



## International Journal of Current Research and Academic Review

ISSN: 2347-3215 Volume 2 Number 1 (January, 2014) pp. 105-120

[www.ijcrar.com](http://www.ijcrar.com)



### Biodiesel characterization and optimization study of used frying palm oil

K.Dhanasekaran<sup>1\*</sup> and M.Dharmendirakumar<sup>2</sup>

<sup>1</sup>Department of Chemical Engineering, Arulmigu Meenakshi Amman College of Engineering, Vadamavandal-604 410, India

<sup>2</sup>Department of Applied Science and Technology, Alagappa College of Technology, Anna University, Chennai-600 025, India

\*Corresponding author

#### KEYWORDS

Biodiesel;  
Used frying oil;  
transesterification;  
Gas Chromatography;  
Palm oil.

#### A B S T R A C T

The utilization of liquid fuels such as biodiesel produced from used frying oil by transesterification process is represents one of the most promising optional sources for the use of conventional fossil fuels. This study describes the characterization and optimization parameters of used frying oil to biodiesel. The process variables were employed i.e., methanol to oil molar ratio (3:1-9:1), catalyst (KOH, NaOH, CH<sub>3</sub>OK, and CH<sub>3</sub>ONa), catalyst (0.5, 1.0 and 1.5 wt %), temperature (50, 60, and 70°C), time (30, 60, 90 and 120 min) and alcohols (CH<sub>3</sub>OH, C<sub>2</sub>H<sub>5</sub>OH, and C<sub>3</sub>H<sub>7</sub>OH) used this experiments. The optimized biodiesel parameters were: KOH Catalyst, 6:1 molar ratio, 1.0 wt % of catalyst concentration, 60°C temperature, and 120 min reaction time. The GC Techniques were used to identify the fatty acid profile of the used frying oil and biodiesel and FTIR Technique is used to identify the peaks formed in final biodiesel product.

### Introduction

The worldwide worry about the protection of environment and the conservation of non-renewable natural resources, has given rise to alternate development of sources of energy as substitute for traditional fossil fuels. The major part of all energy consumed worldwide comes from fossil sources (petroleum, coal and natural gas). However, these sources are limited and will

be exhausted in the near future. Thus, looking for alternative sources of new and renewable energy such as hydro, biomass, wind, solar, geothermal, hydrogen and nuclear is of vital importance. Alternative new and renewable fuels have the potential to solve many of the current social problems and concerns, from air pollution and global warming to other environmental

improvements and sustainability issues (Anitha *et al.*, 2010). So, used frying oil can be used as a biodiesel sources and partially improve the global problems. In addition, the utilization of used frying oils diminishes the problems of contamination, because the reusing of these waste greases can reduce the burden of the government in disposing of the waste, maintaining public sewers, and treating the oil wastewater (Encinar *et al.*, 2007). The use of waste cooking oil as biodiesel feedstock reduces the cost of biodiesel production (Canakci *et al.*, 2007). Since the feedstock costs constitutes approximately 70-95% of the overall cost of biodiesel production (Connemann *et al.* 1998). Hence, the use of waste cooking oils and non-edible oils should be given higher priority over the edible oils as biodiesel feedstock (Arjun B Chhetri *et al.*, 2008). Generally, biodiesel is produced by means of transesterification reaction of a oil with an alcohol to form fatty acid methyl esters (FAME) and a byproduct of glycerol. Considerable research has been conducted to investigate the production of biodiesel from waste oil under acid (Zheng *et al.*, 2006), alkaline (Encinar *et al.*, 2005; Felizardo *et al.*, 2006) and enzyme catalysis (Watanabe *et al.*, 2001; Chen *et al.*, 2006). Waste cooking oils exhibit properties different than those of refined and crude oils.

The high temperatures of particular frying processes and the water from the foods accelerate the hydrolysis of triglycerides and increase the FFA content in the oil. Acid catalysis is more efficient when the amount of FFA in the oil exceeds 1% (Freedman *et al.*, 1984). Though acid-catalyzed transesterification is insensitive to FFA in the feedstock, it requires longer reaction time and higher temperature. Many researchers recommend using acid-catalysis as pretreatment step followed by alkaline

catalyzed step<sup>5</sup>. Wang *et al.*, (2007) were adopted this kind of two-step catalyzed process to prepare biodiesel from used frying oil. In the first step, FFAs of used frying oil were esterified with methanol catalyzed by ferric sulfate. In the second step, the triglycerides in the used frying oil were transesterified with methanol (methanol/oil molar ratio=6) catalyzed by 1.0 wt% potassium hydroxide at 65 °C for an hour. After this two-step catalysis process, the final product with 97.02% conversion of biodiesel was obtained. One alternative reaction conditions suitable for pilot-scale and industrial-scale biodiesel production purposes were found to be: Oil/alcohol molar ratio, 1:6; temperature, 55± 1 °C; NaOH amount, 1% (by the weight of the oil); stirring speed, 40 rpm; pressure, atmospheric; and reaction time, 60 min. Nevertheless, some researchers (Meher *et al.*, 2006; Gerpen *et al.*, 2005 and Tiwari *et al.*, 2007) have pointed out that the Alkali-catalyzed transesterification could be completed as long as the FFA content in the oil is not greater than 1% and all materials should be substantially anhydrous. Basic catalysts lead to higher conversion of methyl esters at low temperature, atmospheric pressure and minimum response time, which reduces the cost of the process considerably (Ghadge *et al.*, 2005).

Alkaline metal hydroxides (KOH and NaOH) are cheaper than metal alkoxides (CH<sub>3</sub>OK, CH<sub>3</sub>ONa), but less active. In this sense, the reactions that used sodium or potassium hydroxides were faster than the reactions with the sodium or potassium methoxides. Nevertheless, they are a good alternative since they can give the same high conversions of vegetable oils just by increasing the catalyst concentration to 1 or 2 mole%. However, even if a water-free alcohol/oil mixture is used, some water is

produced in the system by the reaction of the hydroxide with the alcohol.

In the present study, Used frying oil, which is otherwise wasted, is one of the most economical choices to produce biodiesel. Since one of the major concerns on biodiesel production is the price of feedstock, utilization of used frying oil significantly enhances the economic viability of biodiesel production.

