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A Novel Method Applied to the Production of Biodiesel from Neem Oil

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ABSTRACT: In this study, a new type of process is applied to the conversion of neem oil into biodiesel. This method involves complete saponification of the neem oil by reacting it with sodium hydroxide solution to give sodium salt of fatty acid. This sodium salt of fatty acid called soap when treated with a mineral acid such as concentrated sulphuric acid separates into four layers, the top layer is the free fatty acid, is easily separated and then esterified with ethanol in the presence of a mineral acid which acts as a catalyst to get the final product neem oil biodiesel(NOBD) and hence this new method of production of biodiesel is named SAFALE (Saponification, Fatty Acid Liberation and Esterification). The product obtained by the above process is analyzed and confirmed by GC-Mass spectra and FT-IR spectra. This process does not involve high temperature and pressure. The main advantages of this method are less processes time, less effluent discharge, toxic materials are not involved, more yield, and more environment friendly compared with the only available two-step process in the production of biodiesel from raw neem oil, which usually contains more than 5% of free fatty acid.

Keywords: Neem oil Biodiesel, free fatty acid, saponification,esterification, and Gas chromatographic- Mass spectra.

I. INTRODUCTION

With the desperate search for a suitable replacement of automotive fossil fuels, mainly the petrodiesel, researchers across the world are trying to identify the feedstock for the economical production of biodiesel without making competition for food-based edible vegetable oils.

In countries like India, the non-edible neem oil obtained from the tree called *Azadirachta indica* is a suitable and viable option for conversion to biodiesel, this oil-bearing tree can be grown in various geographical location of India with different percentage of oil content of 30-60% [1-3]. The climatic condition in India is very much favorable for the growth of neem trees and requires less attention and irrigation for the production of neem seeds compared to any other type of oil seed bearing crops, average annual neem oil production in India is about 30000 tons [2]. At present, there is a good market for neem oil due to its use of its bioactive compound called 'Azadirachtin' in organic farming activities as natural pesticides [4]. Azadirachtin can be separated from neem oil during the oil extraction process,and this oil can be used for biodiesel production [5], which has no other applications.

The conversion efficiency and applicability of transesterification of any vegetable oils (triglycerides) to biodiesel mainly depend upon the free fatty acid content. The free fatty acid content of more than 2% in the feedstock will not only decrease the total yield of biodiesel but also makes the much favorite base catalyzed homogenous process more complicated and time consuming due to the formation of soap, forming an emulsion between biodiesel and glycerol making the separation of biodiesel very difficult [6]. The soap formed has to be removed by washing the reaction mixture several times, and the effluent has to be discharged to the environment before getting the final product biodiesel, making this transesterification [25] process very difficult, less economic and environmentally less favorable. Ragit *et al.* carried out transesterification of neem oil to get a biodiesel yield between 41.53 to 87.69%, most of his trial out of 81 gave a low percentage of conversion [7].

To produce biodiesel at a low cost, two factors gain much importance. One is getting low-cost feedstock [26],and another is finding a suitable and economical process to convert that feedstock into biodiesel. Life cycle analysis, in the production of biodiesel by Varanda *et al.,* showed that biodiesel obtained from oil containing a high percentage of free fatty acid is more economical [8]. Generally, the low-cost feedstock contains a higher percentage of free fatty acid and hence, a new type of process for the utilization of this kind of feedstock has to be investigated [9]. The free fatty acid content in oil could vary to a much extent, even for a single batch of oilseeds depending upon the moisture content, the process used in the extraction of oil and conditions of storage [10]. The raw neem oil generally contains 5 to 25% of free fatty acid depending upon the feedstock. Hence many researchers tried synthesizing biodiesel from neem oil using the two-step process [11-15], that is the esterification followed by a transesterification process.

Even though the yield obtained in this two-step process (esterification followed by transesterification process) is better than the alkali-catalyzed transesterification process, the two-step process still suffer from many disadvantages such as long processing time to get the final product, formation of soap when the process temperature is more than 60°C [12], removal of methanol before water washing at the end of the second step to avoid soap formation [5]. The pretreated oil in the first step contains water and the acid catalyst, which has to be removed before going to the second step of the alkali-catalyzed transesterification process [10]. Washing followed by drying of the biodiesel several times will make this process more complicated and less economical in the fabrication of the biodiesel production plant both in the batch as well as the bulk process. Keeping all the above disadvantages as background, a novel method [22] is appliedtothe production ofneem oil biodiesel (NOBD) from neemoil containing any percentage of free fatty acid.

