

FRACTIONAL FACTORIAL DESIGN FOR OPTIMIZATION OF BIODIESEL SYNTHESIS FROM CRUDE PUNGAM OIL

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ABSTRACT

Biodiesel is a mixture of fatty acid alkyl esters that is used mainly in transportation and power sectors. The aim of the present work is to optimize biodiesel production from crude pungam (*Millettia pinnata*) oil with methanol using homogeneous sulphuric acid and sodium hydroxide catalysts by esterification and transesterification, respectively. The factors affecting the biodiesel production from crude non-edible seed oils are molar ratio of alcohol to oil, mass ratio of catalyst to oil, reaction time, temperature, and agitation speed. In this work, transesterification reaction was carried out between crude pungam oil and methanol in the presence of sodium hydroxide as a catalyst. A maximum free fatty acids (FFA) conversion of 66.8% was achieved at the optimum process parameters of molar ratio of alcohol to oil, mass ratio of catalyst (NaOH) to oil, reaction time, temperature, and agitation speed of 9, 4, 1 h, 65 °C and 100 rpm for esterification. During transesterification, a maximum FFA conversion of 83.3% was achieved at the optimum process parameters of molar ratio of alcohol to oil, mass ratio of catalyst (NaOH) to oil, reaction time, temperature, and agitation speed of 6, 1, 40 min, 65 °C and 100 rpm. The results showed that crude pungam oil could serve as potential renewable substrate for biodiesel production.

Keywords: Crude pungam oil, Sodium hydroxide, Esterification, Transesterification, Biodiesel

INTRODUCTION

Biodiesel is a clean-burning alternative fuel derived from domestic and renewable resources. The fuel is composed of alkyl esters of fatty acids derived from either pure edible vegetable oils or pure non-edible vegetable oils, waste vegetable oils, crude vegetable oils, algal oils, animal fats, or recycled fats [1]. Wherever applicable, biodiesel can be used in its purest form with little or no modification in current diesel engines. Biodiesel is simple to use, biodegradable, non-toxic, and almost sulphur- and aromatic-free. It is typically added to petrodiesel as a reducer of particulate matter, carbon monoxide, hydrocarbon, and other pollutants [2]. When added to diesel fuel, it is referred to as B5, B10, or B20. This is the volume ratio of biodiesel to petrodiesel. For instance, B5 indicates a 5:95 blend of biodiesel and petroleum diesel [3].

The production process of biodiesel depends on the FFA of feedstock. If the feedstock has $FFA \leq 4\%$, then the biodiesel production is through single step, transesterification. If the feedstock has $FFA \geq 8\%$, then the biodiesel production is through dual step process, esterification followed by transesterification. Esterification is performed by reacting oil with alcohol in the presence of acid catalyst whereas transesterification is carried out by reacting oil with alcohol in the presence of alkali or heterogeneous catalyst to produce alkyl esters. Biomass-derived alkyl esters can be blended with conventional diesel fuel or utilized as a pure biofuel [4,5]. Oils from plant oils, animal fats and algal sources could be used for biodiesel synthesis. Soybean oil [6], rapeseed oil [7], and palm oil [8] are the most frequent edible oil feedstocks for biodiesel production, accounting for most of the global biodiesel output. Other non-edible basic resources include *Jatropha* [9], mustard [10], flax [11], and hemp [12]. Animal fats, such as sebum [13],

lard [14], yellow fat [15], chicken fat [16], and fish oil [17] derivatives, may contribute a tiny percentage of future biodiesel production, but their availability is limited and raising animals for their fat is not economical.

Biodiesel can be blended with petrodiesel in any proportion, or it can be utilized in its pure form. As with petrodiesel, biodiesel runs on auto-ignition and requires little or no engine modification, as biodiesel shares many of the same qualities as diesel [18]. Because it can be stored in the same way as petrodiesel, it does not require a separate infrastructure. When biodiesel is used in conventional diesel engines, emissions of unburned hydrocarbons, carbon monoxide, and particulates are significantly reduced. Now, a considerable number of biodiesel production plants worldwide are operating at full capacity, and a similar number are under construction or being built to satisfy expanding global demand [19].

