

## Research Article

# Biodiesel Synthesis from Pongamia Pinnata Oil by Acid and Alkaline Transesterification Method

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**Abstract**

The Pongamia tree seed containing 27% - 39% oil was taken in the present study for the production of biodiesel by transesterification using base catalyst (NaOH) and acid catalyst (H<sub>2</sub>SO<sub>4</sub>). The Physico chemical parameters such as Moisture, Iodine value, Saponification value, Viscosity, Acid value and Free Fatty Acid were studied. The observed FFA content of the *Pongamia pinnata* oil was above 3 percent. Therefore two stage transesterification reactions were carried out. Characterization of the fatty acid methyl esters (FAMES) was accomplished by FTIR studies. Various experimental variables, such as catalyst amount, duration, temperature and oil molar ratio were studied. The maximum methyl ester yield was found in 6% wt of catalyst dosage, 60 minutes time duration, 60°C temperature and 30 ml of methanol.

**Keywords:** Biodiesel, Transesterification, pongamia pinnata oil, Methyl ester

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**Introduction**

Recently, developing countries such as India and China have experienced a significant increase in energy demand. In addition, some of the world's largest producers of oil have also suffered from warfare and political and social instability. Diminishing fossil fuel resources, coupled with the steady increase in energy consumption, has spurred research interest in alternative and renewable energy sources. Biodiesel is one among the most promising fossil fuel alternatives [10]. There are at least four ways in which oils and fats can be converted into biodiesel, namely, transesterification, blending, micro emulsions and pyrolysis, transesterification being the most commonly used method [6]. Transesterification refers to a catalyzed (KOH/NaOH) chemical reaction involving oil/fat (triglyceride) and an alcohol (methanol/ethanol) to yield fatty acid alkyl esters (i.e., biodiesel) and glycerol. The main factors affecting transesterification are the amount of alcohol and catalyst, reaction temperature, pressure and time, free fatty acids (FFA) and water content in the oils. Conversion is complicated if oil contains higher amount of FFA (>1% w/w) that will form soap with alkaline catalyst. The soap can prevent separation of the biodiesel from the glycerin fraction [3]. In this above context, we are searching the alternative fuel. Pongamia is one of the best biofuel crops.

Pongamia tree grows up to about 50 to 80 feet tall and is native to subtropical regions. It is a viable non-toxic alternative to jatropha. The Asian varieties reach adult height in 4 or 5 years and start bearing seeds at 4 to 7 years. It can produce 50 to 100 lbs of seed per tree. Assuming 200 trees per acre and 25% oil per pound of seed 100 to 600 gallons of oil per acre can be produced [3]. Genetically modified plants produce more. The tree all over India is used to line roadways and waterways. The seeds can be picked up from these plants. The small quantities of oil expelled in presses in local villages can either be added to diesel used by the village or sold. The oil cake can be used as animal fodder or manure. In the present study it is focused to increase the yield of methyl esters from Pongamia oil.

## Materials and Methods

### Biodiesel production/transesterification reaction

FFA value of oil decides the transesterification process. If free fatty acid content of the oil is lower than 3%, single process is carried out. If it is greater than 3%, double stage process is carried out.

### Acid esterification process

100ml of *Pongamia Pinnata* oil (FFA>3%) was taken in clean conical flask. The moisture content of the given oil was removed by heating the oil at 80°C for 1 hour in magnetic stirrer. After cooling, 30ml of methanol and sulphuric acid was added (volume of H<sub>2</sub>SO<sub>4</sub> = FFA value x 0.05). It was continuously stirred for 1 hour at 65°C. This mixture was separated for overnight. Top layer consisting of acid esterified oil and bottom layer known as residues were found. The bottom layer was separated out from acid esterified oil and used it for biodiesel production.