II. MATERIAL AND METHODS

A. Materials

The raw neem oil which is to be converted to biodiesel was purchased from Sevugamoorthy oil mill at singampunerai, Siiivagangai District, Tamilnadu, India and used as such. This neem oil was titrated and found to contain 21% of free fatty acid and is used as such for the biodiesel conversion process. Methanol and ethanol of 99.9% purity, Sodium hydroxide, and sulphuric acid of laboratory grade were obtained from NICE CHEMICAL and used without any further purification.

B. Analytical methods

Determination of physical properties. The neem oil biodiesel produced using the new process was subjected to the testing of its physical properties, i.e., density, kinematic viscosity, cloud point, pour point, flash point and acid number were determined following the American Society for Testing and Materials(ASTM) methods.

Gas chromatography-Mass spectrometry analysis. The neem oil biodiesel produced by the new process is analyzed by gas chromatography [23] coupled mass spectrometer, model SHIMADZU-QP2020. Helium was used as a carrier gas with a flow rate of 3 mL/min. The column temperature was set to 60°C for 2 minutes and raised to 250°C at the rate of 10°C/min. The injector temperature was set at 250°C, and a sample volume of 1µL in methanol was injected with a split flow of 100mL/minute. The m/z range of the mass spectrometer was set in between 50-500.

Fourier transform–Infrared spectroscopy analysis. The neem oil, the free fatty acids and its biodiesel were also characterized by FT-IR spectrometer, using a Perkin Elmer make, Model-Spectrum Two, scanning between the range of 4000-400 cm^{-1} with a resolution of 1 cm^{-1}.

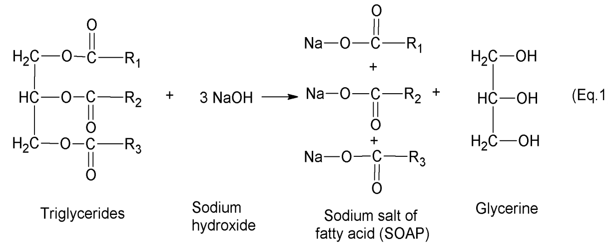
III. EXPERIMENTAL PROCEDURE FOR THE PRODUCTION PROCESS

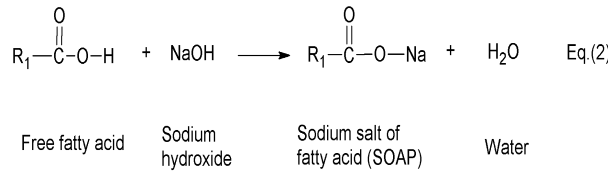
The process of preparation of biodiesel from neem oil comprises the following steps:

A. Step -1 - Saponification

One liter of neem oil is taken in a two-liter glass beaker and mixed with sodium hydroxide solution in the molar ratio 1: 3, preferably at 50-60°C. This mixture was continuously stirred with the help of a motorized stirrer; thereby, all the neem oil is converted into sodium salt of fatty acid (soap). The triglycerides present in the neem oil react with sodium hydroxides to give soap, as shown in the equation (Eq.1).

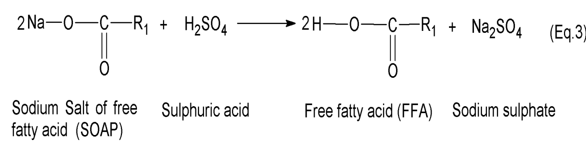
The free fatty acid present in the neem oil is also directly converted into soap (Eq. 2). Thus, at the end of the first step, the whole of the raw neem oil, which contains the triglycerides and the free fatty acids is converted into soap. Since these two reactions (Eq. 1) and (Eq. 2) are irreversible reactions, the products are formed immediately, and the reaction proceeds to completion in 15 minutes.





B. Step -2-Liberation of free fatty acid

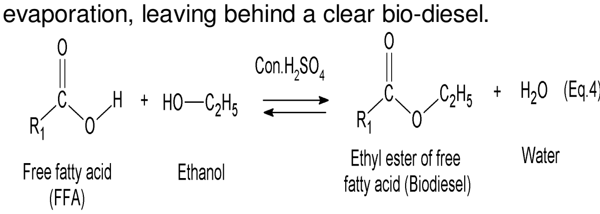
This soap which will be in the form of the highly viscous liquid state is added with 6 Molar sulphuric acids with constant stirring. Now the reaction mixture separates into four distinct layers. The top layer consists of free fatty acids (FFA); the next layer is glycerol, then a layer of water as the third layer and the fourth bottom layer is sodium sulphate crystals. As this reaction of splitting of soap by sulphuric acid is an exothermic and irreversible, the reaction proceeds to completion in a short span of 5 minutes as shown in the equation (Eq. 3).



