Production of Biodiesel from Waste Cooking Oil Using Two Stage Transesterification Process

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Abstract - The fatty acid methyl esters (FAME), known as Biodiesel present the promising alternative substitute to diesel fuels. Biodiesel is renewable, biodegradable, non-toxic, and essentially free of sulfur and aromatics compared to diesel. The higher cost of biodiesel is due to its production mostly from expensive high quality virgin oil. The new process technologies developed during the last years made it possible to produce biodiesel from waste/used cooking oils (WCO) which harmfully affect environment and human's health, comparable in quality to that of fresh vegetable oil with an added attractive advantage of being lower in price. In this experiment, fresh rice bran oil & WCO collected from local restaurant in Indore are used as feedstock. The high level of free fatty acids (FFA) in the WCO was reduced to less than 2% by acid catalyzed esterification using methanol to oil molar ratio of 9:1, 5 wt % of sulfuric acid, at 55 °C with a reaction time of 60 min. Further alkali-catalyzed transesterification was carried using molar ratio of methanol to oil (from the first step) 6:1, 1.5 wt% KOH, at 60 °C with a reaction time of 60 min in the second step. The percentage yield of methyl ester (biodiesel) was 92.5%. Effect of different parameters including effect of temperature (40-80 OC), oil to alcohol ratio (3:1-9:1 molar ratio) and amount of alkaline catalyst(0.75-3 wt%) were studied in order to achieve the optimum condition to obtain the highest conversion. Biodiesel made from WCO using two step transesterification process had viscosity 5.5 mm2/s at 40 OC, density 888 Kg/m3, acid number 0.55 mg KOH/g, flash point 75, cloud point +1oC, pour point -5oC are comparable to Indian Standard for Biodiesel (IS 15607-2005). The Cetane Number which is a measure of ignition quality of diesel fuel found to be 55.85 by empirical formula. The biodiesel made from WCO cost about at Rs. 42.00 per lit. This paper concentrates on the development of economically viable as well as eco friendly substrates such as waste cooking oil for biodiesel production and briefly discusses the factors that can affect the biodiesel production. Reusing of these waste cooking oil not only reduce the burden of the government in disposing the waste, maintaining public sewers and treating the oily wastewater, but also lower the production cost of biodiesel significantly.

KEYWORDS: Waste Cooking Oil, Esterification, Transesterification, Biodiesel, Fuel Characterization

I. INTRODUCTION

The depleting reserves of fossil fuel, increasing demands for diesels and uncertainty in their availability are considered to be the important trigger for many initiatives to search for the alternative sources of energy, which can supplement or replace fossil fuels. Hence it is necessary to look for alternative fuels, which can be produced from materials available within the country. Among the different possible sources, fatty acid methyl esters (FAME), known as Biodiesel present the promising alternative substitute to diesel fuels and have received the most attention now a day. Biodiesel is technically defined as "the mono alkyl esters of long chain fatty acids derived from renewable liquid feedstock, such as vegetable oils and animal fats, for use in compression ignition (CI) engines".

Biodiesel is renewable, biodegradable, non-toxic, and essentially free of sulfur and aromatics. The feed stocks used for biodiesel production primarily include edible oils, non-edible oils, WCO, and animal fats. The various sources used for biodiesel production are shown in Table 1.

Since the prices of edible vegetable oils are higher than that of diesel fuel also use of such edible oil to produce biodiesel in India is also not feasible in view of big gap in demand and supply of such oils, therefore non-edible and used/ waste cooking oil (WCO) are preferred as potential low priced biodiesel sources.

The production of biodiesel from WCO offers a triple-facet solution namely economic, environmental and waste management. One of the major concerns on biodiesel production is the price of feedstock, since the feedstock costs constitutes approximately 60-85% of the overall cost of biodiesel [1]. The use of WCO as biodiesel feedstock reduces the cost of biodiesel production.