### Materials and Methods

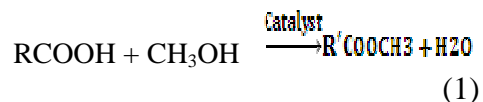
The material sources were: used frying oil is collected from local restaurants with high FFA content and fresh palm oil is purchased from local oil mill. Methyl alcohol of 99.9 % purity, ethyl alcohol of 99.9 % purity, propyl alcohol of 99.9 % purity was purchased from SD Fine Chemicals, Mumbai. The catalysts like KOH pellets of 98.2 % purity, NaOH pellets of 97 % purity, CH<sub>3</sub>OK of 98 % purity, CH<sub>3</sub>ONa of 98 % purity were purchased from Nice Chemicals, Kerala. The gas chromatographic values for methyl esters were obtained from Sargam Laboratory, Chennai and the FTIR Technique values were obtained from Department of Chemistry, Anna University, Chennai, India.

### General procedure

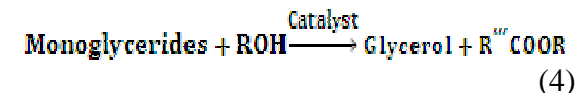
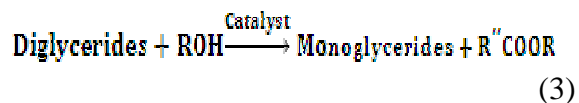
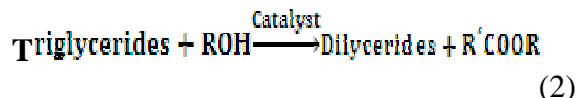
Used frying oil is collected from local restaurants and heated to 110°C and removes the water molecules then filter it by 10µm cloth to separate all food particles and suspended materials. Generally, biodiesel production is strongly influenced by FFA content of the used frying oil and the amount of catalyst had impact of conversion of esters during the transesterification process. Owing to its high FFAs, the transesterification of used frying oil to biodiesel catalyzed directly

used longer reaction time and only lower biodiesel yield can be achieved. So, a two-stage process is followed to convert the biodiesel from used frying oil. First stage, an acid-catalyst (sulfuric acid) was used to esterify (pretreatment) with used frying oil. For that, a three-neck flask with a water-cooled condenser was filled with 200 ml of used frying oil, 40 ml of anhydrous methanol and 4 ml of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). The mixture was vigorously stirred for 1.5h at 50°C. After reaction, the mixture was filtered and the unreacted methanol was separated from the liquid phase via distillation. The pretreated oil is washed three times with sodium chloride solution and then dried using anhydrous sodium sulfate. After the pretreatment, the FFA value of the pretreated oil was lesser than that of freshly used cooking oil.

This esterification reaction was described as below:



The second stage, a homogeneous catalyst (NaOH, KOH, CH<sub>3</sub>ONa, and CH<sub>3</sub>OK) is used to transesterify the pretreated oil:



The reaction procedure was as follows: first, the catalyst was dispersed in methanol using stirring system was connected to it. Then, the pretreated oil was added into the

Strirring mixture and heated to 50°C for three hours. After thorough mixing and kept it overnight; two layers were formed. In that, the upper layer consists of biodiesel whereas the lower layer consists of Glycerin and this layer consist of excess methanol, unreacted catalyst and soap. This unreacted methanol was distilled off under a vacuum condition while biodiesel layer was separated with the help of separating system and finally remaining glycerin layer is separated and then distilled to get 98 % purified glycerin. The separated biodiesel is purified with hot water until the p<sup>H</sup> gets neutral. These purified methyl ester compositions were analyzed by using Gas Chromatograph, FTIR technique. Now, this biodiesel can be characterized with effect of temperature, catalyst, mole ratio of oil to methanol and reaction time.

## **Result and Discussion**

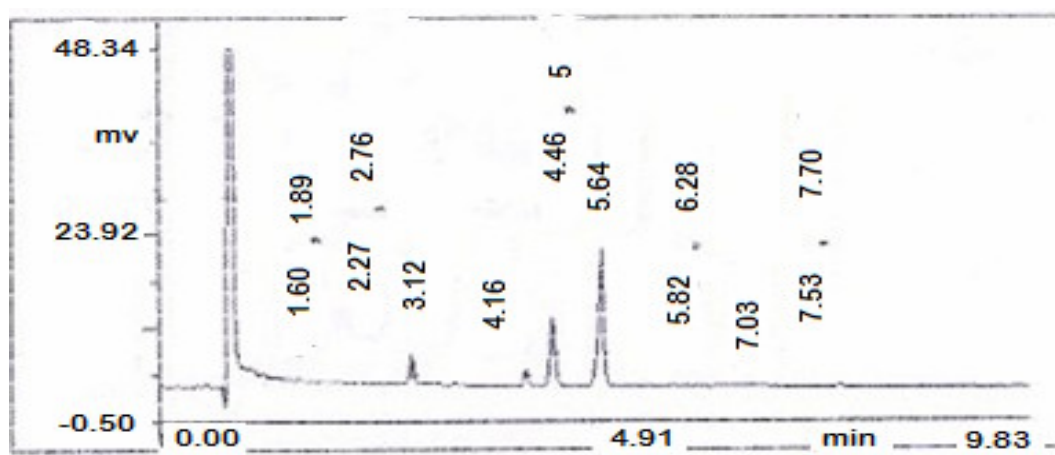
### **Fatty acid profile of used frying oil**

Fatty acid composition of used frying oil was determined by using gas-chromatography. Thus fatty acids are converted into methyl esters using alkaline methanolysis. The chromatogram obtained from the gas chromatographic analysis is

shown in figure 3.1. The fatty acid composition of used frying oil determined using gas chromatography with flame ionization detector is shown in table 3.1. Figure 3.1 and table 3.1 shows that the major fatty acid is Linoleic acid with a mass percentage of 52.50%, followed by Oleic acid with a mass percentage of 28.04%. The results obtained from table 3.1 were used to determine the fatty acid composition according to degree of unsaturation, given in table 3.2.

The results obtained for the other characteristics studied for used frying oil: iodine value, acid value, and water content are given in table 3.3, and discussed here. Iodine value is expressed in grams of iodine which reacts with 100 grams of fat or oil under certain conditions, being a parameter that quantifies the degree of unsaturation of the fat/oil. The iodine value obtained for the used frying oil was 116 g iodine I<sub>2</sub>/100 g of oil, Concerns about possible problems caused by biodiesel on engines were often assigned to high iodine values although studies have shown that biodiesel stability depends, besides the degree of unsaturation, on the content of antioxidants and the production technology used.

