

# Production and characterization of biodiesel from ceiba pentandra seed oil

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## ABSTRACT

*In this research work the production of a biodiesel from ceiba pentandra seed oil by methanol induced transesterification using an alkaline catalyst (KOH) has been examined. The effect of the operating variables such as agitation speed, reaction time, temperature, catalyst concentration and alcohol amount were determined experimentally and found to be 550 rpm agitation rate, 60 min reaction time, 60° C reaction temperature, 14grams of catalysts concentration and 500ml of methanol for 2.5 liters of oil. The yield of biodiesel from the ceiba pentandra methyl ester (CPME) under the optimized conditions is found to be 92%. The properties of biodiesel are measured as per ASTM standards and compared with the base diesel.*

**Keywords:** Ceiba pentandra seed oil, Transesterification, Biodiesel, optimization

## I. INTRODUCTION

Ever increasing demand, not commensurate with the source, of petroleum derived fuels (diesel) and threat to the global environment has given an impetus for researchers to find a viable and eco- friendly alternative fuels for the energy hungry countries to meet energy requirement. The switch to biodiesel brings a sustainable alternative source for diesel fuel. Bio diesel is a renewable and environmental friendly fuel that is derived from vegetable oil. vegetable oils cannot be used as fuel directly owing to the presence of free fatty acids, phospholipids, Sterols, water, Odorant and other impurities which causes potential problems without engine modification and have served disadvantages like poor atomization due to high viscosity, incomplete combustion and carbon deposit build up on several engine parts such as injectors, piston rings, cylinder walls and valve seats. However, the problem of high viscosity can be solved by using a number of techniques like preheating, microemulsion, pyrolysis, blending, super critical method and transesterification methods . Among these, transesterification process has been widely used to prepare bio-diesel from vegetable oil and animal fats. Transesterification process reduces viscosity and it enhances the fuel properties. [1]. In

general, vegetable oil contains 97% of triglycerides and 3% of di- and monoglycerides and fatty acids. Transesterification is the chemical reaction between triglycerides and short chain alcohol in the presence of catalyst to produce mono-ester. The long and branched chain triglyceride molecules are transformed to mono-ester and glycerin. Transesterification or alcoholysis, is the displacement of alcohol from one ester by another alcohol in a process similar to hydrolysis. Transesterification is the process of using an alcohol (methanol or ethanol) in the presence of catalyst, such as sodium hydroxide or potassium hydroxide, to chemically break the molecule of the raw oil into methyl or ethyl esters of the oil with glycerol as a by-product. This process has been widely used to reduce the viscosity of triglycerides. The transesterification reaction requires a catalyst for better efficiency of the process [2]. The transesterification of triglycerides by methanol, ethanol, propanol, and butanol has proved to be the most promising process. Methanol is the commonly used alcohol in this process, due to its low cost. The important variables affecting the esters yield during the transesterification reaction are, catalyst type and concentration, molar ratio of alcohol to oil and type of alcohol, effect of reaction time and temperature and mixing intensity. The experiments for kinetic data on the transesterification reaction. This is achieved by conducting the reaction at various temperatures and molar ratios. They found that acid catalyst transesterification was slower than alkali transesterification. The compared homogeneous and heterogeneous catalytic systems. They reported that homogeneous catalyst process requires mild operation conditioning while heterogeneous catalyst process required high temperature of reaction and higher of methanol to oil ratio. The three principal variables, molar ratio of methanol to oil, amount of catalyst and reaction temperature affecting yield of acid-catalyzed production of methyl ester (bio diesel) from crude palm oil. The biodiesel was extracted in batch and