Free fatty acid (FFA) is separated by decanting and used for the esterification process to produce neem oil biodiesel in the next step.

C. step -3 - Esterification of free fatty acid (FFA) with ethyl or methyl alcohol to obtain neem oil biodiesel

A mixture of ethanol or methanol and sulphuric acid is prepared separately and then added to free fatty acid (FFA) kept at 50°C in the molar proportion of (0.1:1:10) (Sulphuric acid: FFA: methanol or ethanol). The sulphuric acid acts as a catalyst in the reaction (Eq. 4). The reaction mixture is then stirred for about 45 minutes, which results in the formation of two layers consisting of a top layer of the ethyl ester of FFA (bio-diesel) and water. The bio-diesel which is separated from the mixture is cloudy and is heated to 100°C to remove the water by evaporation, leaving behind a clear bio-diesel.



The whole process of production of neem oil biodiesel by the new method is graphically represented as shown in the figure (Fig.1).

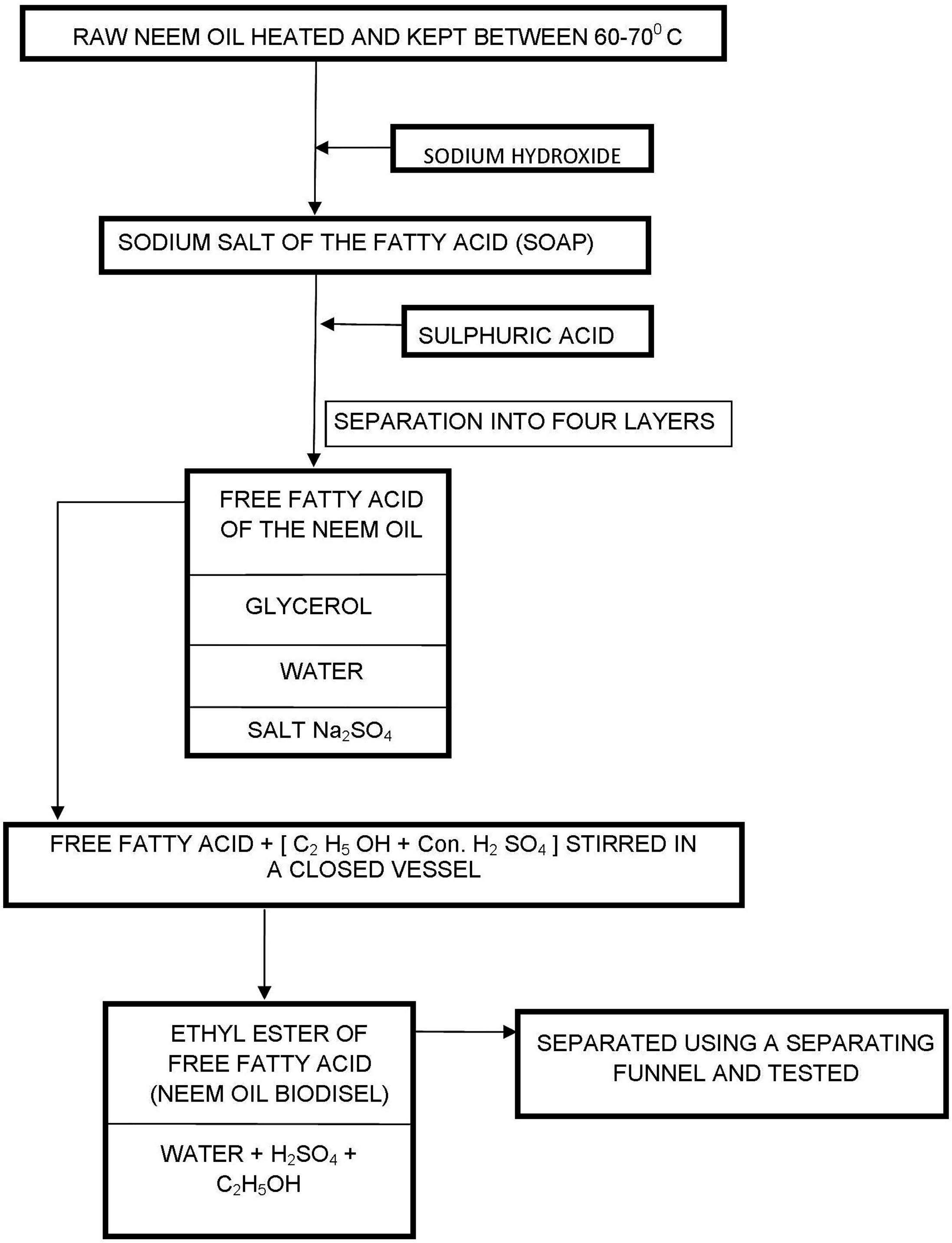


Fig. 1. New process for the production of biodiesel from neem oil.