**Fig.3.1** Chromatogram obtained for used frying oil



**Table.3.1** GC Fatty acid composition of used frying oil

Common Name	C: D	Retention Time, (min)	Content, (%) (m/m)
Lauric Acid	C12:0	4758	0.25
Myristic acid	C14:0	7168	0.38
Palmitic acid	C 16:0	184101	9.84
Stearic acid	C18:0	107263	5.74
Oleic acid	C18:1	524447	28.04
Linoleic acid	C18:2	981802	52.50
Linolenic	C18:3	1255	0.07
Arachidic acid	C20:0	4271	0.22

C=Carbon atoms, D=Double bond

**Table.3.2** Fatty acid composition of used frying oil according to degree of unsaturation

Fatty Acid Composition	Content, (%), m/m
Saturated fatty acids	16.43
Monounsaturated fatty acids	28.04
Polyunsaturated fatty acids	52.57

**Table.3.3** Physico-chemical characteristics of used frying oil

Parameters	Determined value
Iodine value, (g I <sub>2</sub> /100g)	116
Acid value, (mg KOH/g)	4.61
Water content, (mg/kg)	150.2

Acid value is pointing out the presence of free fatty acids or acids formed as a result of the oil degradation and burning (during or after processing). The acid value represents the amount of base required to neutralize the oil sample and is expressed in mg KOH/g sample. This parameter is of great interest, especially for alkaline methanolysis where the presence of free fatty acids must be limited due to soap formation which leads to the formation of emulsions. The acid value of used frying oil used as feedstock for biodiesel production is 4.61 mg KOH/g oil, a value that indicates a high content of free fatty acids. Water is a minor component found in most raw materials for biodiesel production. In

general, high water content causes a decrease in ester yields as undesirable reaction occurs by hydrolysis of triglycerides. The water content determined for the used frying oil used as raw material was 150.2 mg/kg, a value below the maximum value allowed for biodiesel by the European quality standard EN 14214:2010, namely 500 mg/kg.

Free fatty acids (FFAs) content after acid esterification should be minimal or otherwise less than 2% FFAs. These FFAs react with the alkaline catalyst to produce soaps instead of esters. It depicts the effect of FFAs on the yield of methyl ester during alkali catalyzed transesterification. There is

a significant drop in the ester conversion when the free fatty acids are beyond 2% (Naik *et al.*, 2008). In spite of its insensibility to the free fatty acids from the raw materials, it was ignored, especially due to its low reaction rate (Al-Zuhair *et al.*, 2007). From Table 3.3, acid value is 4.61mgKOH/gm and the molecular weight is 833g/mol and this can be calculated using this saponification value (198.34mgKOH/gm) of the used frying oil. Due to High FFA, if this oil was used directly with base catalysts it would give only lower Biodiesel yield (74%) and this can be proved with a trial of 6:1 molar ratio of alcohol to oil with the addition of 1 wt % of each catalyst for 1 hour reaction timings. Figure 3.2 shows that, KOH is showing high biodiesel conversion when compared with other catalyst used for used frying oil and also this catalyst showing is highest catalytic activity than other catalysts used. Always, the fresh oil is shows higher biodiesel conversion when compared with used frying oil.

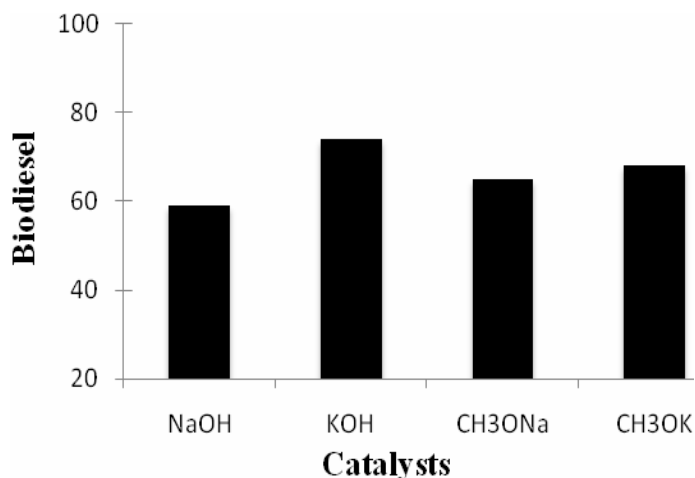
### Effect of Alcohols

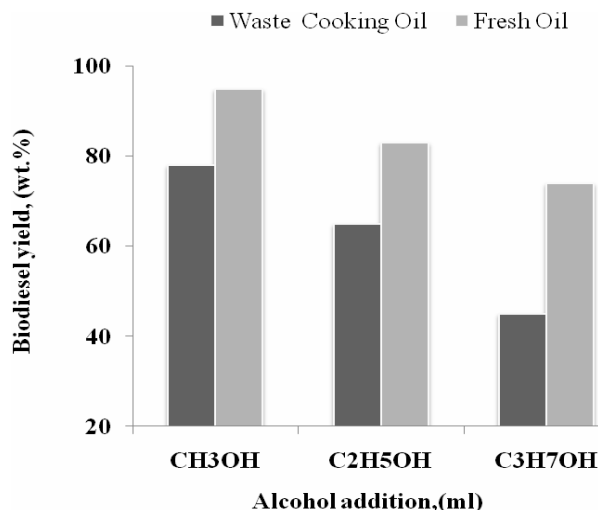
Alcohols are primary and secondary monohydric aliphatic alcohols having 1±8

carbon atoms (Sprules *et al.*, 1950). Among these alcohols, methanol, ethanol, propanol, butanol and amyl alcohol were used in transesterification process. Methanol and ethanol are most frequently used, especially methanol because of its low cost and its physical and chemical advantages (polar and shortest chain alcohol). This methanol can quickly react with triglycerides and NaOH is easily dissolved in it. To complete a transesterification reaction requires stoichiometrically a 3:1 molar ratio of alcohol to triglycerides. In practice, the ratio needs to be higher to drive the equilibrium to a maximum ester yield. So, the solubility of the alcohol is the main criteria to produce biodiesel from various oil sources

In this study, 1 wt % of KOH was used with three alcohols and these alcohols act as a solvent. First take three alcohols in each beaker then add the KOH catalyst into the alcohol containers and precede the solubility process with the help of magnetic stirrer.

Fig.3.2 Biodiesel yield with unesterified condition



**Fig.3.3 Biodiesel yield with catalyst solubility**

In that, methanol shows more solubility than other two alcohols used. Figure 3.3 shows solubility of the catalyst with respect to addition of the alcohols into the reaction vessel and according to literature methanol is more soluble than the other two alcohols because the increase in carbon content then solubility automatically will decrease. Now, in the second step these alkoxides are added into the used frying oil and fresh oil containers then mixed them 1 hour thoroughly and then kept overnight for separation of biodiesel and glycerol layers. Based on the results, biodiesel yield observed on methanol was higher than other alcohols used in this experiment. Nye *et al.*, (1983) described about the amounts of biodiesel produced by using different types of alcohol decreased in the following order: Methanol > Ethanol > Butanol and this result obtained was slightly different with the finding by them. They reported that methanol was the alcohol that can give the highest biodiesel yield, followed by butanol and then ethanol (Hossain *et al.*, 2010). According to Meher *et al.*, (2006), the production of biodiesel by using ethanol in alkali-catalyzed transesterification is more difficult than that by using methanol.