From health point of view, it is advisable to discard WCO after certain time of frying. It is well known fact that, when vegetable oils heated for an extended time (abuse), they undergo oxidation and give rise to oxides. Many of these such as hydro peroxides, epoxides and polymeric substances have shown adverse

health/biological effects such as growth retardation, increase in liver and kidney size as well as cellular damage to different organs when fed to laboratory animals. Also the harmful compounds of WCO may return into the food chain when used as animal feed.
From a waste management standpoint, producing biodiesel from WCO is environmentally beneficial, since it provides a cleaner way for disposing these products. Several billions of gallons of waste vegetable oil are produced every year around the world, mainly from industrial deep fat fryers found in potato processing plants, factories manufacturing foods, and restaurants. Some of this wastage is already being re-used by other industries, such as in animal feed and cosmetics, but the amount that is still being wasted and ending up in land-fill sites is alarming. The oil which reaches the water sources increases its organic pollution load, also forms layer on the water surface preventing the oxygen exchange and alters the ecosystem. The dumping of the oil also causes problems in the pipes drain obstructing them and creating odors and increasing the cost of wastewater treatment.

II. EXPERIMENTAL SETUP, MATERIAL AND METHODS

2.1 *Feedstock fresh rice bran oil and waste/used cooking oil (WCO)*

In this experiment, fresh rice bran oil and used/waste cooking oil (WCO) collected from local restaurant in Indore, are used as feedstock. The temperature observed during frying was in the range of 130^0 C to 175^0C. Depending on the quantity of food used for frying, the oil was discarded sometimes at the end of each day and sometimes in two or three days. Some common physical changes observed in vegetable oil after frying are (i) an increase in the viscosity, (ii) an increase in the specific heat, (iii) a change in the surface tension, (iv) a change in color, and (v) an increase in the tendency of fat to foam. Some of them are linked to the process itself, such as temperature, duration of heating, heating pattern (continuous or intermittent), etc. others to the oil used e.g., degree of unsaturation, initial quality and additives. The negative effects of undesirable compounds can be avoided by pretreatment of the waste cooking oil. Collected WCO was filtered to remove any suspended matter, and burned food bits etc.

2.2 *Basic reaction mechanism involved in the production of biodiesel*

Transesterification :
The most common way to produce biodiesel is by transesterification of triglycerides of refined /edible types of oils using alcohol, in presence of an acid or a basic catalyst. From a chemical point of view, oils from different sources have different fatty acid compositions. The fatty acids vary in their carbon chain length and in the number of unsaturated bonds they contain. Triglycerides are esters of three fatty acids. When the triglycerides react with alcohol in the presence of base catalyst, this is called "transesterification." In this reaction, triglycerides are converted to diglyceride, monoglyceride, and finally converted to glycerol. The following scheme 1 is the general steps of the transesterification of vegetable oil to produce methyl ester of fatty acids (biodiesel).

Fatty acid + Alcohol → Ester + Water
Triglyceride + ROH → Diglyceride + $RCOOR_1$
Diglyceride ROH → Monoglyceride + $RCOOR_2$
Monoglyceride + ROH → Glycerol + $RCOOR_3$
where R is alkyl group.
Scheme 1: Transestrification reaction.

Esterification :
This reaction is very much useful when raw material contains high percentage of free fatty acid (FFA) which esterifies FFA to form esters of free fatty acid (biodiesel).

$$R1\text{-}COOH + ROH \longrightarrow R\text{-}O\text{-}CO\text{-}R1 + H_2O$$

FFA (alcohol) Fatty acid ester

Scheme 2: Esterification reaction.

Side Reaction (Saponification Reaction):
If vegetable oil contains free fatty acid, it will react with homogenous base catalyst to form soap and water. The saponification reaction is represented as below

$$R1\text{-}COOH + NaOH \longrightarrow R_1COONa + H_2O$$

FFA (alcohol) Soap

Scheme 3: Saponification reaction.

2.3 Experimental setup

A 3L capacity three necked glass reactor equipped with magnetic stirrer to provide the mixing in a temperature controlled hot plate is used. In addition, the condenser provides cooling system to control the leakage of methanol by supplying cooled water in the inlet and the hot water was rejected in the outlet. Initially, WCO was preheated at 120°C to remove the moisture content using temperature controlled hot plate for 15 minutes.

The acid number is a direct measure of FFA in biodiesel .The acid number of the waste cooking oil was determined in order to estimate the FFA content and give an idea of how much acid catalyst and methanol would be needed to push the acid esterification chemical towards methyl ester production. Table 2 shows the physicochemical properties of the fresh rice bran oil and waste cooking oil.