continuous acetone-butanol-ethanol formation and its fuel properties were analyzed. The optimized variables were 40:1 methanol/oil (mole/mole) with 5% H<sub>2</sub>SO<sub>4</sub> (volume/weight) reacted at 95 °C for 9 hours gave a maximum ester yield of 97%. The biodiesel prepared from mahua oil by transesterification process using sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) with a catalyst 5% weight/weight, 20:1 molar ratio of ethanol to mahua oil at a temperature of 72- 75 °C for a period of 5 hours. The ester was washed with salt water(5% NaCl solution) and the product was dried at 110 °C in an oven for an hour to remove the traces moisture. The bio diesel produced from palm kernel oil by transesterification process using KOH as a catalyst for 10g of ethanol for 100g of palm kernel oil at a temperature of 60 °C for a period of 100 minutes to produce a yield of 92%. Kaemee and Anju Chadha (2005) reported that KOH catalyzed transesterification of crude pongamia oil resulted in high conversion of 92% in 1.5 hours at 60 °C with a molar ratio 1:10 (oil:methanol). A survey on biodiesel engine performance and exhaust emissions researcher published after the year 2007 determined that 85% of the literature indicates that the use of biodiesel reduced CO, HC, and PM emissions due to the higher oxygen content of biodiesel compared to diesel fuel [3]. From the above literature review, it is clear that majority of the researchers have been focused on transesterification process of various types of vegetable oils (poon, karanja, jojoba, cottonseed, pongamia, ricebran, soyabean, koroch, Honge, orange, castor, coconut, bitter alomond, peanut, okra, camelina,neem, mahduca, sunflower,, rupper, Hoone, cardinal oil,etc..) for production of biodiesel . The main focus of this research is to produce biodiesel from ceiba pentandra seed oil by transesterification process as fuel and also to evaluate the properties of ceiba pentandra biodiesel blends with diesel (B25, B50, B75 and B100) in comparative with diesel fuel.

## II. MATERIALS AND METHODS

In this section, a detailed explanation of the materials used as well as the bio diesel production method will be shown

### 2.1 Material

The ceiba pentandra pods were collected at Kottapalayam, Palamedu village, Tamilnadu, India on October 2014. It was grown naturally in agricultural land. Ceiba pentandra seed occupy about 35- 42% (W/W) of each fruit. The oil extracted by using steam treatment process shadowed by mechanical crushing

process. The oil seed yield was producing on average 2850 kg/ha. Ceiba pentandra seed were low feeding value owing to its higher fiber content. Furthermore, the possibility of kapok (ceiba pentandra) fiber as bio-ethanol feed stock. The found that kapok fiber contains 34 to 64% of cellulose higher potential to produce cellulosic ethanol. Traditionally kapok fiber is utilized as stuffing material for pillows and bed.

### 2.2 Transesterification Reaction:

Transesterification, also called alcoholics, is the displacement of alcohol from an ester by another alcohol in the process similar to hydrolysis except then that an alcohol is used instead of water. This has been widely used to reduce the viscosity of the triglycerides. *2.3 Bio-diesel production unit and procedure for the transesterification process:*

Mixing of reactants is found to be important factor which affects the transesterification and yield of biodiesel. The schematic diagram of the biodiesel production unit as shown in figure 1. Operation was carried out up to a speed of 600 rpm in the first stage of reaction (10min) and a lower stirring speed of 550 rpm for remaining period. A 2.5liters of ceiba pentandra raw oil is filtered using musline cloth to remove the impurities in the oil and poured in to a biodiesel production unit. At room temperature the conversion efficiency is noted to be very low (about 10%) even after two hours of stirring. The required amount of catalyst pellets (KOH) is quickly weighed, protecting it as much as possible from the atmospheric moisture and carbondi-oxide. The pellets are quickly transferred to the dry mixture grinder to convert it in to powder form. The powered catalyst and solvent of methanol solution is then vigorously shaken in a conical flask for homogeneous mixing. At this point dissolved catalyst of potassium hydroxide (KOH) is presumed to have been converted into potassium methoxide. A fixed amount of solvent and catalyst solution is poured into the raw oil container and the container is closed with an air tight lid to mark the beginning of the reaction. Care should take that they maintain temperature around 60 °C. Because methanol evaporates at temperature higher than 65 °C. Once the reaction reaches the preset reaction time, heating and stirring are stopped. Once the reaction period is over the product is allowed to settle overnight. Two distinct liquid phases are identified. Cured esters phase at the top and glycerol phase at bottom. That is biodiesel process turns the oil into esters, separating out the glycerol. The glycerol sinks at the bottom and the biodiesel floats on top and

can be syphoned off. The crude esters phase is separated and glycerol phase is then washed by warm de-ionized (double distilled) water for several times until the washed water becomes clear. The excess methanol and water in the ester phase is removed or recycled by evaporation under atmospheric condition after being measured for product yield calculation.