This is due to the formation of stable emulsion during ethanolysis. For methanolysis, the emulsions formed would break down easily to form a lower glycerol rich layer and upper methyl ester rich layer. While in ethanolysis, the emulsions formed are more stable due to the presence of larger non-polar group in ethanol, making the separation and purification of biodiesel more difficult (Zhou *et al.*, 2003). This explained why the biodiesel yield from ethanolysis was lower than methanolysis in this study. So, finally methanol is alcohol source used for this entire process.

#### Effect of molar ratio

Generally, the stoichiometry of the reaction requires 3 moles of methanol per mole of triglycerides to yield 3 moles of biodiesel and 1 mole of glycerol. Methanol is a commonly used alcohol for transesterification because of its low price and highly reactive nature (Lang *et al.*, 2001). For example, the molar ratio of 1:6 moles of soybean oil to methanol has been found to be the most suitable in the case of alkali-catalyzed transesterification because an excess of alcohol is required to drive the

reaction (Freedman *et al.*, 1986). For acid-catalyzed transesterification, a 1:30 vegetable oil to alcohol ratio is generally used<sup>11</sup>. In this experiment, used frying oil was used with varying molar ratios of methanol and oil (3:1, 6:1 and 9:1) and 1.0 wt% KOH at 60°C as represented in Figure 3.4. As mentioned, the transesterification activity also depends on the molar concentrations of methanol to oil and also associated with the type of catalyst used. If increased from 3:1 to 6:1, methyl esters content increased, then from 6:1 to 9:1, methyl esters content decreased much. Excess of methanol is required to shift the equilibrium favorably during transesterification for better yields of biodiesel. Therefore, 6:1 molar ratio gives a better result than other molar ratios and also repeated same procedure done for fresh oil used with varying molar ratios of methanol and oil (3:1, 6:1 and 9:1) and 1.0 wt % KOH used at 60°C were described by Figure 3.5. Figure 3.5 indicates that 6:1 molar ratio of alcohol to oil gives better biodiesel yield with Fresh Oil. The higher alcohol molar ratio interferes with the separation of glycerol because there is an increase of solubility. In addition, an excess of alcohol was able increase the conversion of di-monoglycerides, but there is

possibility of recombination of esters and glycerol to form monoglycerides because their concentration and also increasing during the course of the reaction, in other words the reactions conducted with low molar ratios. Many researchers have reported an alcohol to oil molar ratio of 6:1 to be the optimal ratio, while Leung and Guo. (2006), reported that the maximum biodiesel production was obtained at a molar ratio of 7:1 in transesterification of used frying oil. In base catalysis, this effect can be observed quickly because the reaction is fast, whereas in acid catalysis requires with high concentration of the reaction. These results are similar that where obtained in the ethanolysis of peanut oil in Feuge and Gros. (1949), Freedman *et al.*, (1986) and Schwad *et al.*, (1987) described about the ethanolysis of sunflower oil. In general, an acid catalyst requires a higher molar ratio of methanol/oil than basic catalysts (Fangrui *et al.*, 1999). In consequence, the alcohol/oil molar ratio is one of the most important variables affecting the esters yield and although the stoichiometry ratio for transesterification required 3 moles of alcohol and 1 mole of triglyceride, excess of alcohol was used in practice.

**Fig.3.4 Biodiesel yield with molar ratios of used frying oil**

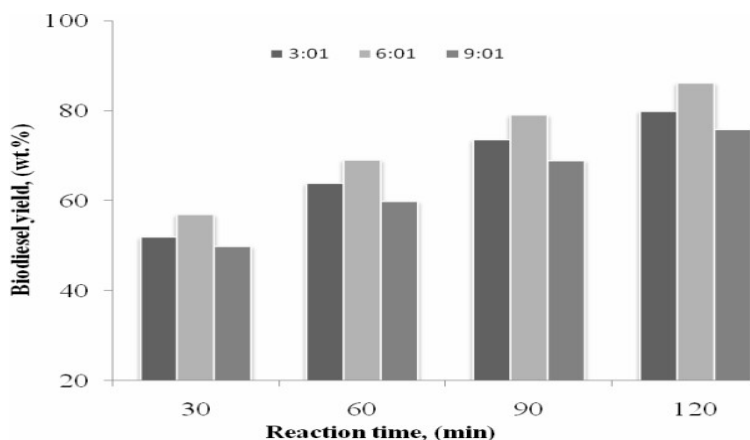




Fig.3.5 Biodiesel yield with molar ratios of fresh oil

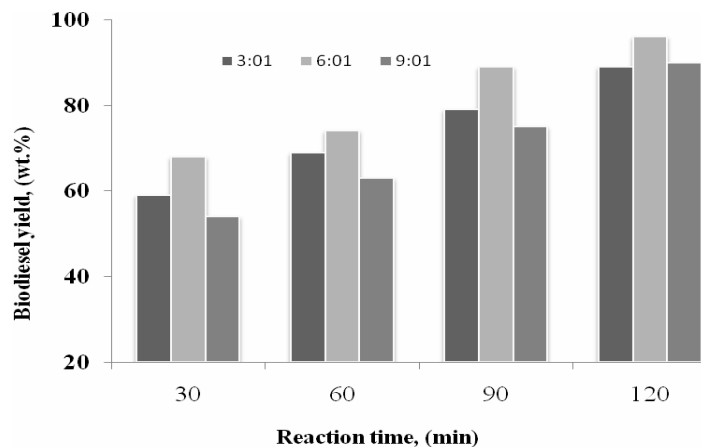
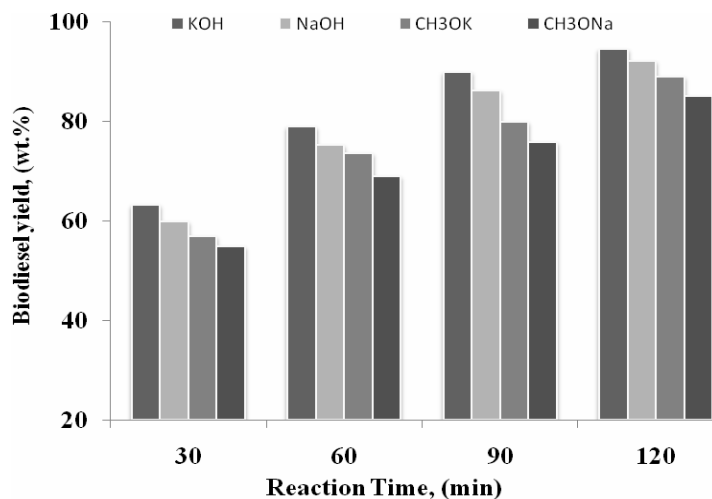


Fig.3.6 Biodiesel yield with catalyst used for used frying oil



Hence, the alcohol molar/oil ratio is an important factor that must be always optimized (Allen, 1998).

