

# A New Process for the Production of Biodiesel from Used Vegetable Oil



A.Balasundaram, A.Cyril

**Abstract:** Production of biodiesel from triglycerides (vegetable oil) is usually done through transesterification with alcohols in the presence of acid or base catalyst. When this oil contains free fatty acid of more than 5 percent due to repeated use for frying, the transesterification process can not be used to produce biodiesel. In this paper, a new method for producing biodiesel even for used vegetable containing higher or any amount of free fatty acid is discussed. This method involves complete saponification of the triglycerides and free fatty acids (FFA) by reacting it with sodium hydroxide solution to give sodium salt of fatty acid (SOAP). This soap when treated with a mineral acid, separates into four layers, the top layer which is also the primary layer is the free fatty acid (FFA), the second layer is the glycerol, the third layer is the water layer, and the bottom layer is the residue of solid sodium salt. The top layer free fatty acid (FFA) is easily separated and then esterified with ethanol in the presence of a mineral acid as a catalyst to get the biodiesel (ethyl ester of FFA). The product obtained by the above process is analyzed and confirmed by FT-IR spectra. This process does not involve high temperature and pressure. The advantages of this new process are that it is much environmentally friendly compared with any other methods of production of biodiesel with less processes time. Since the washing of biodiesel to remove the soap formed due to the presence of free fatty acid content in the feedstock is completely eliminated, there will be no effluent discharge to the environment and at the same time resulting in more yield. The significance of this method is that it can be used to obtain biodiesel from used vegetable oils, raw vegetable oil and tallows and especially for any feedstock material which contains any amount of free fatty acids.

**Keywords:** Biodiesel, esterification, Triglycerides, Free fatty acids.

## I. INTRODUCTION

All vegetable oils and animal fats containing triglycerides and fatty acids, which can be converted into biodiesel (FAME or FAEE) have been an excellent alternative to the diesel derived from fossil fuel. Bio-diesel is a renewable source of energy and also reduces the net production of the greenhouse gas CO<sub>2</sub> when used as a fuel in compression ignition engines. Because of these advantages and the rising fossil fuel prices,

biodiesel produced from vegetable oils and animal fats has gained tremendous attention and use in the past few years. Transesterification is a general chemical process which converts one form of an ester to another form of an ester, here in biodiesel production the triglycerides, which is the ester of glycerol and higher fatty acids are converted into ethyl or methyl ester of fatty acids. There are many ways of producing biodiesel from vegetable oils (triglycerides), but all these processes involve the conversion of triglycerides to ethyl or methyl esters of fatty acids. The transesterification processes are classified on the basis of catalytic and non-catalytic conversion as a) Alkali catalyzed transesterification. b) Acid-catalyzed transesterification. c) Acid and alkali catalyzed two-step transesterification. d) Enzyme catalyzed transesterification and e) Non-catalytic supercritical transesterification. In alkali catalyzed transesterification, the most common method, in which the triglycerides present in the vegetable oils or fats react with alcohol (ethanol or methanol) using NaOH or KOH as a catalyst in a molar ratio of 3:1 to give the fatty acid methyl or ethyl ester (biodiesel) and glycerol. Since the reaction is reversible, excess of alcohol is usually used to get more products [1-4] (biodiesel). Bradshaw [2] carried out the transesterification process using 1.6 times the theoretical amount of methanol, which contained 0.1 to 0.5 % of sodium hydroxide to oil at 80<sup>o</sup> C and got 98% yield, he also observed that the presence of water and free fatty acid inhibit the reaction and decrease the yield. Sprules [3] and price in their patented process had used a higher molar ratio of 45:1, mainly when the triglycerides contained a large amount of free fatty acids. The main feature of using an alkali catalyst is the shorter reaction time at a relatively low temperature with less amount of catalyst, and because of these reasons, more than 60 % of the industrial plants use this catalyst [4]. But if the raw materials used for biodiesel production contain any water content [5] and free fatty acid of more than 1 %, soap formation will happen, and it will interfere with glycerol separation due to the formation of emulsion and also decrease the yield of biodiesel production. Heterogeneous catalyst such as MgO [6] and mixed oxides have also been used for the transesterification but with the yield of around 60 % at 130<sup>o</sup> C. Acid-catalyzed transesterification [7] is the process in which the triglycerides present in the oils and fats are treated with alcohol in the presence of inorganic acids such as sulphuric acid, hydrochloric acid, and nitric acid. Nye et al. [8] transesterified the used frying oil with methanol, ethanol, propanol, and butanol using both sulphuric acid and KOH as a catalyst, and the result showed that the acid-catalysed process had a relatively better yield than the base-catalysed process.