1. *Esterification :*

An acid-catalyzed esterification process before the base-catalyzed transesterification process will eliminate most of the free fatty acids (FFA) from the waste oil. In this study, FFA was reduced to less than 2% by acid catalyzed esterification using methanol to oil molar ratio of 9:1, 5 wt % of sulfuric acid, at 55 °C with a reaction time of 60 min. The stirring speed 200 rpm were used to mix the solvent.

2. *Separation I*:

Separation needed 3 hour to get the top methanol and bottom oil layers was mainly triglyceride. Two layers could clearly be seen after successful esterification of biodiesel. These processes reduce free fatty acid below 2%. The density of the methanol is less than that of bottom triglyceride.

3. *Transesterification:*

The two layers separated clearly in upper layer of methanol and lower layer of triglyceride. The top layer of methanol was removed and further alkali-catalyzed transesterification was carried using molar ratio of methanol to oil (from the first step) of 6:1, 1.5 wt% KOH, at 60 °C with a reaction time of 60 min. After adding the methanol/potassium hydroxide to oil layer, stirring speed 200 rpm were used to mix the solvents.

4. *Separation II:*

Separation process needed at least 8 hours to get the top layer of methyl ester and bottom layer of glycerol .The top layer was mainly composed of free fatty acid methyl esters. The bottom deposit was mostly made up of glycerol, salts, soap, other impurities and excess methanol as it is a very polar compound i.e. it partitions more with polar glycerol as opposed to the non-polar methyl ester.

5. *Washing* :

The top methyl ester (biodiesel) layer was separated from the glycerin and removed. Once separated, the biodiesel is sometimes purified by washing gently with warm water to remove residual catalyst or soaps and then sent to storage. In some processes this step is unnecessary. This is normally the end of the production process resulting in a clear amber-yellow liquid with a viscosity similar to petro diesel.

III. OBSERVATION, RESULT AND DISCUSSION

The biodiesel yield was estimated before and after the purification step. The produced biodiesel weight relative to the initial used WCO was taken as the biodiesel yield. Table 3 shows the physicochemical properties of the biodiesel obtained from fresh rice bran oil and waste cooking oil and are compared with Indian standard for biodiesel specification. It was observed that the yield of produced biodiesel was influenced by alcohol/ oil molar ratio, temperature, the type and amount of catalyst, and in order to fix the optimum reaction conditions the same transesterification procedure was followed to study the effect of each of these factors on production yield.

3.1. Effect of reaction temperature

Transesterification can occur at different temperatures depending on the properties of waste oil. Transesterification of waste cooking oil was carried out using methanol as alcohol and KOH as alkaline catalyst at different temperatures and the yield percentage was calculated and the effect of temperature change on yield was shown in the Fig 1. It was found that at 60°C the yield of produced biodiesel was the highest. The higher

reaction temperature accelerates the saponification of triglycerides and causes methanol to vaporize resulting in decreased yield. This may be due to temperature increases the energy of the reacting molecules and also improves the miscibility of the alcoholic polar media into a non-polar oily phase, resulting in much faster reactions. The effect of temperature firstly explains that the transesterification is an equilibrium process and this equilibrium is dependent on concentration of alcohol and glycerol on the two sides of the reaction.

3.2. Effect of methanol to oil ratio

The stoichiometric ratio for transesterification reaction requires 3 mole of alcohol and 1 mole of triglyceride to yield 3 mole of fatty acid ester and 1 mole of glycerol as shown in scheme1. The production yield of WCO biodiesel was studied using different mole ratios of methanol to WCO at the previously fixed optimum temperature (60°C) using KOH as alkaline catalyst and the yield percentage is calculated and the effect of methanol to oil ratio on yield is shown in the Fig 2. Biodiesel yield could be elevated by introducing an excess amount of methanol to shift the equilibrium to the right hand side. The best yield (92.5%) was achieved at 6:1 methanol to oil mole ratio. This means that 6:1 mole ratio provides sufficient excess of methanol to shift transesterification equilibrium toward biodiesel product.