### Transesterification process

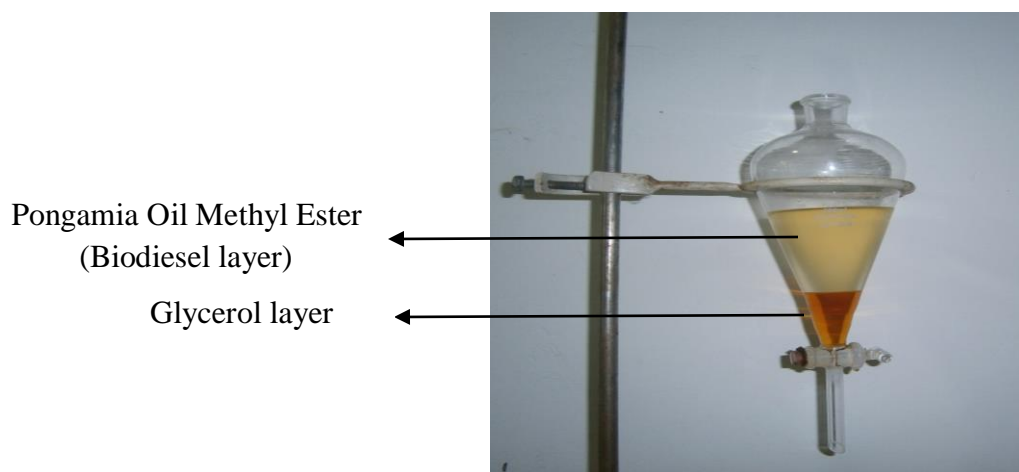
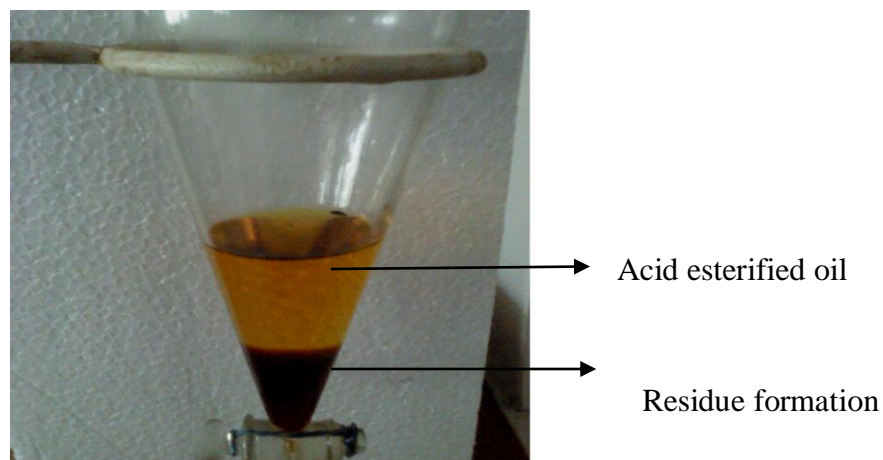
100ml of transesterified oil (FFA < 3%) was taken in a clean conical flask. The moisture content of the given oil was removed by heating it at 80°C for 1 hour in magnetic stirrer. After cooling, 20 ml of methanol and 0.4 g of NaOH were added. It was continuously stirred for 1 hour at 65°C. This mixture was separated for 2-3 hour. Top layer containing biodiesel and bottom layer glycerin were separated.

(Ref: *National Renewable Energy Laboratory – Biodiesel Analytical Methods 2004*)

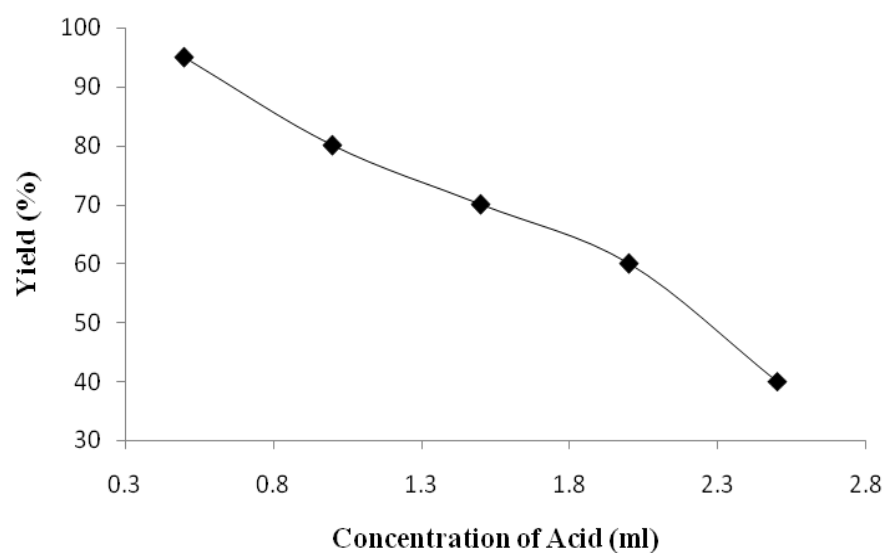
**Table 1** The physico-chemical properties of the *Pongamia* oil