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## A New Process for the Production of Biodiesel from Used Vegetable Oil

But the acid-catalysed process took a long time and relatively higher temperature for the completion of the process. Ripmeester [9] transesterified the used cooking oil with a sulphuric acid concentration of 1.5 -3.5 mol % at 70°C, in a molar ratio of 50:1 (methanol to oil). Canakci and Gerpen [7] found that the process of transesterification, using the acid-catalysed gets stopped in many cases at an acid value well above 2mg KOH/g target, and this is due to the fact the water produced when free fatty acid reacts with alcohol reverses the equilibrium and stops the formation of biodiesel. The main advantage of the acid-catalysed process is that it is insensitive towards the presence of water content and the free fatty acid<sup>10</sup>, avoiding the formation of undesired soap, but requires more reaction time and the reactant alcohol. In acid and alkali catalysed two-step transesterification process, the first step is the esterification process in which the free fatty acid present in the raw material (used vegetable oil and tallows) is treated with the alcohol in the presence of acid catalyst to give the product biodiesel (FATTY ACID ETHYL OR METHYL ESTERS), so that the total free fatty acid of the reactant mixture decreases below 1 %. Now in the second step, the reaction mixture is transesterified with an alkali catalyst to convert the triglycerides into biodiesel and glycerol. Fan [11] et al. carried out the transesterification of recycled canola oil by following the two-step process of using acid catalyst first and followed by alkali catalyst, they found out that the optimum condition to reduce the free fatty acid level from 11 % to 0.42 % was to use a 40:1 molar ratio of methanol to oil with 5 % sulphuric acid catalyst at 55° C after 1.5 hours reaction time. After that the transesterification process was carried out with 1% potassium hydroxide catalyst in a 6:1 molar ratio of methanol to oil, he also mentioned about the disadvantage of the final purification step such as the traditional water washing and drying to remove the glycerol, soap, methanol, triglycerides and sodium hydroxide catalyst without which it can corrode the engine parts and choke the fuel injector, which adds to the increase in production cost and the time involved in getting the final product biodiesel. Wang [12] et al. compared the acid-catalysed transesterification with the two-step process in which the solid acid catalyst is used in the first step, followed by alkali catalysed transesterification in the second step. They found out that the optimum condition for the acid-catalysed process to get 90 % yield was to use the ratio of 20:1 (methanol: oil) at 95° C for 10 hours reaction time, and for the two-step solid acid-catalysed process the yield was 97 % in 4 hours reaction time using methanol to oil ratio 10: 1, 2 wt% ferric sulphate acid catalyst in the first step and 1 wt% KOH in the second step. Even though these type of process as claimed by many researchers is better than the acid catalysed or base catalysed transesterification for obtaining biodiesel from raw material containing higher free fatty acid, as in yellow grease (FFA < 15 %) and brown grease (> 15 %), there will be a small amount of free fatty acid left at the end of the first step because of the incomplete esterification process as a result of water formation, this free fatty acid and water will react with the alkali catalyst added in the second step of the process leading to soap formation and require a traditional water wash and drying. Therefore the most challenging part in this type of process will be the complexity of the instrumentation, removal, and recovery of the catalyst, recovery of the excess of methanol used, traditional water washing and neutralization of the acid catalyst leading to the higher cost of

