

## Production and Testing of Methyl Esters of Used Cooking Oil

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### ABSTRACT

*Biodiesel, defined as the mono-alkyl esters of vegetable oils or animal fats is an alternative diesel fuel that is becoming accepted in a steadily growing number of countries around the world. The vegetable oils have shown good performance for short-term operation. The major draw backs associated in using vegetable oil directly as fuel for diesel engine for longer operations are clogging of fuel lines and filters due to fine particles, gums, waxes, carbon deposits and higher smoke formation. These problems occur due to the higher viscosity of vegetable oils. Transesterification process is the conventional method, which eliminates the above-mentioned drawbacks. Among the vegetable oils, the non-edible oils would be less expensive and also would not put pressure on cooking oil prices. The non-edible oils such as jatropha oil and karanja oil, even though attractive due to their low cost, requires considerable amount of land and time for cultivation. These uncertainties can prevent large-scale production and marketing of biodiesel. Used cooking oils provide a viable alternative to diesel, as they are easily available. The main objective of this work is to optimize the transesterification process for methyl ester production and testing its performance in diesel engine. Used cooking oil is used as feedstock for the production of methyl esters in this work as they provide a viable alternative to diesel, due to its availability. The effect of excess methanol, catalyst amount, temperature, time of reaction was studied to optimize the transesterification process. All the reactions were carried out under the same experimental conditions. The performance and emission characteristics of used cooking oil methyl esters and its blend with diesel oil were analysed in a diesel engine. The minor decrease in thermal efficiency with significant improvement in the reduction of particulates, carbon monoxide, unburnt hydrocarbon was observed compared to diesel.*

**Key Words:** Biodiesel, Castor Oil, Methyl Ester

Received 25.05.2013 Accepted 17.06.2013

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### INTRODUCTION

The generated waste frying oils from household, restaurants, food processing industries and fast food shops everyday is a growing problem throughout the world. Reusing of these Used cooking oil (UCO) can not only reduce the burden of the government in disposing the waste, maintaining public sewers and treating the oily waste water, but also serves as a raw material in the production of soaps or energy by anaerobic digestion, thermal cracking and more recently in the production of biodiesel, a fuel which fulfills all the environmental and energy security needs. Biodiesel is a renewable, biodegradable and eco-friendly mineral diesel substitute for engines. There are various ways through which these oils can be converted to biodiesel namely, transesterification, blending, microemulsions, pyrolysis and transesterification. Transesterification being the most commonly used methods [1,2]. Transesterification is a process of displacement of an alcohol group from an ester by another alcohol. A catalyst is usually used to improve the reaction rate and yield. Because the reaction is reversible excess alcohol is used to shift the equilibrium to the product side. Alcohols like methanol, ethanol, propanol, butanol, and amyl alcohol can be used, of which methanol most frequently is used due to its low cost and its physical and chemical advantages. In vegetable oil almost 90 – 95% is glycerides, which are basically esters of glycerol and fatty acids [3].

The production of biodiesel has been actively investigated in the past 20 years. Meher et al. [4] reviewed the biodiesel production processes by transesterification. Marchetti et al [5] discussed various methods to produce biodiesel. It is reported that the alkali catalyzed reaction process has higher yield compared to acid catalyzed reaction. Time required for reaction is found to be lower for alkali catalyzed reaction compared to acid catalyzed reaction. However, recovery of glycerol was difficult in case of alkali catalyzed reaction. Pedro Felizardo et al [6] produced biodiesel from used frying oils by base catalyzed transesterification. The results showed that after 1 hour of reaction, a waste frying oil/methanol ratio of 4.8 and a catalyst/ waste frying oil weight ratio of 0.6% gives the highest yield of methyl esters and allows an efficient separation/ purification of the methyl esters phase, as shown by the viscosity reduction and

increased purity of this layer. Tomasevic et al [7] investigated the effect of molar ratio 4.5:1, 6:1 and 9:1 on ester yield and its quality. With 1% potassium hydroxide, temperature at 25°C, molar ratio 6:1 and 30 min, refined sunflower oil and used frying oils were sufficiently transesterified. Jon Van Gerpen [8] discussed effects of reaction time, reaction temperature on quality and quantity of esters. It is concluded that a trade-off between reaction time and temperature as reaction completeness is the most critical fuel quality parameter. Contaminant in the feedstock such as water and Free Fatty Acids (FFAs) affects the reaction process and reduce the quantity of esters. Most researchers have reported that vegetable oil esters performed well in diesel engines. However the cost has been the major obstacle for commercializing biodiesel produced from neat vegetable oils as compared to the petroleum based diesel fuel. This study was initiated to examine the possibility of transesterifying a UCO for the production of biodiesel. The UCO is an appealing raw material since it serves as a cheap source and its utilization serves the environmental cause. In particular the aspects of catalyst concentration, excess alcohol, reaction temperature, time of reaction and agitation were investigated as to their effect on conversion efficiency. The intention was to optimize the yield in terms of these fundamental process variables.