Parameter	Characteristic
Moisture (%)	0.06
Iodine value (gram of iodine /100g)	89
Saponification value (mg of KOH/g)	195
Viscosity <sub>40°C</sub> (mm <sup>2</sup> /s)	48.5
Acid value (mg of NaOH/g)	12.5
Free fatty acid value (%)	6.25

Source: ASTM methods [1]



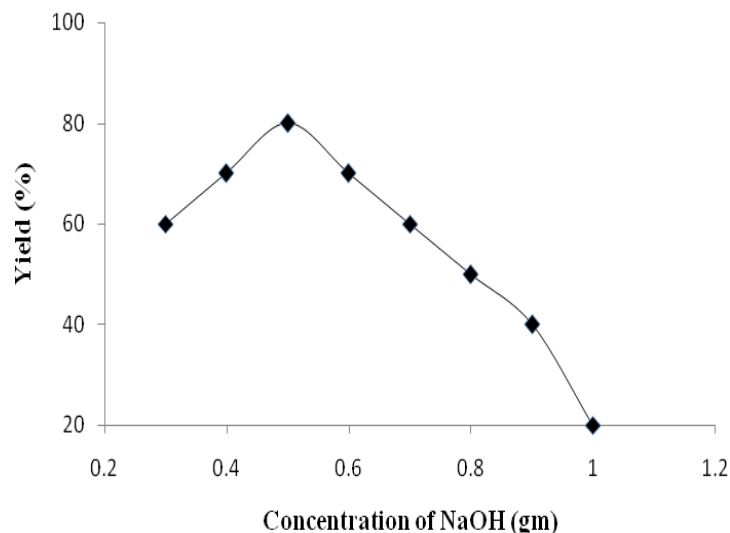
**Figure 1** Two stage transesterification process



**Figure 2** Effect of acid on esterification yield

### Effect of acid on esterification yield

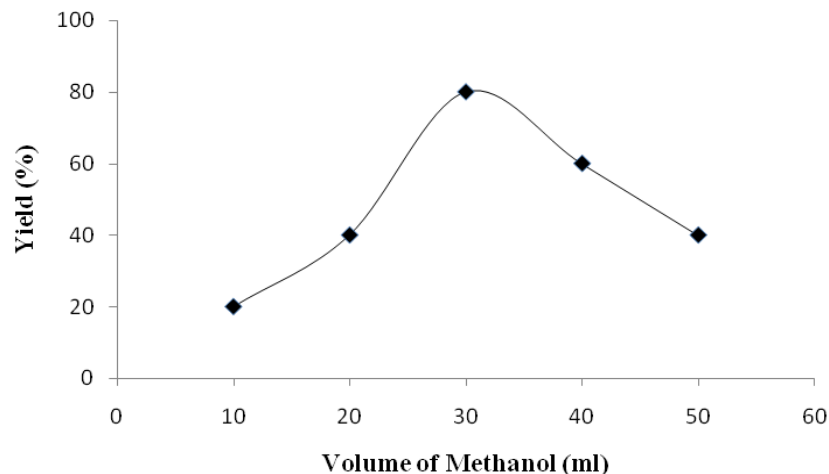
The sulfuric acid-catalyst amount was varied in the range of 0.25–2.5 wt%. These percentages are based on the volume of the oil used for the acid esterification reaction [4]. The pretreatment of high FFA Pongamia oil using acid-catalyzed esterification was shown in Figure 2. It was also observed during the present experiments that addition of excess sulfuric acid darkens the color of the product but yields remains the same for oil. The maximum esterified yield was obtained 95 %.