production of biodiesel. In Enzyme catalysed transesterification process, the triglycerides and alcohol are treated with an immobilized lipase, which acts as a catalyst to produce biodiesel. Some of the merits of this type of process are, no by-product formation, temperature of operation is low, the enzymes can be used several times and one more main advantage is that it is not sensitive to the presence of free fatty acid and water, which enables us to transesterify waste cooking oil with a high percentage of free fatty acids [13]. There are various types of enzymes of lipase that is active in transesterifying the triglycerides and fatty acids into biodiesel such as *Thermomyces lanuginosus* [14], *Rhizopus oryzae* [15], *Pseudomonas fluorescens* [16], *Candida antarctica* [17], *Pseudomonas cepacia*[18] and *Novozym* [19]. Alkyl esters were produced from tallow and grease using two different alcohols (ethanol and methanol) with lipase, which is immobilized in pylosilicate sol-gel [20]. Biodiesel production was good (84-94%) when using ethanol, whereas when methanol was used, the yield was low(47-89%). Waste cooking oil was transesterified to biodiesel by immobilized lipase on hydrotalcite and zeolite catalyst [21] (4wt%) using 4:1 molar ratio of methanol to oil getting the highest yield of 95% with the reusability of 7 times for the catalyst. It is to be understood from the above studies that even though the enzymatic transesterification has some advantages compared to the other methods of biodiesel production the cost of production by enzymatic process is high [22], and also it suffers from the following limitations like, inhibition of enzyme due to glycerol, presence of excess of water, more reaction time and loss of yield due to excess of methanol if not added in steps [23]. In Non-catalytic supercritical transesterification process, the triglycerides and free fatty acids are treated with alcohol at a temperature and pressure above the critical point of the mixture to give biodiesel and glycerol in a short period with an almost near-total conversion. Saka [24] and Kusdiana made the fundamental study on the transesterification of the rapeseed oil in supercritical methanol with the critical temperature range of 350-400°C and critical pressure range of 45-65MPa in a molar ratio of 1:42 ( oil to methanol).since this process involves very high pressure and temperature construction and operation of large scale industrial plants are less feasible, and therefore further research work has been focussed on reducing the temperature and pressure of operation using some co-solvents [25] and solid metal oxide [26] catalyst. Jian-Zhong [27] et al. compared the transesterification process of the supercritical method with and without co-solvent (hexane or carbon dioxide) and potassium hydroxide. Under supercritical condition the biodiesel yield was 98% at the temperature range of 260° C to 350° C in a methanol to oil ratio of 42:1,when carbon dioxide or hexane is used as co-solvent along with 0.1wt% of potassium hydroxide and methanol to oil ratio of 24:1 the yield was 98 % but at 160° C. A patented process by Boocock [28] using tetrahydrofuran as a co-solvent gave a biodiesel yield of 99%, even with triglycerides containing up to 10% of free fatty acid at 30° C. In spite of having these advantages such as getting good yield in short time, this method suffers from some severe demerits such as operating at very high temperature and pressure,

presence of free glycerol above the international standards and carcinogenic effect of some co-solvents like tetrahydrofuran. All the above process of production of biodiesel suffers from some sort of disadvantages, still there is a need for inventing a better process which can handle any type of feedstock containing any percentage of free fatty acids, involving simple steps which can be done in small level of plants as well as scaled up to an large scale industry level and does not involve any discharge of toxic substance and waste in to the environment.

## II. ANALYTICAL METHODS

### A. Determination of physical properties

The biodiesel produced using this new process was tested for 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.