### Effect of catalyst

In this case, the experiments were carried out at 6:1 molar ratio of alcohol to oil used 1 wt % of KOH, NaOH, CH<sub>3</sub>OK, and CH<sub>3</sub>ONa with varying temperature ranges at 30, 60, 90 and 120 min for both used frying oil and fresh oil. Figure 3.6 and 3.7 shows the biodiesel yield with catalysts used with varying time in the molar ratio of 6:1 at 60°C for used frying oil and fresh oil.

Figure 3.6 and 3.7 show that KOH catalyst was the better biodiesel yield behavior than other catalysts used here (i.e., NaOH, CH<sub>3</sub>OK, and CH<sub>3</sub>ONa) and also higher time of mixing had proved higher biodiesel yield and the figure also indicates that hydroxides gave rise to higher percentages than the corresponding methoxides. These results are very similar to those found in the literature in transesterification processes of used frying oil with methanol and other alcohols that also concluded that potassium hydroxide was the best catalyst (Tomasevic *et al.*, 2003; Xiaohu *et al.*, 2009). The reaction can be catalyzed by alkalis, acids,

or enzymes. The alkalis include NaOH, KOH, carbonates and corresponding sodium and potassium alkoxides, such as, sodium methoxide, sodium ethoxide, sodium propoxide and sodium butoxide. Acid catalysts are usually sulfuric acid; sulfonic acids and hydrochloric acid. Alkali-catalyzed transesterification is much faster than acid-catalyzed transesterification and is more often used commercially.

Several researchers found that alkali-catalyzed transesterification proceeds much faster than acid catalyzed transesterification. Based on the above results KOH was the best catalyst compared with other base catalyst and also studied with 0.5, 1.0 and 1.5 wt % of KOH catalyst for both used frying oil and fresh oil at 60°C with 6:1 molar ratio of alcohol to oil. Figure 3.8 and 3.9 show that 1.0 wt % showed the higher yield than 0.5 wt % and 1.5 wt % of KOH used. In general, as the catalyst concentration increased, the conversion of triglycerides also increased. This is because an insufficient amount of catalyst results in an incomplete conversion of triglycerides into fatty acid esters<sup>20</sup>. In basic catalyst, a slight decrease in ester

content was observed in the experiments with 1.5 wt % of catalyst with regard to the experiments with 1 wt %. This is because the addition of excess alkaline catalysts caused more triglycerides participation in the saponification reaction, resulting in increased production of soap and mentioned reduction of the esters yield (Krishnangura *et al.*, 1992). So, any increase in concentration of catalyst beyond the neutralization limit results in decrease in biodiesel conversion.

### Effect of Temperature

In this process, the temperature ranges were used 50, 60 and 70°C and in order to optimize biodiesel yield and that temperature can influence the rate of reaction. For all temperatures, alcohols to oil molar ratio was 6:1 with KOH concentration of 1 wt. % for used frying oil as well as fresh oil. Figure 3.10 shows 60°C higher biodiesel yield than the temperatures of 50 and 70°C for used frying oil. After 20 min, biodiesel yields were 54, 57 and 51 wt % with respect temperatures of 50, 60 and 70°C. After 120 min, the biodiesel yields were 85, 91, and 79 wt % of biodiesel yield for the temperatures of 50, 60 and 70°C.