3.3 Effect of (KOH) catalyst amount

Under the two fixed optimum values of temperature (60°C) and methanol: oil mole ratio (6:1) the production yield of sesame biodiesel was examined by using different amounts of KOH and the yield percentage was calculated and the effect of catalyst amount on yield was shown in the Fig 3. It was observed that 1.5 %KOH to oil was enough to catalyze the transesterification process and gave desired alkaline medium since excess hydroxyl anion may increase the hydrolysis of ester linkage of biodiesel.

3.4. Effect of catalyst type

In another experiment sodium hydroxide (NaOH)was used instead of KOH as alkaline catalyst under the previously described optimum conditions in order to investigate the effect of alkaline catalyst on production yield of biodiesel. It was observed that when 1.5 wt % of NaOH was used; the highest production yield (90%) was obtained which was relatively lower than the production yield when KOH was used as catalyst (92.5%).

3.5. Fuel characterization

The fuel properties of the esters produced from the transesterification can vary depending on the types of vegetable oils used [2].

(i) Total Acid Number

The total acid number (TAN) is "The quantity of base, expressed as milligrams of potassium hydroxide per gram of sample, required to titrate a sample to a specified end point." The acid number is a direct measure of FFA in biodiesel. Higher acid number could cause degradation of rubber parts in older engines resulting in filter clogging. The total acid number can also be determined by titration. 25ml of diethyl ether and ethanol mixture was added to 5 gm of oil in a 250 ml conical flask and the solution was titrated with 0.1N ethanolic KOH solution in the presence of 5 drops of phenolphthalein as indicator until the endpoint (colorless to pink) is recognized with consistent shaking [46]. The total acid number of oil in mg KOH/ gram was calculated using the following equation:

TAN = $56.1 \times N \times V/W$

Where, V = the volume expressed in milliliter of 0.1N solution of ethanolic KOH.

W = the weight of oil sample (the mass in gram of the test portion)

N = concentration of ethanolic KOH

(ii) *Cloud Point and Pour Point*

The cloud and pour point are also the important properties of biodiesel fuel. Cloud point is the temperature at which a cloud of wax crystals first appear in the oil when it is cooled. The pour point is the lowest temperature at which the oil sample can still be moved. These properties are related to the use of biodiesel in the cold temperature.

(iii) Viscosity

Kinematic viscosity: "the resistance to flow of a fluid under gravity". The kinematic viscosity is equal to the dynamic viscosity/density. The kinematic viscosity is a basic design specification for the fuel injectors used in diesel engines. Too high a viscosity and the injectors do not perform properly.

(iv) Density

Density is important mainly when the oil is combusted in a CI engine because it influences the efficiency of atomization of the fuel.

(v) *Flash Point*

Flash point of a fuel is the temperature at which it will ignite when exposed to a flame or spark. The flash point of WCO biodiesel is found to be 75.

(vi) Cetane Number

Cetane numbers are the indicators of ignition properties of the diesel fuel. Because of the higher oxygen content, biodiesel has a higher cetane number compared to petroleum diesel. The higher cetane number indicates the higher engine performance resulting in lower emission of all pollutants other than oxides of nitrogen (NOx). The oxidative stability of biodiesel fuels also increases due to the presence of higher amount of saturated fatty acids. However, the drawback of higher amount of saturated fatty acid content in biodiesel fuel is that the cold filter plugging point occurs at a higher temperature.

The cetane number of WCO biodiesel was calculated using empirical formula given by **Sivaramkrishnan et al [35]**as follows :

Empirical Equation:

$CN = K_5 + K_4 v + K_3 HV + K_2 FP + K_1 \rho$ ……(1)

HHV (for biodiesel) = $0.4625v + 39.450$……(2)

Where K_1, K_2, K_3, K_4, K_5 are constants and v is kinematic viscosity (mm^2/sec), HV is heating value (MJ/kg), FP is flash point (^{0}C), ρ is density (kg/l)

In case waste cooking oil biodiesel the values are as follows:

$K_1 = 140.337$, $K_2 = -0.0258$, $K_3 = -0.202$, $K_4 = 0.071$, $K_5 = -56.464$, $v = 5.80$ mm^2/sec,