18. Shaft

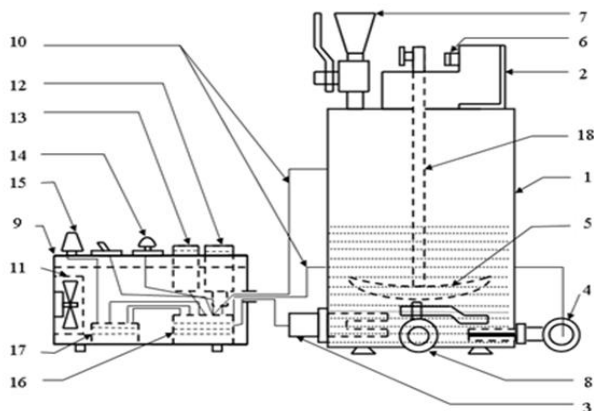


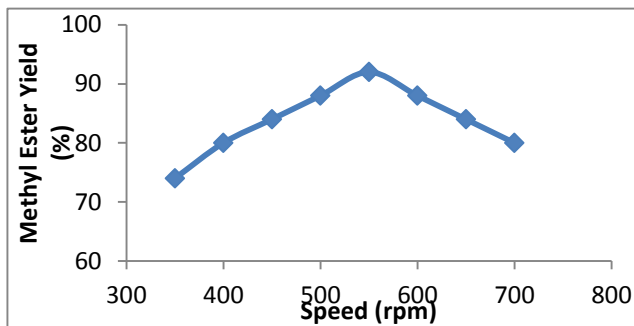
Figure1 Schematic of the bio-diesel production unit

- |  |  |
|--|--|
| 1. Container   | 11. Cooling fan for microprocessor kit |
| 2. DC motor  | 12. Temperature indicator              |
| 3. Heater  | 13. Speed (rpm) indicator              |
| 4. Thermocouple  | 14. ON/OFF switch                      |
| 5. Blades  | 15. Speed regulator for DC Motor       |
| 6. Speed sensor  | 16. Transformer                        |
| 7. Inlet valve   | 17. Sensor controller                  |
| 8. Outlet valve  |  |
| 9. Microprocessor kit  |  |
| 10. Electrical connection for motor, heater and thermocouple |  |

### III Optimization Process

#### 3.1 Effect of speed of the stirrer

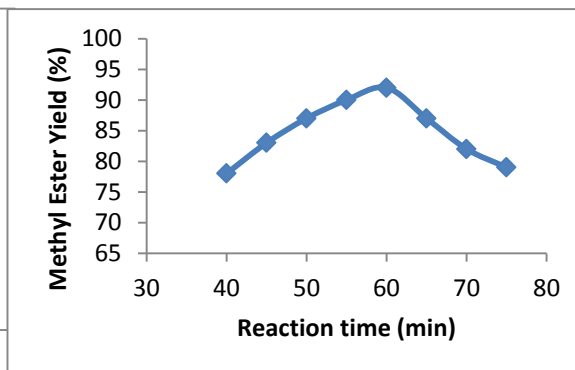
Mixing is very important in the transesterification reaction, as an oil and fat are immiscible with potassium hydroxide-methanol solution. Once the two phase of mixed the reaction is started. Methanolysis was conducted with different stirrer rates such as 350, 400, 450, 500, 550 and 600 revolutions per minutes (rpm). The variation of methyl esters yield with different rate of stirrer is shown in the figure 2. It was observed from the figure 2 the reaction was incomplete up to 500 rpm, and rate of mixing was insufficient for methanolysis. Further increasing the speed of 550 rpm was 92% and again further increase in the speed of stirrer to 600 rpm. The yield of methyl esters was reduced to 88%. Therefore, the speed of 550 rpm is sufficient for production of maximum yield of bio-diesel for this batch type production unit.