**Fig.3.7 Biodiesel yield with catalyst used for fresh oil**

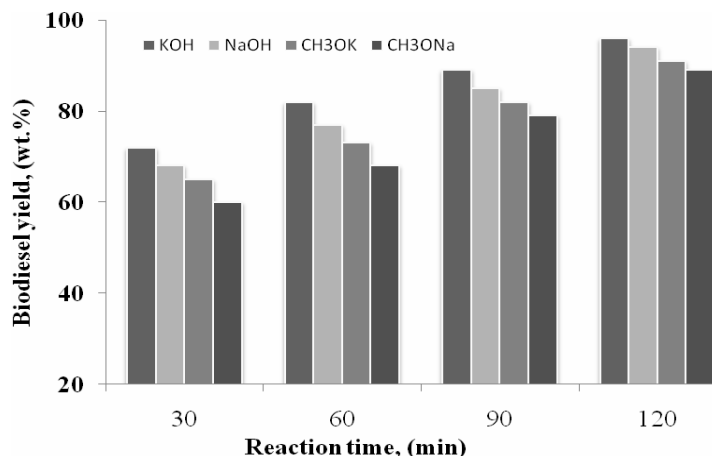


Fig.3.8 Biodiesel yield with concentration of catalyst for used frying oil

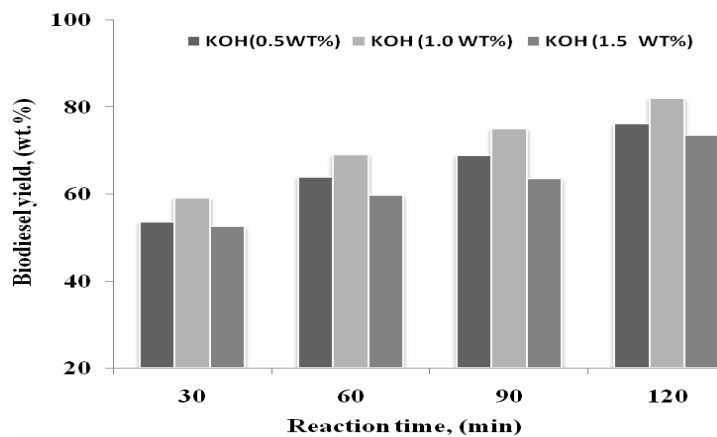


Fig.3.9 Biodiesel yield with concentration of catalyst for fresh oil

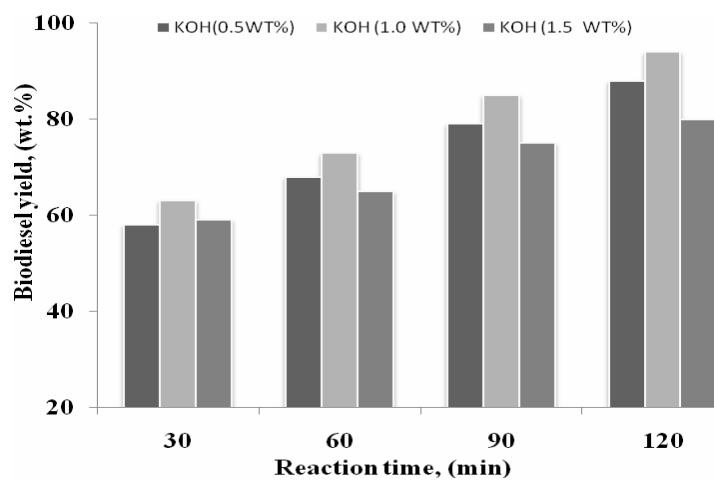


Fig.3.10 Biodiesel yield with 1.0 wt %, KOH for 6:1 molar of used frying oil

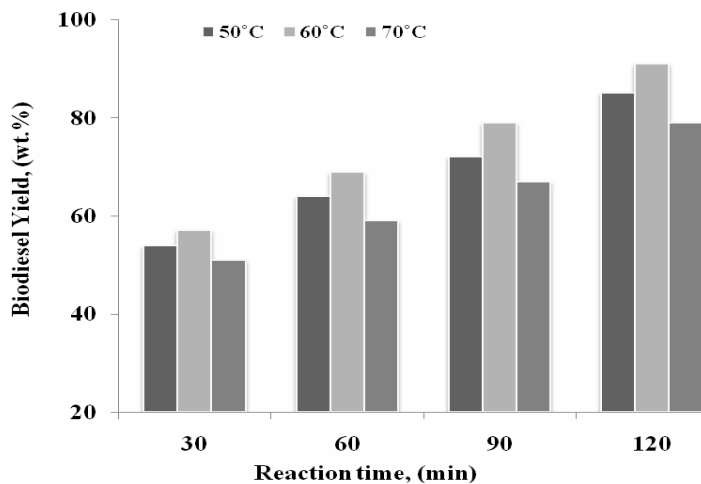
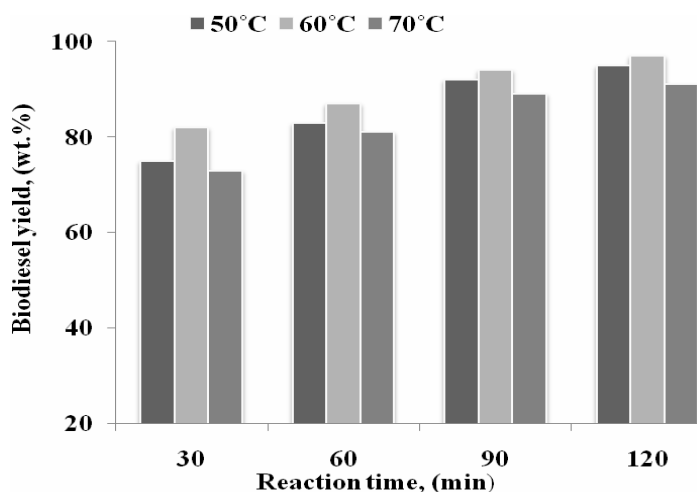


Fig.3.11 Biodiesel yield with 1.0 wt %, KOH for 6:1 molar of fresh oil



Therefore, the optimum temperature for used frying oil process was 60°C. Similarly, Figure 3.11 shows 60°C showing higher biodiesel yield than the temperatures of 50 and 70°C for fresh oil. After 20 min, the biodiesel yields were 75, 82 and 73 wt % with respect to temperatures of 50, 60 and 70°C. After 120 min, the biodiesel yields were 95, 97, and 91 wt % of methyl esters obtained for the temperatures of 50, 60 and 70°C. Therefore the optimum for the same; that is, the equilibrium concentration increased as the temperature increased. Encinar *et al.*, (2007) explained three temperatures like 35, 60, 78°C for used frying oil in 12:1 molar ratio of methanol-oil condition with 1 wt. %. But in that 60, 78°C temperatures show almost same curve, whereas in the present case three temperatures show different curve with 6:1 ratio and 1 wt % of KOH used. Encinar *et al.*, (2010) explained about castor oil with temperatures (25, 35, 45, 55, 65°C) and methanol/oil ratio (3:1, 6:1, 9:1, 12:1) with different catalyst concentrations. Prafulla *et al.*, (2009) explained about used frying, *Jatropha Curcus* and *Camelina* oils with up to maximum temperature 140°C and in that, maximum biodiesel obtained at

temperature of fresh oil was 60°C. The primary advantage of higher temperatures influences to shorter reaction time. However, higher reaction temperatures causes methanol to vaporize resulting in decreased yield (because the boiling point of methanol is 65°C). When the temperature was decreased, the rate of reaction was also decreased. Therefore, the equilibrium concentration was strongly conditioned by the temperature and favored 100°C because it contained high FFA value compared with this process used frying oil.

### Effect of Reaction Time

According to many researchers, the biodiesel yields are directly proportional to the reaction times used. The experiment was conducted from 30-120 min of reaction time with 50-70°C of reaction temperature for 1 wt % of catalyst used in 6:1 molar ratio of used frying oil and fresh oil. Based on the results, Figure 3.10 and 3.11 show that effect longer mixing gives higher yield than using shorter time. So, 120 min of reaction time gave a good result than other reaction times used here. In other words, the biodiesel yields increases with

increasing the reaction time. However, based on the results, it shows that the biodiesel yields were higher when reaction time of 120 min was used. This undesirable result may be due to the higher soap formation when longer reaction time was used. Thus, the rate of soap formation was also increased.

### **Bio diesel Confirmation**

In this study, FTIR technique is used to identify the frequency peaks are formed with respect to the % transmittance. A detailed description was given for waste cooking palm oil with biodiesel peaks result of KOH catalyts. Figure 3.12 shows the FTIR spectrum of waste cooking palm oil. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of carboxyl group at  $2675.6\text{ cm}^{-1}$ , along with the O-H stretching of the hydroxyl bonded with alcohol at  $3461.3\text{ cm}^{-1}$  and Aldehyde, Ketones(C=O) group along with carboxylic group at  $1747.2\text{ cm}^{-1}$ . C-O group combined with carboxylic group at  $1238.9$ ,  $1165.4\text{ cm}^{-1}$ . Finally Alkanes groups strongly stretched at  $2905.5$ ,  $2854.8$  and  $1458.2\text{ cm}^{-1}$ . Figure 3.13 shows the FTIR spectrum of biodiesel for KOH Catalyst. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of carboxyl group at

$3000\text{-}2500\text{ cm}^{-1}$ , along with the O-H stretching of the hydroxyl bonded with alcohol at  $3600\text{-}3200\text{ cm}^{-1}$  (broad) with Alcohols, Phenols groups. Aldehyde, Ketones (C=O) group along with carboxylic group, Ester group strongly stretched found at  $1744.0\text{ cm}^{-1}$  ( $1470\text{-}1670\text{ cm}^{-1}$ ). C-O group combined with carboxylic group, Ester group strongly stretched at  $1169.9\text{ cm}^{-1}$  ( $1260\text{-}1000\text{ cm}^{-1}$ ). Finally, Alkanes (C-H) groups strongly stretched at  $1458\text{-}1377.7\text{ cm}^{-1}$ .