$FP = 70\ ^0C$, $\rho = 0.8709$

Using Eq. (1) and (2):-

HHV = 42.13 MJ/ kg

Cetane number for waste cooking oil biodiesel = 55.85

The Cetane number of biodiesel from fresh rice bran oil is calculated as follows:

$K_1 = -192.86$, $K_2 = -0.0124$, $K_3 = -2.231$, $K_4 = -0.127$, $K_5 = 309.87$, $v = 6.80$ mm^2/sec,

$FP = 65\ ^0C$, $\rho = 0.8756$

Using Eq. (1) and (2):-

HHV = 42.59 MJ/ kg

Cetane number of biodiesel from fresh rice bran oil = 45.32

3.6 Tentative production cost

The tentative production cost per liter of WCO biodiesel are calculated in the Table 4.The cost of WCO biodiesel was found Rs.42.00 per lit. The cost found higher because of small scale production calculation. If large scale production have been made than the production cost of produced biodiesel may be lesser than as obtained above.

IV. CONCLUSION

The findings of this work clearly indicate that biodiesel derived from waste cooking oil (WCO) is perhaps the greenest liquid fuel available because of the primary ingredient being a post consumer waste product. Public should be aware of ill effects that WCO (overused or abused) oil has on health & utilizing the recycled WCO for human consumption in any way is not advisable from health point of view .A chemical route to convert this WCO into WCO biodiesel is recommended in this paper and briefly discusses the factors that will affect the biodiesel production. On the basis of this article following conclusion has been drawn.

- The highest biodiesel yield obtained from transesterification of waste cooking oil was 92.50% at optimum temperature of 60 ^{0}C, at methanol to oil ratio was 6:1 and at 1.5 wt % of KOH.
- Biodiesel made from WCO using two stage formulation process had viscosity, density, flash point, pour point similar to Indian Standard Biodiesel Specifications ((IS 15607-2005). Cetane number which is a measure of ignition quality of diesel fuels, found to be 55.85 by empirical formula.
- The waste cooking biodiesel cost comes about at Rs. 42.04 per lit.

  Further studies are recommended on issues such as engine combustion characteristics, emission parameters, environmental impact before it can be considered for commercial application.