**Figure 2 Influence of stirrer speed on methyl ester yield. Methanol 500ml, 14g catalyst amount, 60°C reaction temperature and reacted time of 60 min.**

### 3.2 Effect of reaction time

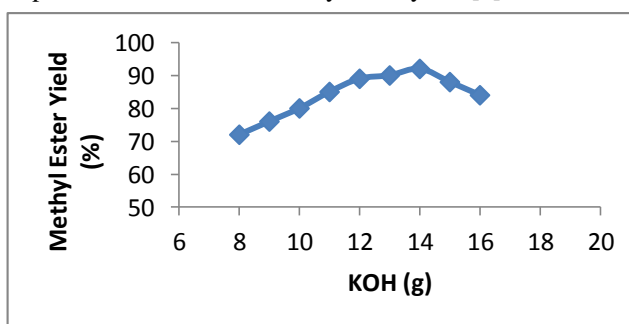
The reaction time is required for the completion of alkaline-catalyst transesterification depends not only on the reaction temperature, but also on the degree of mixing in the process. The reaction rate of transesterification increase with increase temperature and degree of mixing. The reaction time of transesterification process is conducted at 60° C ie. Nearer to the boiling point of the methanol. The variation of methyl ester yield with different reaction time as shown in figure 3. From the figure yield of biodiesel is less at the beginning and reaches a maximum at the reaction time of 60 minutes (min) at 60° C and relatively decrease afterwards. The reaction time of the product yield after more than 65 minute to 75 minute has no significant effect on the conversion triglycerides, but leads to a reduction in the product yield. This is because, longer reaction time enhances the hydrolysis of esters (reverse reaction of transesterification) which results in the loss of esters as well as causing more fatty acids to form soap. The gradual increase of reaction time shows the negative effect on the product yield [4].



**Figure 3 Influence of reaction time on methyl ester yield. Methanol 500ml, 14g catalyst amount, 60°C reaction temperature and stirrer speed 550 rpm.**

### 3.3 Effect of Catalyst concentration

The variation of methyl esters yield with different quantities of catalyst. The yield of methyl esters varies with different quantities of catalysts. It was observed from the figure 4 that the catalyst concentration of 14g of KOH was optimal in the reaction with the yield of 92% in 60 minutes. With 15g of catalyst, the yield of biodiesel was 88%. It is clearly showed 14g of KOH was optimal for producing the methyl esters of biodiesel. Excess amount of catalyst was apt to form emulsion which increased the viscosity and led to the gels. The formation of emulsion will therefore block the reaction (lin lin et al.,2009). No clear separation occurs during washing with warm deionized water, and addition of excess alkaline catalyst caused more triglycerides participation in the saponification reaction with potassium hydroxide, resulting more amount of soap and reduction of the methyl ester yield [5].



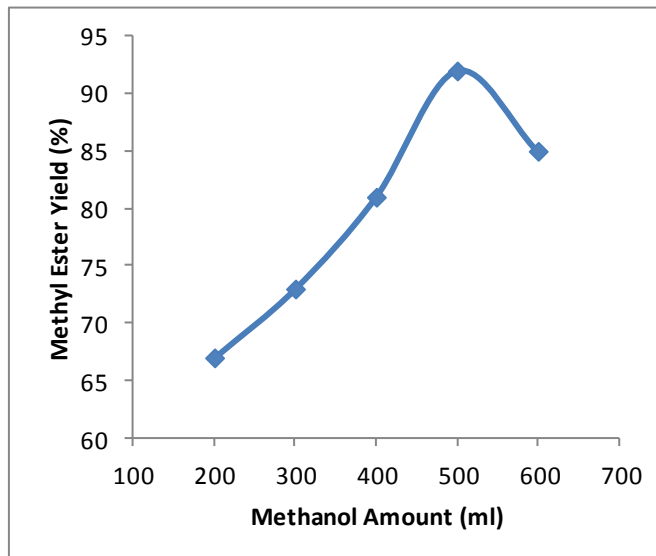
**Figure 4 Influence of catalyst concentration on methyl ester yield. Methanol 500ml, 60°C reaction**

**temperature, stirrer speed 550 rpm and reacted time of 60 min.**

### 3.4 Effect of methanol to oil ratio

The methanol to oil ratio is one of the most important factor that affect the transesterification reaction process. The reaction temperature is set at 60°C. The stoichiometric molar ratio of alcohol triglyceride is 3:1. However, the transesterification is an equilibrium reaction and an extra amount alcohol is required to drive the reaction to the right side of reaction products [6]. Figure 5 shows the methyl ester yield increases as the quantity of methanol was increased up to an optimal value of 500ml and at that point a maximum yield of 92% was observed. Further increase in the methanol amount and decrease the product yield.