### **Conclusions**

Used frying oil may be an easily available resource for the transesterification of biodiesel. In that best condition of reaction, esters content was 96%, close to 96.5% required by European Standard EN 14214. Consequently, the use of used frying oil is an effective way to reduce the raw material cost. In addition, the pollution problems could be reduced. The methanol/oil molar ratio was one of the variables that had more influence on the process. Within the range of molar ratios employed (3:1-9:1), the best results were obtained for a 6:1 molar ratio. Potassium hydroxide gave the highest biodiesel yield, although the yields with sodium hydroxide, potassium methoxide, and sodium methoxide were acceptable.

**Fig.3.12 FTIR Spectrums for used frying oil**

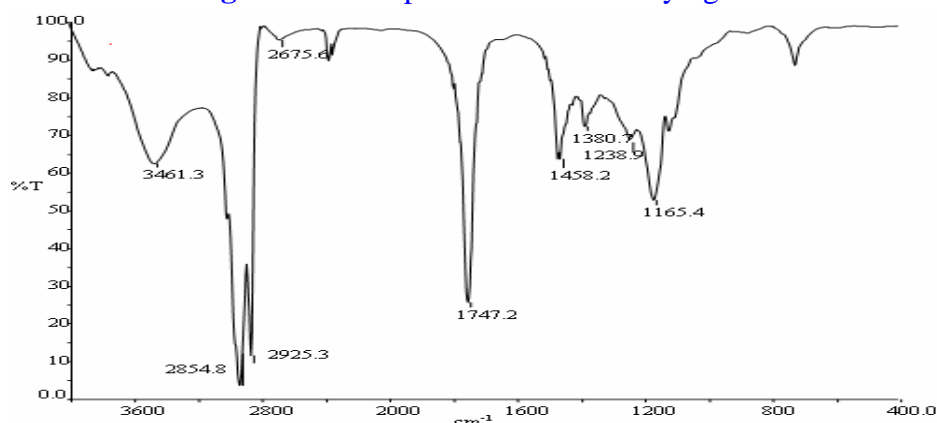
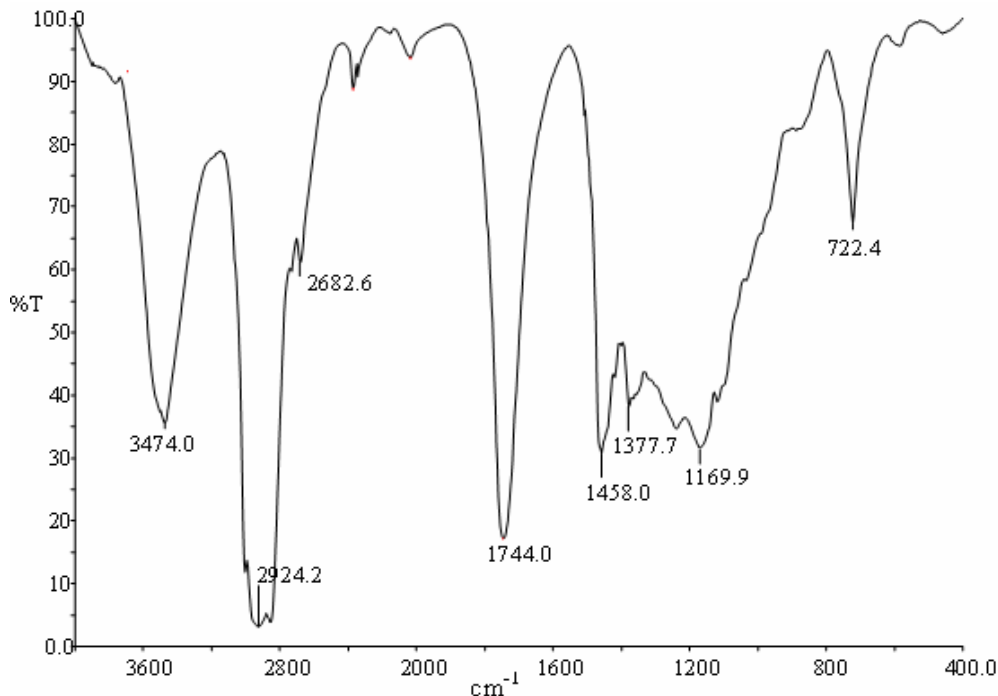


Fig.3.13 FTIR Spectrums of biodiesel for KOH Catalyst



The catalyst concentration of 0.5, 1.0 and 1.5wt % were employed and the optimum concentration of 1.0 wt. %. The temperature ranges of 50, 60, and 70°C used for biodiesel production and the optimum temperature is 50°C. The FTIR technique identified, used frying oil is obtained the carboxyl group at 2675.6  $\text{cm}^{-1}$ , along with the O-H stretching of the hydroxyl bonded with alcohol at 3461.3  $\text{cm}^{-1}$  and carboxylic group at 1747.2  $\text{cm}^{-1}$ . C-O group combined with carboxylic group at 1238.9, 1165.4  $\text{cm}^{-1}$ . FTIR spectrum of biodiesel for KOH Catalyst show asymmetric and symmetric strong stretching vibrations of carboxyl group at 3000-2500  $\text{cm}^{-1}$ , along with the O-H stretching of the hydroxyl bonded with alcohol at 3600-3200  $\text{cm}^{-1}$  (broad) with Alcohols. Ester group strongly stretched found at 1744.0  $\text{cm}^{-1}$  (1470-1670  $\text{cm}^{-1}$ ). C-O group combined with carboxylic group, Ester group strongly stretched at 1169.9  $\text{cm}^{-1}$  (1260-1000  $\text{cm}^{-1}$ ).

### Acknowledgements

I am sincerely thanks the my supervisor, Anna University, Chennai and Arulmigu Meenakashi Amman College of Engineering, Vadamavandal, Tamilnadu, India

### References

- Allen C.A.W., Prediction of Biodiesel Fuel Atomization Characteristics Based on measured properties. PhD. Thesis, Faculty of Engineering, Dalhousie University, 1998.
- Al-Zuhair, S., Production of biodiesel: possibilities and challenges, *Biofuels, Bioprod. and Biorefi.*, 1, 57-66 2007.
- Anitha A. and Dawn S.S., Performance Characteristics of Biodiesel Produced from Waste Groundnut Oil using Supported Heteropolyacids, *Int. J. of Chemical Engg. and Applns.*, 13, 261-265 2010.

