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COMPARATIVE STUDY OF BIOFUEL PRODUCTION FROM VARIOUS EDIBLE OILS

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AUTHORS' CONTRIBUTIONS

This work was carried out in collaboration between both authors. Author AS designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author CM managed the analyses of the study. Author AS managed the literature searches. Both authors read and approved the final manuscript.

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ABSTRACT

Biodiesel is a clean renewable fuel and hence has recently been considered as the best substitute for diesel. Chemically, biodiesel is a mixture of methyl ester with long chain fatty acids. It is produced from renewable biological sources such as vegetable oil and animal fats by simple Trans-esterification reaction. Among the energy resources biodiesel as a received more attention recently. In the present study, the potential impact of biodiesel production from edible oil such as sunflower oil and sesame oil was studied. The total acid number of sunflower oil was found to be 0.3 whereas TAN Number of sesame oil was recorded as 0.4. The results revealed that the biodiesel yield for sunflower oil was found to be 74.9% whereas in sesame oil the biodiesel yield was 71.4%. The effect of methanol to sunflower oil and sesame oil with different ration was studied indicated that three molar of triglycerides produce three molar of glycerol. The different ratio showed the concordant value was studied. Trans-esterification process consists of two step namely acid esterification and alkali esterification. Acid esterification was performed with H2SO4 and alkali Trans-esterification was performed with NaOH. In the present investigation purification performed in post treatment process. FTIR is useful for quantifying various conversion of Trans-esterification reaction. FTIR spectrum for sesame oil and sunflower oil produced biodiesel.

Keywords: Biofuel; alkaline; acid trans-estrification; total acid number; FTIR study; sesame and sunflower oil.

1. INTRODUCTION

Biodiesel is defined as a fuel comprised of mono alkyl ester of long chain fatty acids derived from vegetable oils and animal fats. It is nontoxic biodegradable and high oxygen content (10 to 11%). It does not contain sulfur aromatic compounds [1, 2].

Vegetable oil is one of the renewable fuels which have become more attractive recently because of its environmental benefits and fact that it is made from renewable resources [3].

Alkali process yield high quantity and high purity biodiesel in shorter reaction time. Therefore a two step trans-esterification process (acid esterification

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followed by alkali trans-esterification) was developed to remove high free fatty acids (FFA) content and to improve the biodiesel yield [4].

Petroleum is a non renewable energy source, which means that the resources of this kind of fossil fuel are finite and would be run out upon continuous use. Both of the shortage of resources and increase of petrol price have led to the finding of new alternative and renewable energy sources [5].

Waste cooking oil, which is much less expensive or sometimes available at free of cost, can be a promising alternative to edible vegetable oil [6]. In the conventional trans-esterification process, sunflower seed oils, methanol and NAOH in various concentrations were refluxed together in a 500 ml glass reactor equipped with a glass anchor shaped mechanical stirred a water condenser and funnel after the complete conversion of the vegetable oil. The reaction was stopped and the mixture was allowed to stand for phase separation, using a separator funnel, the ester mixture was dried over anhydrous sodium sulfate and analyzed [7].

Vegetable oils are always triglyceride, generally with a number of branched chains of different length and different degrees of saturation. Vegetable oils have about 10% lower heating value than mineral diesel due to the oxygen present in their molecules, with vegetable oil as a fuel, short term engine performance results with mineral diesel [8, 9].

In the present study, to collect oil from different sources in order to produce biodiesel (sunflower oil and sesame oil) were performed with Acid catalyzed Trans-esterification, alkali Trans-esterification and post treatment process, further analysis of biodiesel by FTIR method to determine the total acid number and types of catalyst were analyzed.

2. MATERIAL AND METHODS

2.1 Collection Sample

Sunflower oil and sesame oil sample were collected from the local market of Salem-D.T.

2.2 Materials

Industrial grade sunflower oil was procured from a local oil trader of Salem. Sesame oil was purchased from local market. The magnetic stirrer with hot plate was purchased from Telco chemicals PVT LTD at Salem. Chemicals are used in trans-esterification process like potassium hydroxide, methanol, concentrated sulphuric acid and sodium hydroxide.