When 1 liter (900g) of the neem oil was subjected to the above process, the yield of free fatty acid was 855.60g, which was esterified with methanol to get the neem oil biodiesel. The theoretical maximum amount (100%) of free fatty acid that can be produced from 900g of neem oil is 863.62g, that is if all of the neem oil is converted in to free fatty acid.

The total yield of the neem oil biodiesel produced using the new process was calculated by using the equation (Eq.5)

Percentage yield =

$$\frac{Grams\ of\ free\ fatty\ acid\ produced}{(Grams\ of\ theroritical\ maximum\ amount\ of\ free\ fatty\ acid)} \times 100 \quad \text{(Eq. 5)}$$

$$= \frac{855.60}{863.62} \times 100$$

$$= 99.00$$

IV. ANALYSIS OF THE PRODUCT

The product neem oil biodiesel obtained by the above process is analyzed and confirmed by Gas chromatography-Mass spectrometry and FT-IR spectroscopy. The critical physical properties such asdensity, kinematic viscosity, flash point, cloud point, pour point, and acid number of the neem oil biodiesel synthesized using the new process were determined as per ASTM methods and tabulated (Table 1).

Table 1: Physical properties of the neem oil biodiesel.

Physical properties	ASTM method used	ASTM D6751 Biodiesel	Neem oil Biodiesel
Density (40° C, g cm^{-3})	*D5002*	0.86 – 0.90	0.88
Kinematic viscosity (40°C, mm^{2}s^{-1})	D445	1.9 – 6.0	5.8
Acid number (mg KOH g^{-1})	D664	0.5	0.12
Flash point (°C)	D93	100 -170	126
Cloud point (°C)	D2500	-3.0 to 12	4
Pour point (°C)	D97	-15 to 16	-10
Water and sediment content (% vol. max.)	*D2709*	0.050	0.045

V. RESULT AND DISCUSSIONS

This new process was carried out with 1 liter (900g) of neem oil. It resulted in the production of 855.60g of free fatty acid. Hence almost 99.00 percentage of the neem oil molecule is converted in to free fatty acid. The conversion efficiency that was expected or that may have occurred might be 100 percent, but the loss of one percent may be due to the free fatty acid sticking to the reaction container and the separating funnel used during the separation of glycerol from the fatty acid. The reported percentage yield values are the average of three measurements. This free fatty acid is esterified with methanol or ethanol (1:10 ratio) in the presence of concentrated sulphuric acid acting as a catalyst to yield methyl or ethyl ester of fatty acid. The conversion of free fatty acid to bio-diesel also depends on how good the equilibrium (Eq.6) is shifted; that largely depends on how far we can remove the water produced during the esterification process.

$$\text{Free fatty acid(FFA)} + \text{Ethanol} \rightleftharpoons \text{Ethyl ester of free fatty acid (Biodiesel)} + \text{Water} \quad \text{(Eq.6)}$$

Since the esterification reaction is a reversible reaction, getting 100% yield may not be possible. Even if the final product bio-diesel contains some traces of unreacted free fatty acid (which is within the ASTM standards), this does not affect the quality of the fuel because this unreacted FFA mixes very well with the ester of FFA and possesses no phase separation or combustion problems. In this process, there is no wastage of oil in the form of soap, and because of this advantage, a very high yield is obtained. The whole process is completed in less than one hour since the first two steps in this new process involve the irreversible reaction giving the products instantaneously and making the reaction to completein a very short time compared to many other processes operating at this temperature and pressure for the production of bio-diesel.

A. Physical properties of the produced neem oil biodiesel

The neem oil biodiesel produced using the new process was tested for its physical properties employing the ASTM methods and compared with the ASTM D6751 specification of biodiesel and listed in the (Table 1).

The density of the neem oil biodiesel determined at 40°C employing D5002 method was 0.88g cm^{-1}, which is within the range of the ASTM D6751 specifications.

Kinematic viscosity is the most crucial property of any biodiesel which has the direct influence on the fuel injection pattern, and combustion of the fuel inside the engine, the kinematic viscosity of the neem oil biodiesel was determined using D445 method at 40°C which was 5.8 mm^{2}s^{-1} and found to be within the ASTM D6751 range.