- Arjun B., Chhetri, K., Chris Watts and Rafiqul Islam M., Waste Cooking Oil as an Alternate Feedstock for Biodiesel Production, *Energies*, 1, 3-18 2008.
- Canakci M., the Potential of Restaurant Waste Lipids as Biodiesel Feedstock's, *Biores. Techno.*, 98, 183-190 2007.
- Chen G, Ying M. and Li W., Enzymatic conversion of waste frying oils into alternative fuel-biodiesel, *Appli. Biochemis. Biotechno.*, 129-132, 911-921 2006.
- Connemann J. and Fischer J., Biodiesel in Europe 1998; Biodiesel processing technologies. Paper presented at the Int. Liquid Biofuels Congress, Brazil, pp.1-16, 1998.
- Encinar J.M., Gonzalez J.F. and Rodriguez-Reinares A., Ethanolysis of used frying oil. Biodiesel preparation and characterization, *Fuel Pro. Techno*, 88, 513-522 2007.
- Encinar, J.M., Gonzalez, J.F. and Rodriguez-Reinares A., Biodiesel from used frying oil; Variables affecting the yields and characteristics of the biodiesel, *Ind. & Eng. Chem. Res.*, 44, 5491-5499 2005.
- Fangrui Ma. and Hanna A. Milford, Biodiesel production: a review, *Biores. Techno.*, 70, 1-15 1999.
- Felizardo P., Correia M.J., Raposo I., Mendes J.F., Berkemeier R. and Bordado J.M., Production of biodiesel from waste frying oils, *Waste Manage.*, 265, 487-494 2006.
- Feuge R.O. and Gros A.T., Modification of vegetable oils. VII Alkali catalyzed interesterification of peanut oil with ethanol, *J. Am. Chem. Soc.*, 26, 97-102 1949.
- Freedman B, Pryde E.H. and Mounts T.L., Variables affecting the yields of fatty esters from transesterified vegetable oils, *J. Am. Chem. Soc.*, 61, 1638-1643 1984.
- Freedman B., Butterfield E.H. and Pryde, Transesterification kinetics of soybean oil, *J. Am. Chem. Soc.*, 63, 1375-1380 1986.
- Gerpen J.V., Biodiesel processing and production, *Fuel Pro. Techno.*, 86, 1097-1107 2005.
- Ghadge S.V. and Raheman H., Biodiesel production from mahua *Madhuca indica* oil having high free fatty acid, *Biomass and Bioenergy*, 28, 601-605 2005.
- Hossain, A.B.M.S., Boyce, A.N., Sallesh, A. and Chandran, S., Impacts of alcohol type, ratio and stirring time on the biodiesel production from waste canola oil, *Afri. J. of Agri. Res.*, 514, 1851-1859 2010.
- Krishnangura K. and Simamaharnnop R., Continuous transmethylation of palm oil in an organic solvent, *J. Am. Chem. Soc.*, 69, 166-169 1992. Encinar J. M., Gonzalez J. F., Martinez G., Sanchez N. and Gonzalez C. G., Synthesis and characterization of biodiesel obtained from castor oil transesterification, Int. Conf. on Renew. Energies and Power Quality ICREPQ'11, Spain, 13 to 15<sup>th</sup>, 2010.
- Lang, X., Dala, A. K., Bakhshi, N. N., Reaney M. J. and Hertz, P. B., Preparation and characterization of biodiesels from various bio-oils, *Biores. Techno.*, 80, 53-62 2001.
- Leung D.Y.C. and Guo Y., Transesterification of neat and used frying oil: Optimization for biodiesel production, *Fuel Pro. Techno.*, 87, 883-890 2006.
- Meher L.C., Vidya Sagar D. and Naik S.N., Technical aspects of biodiesel production by transesterification - a review, *Renew. Sustain. Energy Reviews*, 10, 248-268 2006.
- Naik, M., Mehar, L.C., Naik, S.N, Das, L.M., Production of biodiesel from high

- free fatty acid Karanj oil, *Biomass and Bioenergy*, 324, 354-357 2008.
- Nye, M.J., Williamson, T.W., Deshpande, S., Schrader, J.H., Snively, W.H., Yurkewich, T.P. and French C.L., Conversion of used frying oil to diesel by transesterification: preliminary tests, *J. Am. Chem. Soc.*, 8, 1598-1601 1983.
- Prafulla D., Patil Veera, Gnaneswar Gude and Shuguang Deng, "Biodiesel Production from Jatropha Curcas, waste cooking, and Camelina Sativa Oils", *Ind. & Eng. Chem. Res.*, 48, 10850-10856 2009.
- Schwad A.W., Bagby M.O. and Freedman B., Preparation and properties of diesel fuels from vegetable oils, *Fuel*, 66, 1372-1378 1987.
- Sprules, F.J. and Price D., Production of fatty esters, US Patent 2; pp. 366-494, 1950.
- Tiwari A.K., Kumar A. and Raheman H., Biodiesel production from Jatropha oil Jatropha curcas with high free fatty acids: An optimized process, *Biomass and Bioenergy*, 31, 569-575 2007.
- Tomasevic A.V. and Siler-Marinkovic S.S., Methanolysis of used frying oil, *Fuel Pro. Techno.*, 81, 1-6 2003.
- Wang Y, Ou S.Y., Liu P.Z. and Zhang Z.S., Preparation of biodiesel from waste frying oil via two- step catalyzed process. *Energy Conv. and Manage.*, 48, 184-188 2007.
- Watanabe Y., Shimada Y., Sugihara A. and Tominaga Y., Enzymatic conversion of waste edible oil to biodiesel fuel in a fixed-bed bioreactor, *J. Am. Chem. Soc.*, 787, 703-707 2001.
- Xiaohu Fan, Rachel Burton and Greg Austic, Preparation and Characterization of Biodiesel Produced from Recycled Canola Oil, *the Open Fuels and Ener. Sci. J.*, 2, 113-118 2009.
- Zheng S., Kates M., Dube M.A. and McLean D.D., Acid-catalyzed production of biodiesel from waste frying oil, *Biomass and Bioenergy*, Vol.30, pp.267-272, 2006.
- Zhou, W., Konar, S.K. and Boocock, D.G.B., Ethyl esters from the single-phase base- catalyzed ethanolysis of vegetable oils, *J. Am. Chem. Soc.*, 804, 367-371 2003.