2.3 Equipmemt

A round bottomed conical flask is used as reactor for these experimental purposes. A magnetic stirrer with hot plate arrangement is used for heating the mixture in the flask. The mixture is stirred at the same speed for all test runs. The temperature ranges of 40 - 60◦c is maintained during experiment and it is monitored by thermometer. The separating funnel is used to separate the methanol water mixture after acid pretreatment and the glycerol after trans-esterification. Four set off trail runs are carried out for each combination of parameter.

2.4 Biodiesel Production Process

The production of biodiesel has been conducted using two oil samples, sunflower oil and sesame oil. The parameter that has been varied the mode of the trans-esterification reaction using base catalysis and then two stage acid base catalysis. The different base catalysts employed are sodium hydroxide or potassium hydroxide and sodium methoxide.

2.5 Oil Filtration

Sunflower oil and sesame oil have higher moisture content and some other impurities. So in order to remove the moisture and impurities from the sesame oil it should be refined. The purification can be done by boiling oil with about 20% of water. The boiling should continue until no bubbles of water vapor any more. After one hour, the oil becomes clear. This refined sesame oil is taken as raw material for trans-esterification process.

2.6 Three Stages

2.6.1 Primary transesterification

2.6.1.1 Acid catalysed transesrification

The first stage is considered as the pretreatment process. The entire oil was preheated first. Then acid esterification was carried out for the whole feed stock under a recommended condition of 50 ml v/v oil and methanol and 1% concentrated H_2SO_4 at 60^0c for 60 minutes with constant mixing . When the first stage of the acid esterification was completed, the reaction mixture was transferred to a separating funnel and allowed to settle for about 90 minutes. The bottom layer was taken for the alkali trans-esterification. The top layer containing excess methanol, acid and impurities was discarded.

2.6.2 Secondary trans esterification

2.6.2.1 Alkaline catalysed trans esterification

The bottom layer product from the first stage containing oil with FFA less than 3% was subjected to alkali trans-esterification using 1% of NAOH as the base to avoid moisture absorption by the catalyst. The sodium meth oxide prepared was added to the preheated product of the acid esterification. The mixture was continuously stirred at constant speed keeping temperature constant at 60◦c. The Temperature was not allowed to rise above this specified limit to avoid the methanol loss.

After wards the mixture was transferred to a separating funnel and kept undisturbed for a settling period of 60 minutes. On settling, the biodiesel forms the top layer and glycerin along with any impurities move to the bottom layer.

2.6.3 Post Treatment Process

The bottom layer was removed and biodiesel was collected for post treatment process. It was washed with distilled water at $60^0$to remove the presence of any excess methanol, Soap and impurities like catalyst. The mixture was allowed to settle under gravity. The settled layer of mixture with impurities was drained out. Hot water wash was repeated two or more times. Water content was removed by addition of Na_2So_4 salt and any other remaining impurities present in biodiesel were removed by filtration through muslin cloth. Biodiesel was then heated to 110^0c for 10 minutes to remove any moisture present in it. Finally, the finished biodiesel having a golden yellow color was obtained.

2.6.4 Purification

The product was washed with double distilled water to remove the un reacted alcohol, catalyst or present in the biodiesel further the water washed fuel was heated to 110◦c in an open container until there was no more steam in the fuel. The resultant fuel should be a clear liquid.