The acid number of biodiesel is an excellent indicator to determine the conversion percentage for this new process used for the production of biodiesel from neem oil, and it is also an essential parameter in determining the storage stability of the biodiesel. The acid value of the neem oil biodiesel was determined using the D664 method and was found to be 0.12 mg KOH g^{-1}, which is well below the maximum value of ASTM D6751 standard for biodiesel. The flash point of the neem oil biodiesel was found to be 126°C, which is also within the ASTM D6751 range and may not have any combustion problem with the compression ignition engines. The cloud point and pour point of the neem oil biodiesel was also determined using the D2500 and D97 methods, which asses the cold flow properties of the fuel and its usage in colder climates. The cloud point of the neem oil biodiesel was 4°C, and the pour point was found to be -10°C, which is well within the limits of ASTM D6751 standards. Water and sediment content of the neem oil biodiesel were determined using D3709 methods and found to within the ASTM D6751 limits.

B. FT-IR spectral studies

The new process of conversion of neem oil to biodiesel has been confirmed by taking the FT-IR spectra for the following three samples, the raw neem oil, the free fatty acids produced in the second step of the process and the neem oil biodiesel produced in the final stage of the process. The frequency of C=O stretching depends upon the nature of the substituent attached to it in an FT-IR spectrum [16,17]. The presence of ester functional group is characterized by a strong absorption band around 1750-1730 cm^{-1} and 1300-1000 cm^{-1} due to antisymmetric axial stretching and asymmetric axial stretching of the C=O and C-O groups respectively [18,19].

FT-IR spectra for the raw neem oil. The spectra for the raw neem oil (Fig. 2) shows a strong absorption band at 1752cm^{-1} is because of C = O stretching of the tri-ester in the neem oil. The peak at 3480 cm^{-1} is due to the overtones produced by C=O group of the esters, and the

C–H stretching of the long alkyl chain present in the triglycerides shows the peak at 2982 cm^{-1} and 2918 cm^{-1}. A decrease in absorbance between 3400 cm^{-1} and 3100cm^{-1} is due to the ester groups of the triglycerides (the neem oil).

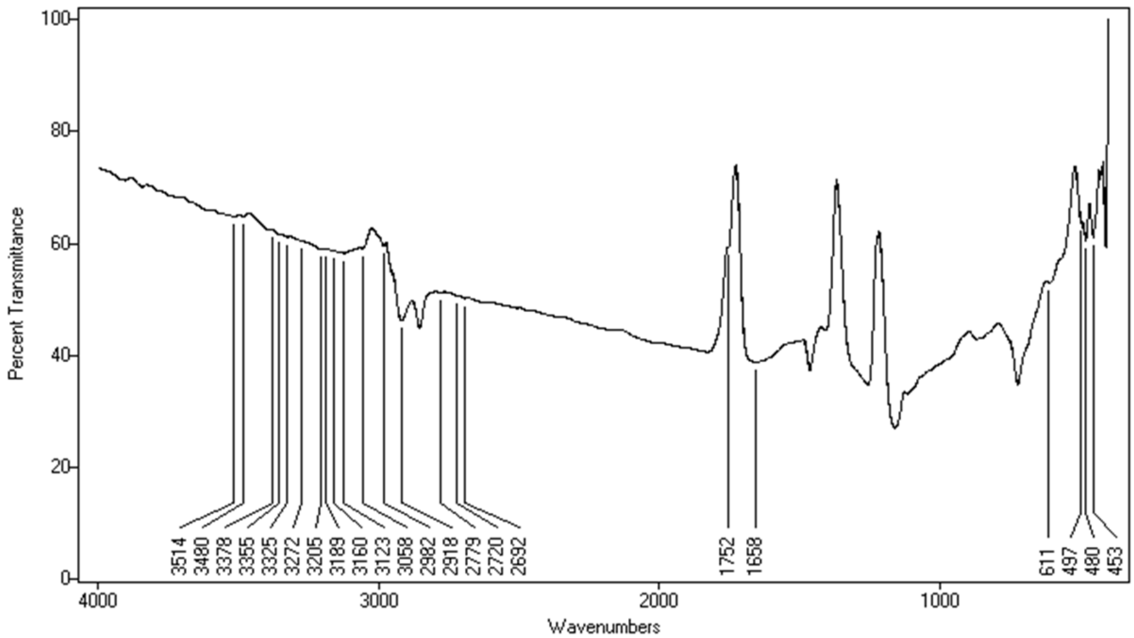


Fig. 2. FT-IR spectra of the raw neem oil.