**Figure 3** Effect of alkali on biodiesel yield

### Effect of alkali on biodiesel yield

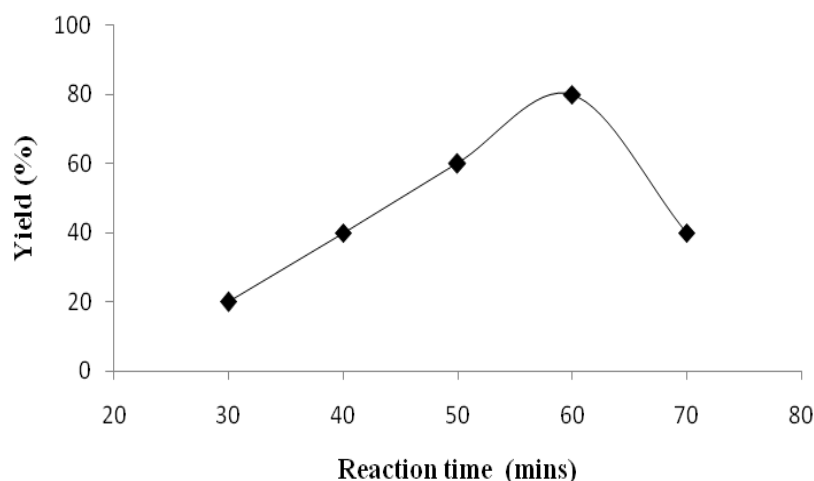
Transesterification of oil was carried out with NaOH as a catalyst at a concentration of 0.3–1% (w/w oil). Figure 3 shows the yield of biodiesel versus NaOH concentration. The lower catalytic concentration of 0.3% of NaOH was insignificant to catalyze the reaction to complete. It was found that the biodiesel yield increased as the amount of catalyst concentration increased from 0.3% to 0.6% in this study. Excess amount of catalyst was quick to form emulsion which increased the viscosity and led to the formation of gels. The formation of emulsion will therefore block the reaction [7]. The maximum methyl ester yield was obtained 90 %.



**Figure 4** Effect of methanol on biodiesel yield

### Effect of methanol on biodiesel yield

The methanol/oil ratio is one of the most significant factors that influence the reaction process. The stoichiometric ratio for transesterification requires three moles of alcohol and one mole of triglyceride to yield three moles of fatty acid alkyl esters and one mole of glycerol. However, in practice, transesterification is an equilibrium reaction in which excess of alcohol is required to drive the reaction to the right side of reaction products. The methanol to oil is one of the most important variables affecting the ester yields [2]. In the present study, the methanol to oil was varied from 10ml to 50ml. The optimum yield was obtained at 30 ml methanol concentration. Figure 4 shows the effect of methanol to oil molar ratio on yield of biodiesel. The percentage increase in biodiesel yield decreased with increase in the molar ratio as shown in the figure 4. The maximum methyl ester yield was obtained 80 %.



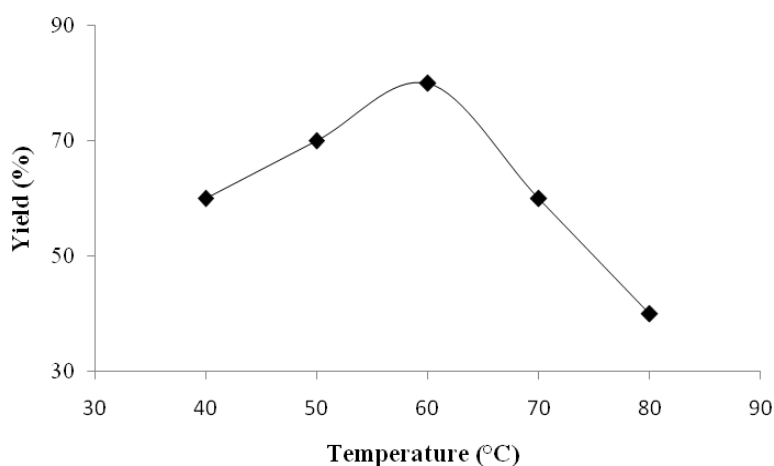
**Figure 5** Effect of reaction time on biodiesel yield

### Effect of reaction time on biodiesel yield

Studies were carried out at different reaction time such as 40, 50, 60, 70, 80 and 90 minutes. The yield of biodiesel versus reaction time at different NaOH catalyst and methanol to oil molar ratio of 30 ml for a reaction temperature of  $60 \pm 1^\circ\text{C}$  is shown in Figure 5. It was observed that biodiesel yield increased with increase in reaction time from 40 to 60 minutes. As the reaction time increased beyond 60 minutes, the biodiesel yield decreased. However, for reaction time less than 40 min, the biodiesel yield was insignificant. Hence it was not considered. Optimum reaction time in order to get maximum biodiesel yield found to be 60 minutes [5]. The maximum methyl ester yield was obtained 80 %.