2.6.5 To Analysis Biodiesel by Ftir

Add 0.35 g of finely ground anhydrous NaOH into 20 ml of pure (99% or higher purity) methanol in a 250 ml Erlenmayer flask containing a magnetic stir bar. Put the flask on a magnetic stir plate, and stir vigorously until all of the NaOH is dissolved. This flask now contains sodium meth oxide. Warm up 100 ml of 100% pure vegetable oil to about 40^0Cin a 250 ml beaker. Warming the oil up is not necessary, but increases the reaction rate. When all of the NaOH is dissolved, pour the 100 ml of oil into the meth oxide solution while continually stirring. At first the mixture will become cloudy, but should soon separate into two layers. Stir for 15-30 minutes on high. Transfer the contents of the flask into a 250 ml separator funnel. The mixture will separate into two layers. The glycerol will fall to the bottom, and the methyl ester (biodiesel) will float to the top. Since about 75% of the separation occurs within the first hour, you will be able to see immediate process. Allow the experiment to sit for about an hour. Open the stop clock of the separator funnel and allow the glycerol to drain into a small beaker. Make sure not to get any biodiesel in the glycerol or glycerol in the biodiesel. Use the IR spectrometer to identify your products. Print out the spectra's and compare with known spectra. The biodiesel may be hard to compare, since most of oils are comprised of different length carbon chains. Comparing to known spectra can easily identify the glycerol. The presence of glycerol indicates a successful reaction.

2.7 Determinaton of Total Acid Number (TAN)

The determination of TAN number is very essential for Trans-esterification of oil. The result of TAN analysis showed that the TAN values of most samples were valid. Total acid number in activity was measured by titration method using sunflower oil emulsion in 5% methanol was used as a substrate. The reaction mixture composed of 5 ml oil, 5ml ethanol, and add 1ml potassium hydroxide take with burette solution. The prepared was 10 methanol and 1% phenolphthalein indicator take with conical flask. Reaction was carried out at 600 for 60 minutes. The liberated fatty acid was titrated with 0.05 NAOH using phenolphthalein indicators. The amount of NAOH required achieving end point pink color was recorded.

The result showed that the biodiesel with lowest TAN was obtained when the methanol to molar ratio of 1:4 gave the highest TAN in produced biodiesel. Table 1 showed the effect of types of catalyst used on the biodiesel TAN value. It was shown that the sodium hydroxide–catalyzed Trans esterification using potassium hydroxide. It was showed that the values of 1:3. TAN increased with increasing NAOH catalyst concentration. The effect of different methanol to molar ratio on biodiesel yield. (Oil was heated at 60◦c for 60 minutes in the water bath was studies. In the study, the TAN value of most excessive acid value which means that the acid contents of these samples were either too low to be detected or too high [10].

It was also reported the acid numbers of the biodiesel were influenced by reaction times, in which increasing reaction times causes increase in the value of acid number [11]. Their result showed that the TAN values for biodiesel produced using sodium hydroxide were higher than those produced using potassium hydroxide [4].

2.8 Tritimetric Method of Catalyst

Type of catalyst was measured by titrimetric method using sunflower oil and sesame oil as a substrate two type of catalyst is used in KOH/NAOH. In KOH in 5% is prepared in standard of 10ml ethanol + 1ml of phenopthaline indicator. Then burette in 0.14g of KOH and add 25ml of methanol. In NAOH and then adding amounts of catalyst in methanol to oil molar ratio in using 1:1 and 1:2, 1:3, 1:4 the reaction time in 60◦c in 60 minutes, mixing intensity in 250 rpm. The amount of NAOH and NAOH required to achieve in end point of colorful to pink was recorded. They are produced in colorful of pink color they are produced in high molar ratio in 1:1 and the ratio was decreased in 1:4.

2.9 Determination of Acid Value

Free fatty acids occur naturally in vegetable oils and thus are carried over into the final product after Trans-esterification. The fatty acids present in biodiesel depend primarily on the type of feed stock used although most of them are removed during the refining of the feed stock oil before the Trans-esterification reaction. High free fatty acid levels in biodiesel can cause fuel system deposits and is also an indication that the fuel will act as a solvent resulting in the determination of the rubber components of a fuel system. In 5ml oil and 10 ml of methanol and added 1% of phenolphthalein indicator and conical flask was filled in 0.14 KOH and 15ml methanol and titrimetric method. The result was obtained in colorful pink. So the standard established by the maximum of mg KOH/g of solvent.