### B. Fourier transform-infrared spectroscopy analysis

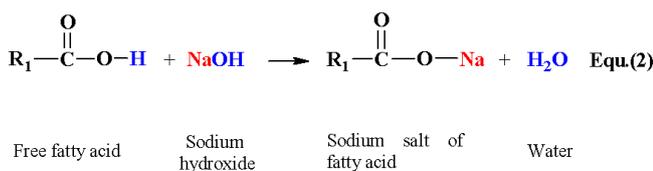
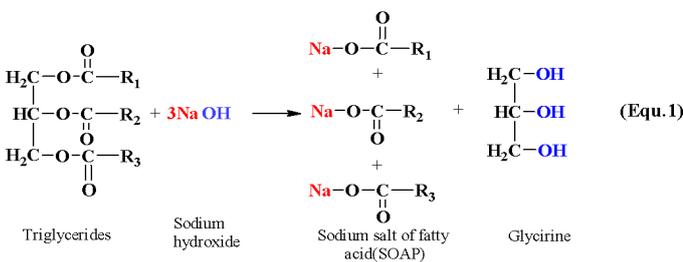
The used vegetable oil, the free fatty acids and its biodiesel were characterized by FT-IR spectrometer, using a Perkin Elmer make, Model-Spectrum Two, scanning between the range of 4000- 400 cm<sup>-1</sup> with a resolution of 1 cm<sup>-1</sup>.

## III. EXPERIMENTAL PROCEDURE FOR THE PRODUCTION PROCESS

### A. The new process for the preparation of biodiesel consists of the following steps:

#### Step -1 - Saponification:

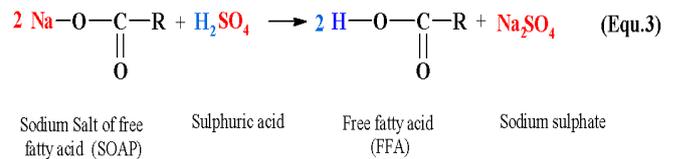
About 250 ml of the cooked vegetable oil is taken in a one-liter glass beaker and mixed with sodium hydroxide solution in the molar ratio 1: 3, preferably at 55-60°C. This mixture was continuously stirred with the help of a mechanical stirrer; thereby, all the cooked vegetable oil is converted into sodium salt of fatty acid (soap). The triglycerides and the free fatty acid present in the cooked vegetable oil react with sodium hydroxides to give soap, as shown in the equation (Equ.1 & Equ.2).



Thus, after the first step, all the used vegetable oil, which contains the triglycerides along with the free fatty acids, is converted into soap. Since these two reactions (Equ.1) and (Equ.2) are irreversible reactions, the products are formed immediately, and the reaction proceeds to completion in 15 minutes.

### Step -2-Liberation of free fatty acid

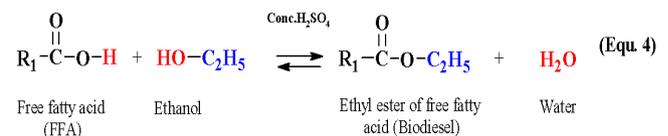
This highly viscous soap which will be in the liquid state is mixed with 6M sulphuric acids with constant stirring. After which the reaction mixture separates into four distinct layers. The top layer is free fatty acids (FFA); below this layer is glycerol, then water as the third layer and the fourth bottom layer consists of solid sodium sulfate crystals. Since this reaction of soap with sulphuric acid is irreversible and exothermic, the reaction gets completed within 4 to 5 minutes as represented in the equation (Equ.3)



The free fatty acid (FFA) is separated using a separating funnel and used for the esterification process to produce biodiesel.

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

A mixture of ethanol and sulphuric acid is taken and then added to the free fatty acid (FFA) at 55°C in the molar ratio of (0.05:1:10) (Sulphuric acid: FFA: ethanol). The small amount of sulphuric acid acts as a catalyst as shown in the equation (Equ.4), the mixture is then stirred for about 50 minutes, which results in the formation of the ethyl ester of FFA (bio-diesel) and small quantities of water. The bio-diesel which is separated from the mixture is cloudy due to the presence of small amounts of water, which is heated to 100°C for few minutes with stirring to remove the water by evaporation, leaving a pure form of bio-diesel.