Numerous scientific papers on the generation of biodiesel from pure edible vegetable oils [6-8], pure non-edible vegetable oils [9-12], waste vegetable oils [20-22], algal oils [23-25], and animal fats [13-17] are accessible in the literature. However, there is a dearth of literature on the generation of biodiesel from crude vegetable oils [26-27] and recycled fats [28-29]. Thus, the current work focuses on optimizing process parameters for the synthesis of biodiesel from crude pongam oil with methanol through catalytic esterification and transesterification.

MATERIALS AND METHODS

Materials

Pongam seeds were collected from the local agricultural farm in Tamil Nadu, India. The seeds were washed, ground and the mixture were subjected to solvent extraction in Soxhlet apparatus. The crude pongam oil was collected from the assembly, stored, and used for further experimental studies. All the chemicals used in the work are of analytical grade and the products of VWR International. Double distilled water was used in this study.

Methods

Esterification of crude pongam oil

Esterification reaction was carried out in a three necked flat-bottomed flask. The top neck is used for fitting condenser to condense the vapours formed during reaction. The right neck is used for fitting thermocouple for monitoring temperature. The left neck is used for periodic sample collection for analysing FFA. The flask was loaded with 3 L of crude pongam oil, 1.125 L of methanol, and 40 mL of sulphuric acid. Then, the mixture was heated at 65 °C for 1 h with mixing at 100 rpm. After the time is completed, the mixture was allowed to settle for two distinct layer separation of esterified oil and excess reactants. The esterified oil was washed thoroughly with hot water to remove excess reactants, and finally dried to remove moisture from esterified oil. Finally, FFA of crude and esterified oils were measured and the percentage conversion was calculated using the Equation (1) as given below:

$$\% \text{ conversion} = \frac{(\text{FFA of crude oil} - \text{FFA of esterified oil})}{\text{FFA of crude oil}} \times 100 \quad (1)$$

Transesterification of esterified pongam oil

Transesterification reaction was carried out in the same setup used for esterification. The flask was loaded with 2 L of esterified pongam oil, 0.5 L of methanol, and 18 g of sodium hydroxide. Then, the mixture was heated at 65 °C for 40 min with mixing at 100 rpm. After the time is completed, the mixture was allowed to settle for two distinct layer separation of biodiesel and glycerol. From the top layer, the mixture of biodiesel and water with little quantity of methanol was washed thoroughly with hot water to remove excess methanol, and finally dried to remove moisture from biodiesel. Finally, FFA of esterified oil and biodiesel were measured and the percentage conversion was calculated using the Equation (2) as given below:

$$\% \text{ conversion} = \frac{(\text{FFA of esterified oil} - \text{FFA of biodiesel})}{\text{FFA of esterified oil}} \times 100 \quad (2)$$

Fractional factorial design approach for optimization of biodiesel synthesis from crude pongam oil

Fractional factorial design approach is a strategy for designing experiments that involves $m^f - n$, where m is the number of levels of each factor investigated, f is the number of factors investigated, and n describes the size of the fraction of the full factorial used. Formally, n is the number of generators, assignments as to which effects or interactions are confounded, i.e., cannot be estimated independently of each other. The fractional factorial design approach is used for optimization of both esterification and transesterification.

During esterification, the effect of molar ratio of methanol to oil (5-11) was studied by fixing mass ratio of catalyst to oil, time, temperature, and agitation speed at 4, 1 h, 65 °C and 100 rpm, respectively. The effect of mass ratio of catalyst to oil (2-5%) was studied by fixing mole ratio of alcohol to oil, time, temperature, and agitation speed at 9, 1 h, 65 °C and 100 rpm, respectively. The effect of time (0 – 2 h) was studied by fixing mass ratio of catalyst to oil, mole ratio of alcohol to oil, temperature, and agitation speed at 4, 9, 65 °C and 100 rpm, respectively. The effect of temperature (55-75 °C) was studied by fixing mass ratio of catalyst to oil, mole ratio of alcohol to oil, time, and agitation speed at 4, 9, 1 h and 100 rpm, respectively. The effect of agitation speed (50-150 rpm) was studied by fixing mass ratio of catalyst to oil, mole ratio of alcohol to oil, time, and temperature at 4, 9, 1 h and 65 °C, respectively.