Acid value in formula,

$[(A - B)] \times M.\ wt] / weight$

A= KOH required for titration of sample
B= KOH required for blank titration
M= Molarity of the KOH solution
W= Weight of sample used.

2.10 Determine of Catalyst for Enhance Biodiesel Production

2.10.1 By trans esterification method

Trans-esterification is a common method for biodiesel production from vegetable oils and animal fats and usually preferred instead of direct esterification [12]. In trans-esterification or alcoholysis, fats or oils react with alcohol in presence of a catalyst to form alky esters and glycerol The trans-esterification process reduce the acid value of oils which is higher than petro-diesel .Selecting a suitable alcohol and catalyst is important for trans-esterification method.

Various alcohols such as methanol, butanol, ethanol, propanol and amyl alcohol can be used for Trans-esterification. Methanol is used widely because it is relatively cheaper than other alcohols and has chemical and physical advantages over other alcohols. In theory 3 moles of alcohols are required to neutralize 1 mole of triglyceride to produce 3 Moles of fatty acid methyl ester (FAME) and1 mole of glycerin. A good Catalyst is also needed to obtain a reasonable rate for trans-esterification of triglycerides and its conversion to biodiesel.

High water and free fatty acid in oil reduce the effectiveness of catalysts, produce soap and require considerable amounts of catalysts. Free fatty acids (FFAs) and water in oil needs to be removed before applying base catalysis process. [13] used acid catalyst which eliminated the above-mentioned problems. They stated that acid catalysts act better than base catalysts, because acid catalysts are able to convert higher percentage of free fatty acids (FFAs) to triglyceride. The first choice for acid catalysts is sulfuric acid which was used by several researchers [14]. In addition to the acid and base catalysts, enzyme catalysts are also considered for biodiesel production. The enzyme catalysts are gaining more interest in recent years because they don't constitute soap and their process is simple to complete. Enzymatic catalysts are currently not feasible for commercial productions since they have higher cost and need longer reaction time [15].

3. RESULTS AND DISCUSSION

Trans esterifies vegetable oils are a viable alternative engine fuel with characteristic similar to those of diesel. Trans-esterification means taking a triglyceride molecule for a complex fatty acid, and removing the glycerin and creative on alcohol ester. Biodiesel productions incorporate use of conventional catalyst like acid and alkali. The present study on acid esterification was study for two different cooking oils.

3.1 Determination of Total Acid Number

The present study on the Trans-esterification of waste cooking oil (sunflower oil was catalyst with acid. The result showed TAN number of 0.3 ml was sunflower oil. Table-1 showed determination of TAN number was sesame oil evaluated. It is found at that TAN number of 0.4 ml was recorded in Table-2 and Plate: 1 upto 5.

3.2 Effect of Methanol to Sunflower Oil Molar Ratio

The molar ratio of methanol to oil is a very important factor as it affect the yield and also then cost of reaction. According to stock in chenetrometric ratio three molar of triglyceride produce three molar of ester and one molar of glycerol in Tran's esterification process. The presence study sunflower oil showed two different layers at the molar ratio of 1:1 (5 ml +5 ml sunflower oil).Showed the concordant value of 1:1 in Table-3. Similarly the effect of methanol to oil molar ratio oil 1:2 ratio (5 ml methanol+10 ml sunflower oil). The concordant value was recorded as 1:2 in Table-4. Similarly the ratio was modified as 1:3 ratios (5 ml methanol +15 ml sunflower oil). The produced yield of 1:3 ratios in Table-5 and Plate: 6 upto 10.