FT-IR spectra for the free fatty acids. The second step of the process which is the liberation of the free fatty acid is confirmed by taking the FT-IR spectra (Fig. 3) of the top layer free fatty acid produced by reacting the soap formed in the first step with sulphuric acid. The peak at 1710 cm^{-1} is due to the C=O stretching of the carboxylic acid group in the free fatty acid. The peak around 3000 cm^{-1} (O–H stretching of the carboxylic group) begins at about 3300cm^{-1} and slopes into the aliphatic C–H absorption bands of the long alkyl chain at 2921 cm^{-1} and 2854cm^{-1} and thus centered around 3000 cm^{-1}.

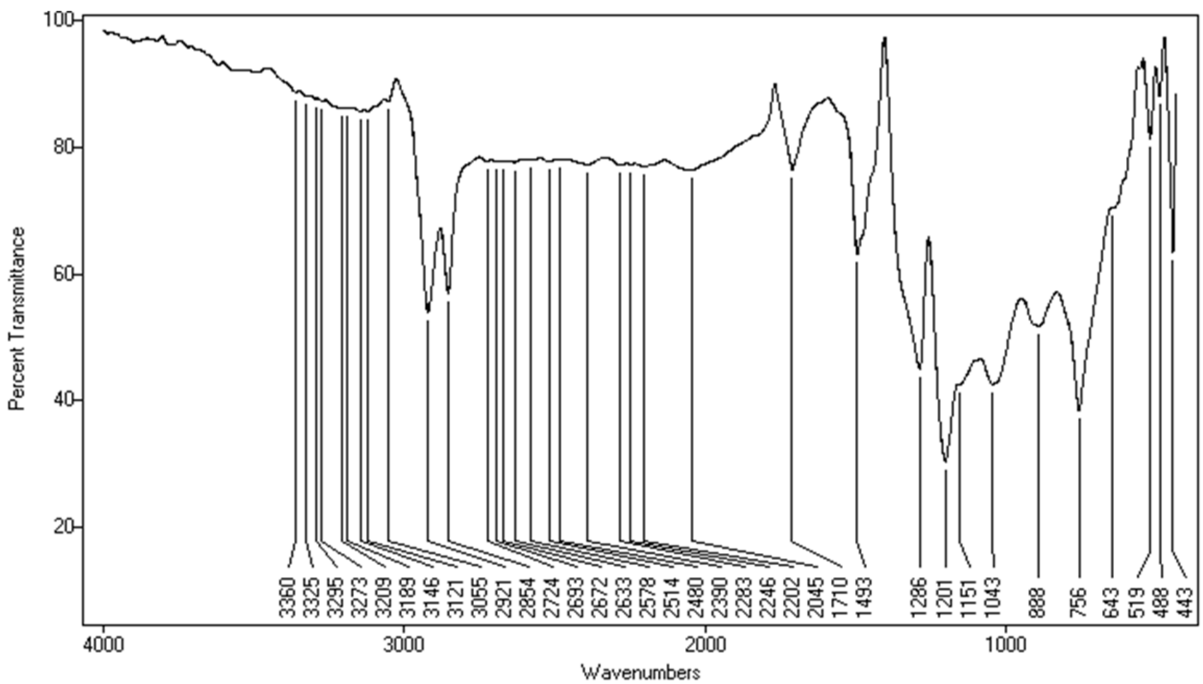


Fig. 3. FT-IR of free fatty acid of the neem oil.

FT-IR spectra for neem oil biodiesel. The final product neem oil biodiesel produced by the esterification of the free fatty acid in the third step of the process was confirmed by taking the FT-IR spectra (Fig. 4) of the product neem oil biodiesel. The peak at 1745 cm^{-1} is due to the C = O stretching of the ester group. The overtones at 3483cm^{-1} have appeared, indicating the presence of fatty acid esters. (BIO-DIESEL).

