR oil was used for production of LRME.100 ml of esterified LR oil was poured into the reaction flask and heated to 60^0 c temperature. Then 35 ml methanol and 1.5% w/w sodium methoxide were mixed separately and then added to the preheated oil. The whole reaction mixture was vigorously stirred at 600 rpm for 3 hr under reflux condition as shown in figure 3.1. After completion of reaction, the whole reaction mixture was transferred into separating funnel to allow clear separation of the biodiesel from glycerol for 12 hr. Two layers was separated out, upper layer was organic layer which contained LRME while lower layer was aqueous layer containing glycerol. The organic phase was separated from aqueous phase and washed by distilled water 2-3 times to remove residual catalyst or soaps thus clear bio diesel was obtained.

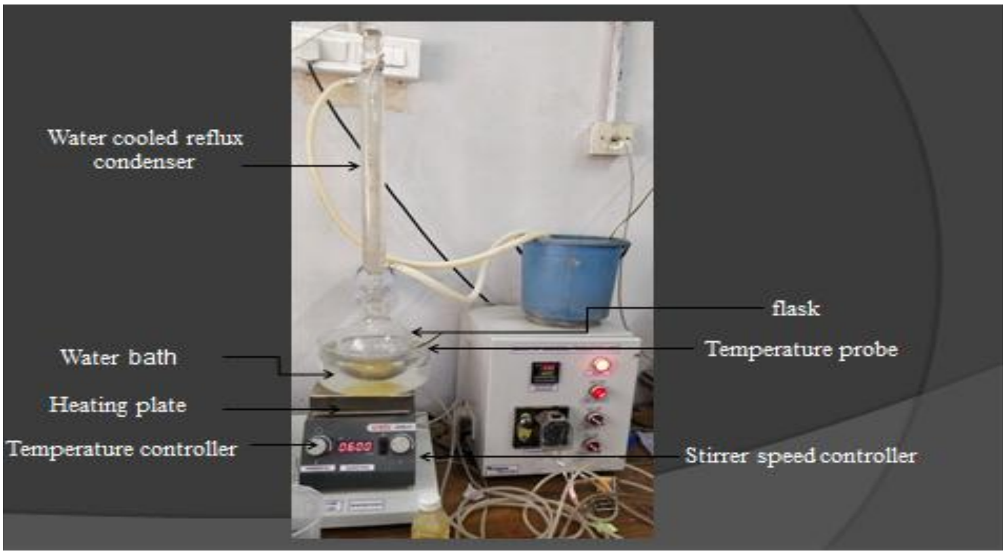

Figure 1 Transesterification with reflux condition

4. Result and discussion

The biodiesel yield depends not only on the type of feedstocks used, but also depends on molar ratio of alcohol to oil, the catalyst type and its concentration and reaction conditions such as temperature, duration and Stirrer speed.

In first trial, 100ml LR oil, 35 ml methanol and 1.5%v/v sodium methoxide were taken for transesterification process for 3 hr, but reaction was not completed because the whole reaction mixture was vigorously stirred without reflux condition as shown in figure 2. During second trial, same quantity of materials were taken for transesterification process for 3 hr with reflux condition; however, separation was not completed and there was shop formation because oil have high FFA contain which not suitable for direct transesterification process as shown in fig 3. Apart from this more than ten trial were taken to overcome such difficulty after that in last trial LRME was successfully produced from LR oil by following two step transesterification process with maximum 78% yield as shown in table 4.1. The percentage yield of biodiesel was calculated by the following equation:

Yield = (Weight of LRME produced /Weight of LR oil used) × 100%

Table 2 Proportion of Acid & Alkali catalyzer

Acid-catalyzed esterification		Alkali-catalyzed esterification	
LR oil	100 ml	Pre-treated LR oil	100 ml
Methanol	45 ml	Methanol	35 ml
Catalyst	H_2SO_4	Catalyst	CH_3NAO
Catalyst concentration	1%w/w	Catalyst concentration	1.5%w/w
Stirrer speed	600 rpm	Stirrer speed	600 rpm
Reaction temperature	60^0 C	Reaction temperature	60^0 C
Reaction time	2 hr	Reaction time	3 hr
Separation time	8 hr	Separation time	12 hr

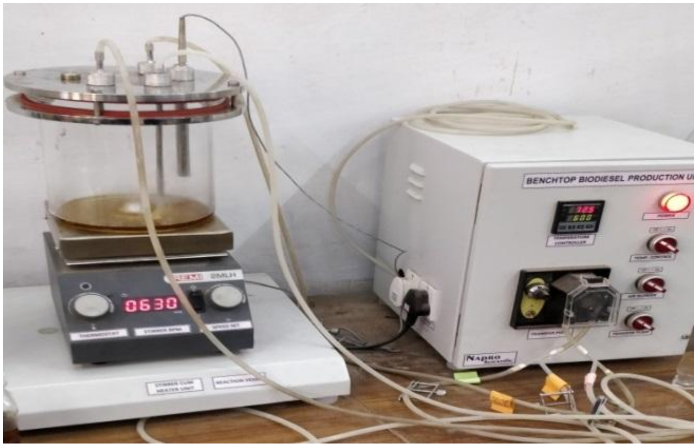

Figure 2 Transesterification without reflux condition

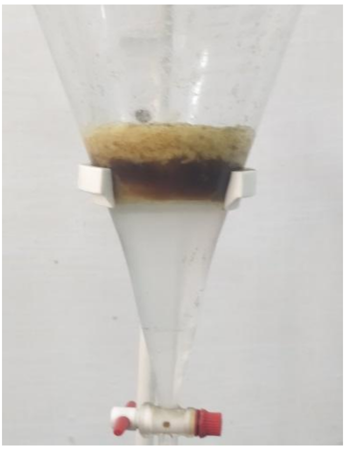

Figure 3 Shop formation during washing problem

Conclusion

In present work biodiesel is produced by non-edible oil namely Leptadenia reticulata oil was selected. These oils are available all over the year in India. In the present study, CH3NAO was used as a catalyst to understand its catalytic activity in transesterification of Leptadenia reticulata oil. Based on the results from the study, the following conclusions are made.

- The LRME yield that resulted from the optimum reaction was 76.2 % at the optimized values of process parameters for the transesterification of FFA present in LR oil which are methanol to oil ratio 6:1, catalyst amount 1.5 %(v/v), reaction time 180 min and reaction temperature 60 ^{0}C.
- Transesterification reduced the viscosity, acid value and water content of the FAME produced thereby improving the quality of biodiesel and enhancing energy efficiency of the engine running on such fuel.

Nomenclature

FFA	Free fatty acid
LR Oil	Leptadenia reticulata Oil
LRME	Leptadenia reticulata Methyl ester
H_2SO_4	Sulphuric acid
CH_3NAO	Sodium methoxide
NAOH	Sodium hydroxide

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