## METHODS AND MATERIALS

The optimization procedure for Transesterification process and engine testing procedure presented in this section.

### Optimization of biodiesel production

#### Pretreatment of the frying oil

The UCO from the local restaurants was collected. The used cooking oil was filtered to remove food residues and solid precipitates in the oil. Minimal amounts of water present in the UCO had to be removed as every molecule of water will destroy a molecule of catalyst. Hence the filtered oil is subjected to heating at 60°C for 10 min. The quality of oil is expressed in terms of the properties such as density, kinematic viscosity, flash point, Heating value and Acid value.

Table 1 : Quality of Oil

Property	Unit	Used cooking oil	Used cooking oil methyl ester	IS for biodiesel	Diesel
Density	kg/m <sup>3</sup>	920	885	860-900	830
Kinematic viscosity	CSt	42.91	4.73	2.5 - 6.0	3.52
Flash point	°C	290	200	> 120	49
Heating value	kJ/kg	36,750	38650	-----	44136
Acid value	mg KOH/g	3.05	0.13	0.5 max.	0.2

#### Transesterification procedure

The optimization setup consists of a three-necked round-bottom flask. The flask is kept in an oil bath heated by an electrical heater. The motor driven stirrer is inserted into the flask through the center neck. The stirrer passes through an airtight gland, which prevents the methanol from escaping out of the flask. A thermocouple is inserted through the other neck for measuring the temperature of the oil inside the flask. The thermocouple in turn is connected to a temperature controller relay, which turns the heater on / off to maintain constant temperature of the oil. A variable voltage regulator was also used to regulate the voltage applied to the heating coil. The third neck is used for pouring the UCO and methoxide (mixture of catalyst and methanol) into the flask. The UCO is heated to the desired temperature. The stirrer enables uniform mixing of the oil to ensure uniform temperature and also improves contact between the oil and methanol molecules. The Sodium Hydroxide required is precisely weighed and dissolved in the methanol to form the methoxide solution. This methoxide solution prepared is poured into the flask. The reaction is allowed to continue for selected time. The reaction mixture is then taken out and is allowed to separate in a separating funnel. The cloudy looking part of the product is glycerin, which sinks to the bottom, and methyl ester, which is a translucent liquid, floats on top. The reaction was arrested in each sample by

adding ten to fifteen drops of water. The reaction mixture was poured into a separating funnel. The mixture should be allowed to settle for at least 8 hours. The separation of methyl esters and glycerol occurs due to the density difference between methyl esters and glycerol. The methyl esters obtained is separated and water is added to the methyl esters gently over the sides so that traces of glycerol present along with the methyl esters mixes with water to form soap and settles down. The process is repeated until all the impurities are removed completely. The methyl ester is then mixed with water and it is gently agitated through a bubbling process in which air is pumped into the methyl ester-water mixture. The bubbling process is done for about 30 minutes. Thus, the methyl ester obtained after the bubbling process is completely devoid of glycerol. The variables affecting transesterification such as catalyst concentration (0.47- 0.58% wt. of oil), methanol/oil ratio (0.22 - 0.37 v/v), reaction temperature (40 - 65°C) and reaction time (30 - 90 min.) were studied to get higher conversion.

#### Engine testing procedure

A single cylinder four stroke direct injection air-cooled diesel engine normally used for agricultural applications was employed. The details of this engine are given in table.2. The engine was coupled to an electrical dynamometer to provide brake load. The fuel consumption was measured with the aid of glass burette and stopwatch on the volume basis. A Chrommel alumel thermocouple was used along with a digital temperature indicator for measuring the exhaust gas temperature. The MRU Delta 1600L determines the emissions of CO (carbon monoxides), CO<sub>2</sub> (carbon dioxides), HC (hydrocarbons) with means of infrared measurement and O<sub>2</sub> (oxygen) and NO<sub>x</sub> (nitrogen oxides) with means of electrochemical sensors. Smoke levels were measured with help of an AVL 415 smoke meter.