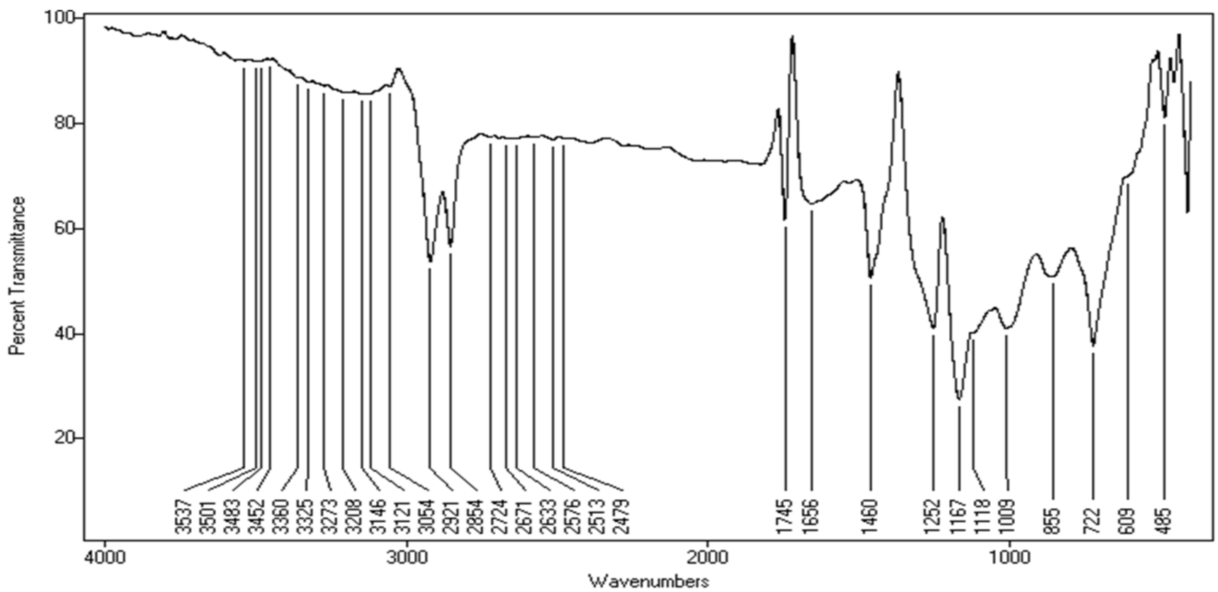


Fig. 4. FT-IR of the neem oil biodiesel.

By comparing the three spectra (Fig. 2, Fig. 3, and Fig. 4), it is found that in the spectra (Fig. 3) and (Fig. 4), transmittance between 3400 to 2854cm^{-1} has a noticeable change compared to spectra (Fig. 2), this indicates the separation of fatty acid attached to the triglycerides into biodiesel. Hence from the FT-IR spectral study, we can conclude that this new process has taken place successfully yielding bio-diesel.

C. Gas chromatography-Mass spectral studies

The composition of the final product, neem oil biodiesel produced was also confirmed by GC-Mass spectra (Fig. 5). The spectra showed eleven major peaks in the analysis representing the fatty acid methyl ester content of the neem oil biodiesel, out of these eleven peaks the relative abundance was good for five peaks at m/z = 242, 270, 294, 296 and 298, these peaks corresponding to the respective fatty acid methyl ester were identified using the library match software (NIST14.lib and WILEY8.LIB). The base peak at m/z = 74 which is a product of Mclafferty rearrangement [20] and characteristics of the saturated fatty acid methyl ester was observed, another base peak at m/z = 55 which is the characteristic fragmentation pattern of unsaturated fatty acid methyl esters was also observed [17]. The peak at, m/z = 242 corresponds to methyl tetradecanoate, m/z = 270 corresponds to methyl hexadecanoate, m/z = 294 corresponds to methyl-9,12-octadecadienoate,m/z = 296 corresponds to methyl-9-octadecenoate and m/z = 298 corresponds to methyl stearate. The relative abundance, as found in the GC-MS of the neem oil biodiesel (Fig. 5) agrees well with the fatty acid composition of the neem oil [21].