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8. TABLES AND FIGURES

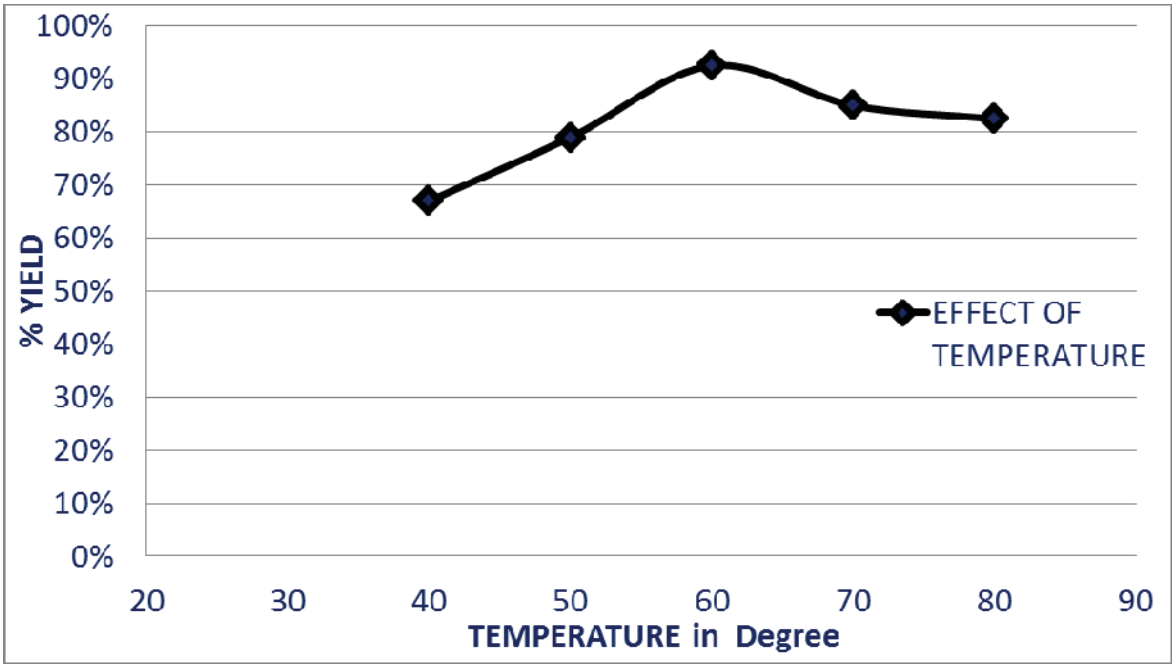

Fig.1: Effect of temperature change on % biodiesel yield

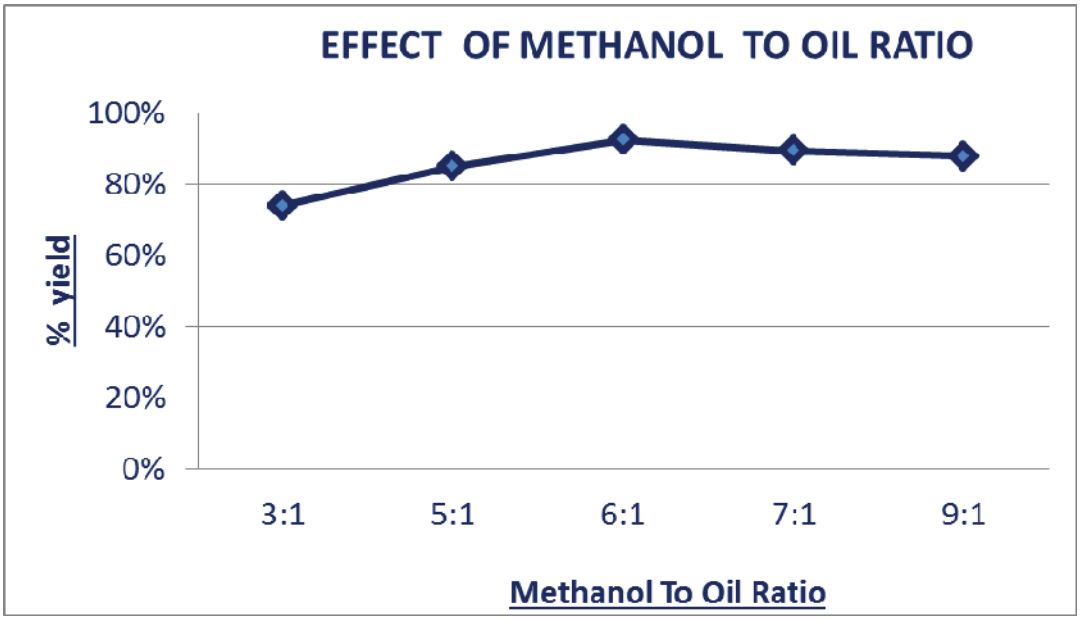

Fig.2 Effect of methanol to oil ratio on % biodiesel yield

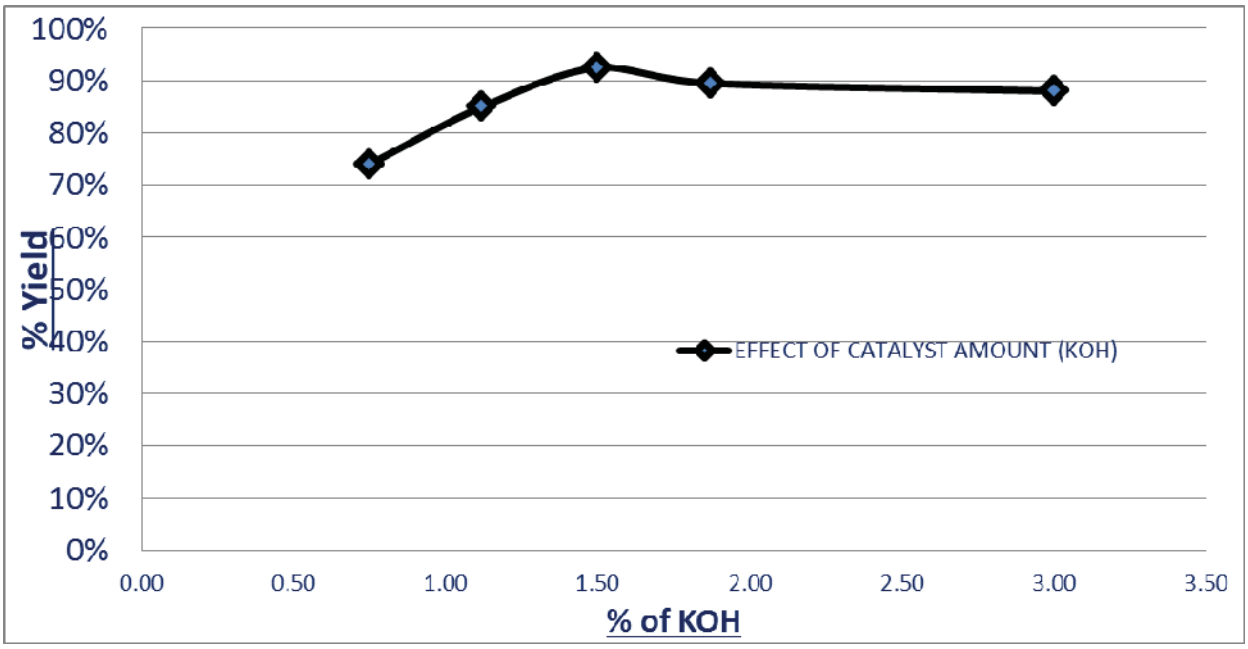

Fig.3 Effect of catalyst KOH amount on %biodiesel yield

Edible vegetable oil	Non Edible oil	Other sources
Soybean	Jatropha curcus	Beef tallow
Palm	Mahuaa	Algal oil
Rapseed	Karanja	Micro algae oil
Canola	Terebinth oil	Waste cooking oil
Castor bean oil	Neem oil	
Sesame seed oil	Moringa oleifera	
Rice bran oil	Rubber seed oil	
Sunflower oil		

Table 1: Sources of biodiesel production

Properties	Fresh rice bran oil	Used cooking oil/WCO
Acid value mg KOH/gm	0.3	4.03
Calorific value J/gm		39658
Saponification Value mg KOH/gm	194	178
Density gm/cm^3	0.898	0.9013
Kinematic viscosity mm^2/s	39.994	44.956
Flash point ^{o}C	161	222
Moisture content wt %	0.101	0.140

Table 2: Physicochemical properties of fresh rice bran oil and waste cooking oil