**Table.2: Engine Specifications**

Engine Type	Four stroke, stationary, direct injection, diesel engine
Make	Kirloskar
Model	TAF1
Maximum Power	4.4 kW @ 1500 RPM
Maximum Torque	28 N-m @ 1500 RPM
Bore	87.5 mm
Stroke	110 mm
Compression Ratio	17.5:1
Injection Timing	23.4° bTDC
Loading Type	Electrical Dynamometer

## RESULTS AND DISCUSSION

### Optimization of biodiesel

The percentages of yield of methyl esters are presented as graphs for the different conditions of molar ratios of methanol to vegetable oil, reaction temperatures and catalyst amounts. The percentage yield of methyl esters is calculated by using equation (1)

$$\% \text{ of yield} = \left[ \frac{\text{methyl esters (in gms)}}{\text{vegetable oil (in gms) in the reaction}} \right] \times 100 \quad \text{--(1)}$$

#### Effect of Methanol/UCO ratio:

Fig.1 shows the variation of % of yield of methyl esters with Methanol/UCO for various catalyst/UCO ratios at reaction temperature 65°C for reaction time of 1 hr at stirring speed of 250 rpm. It is clear from the fig that the yield of esters increases directly with the excess of methanol/UCO ratio but there was negligible increase in yield beyond the methanol/UCO ratio of 0.294. The percentage of yield of ester increased with increase in the catalyst/UCO ratio up to 0.52% and further increase in catalyst/UCO ratio, results in decreased yield of methyl esters. This is due to the formation of soap at higher catalyst amounts.

#### Effect of Catalyst/oil ratio:

The amount of catalyst used in the process is an important variable because it determines the reaction rate and also interferes in the separation of glycerol and esters. Methanolysis of UCO was carried out with NaOH as a catalyst at a concentration of 0.47 - 0.574 % wt of oil at stirring speed at 250 rpm, Methanol/oil ratio of 0.294, reaction time 1 hr. The Fig 2 shows the yield of methyl esters versus catalyst/UCO ratios at different temperatures. It was observed that temperature had positive influence on methanolysis of UCO.

#### Effect of Reaction time:

The variation in the yield of Methyl Esters with Reaction time for different Catalyst/UCO ratio is shown in Fig 3. It is observed that the yield of Methyl esters is very less for lesser reaction timings and also the % yield of Methyl Esters increases with increase in Reaction time for all Catalyst / UCO ratios. It is depicted

that the variation in % of yield of methyl esters is negligible at higher Reaction timings.

**Effect of Reaction temperature:**

Fig 4. shows the variation of % of yield of methyl esters with reaction temperature for different with methanol/UCO ratio of 0.294 and catalyst/UCO ratio 0.52% at a stirring speed of 250 rpm. It is observed that, the percentage of yield of methyl esters increase with increase in reaction temperature. The reaction temperature above boiling point of methanol is incised since at high temperature it tends to accelerate the saponification of glycerides by the catalyst before completion of the alcoholysis.

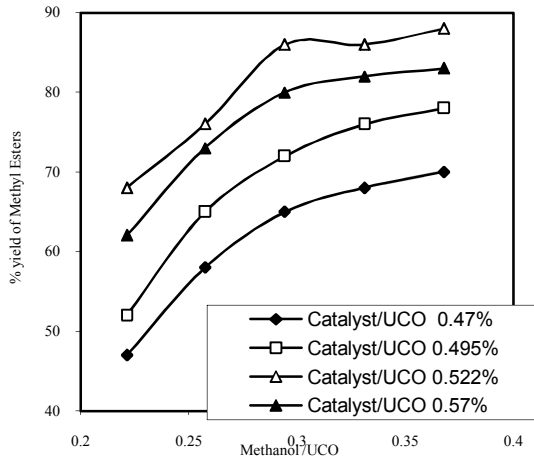


Fig 1: Variation in the Yield of methyl esters with Methanol/UCO ratio for various catalyst/UCO ratio (Rn Temperature : 65°C, Reaction Time : 1 Hr)

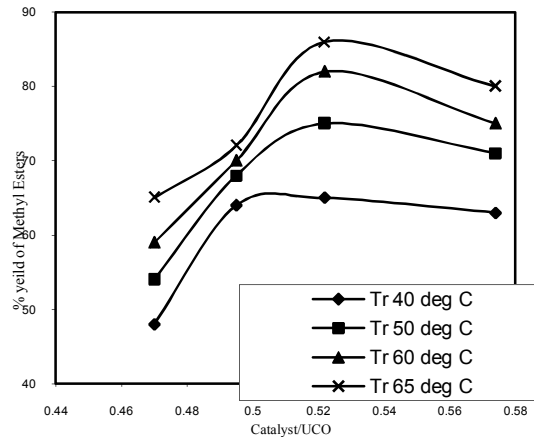


Fig 2: Variation in the Yield of methyl esters with Catalyst/UCO ratio for various Temperatures (Rn Time : 1 Hr, Methanol/UCO : 0.294)

**Performance and emission characteristics of diesel engine**

Figure 5 shows that brake thermal efficiency of UCME and its blend is lower compared to that of diesel. At rated load the BTE of UCME is lower than that of diesel by 2.5%. The BTE of blend of UCME lie between those of diesel and UCME at all loads. . Since the engine is operated under constant injection advance and UCME has a smaller ignition delay, combustion is initiated much before TDC is reached. This increases compression work and more heat loss and thus reduces the brake thermal efficiency of the engine.