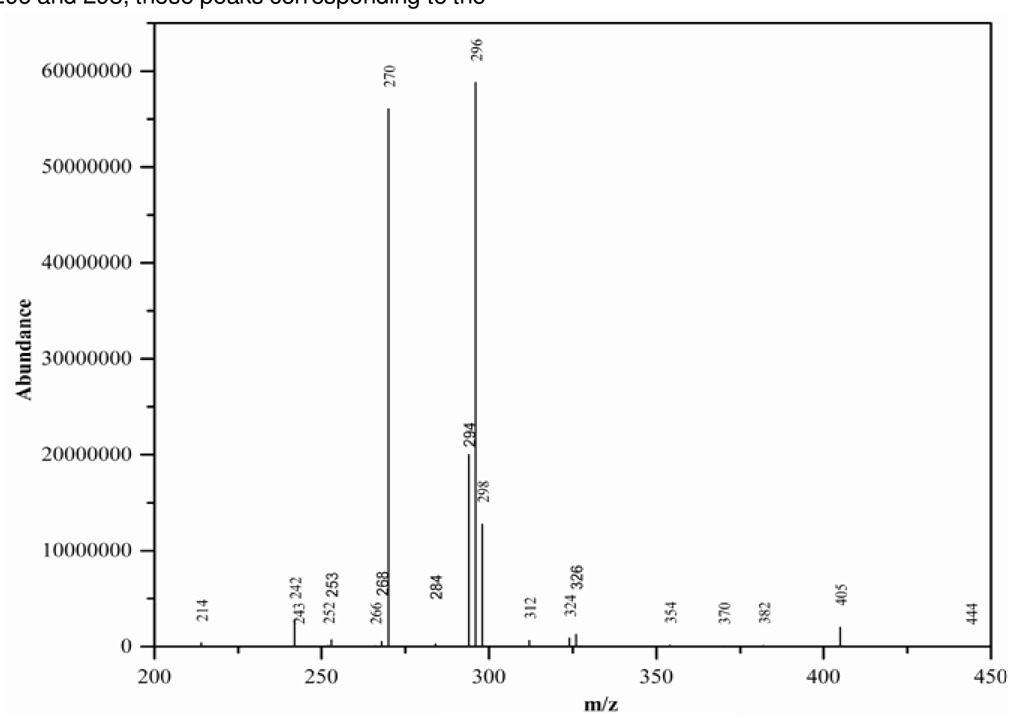


Fig. 5. Mass spectra of neem oil biodiesel.

VI. CONCLUSIONS

In this new process named as SAFALE (Saponification, Fatty Acid Liberation and Esterification), we have shown that there is one more way of producing biodiesel which is much superior to other types of production of biodiesel (Table 2).

This new process gives good yield, and the processing time is also short compared to other methods of biodiesel production from neem oil. Since the first two reaction steps involved in this new process are irreversible reactions, it proceeds to quick completion, and the complexity of the reversible nature of the reaction as found in esterification and transesterification is avoided. The steps involved in this process are simple and does not require any skilled manpower. This process may also be carried out by using ethanol for esterification and avoid toxic materials like methanol. Since this new process, unlike other methods, does not generate any toxic by-product, the process is eco-friendly, and disposal of waste is not required as compared to the transesterification process or the two-step esterification followed by transesterification process, which involves washing of the biodiesel several times resulting in the discharge of effluent into the environment.

This new process is also more economical compared to the other methods like the two-step (esterification followed by transesterification) process since the latter process requires the use of an excess of methanol or ethanol to shift the equilibrium in the esterification step and its recovery by distillation before going to the second step of transesterification. The by-products of this new process such as glycerol and sodium sulphate can be used for various other applications. This process does not require high temperature and pressure, compared to some other methods of production of bio-diesel. This process can also be used for the production of bio-diesel from neem oil, which has very high or containing any percentage of free fatty acids. This process can be done in small batch sizes as well as large batch sizes for industrial production of bio-diesel. The final step, in this process (i.e., esterification of free fatty acid.), may not proceed to give 100 percent ethyl ester of fatty acid, this is because the shifting of the equilibrium largely depends on the continuous removal of water. Even if the conversion is around 99 percent, it will not affect the quality and quantity of the bio-diesel produced, because the FFA will exist as a perfect blend with the ethyl ester of FFA. GC-MS and FT–IR spectral studies confirm the products and the new process of production of bio-diesel by this method. The future scope of this work is that it can be applied for the production of biodiesel of any non-edible oils [24-27] like neem oil.

Table 2: Comparison of different types of process and their yield of biodiesel production from neem oil.

Authors	Temperature (°C) & Pressure (atm)	Process type	Washing of the biodiesel	Time required to get the final product (Hours)	Conversion yield (%)
Ali, *et al.* [11]	55- 61& 1	Transesterification	Required	6-7	95
Awolu and Layokun [14]	60& 1	Two step esterification and transesterification.	Required	1-4	85
Radha and Manikandan, [15]	40 -70 & 1	Transesterification	Required	26	55-63
Sekhar *et al.* [12]	50-55 &1	Two step esterification and transesterification.	Required	2.5	75
Dhar *et al.* [5]	60 & 1	Two step esterification and transesterification.	Required	11	*
Sathya and Manivannan [13]	50-55 &1	Two step esterification and transesterification	Required	2	90
Sathya *et al.* [1]	60&1	Esterification	Required	1	92.5
Present work	50-55 & 1	SAFALE	Not required	1	99

*Not mentioned

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Conflict of Interest: We the authors of this research artical has no conflict of interst.

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