### Effect of temperature on biodiesel yield

Catalytic transesterification of vegetable oil is normally investigated close to the boiling point of the methanol. Experiments were carried out at different temperatures such as  $40^\circ\text{C}$ ,  $50^\circ\text{C}$ ,  $60^\circ\text{C}$ ,  $70^\circ\text{C}$  and  $80^\circ\text{C}$ . The biodiesel yield follows an increasing trend with increase in reaction temperature up to  $60 \pm 1^\circ\text{C}$ . The reaction temperature above boiling point of methanol ( $65^\circ\text{C}$ ) should be avoided since at higher temperature, it tends to loss methanol and accelerates saponification of the glycerides by the alkaline catalyst before completion of the alcoholysis [12]. The maximum methyl ester yield was obtained 85 %

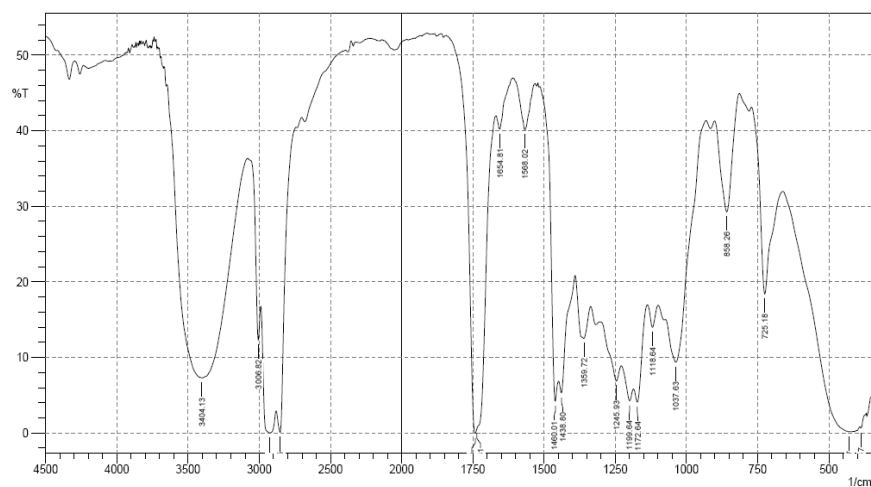


**Figure 6** Effect of temperature on biodiesel yield

### Analysis of pongamia oil methyl ester in FTIR

FTIR finds a feasible solution of all biodiesel components without derivation. FTIR is useful for quantifying the various conversions of transesterification reactions. Generally, raw oil contains a mixture of low and high molecular weight components spread over different reaction time in chromatograph with higher molecular mass being detected at higher retention time [8, 7]. In the chromatograph the esters appear in the order of increasing number of carbon atoms and increasing unsaturation level. Number of peaks corresponding to saturated and unsaturated fatty acids of biodiesel components was less than oils and it is due to reduction in molecular mass of individual components of the biodiesel due to transesterification process [11]

The infrared spectrum of biodiesel showed the presence of pronounced functional groups, which indicated the presence of alkanes and poly-aromatic groups. Different bonds were present in biodiesel (C-C, C=C, C-O, C=O, O-H, N-H etc.) with different vibrational frequencies. The presence of these bonds in biodiesel could be detected by identifying characteristic frequencies as absorption bands in the infrared spectrum. Functional group vibrations were made for most of the dominant peaks. The typical FT-IR spectrum of the transesterified oil showed that it contained significant amount of esters. Transformation of vegetable oils into biodiesel was confirmed from the IR bands in the region 1188–1200 for O-CH<sub>3</sub> stretching in all biodiesel IR spectrums as seen in Figure 7. The value suggests this spectrum corresponds to pongamia biodiesel.



**Figure 7** FTIR Spectrum result of Pongamia oil biodiesel

## Conclusion

Because of fossil fuel shortage and emissions, we are now looking for the various source of renewable energy. Of the various sources; biodiesel is the promising source of energy substituting petro diesel. Various studies were carried out to optimize the yield of biodiesel. In the present study, the overall optimum conditions for the biodiesel yield were determined with respect to different parameters. The maximum biodiesel yield of 90% was found out at the catalyst dosage of 6%, reaction time of 60 min and reaction temperature of 60 °C and the methanol was 30 ml.

## Acknowledgement

The financial assistance received from the University Grants Commission (UGC) in the form of UGC-BSRF is gratefully acknowledged. The facilities provided in the DST – Purse program is greatly acknowledged.

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### Publication History

Received 23<sup>rd</sup> Jan 2015  
Revised 04<sup>th</sup> Feb 2015  
Accepted 25<sup>th</sup> Feb 2015  
Online 30<sup>th</sup> Mar 2015