Figure 6 shows the variation of nitrogen oxide (NO<sub>x</sub>) emission with brake power for diesel, 20%UCME and UCME. It is observed that the Nox emissions are higher for UCME and its blend compared top diesel. The NO<sub>x</sub> increase for UCME may be associated with the oxygen content of the UCME, since the oxygen present in the fuel may provide additional oxygen for NO<sub>x</sub> formation. Another factor causing the increase in NO<sub>x</sub> could be the possibility of higher combustion temperatures arising from improved combustion. However NO<sub>x</sub> can be controlled by adopting Exhaust Gas Recirculation and by employing suitable catalytic converters.

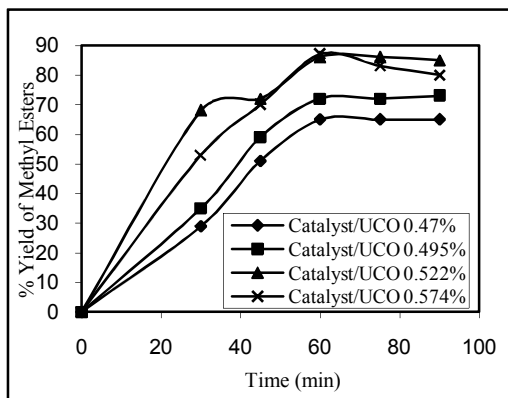


Fig 3 : Variation in the yield of Methyl Esters with time for various catalyst/UCO ratios.(Reaction Temperature : 65°C, Methanol/UCO : 0.294)

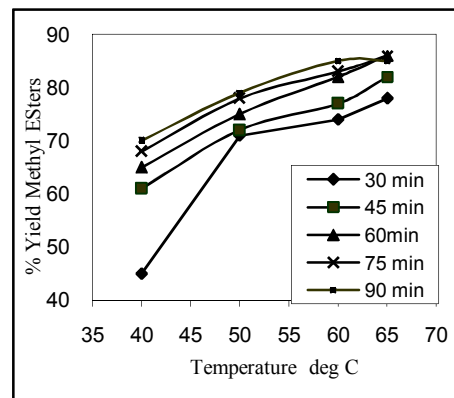


Fig 4: Variation in the Yield of methyl esters with Temperature for various Reaction timings. (Catalyst/UCO : 0.52%, Methanol/UCO : 0.294)

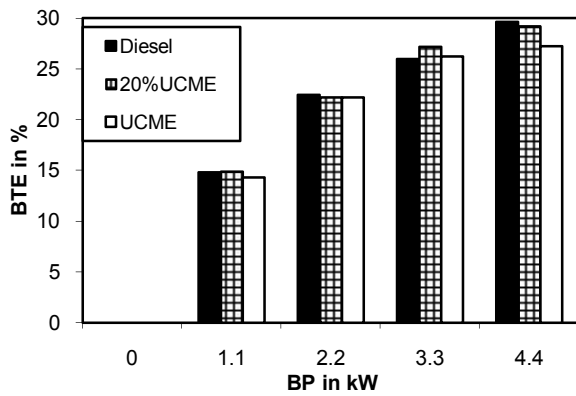


Fig.5 Comparison of brake thermal efficiency for UCME - diesel blends

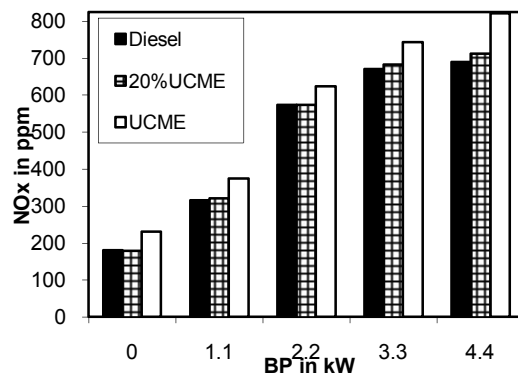


Fig.6 Comparison of Nitrogen Oxides for UCME - diesel blends

Figure 7 shows that carbon monoxide emissions are greatly reduced with the addition of UCME to diesel. The emission of CO is reduced by 15% for 20%UCME and by 50% for UCME when compared to diesel at rated load condition. CO is predominantly formed due to the lack of oxygen. Since UCME is an oxygenated fuel, it leads to better combustion of fuel resulting in the decrease in CO emission. Reduction in CO emissions is a strong advantage in favor of UCME.