The new process of production of biodiesel from vegetable oil is graphically represented as shown in the flow diagram (Fig.1)

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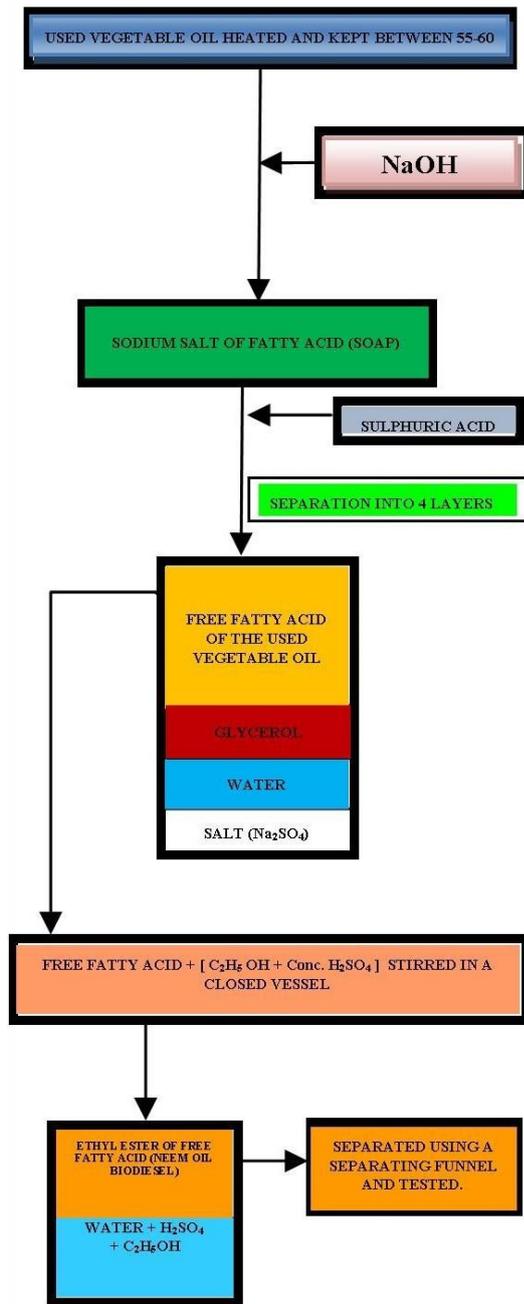


Fig.1-New process for the production of biodiesel from the used vegetable oil.

## B. Calculation of yield of biodiesel obtained by the new process from used vegetable oil

When 250ml of the used vegetable oil was subjected to this new process of biodiesel production, the yield of biodiesel was 231.7ml and 16.1 ml of glycerin. The total yield biodiesel produced using the new process from the used vegetable oil was calculated by using the equation (Eq.5)

$$\begin{aligned} \text{Percentage yield} &= \frac{\text{Volume of biodiesel produced}}{(\text{Volume of oil taken} - \text{Volume of glycerin})} \times 100 \quad (\text{Eq.5}) \\ &= \frac{231.7}{(250 - 16.1)} \times 100 \\ &= 99.00 \end{aligned}$$

## IV. ANALYSIS OF THE OBTAINED BIODIESEL

The product biodiesel obtained from the used vegetable oil by the new process is analyzed by FT-IR spectroscopy. The most important physical properties such as density, acid number, flash point, cloud point, kinematic viscosity, pour point, and of the used vegetable oil biodiesel produced by using the new process were determined as per ASTM methods (Table 1).