Table 1. Determination of total acid number for biodiesel obtained from sunflower oil

S.NO	Neutralized ethanol (ml)	Titration 0.1N methanolic (KOH) (g)	Initial reading (ml)	Final reading (ml)	Acid velocity TAN concordant value
1.	10 ml	0.14	25.0	25.3	0.3
2.	10 ml	0.14	25.0	25.3	

Table 2. Determine of total acid number for biodiesel obtained from sesame oil

S.NO	Neutralized ethanol (ml)	Titration 0.1N methanolic (KOH) (g)	Initial Reading (ml)	Final Reading (ml)	Acid velocity TAN concordant value
1.	10 ml	0.14	25.0	25.4	0.4
2.	10 ml	0.14	25.0	25.4	

Plate 1. Acid catalyzed Trans-esterification process for sunflower oil

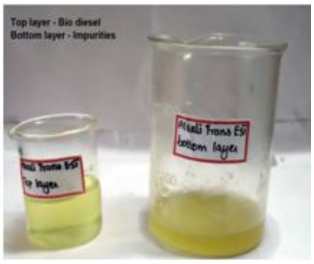

Plate 2. Alkali Trans-esterification process for sunflower oil

Plate 3. Post treatment process for sunflower oil

Plate 4. Post treatment process for sunflower oil

Plate 5. Sunflower oil

Table 3. Effect of methanol to sunflower oil molar ratio (1:1)

S.NO	Neutralized methanol (ml)	Titration 0.1N methanolic KOH(g)	Initial Reading (ml)	Final Reading (ml)	Acid velocity TAN concordant value
1.	10 ml	0.14	25.0	23.9	1.1
2.	10 ml	0.14	25.0	23.9	

Table 4. Effect of methanol to sunflower oil molar ratio (1:2)

S.NO	Neutralized methanol (ml)	Titration 0.1N methanolic KOH(g)	Initial Reading (ml)	Final Reading (ml)	Acid velocity TAN concordant value
1.	10 ml	0.14	25.0	23.8	1.2
2.	10 ml	0.14	25.0	23.8	

Table 5. Effect of ethanol sunflower oil molar ratio (1:3)

S.NO	Neutralized methanol (ml)	Titration 0.1N methanolic KOH(g)	Initial Reading (ml)	Final Reading (ml)	Acid velocity TAN concordant value
1.	10 ml	0.14	25.0	23.7	1.3
2.	10 ml	0.14	25.0	23.7	

Plate 6. Acid catalyzed Trans-esterification process for Sesame oil

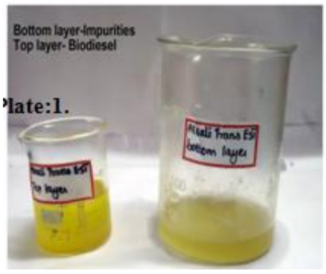

Plate 7. Alkali Trans-esterification process for Sesame oil

Plate 8. Post treatment process for Sesame oil

Plate 9. Post treatment process for Sesame oil

Plate 10. Sesame oil

Table 6. Effect of methanol to sesame oil molar ratio (1:1)

S.NO	Neutralized methanol (ml)	Titration 0.1N methanolic KOH(g)	Initial Reading (ml)	Final Reading (ml)	Acid velocity TAN concordant value
1.	10 ml	0.14	25.0	23.9	1.1
2.	10 ml	0.14	25.0	23.9	

Table 7. Effect of methanol to sesame oil molar ratio (1:2)

S.NO	Neutralized methanol (ml)	Titration 0.1N methanolic KOH(g)	Initial Reading (ml)	Final Reading (ml)	Acid velocity TAN concordant value
1.	10 ml	0.14	25.0	23.8	1.2
2.	10 ml	0.14	25.0	23.8	

Table 8. Effect of methanol to sesame oil molar oil ratio (1:3)

S.NO	Neutralized methanol (ml)	Titration 0.1N methanolic KOH(g)	Initial Reading (ml)	Final Reading (ml)	Acid velocity TAN concordant value
1.	10 ml	0.14	25.0	23.7	1.3
2.	10 ml	0.14	25.0	23.7	

Table 9. Determination of biodiesel production in trans esterification

S. No	Variety of natural oil used	Normal Biodiesel Yield	Biodiesel produced after trans esterification
1	Sunflower oil	65%	76.9%
2	Sesame oil	70%	71.4%

3.3 Effect of Methanol to Sesame Oil Molar Ratio

In this study effect of methanol with 1:1 ratio was investigated. The results were depicted in Table-6. Effect of methanol and sesame oil 1:2 ratio showed in Table-7. Owing to analyze of 1:3 molar ratio (5 ml methanol+ 15 ml sesame oil were carried for 24 hours) the results exhibited in Table-8. The concordant value review 1:3 ratio exhibited.