The unburnt hydrocarbon (UBHC) emissions with UCME and its blend are compared with diesel in Figure 8. UBHC emissions are reduced over the entire range of loads for UCME and its diesel blend. Since the UCME is an oxygenated fuel, it promotes combustion and results in reduction in UBHC emissions.

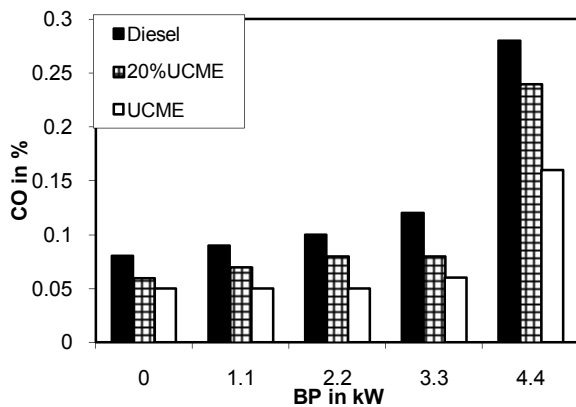


Fig.7 Comparison of carbon monoxide for UCME - diesel blends

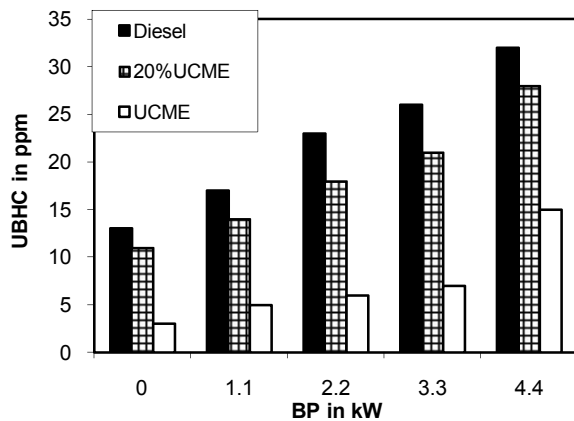


Fig.8 Comparison of UBHC emissions for UCME - diesel blends

Smoke or soot primarily comprises of carbon particles. The improved combustion characteristics of UCME may lead to fewer unburnt fuel particles impinging on cylinder walls (wall quenching). Table 3 presents the smoke intensity of Diesel, UCME and its blends. A vast reduction in smoke intensity is observed with increase in percentage of UCME in the blend, especially at high loads. UCME and its blends as fuel in diesel engines significantly reduce smoke.

Brake Power (kW)	Smoke intensity in mg/m <sup>3</sup>		
	Diesel	20%UCME	UCME
0	2	2	2
1.1	9	6	7
2.2	19	16	15
3.3	82	37	29
4.4	206	115	72

Table 3. Comparison of Smoke intensity

## CONCLUSION

In this study, Biodiesel was prepared from used cooking oil and tested in single cylinder diesel engine and compared with diesel. It was found that with biodiesel the engine operated smoothly without notable problems.

- The yield variations for change in alcohol volume, catalyst concentration and temperature have been studied and the optimal conditions for the parameters are found to be Methanol to used cooking oil – 0.294, Reaction temperature – 65°C, Catalyst to used cooking oil – 0.52% and Reaction time –1 hour.

The properties of methyl esters satisfied the standards of IS specified in literature.

- Blending UCME with diesel causes BTE to drop. Brake Specific Fuel Consumption (BSFC) is increased due to lower BTE and lower calorific value of UCME compared to diesel. BSFC increases gradually with increase in percentage of UCME and uniformly throughout the range of engine outputs.
- As far as emissions are concerned, UCME recorded greater values of NO<sub>x</sub> with respect to diesel at rated loads. Blending of UCME increases NO<sub>x</sub> emissions. However the increase is negligible for 20% blend over the lower range of engine outputs. Emissions of CO and HC decrease with increase in percentage of UCME in blend. It is also observed that there is a great reduction in smoke density especially at greater engine outputs even if 20% UCME is blended with diesel.

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**Citation of Article:** S.Sreenatha Reddy. Production and Testing of Methyl Esters of Used Cooking Oil. *Int. Arch. App. Sci. Technol.*, Vol 4 [2] June 2013: 76- 81

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