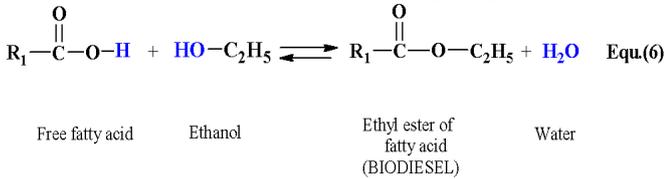
Table 1 Properties of the biodiesel produced from used vegetable oil using the new method

Physical properties	ASTM method used	ASTM D6751 Biodiesel	Used vegetable oil Biodiesel
Density (40° C, g cm <sup>-3</sup> )	D5002	0.86 – 0.90	0.86
Kinematic viscosity (40°C, mm <sup>2</sup> s <sup>-1</sup> )	D445	1.9 – 6.0	5.7
Acid number (mg KOH g <sup>-1</sup> )	D664	0.5	0.13
Flash point (°C)	D93	100 -170	131
Cloud point (°C)	D2500	-3.0 to 12	3
Pour point (°C)	D97	-15 to 16	-11
Water and sediment content (% vol. max.)	D2709	0.050	0.04

## V. RESULT AND DISCUSSIONS

The production of biodiesel by the new process was carried out with 250 ml of used vegetable oil. It resulted in the production of 231.7ml of free fatty acid and 16.1ml of glycerin. Hence around 99.00 percentage of the used vegetable oil taken, is converted in to free fatty acid and then into biodiesel. The conversion efficiency should be 100 percent, since the whole of the oil taken, was converted into (sodium salt of fatty acid) soap and all the soap is converted back into free fatty acid. The loss of one percent of free fatty acid maybe because of the incomplete separation from the four-layer mixture got in the

second step and some free fatty acids sticking to the walls of the container and separating funnel. The reported yield values are the average of five trials conducted. This free fatty acid is esterified with ethanol (1:10 ratio) in the presence of a small amount of the catalyst concentrated sulphuric acid to give ethyl ester of fatty acid (Biodiesel). The esterification reaction to give bio-diesel is equilibrium dependent (Equ.6). The shifting of the equilibrium to the product side largely depends on the removal of water produced during the process.



Since the final step of production of biodiesel (esterification) is a reversible reaction, getting a hundred percentage yield may not be possible. But even though, if the bio-diesel contains some traces of unreacted free fatty acid (within 0.5 mg of KOH g<sup>-1</sup>, as per the ASTM standards), it will not affect the quality of the biodiesel because the free fatty acid present mixes well with the biodiesel and does not give rise to phase separation or combustion problems. In this new process, there is no wastage of the feedstock in the form of soap compared to other process followed which involves washing of soap and discharging into the environment and responsible for the loss of yield. The whole process is completed in less than forty-five minutes. Since the first two steps involved in this new process is an irreversible reaction formation of the products and completion of the reaction takes place in a very short time compared to other processes which involve reversible reactions operating at this temperature and pressure for the production of bio-diesel.

#### A. Physical properties of the produced neem oil biodiesel

The physical properties of the used vegetable oil biodiesel produced using the new process were tested using ASTM methods and compared with the ASTM D6751 specification for biodiesel. (Table 1).

Kinematic viscosity is the most important property of any biodiesel which affects the fuel injection pattern, and hence the combustion of the fuel inside the combustion chamber of the engine, the kinematic viscosity of the biodiesel produced from the used vegetable oil was measured using the D445 method at 40°C which was 5.7 mm<sup>2</sup>s<sup>-1</sup> and is within the ASTM D6751 range. The density of the produced biodiesel was determined by employing D5002 method at 40°C, which is 0.86g cm<sup>-3</sup> and is within the ASTM D6751 range.

The flashpoint of the used vegetable oil biodiesel was 126°C and found within the ASTM D6751 standards, which will not have any problem with the combustion process. The acid value of the biodiesel produced using the used vegetable oil was 0.13 mg KOH g<sup>-1</sup>, estimated using the D664 method and is well below the maximum value of 0.5 mg KOH g<sup>-1</sup> of ASTM D6751 standard for biodiesel. The cloud point and pour point of the used vegetable oil biodiesel was also measured using the D2500 and D97 methods, which predicts the cold flow properties and its usage in various climatic conditions. The pour point of the produced biodiesel was -11°C, and the cloud point was found to be 3°C, which is also within the ASTM D6751 standards. Water and sediment content of the used vegetable oil biodiesel were also within the ASTM D6751 limits, determined using D3709 methods.