Characteristics	**Unit**	**Fresh rice bran oil Biodiesel**	**Waste cooking oil Biodiesel**	**Indian Standards for Biodiesel (IS 15607-2005)**
Density	kg/ m^3	875	888	860-900
Viscosity at 40°C	mm^2/sec	4.90	5.50	3.5-5.0

Flash Point	^{o}C	70	75	>101
Cloud Point	^{o}C	+4	+1	----
Pour Point	^{o}C	-3	-5	0
Acid Value	mg KOH/g	0.5	0.58	0.5
Carbon Residue	%(m/m)	0.4	0.5	0.3
Water Content	mg/kg	1100	800	500
Net calorific value	MJ/Kg	42.59	42.13	35
Cetane No.		45.32	55.85	51

Table 3: Physicochemical properties of biodiesel obtained from fresh rice bran oil and waste cooking oil and Indian standard of biodiesel specification

Tentative production cost analysis of biodiesel from waste cooking oil			
Sr. No.	**Particulars**	**Quantity**	**Unit**
1	Market price 10 lit waste cooking oil	100	Rs
2	Cost of methanol req. for 10 lit WCO	120	Rs
3	Cost of KOH for 10 lit	58	Rs
4	Additional expenses for labour, electricity , miscellaneous cost	100.00	Rs
5	Cost Req. for making bio diesel of 9 lit (taking 90% yield)	378.00	Rs.
6	**Cost per lit of biodiesel**	**42.00**	**Rs. /lit**
7	Diesel price in Indore as on 1st Jan 2015	49.39	Rs. /lit

Table 4: Tentative production cost per liter of WCO biodiesel