3.4 Determination of Biodiesel Production

Table-9 depicts the biodiesel yield after Tans-esterification with cooking such as sunflower oil, Sesame oil. The result showed that biodiesel yield for sunflower oil was found to be 65% during normal biodiesel analysis whereas after trans-esterification a significant increase in biodiesel yield of sunflower oil in 76.9% was recorded similarly the biodiesel analysis for studied was sesame oil will required 71.4% of normal biodiesel yield with trans-esterification. However the slight increase in biodiesel yield was found to be 76.9 %.

3.5 Calculation

3.5.1 Sunflower oil

$$\text{Oil yield \%} = \frac{\text{Weight of oil extracted}}{\text{Weight of oil used}} \times 100$$

$$= \frac{0.3}{50} \times 100 = 0.6\%.$$

$$\text{Yield \%} = \frac{\text{Weight of oil used in reaction}}{\text{Weight of biodiesel produced}} \times 100$$

$$= \frac{50}{65} \times 100 = 76.9\ \%.$$

3.5.2 Sesame oil

$$\text{Oil yield} = \frac{\text{Weight of oil extracted}}{\text{Weight of oil used}} \times 100$$

$$= \frac{0.4}{50} \times 100 = 0.8.$$

$$\text{Yield \%} = \frac{\text{Weight of oil used in reaction}}{\text{Weight of biodiesel produced}} \times 100$$

$$= \frac{50}{70} \times 100 = 71.4\%.$$

3.6 Analysis of Vegetable Oil in Sunflower Oil and Sesame Oil in FTIR

FTIR spectrum for sesame oil revealed a peak of 1714.8 cm^{-1}, this band conforms that ester groups are present in biodiesel. The results were recorded in fig:1 depicted the peak in the band of 1174.7cm^{-1} corresponds to a peak of sesame oil biodiesel. Another prominent peak of 2859 cm^{-1} and 3004.26 cm^{-1}, these values such as this spectrum correspond to sesame oil of biodiesel. Fig:2 exhibit the spectral analysis of FTIR pertaining to sunflower oil biodiesel revealed the prominent the band of 1744 cm^{-1}, which such as that this band was conformed be ester group present in the sunflower oil biodiesel. Similar peak vibrate as varies peaks depending an ailment and type of band. The sunflower oil biodiesel spectral vibrations at 1164 cm^{-1}, 1365 cm^{-1} and 2860 cm^{-1}, these values or peaks confer that spectrum correspond to sunflower oil biodiesel.

Studies of Trans esterification of sunflower oil and sesame oil with catalyst as acid value determine as TAN number. In the present study 0.4 ml with sesame oil was established. Similarly the TAN number of was