#### B. FT-IR spectral studies

The new process employed for the conversion of the used vegetable oil to biodiesel has been confirmed by FT-IR spectra. The spectrum was taken for the used vegetable oil, the free fatty acids produced in the second step of the process, and the used vegetable oil biodiesel produced in the final stage of the process. The carbonyl group present in the ester functional group can be an evidence for the formation of biodiesel which is alkyl ester of free fatty acid, the stretching frequency of C = O group in an ester depends upon the presence of the substituent attached to it in an FT-IR spectrum [29]. A strong absorption band around 1700-1750 cm<sup>-1</sup> arises out of asymmetric axial stretching and antisymmetric axial stretching of the C-O and C=O groups respectively [30,31] confirming the ester functional group.

#### FT-IR spectra for the used vegetable oil

The FT-IR spectra show a strong absorption band at 1752 cm<sup>-1</sup> for the used vegetable oil (Fig.2) is due to the C = O stretching of the tri-ester present in the used vegetable oil. The overtones produced by the C = O group of the esters is shown by the peak at 3480 cm<sup>-1</sup>, and the presence of long alkyl hydrocarbon chain is shown by the C – H stretching at 2982 cm<sup>-1</sup> and 2918 cm<sup>-1</sup>. The ester group of the triglycerides in the used vegetable oil also shows a decrease in absorbance between 3400 cm<sup>-1</sup> and 3100cm<sup>-1</sup>.

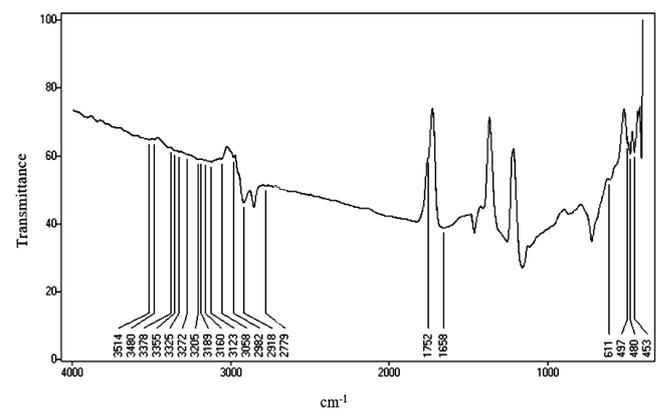
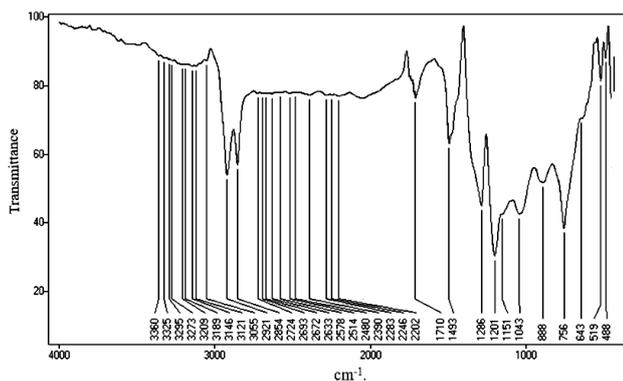