The separation of esters from glycerol becomes difficult when the methanol to oil ratio is increased above 500 ml. The presence of unreacted methanol, can suppress the gravity decantation. So that the apparent yield of biodiesel decreases because traces of glycerol may remain in the biodiesel phase. The glycerol remaining in the solution drives the equilibrium back to the left side of reaction produced in the lower yield of esters. Similar results also obtained by [7]. From raw ceiba pentandra seed oil the ester is prepared by using transesterification unit with help of alcohol and catalyst. The physical properties of the biodiesel were tested as per ASTM standards and also the relevant physic-chemical properties were experimentally evaluated and compared with the base diesel. The viscosity of raw oil as well as esterified oil was measured by red wood viscometer (ASTM D445), density by hydrometer (ASTM D1298), calorific value by bomb calorimeter (ASTM D2015), flash and fire point are measured with the help of open cup method (ASTM D93), cetane number (ASTM D613), pour point (ASTM D97), acid value (ASTM D664)[8].



**Figure 5 Influence of methanol to oil ratio on methyl ester yield. 14g catalyst amount, 60°C reaction temperature, stirrer speed 550 rpm and reacted time of 60 min.**

### IV. Ceiba pentandra methyl ester Properties

The physical properties of diesel, biodiesel-diesel blends were compared in table I. It was clearly proved from table I that the transesterification reduces the viscosity, density, flash point and fire point of biodiesel (B100) compared with raw oil. When the biodiesel blends ratio increases, increased specific gravity, kinematic viscosity, flashpoint, and acid value, when compared to diesel. It may be due to attribution of the higher molecular weight of triglyceride molecules present in the oil. Higher molecular structure of the oil jointed with the saturated and unsaturated fatty acid structure present in the bio diesel. On the other hand, the net calorific value of the bio diesel mixtures proportionally reduced with increases biodiesel percentage in blends. These may be due to oxygen content in the biodiesel and difference in the percentage of hydrogen and carbon content in the esters.

Table I Physical and chemical properties of CPME

Properties	Diesel	B25	B50	B75	B100
Kinematics Viscosity(cSt) at 40°C	3.20	3.51	3.90	4.22	4.58
Specific gravity	0.838	0.848	0.861	0.870	0.882

Density (Kg/m <sup>3</sup> )	838	848	861	870	882	for biodiesel production. Energy Conversion and Management, 79, 246.
Calorific value (MJ/kg)	42.800	42.092	41.396	40.684	40.016	[6] Lin, L., Dong, Y., Sumpun, C., & Saritporn, V. (2009). Biodiesel production from crude rice bran oil and properties as fuel. Applied Energy, 86, 681.
Flash point ( °C)	53	62	85	104	148	[7] Arumugam, A., & Ponnusami, V. (2014). Biodiesel production from Calophyllum oil using lipase producing Rhizopus oryzae cells immobilized within reticulated foams. Renewable Energy, 64, 276.
Fire point ( °C)	65	76	101	119	163	[8] Panneerselvam, N., Murugesan, A., Vijayakumar, C., Kumaravel, A., Subramaniam, D & Avinash, A. (2015). Effects of injection timing on biodiesel fuelled engine characteristics- An overview. Renewable and Sustainable Energy Reviews, 50, 17.
Cetane number	53	-	-	-	55.4	
Acid Value (mg KOH/g)	0.16	0.19	0.25	0.31	0.37	

## V. Conclusion

Production of ceiba pentandra oil methyl esters (CMME) as biodiesel was carried out by using transesterification process. The optimum conditions for transesterification process were stirrer speed 550rpm, reaction time 60 min, catalyst concentration 14g and methanol amount 500ml with the conditions the optimal CPME yield achieved was 92%. The biodiesel product was blended with diesel to prepared (B25 to B100), physical and chemical properties biodiesel blends and compared with ASTM standards. This result is very close to diesel.

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