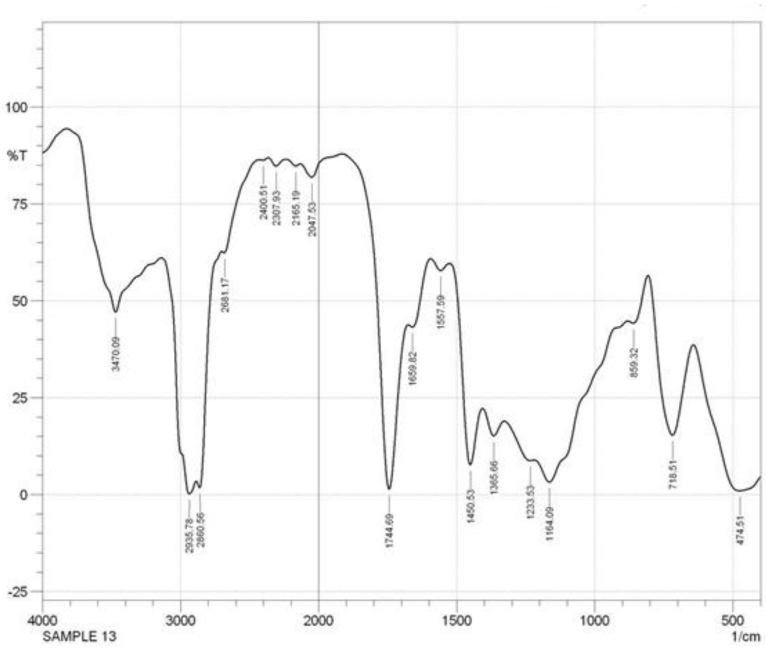

Fig. 1. FTIR analysis of vegetable oil in sesame oil

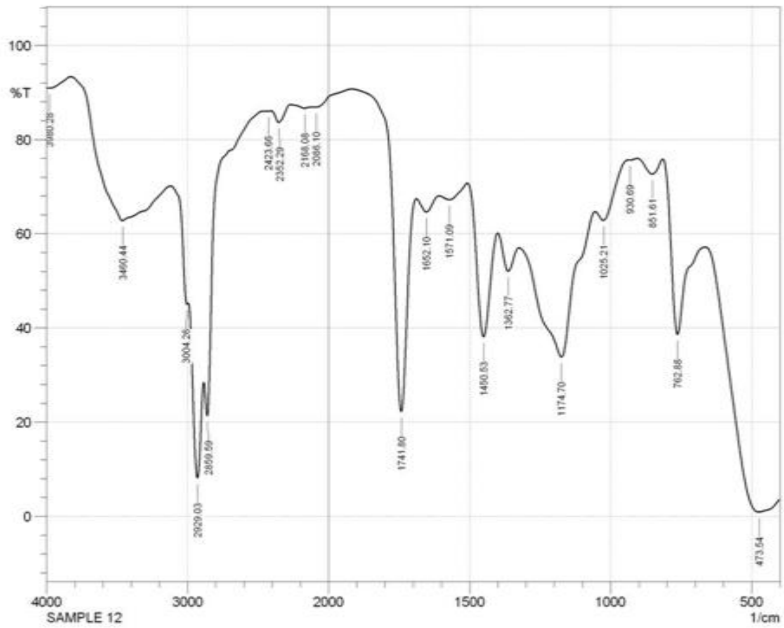

Fig. 2. FTIR analysis of vegetable oil in sunflower oil

found out 0.3 ml sunflower oil. On comparison among the two different oils sesame oil was found to be high [16].

The effect of methanol to sunflower oil with different ratio was studied indicated that three molar of triglyceride produce three molar of glycerol. The 1:1 ratio showed the concordant value 1:1. Similar pattern of study was recorded for 1:2 and 1:3 ratio [17].

Studies to prove the effect of methanol to sesame oil molar ratio revealed 1:2 ratio produced the concordant value of 1:2. The similar report was reported [18]. FTIR is device quantify the functional group changes. It is also useful for quantifying various conversion of trans esterification reaction [19].

The FTIR spectra of biodiesel production with sunflower oil revealed different type of bands presumed to be free fatty acids and esters present in the biodiesel [20]. The calculated acid value from sunflower oil 76.9% mg NaOH/ g were as the acid value for sesame oil was found to be 71.4% mg NaOH / g respectively biodiesel yield. Water is not required oil sample since it is instead of Trans-esterification process. The acid value for sunflower was comparison of more than sesame oil [21].

4. CONCLUSION

In the present study was concluded that sunflower oil and sesame oil was one of the option for biodiesel production at the large scale. The sunflower oil after trans-esterification process yield at the maximum conversion frequencies was analyzed. On analysis of results revealed the presence of ester group in the biodiesel from sunflower oil. Our current research findings were exhibited potential impact of biodiesel production from edible oil such as sunflower oil and sesame oil was studied.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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