Fig.2-FT-IR spectra of used vegetable oil

#### FT-IR spectra for the free fatty acids produced from used vegetable oil

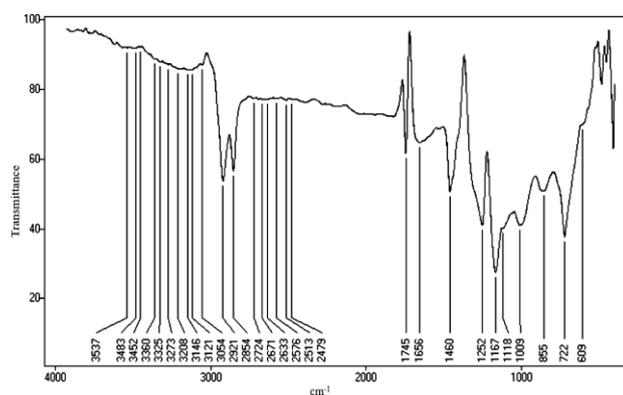
The free fatty acid liberated from the sodium salt of fatty acid of the used vegetable oil in the second step of the process is confirmed by FT-IR spectra (Fig.3). The C=O stretching of the carboxylic acid group in the free fatty acid is got at 1710 cm<sup>-1</sup>. The O–H stretching of the carboxylic group is got around 3000 cm<sup>-1</sup> which begins at about 3300cm<sup>-1</sup> and slopes into the aliphatic long alkyl chain C–H absorption bands of 2921 cm<sup>-1</sup> and 2854 cm<sup>-1</sup> and centered around 3000 cm<sup>-1</sup>.



**Fig.3-FT-IR spectra of free fatty acid of the used vegetable oil.**

### FT-IR spectra for the biodiesel produced from the used vegetable oil

The biodiesel produced from the free fatty acid of the used vegetable oil by esterification in the third step of the new process was confirmed by FT-IR spectra (Fig.4). The ester group of the ethyl ester of fatty acid showed a peak at  $1745\text{ cm}^{-1}$ , which is due to the C=O stretching. The peak at  $2921\text{ cm}^{-1}$  and  $2854\text{ cm}^{-1}$  due to C – H stretching of the alkyl chain and the overtones at  $3483\text{ cm}^{-1}$  confirms the presence of fatty acid of ethyl esters. (BIO-DIESEL).



**Fig.4 –FT-IR spectra of ethyl ester of fatty acid (BIO-DIESEL)**

By comparing the three spectra of used vegetable oil, free fatty acid, and biodiesel (Fig.2, Fig.3, and Fig.4), it is found that in the spectra of free fatty acid and biodiesel, transmittance between  $3400\text{ to }2854\text{ cm}^{-1}$  has a noticeable difference compared to spectra of used vegetable oil, this indicates the detachment of fatty acid part from the triglycerides of the used vegetable oil into biodiesel. Hence the new process has been confirmed yielding bio-diesel from the FT-IR spectral study.

## VI. CONCLUSIONS

Since this new process involves the production of biodiesel by saponification, fatty acid liberation, and esterification, it is named as SAFALE. In addition to the different methods of production of biodiesel such as transesterification, two-step esterification followed by transesterification, enzymatic transesterification, and supercritical transesterification

process we have invented one more method of producing biodiesel which is much better in handling feedstock containing higher fatty acids. There is a possibility of getting close to hundred percent yield at low temperature and pressure in this new process compared to the transesterification and two-step esterification followed by transesterification process because in the older process, the higher percentage of free fatty acids leads to soap formation which has to be removed by washing the product and discharging it as an effluent in to the environment, these two drawbacks are completely eliminated in this new process and hence it is totally ecofriendly. The time taken for the completion of the process is also less compared to the transesterification process and the two-step process because the reactions involved in the first and the second step of the new process are irreversible reactions, and hence the products are formed completely without getting reversed as in the case of transesterification and esterification process involving reversible reactions. This new process is also more economical in terms of energy and material consumption, since in the, two-step process (esterification followed by transesterification) for biodiesel production from feedstock containing higher free fatty acids requires the use age of excess of ethanol for esterification using the acid catalyst to convert all the free fatty acid into biodiesel. This excess of ethanol has to be removed by fractional distillation before proceeding to the transesterification process to produce biodiesel, which also makes the instrumentation of the biodiesel production in the bulk process more complex. This new process called SAFALE can be applied for the production of biodiesel from feedstock containing triglycerides, triglycerides with any percentage of free fatty acids and tallows.

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