During transesterification, the same levels were maintained for time (0 – 2 h), temperature (55-75 °C), and agitation speed (50-150 rpm). But, the effect of mass ratio of catalyst to oil and molar ratio of methanol to oil (0.5 – 2%) and (4-10) were studied by fixing time, temperature, and agitation speed at 40 min, 65 °C and 100 rpm, respectively.

Analytical procedures:

FFA was estimated following the procedure as follows [30]: Standard solvent was prepared by mixing 25 mL diethyl ether and 25 mL 95% ethanol and titrated against 0.1 N KOH using 1 mL of 1% phenolphthalein solution as an indicator. 5 g of oil

was dissolved in 50 mL of standard solvent in a 250 mL Erlenmeyer flask. The contents are titrated against 0.1 N KOH using few drops of phenolphthalein as an indicator. The end point is the appearance of pink colour that lasts for 15 s. Then, FFA was calculated using the equation as below:

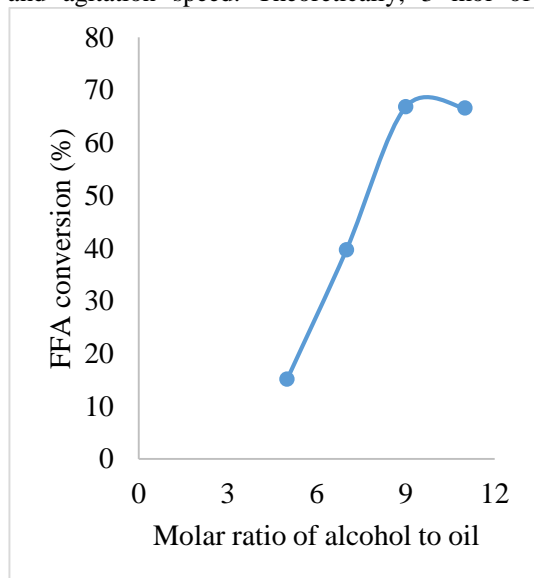
$$\text{Free fatty acid value (mg KOH/g oil)} = \frac{\text{Titre value} \times \text{Normality of KOH} \times 28.05}{\text{Mass of oil in g}}$$

The properties of various samples were determined for density, dynamic viscosity, flash and fire points, and cloud and pour points were determined using standard operating procedures followed for pycnometer, Fungilab ViscoLead rotational viscometer, Pensky-Martens apparatus, and Koehler K46100 cloud and pour point bath, respectively. Kinematic viscosity was calculated as a ratio between dynamic viscosity and density and expressed as mm²/s.

RESULTS & DISCUSSION

Esterification of crude pungam oil

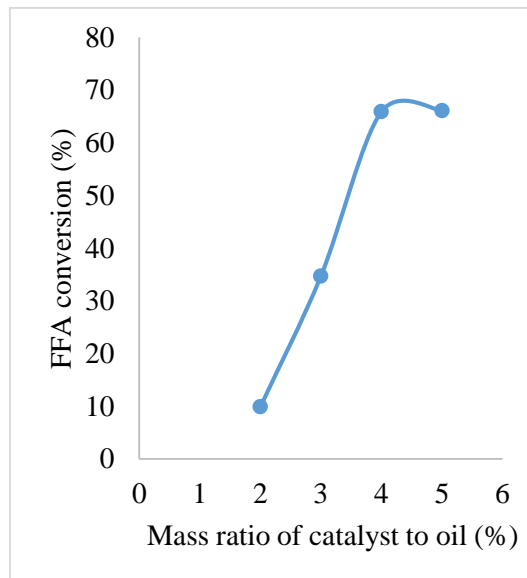
Figure 1(a) shows the effect of molar ratio of alcohol to oil on FFA conversion at constant mass ratio of catalyst to oil, time, temperature, and agitation speed. Theoretically, 3 mol of



(a)

alcohol is required for esterification of triglyceride. But, being an excess reactant, alcohol is added always in excess to that of stoichiometric value. Hence, molar ratio of alcohol to oil was varied from 5 to 11. The % FFA conversion initially increased with increase in molar ratio of alcohol to oil from 5 to 9. Above the molar ratio of 9, conversion started to remain constant. This is because high content of methanol has no effect on reaction conversion [31].

Figure 1(b) shows the effect of mass ratio of catalyst to oil on FFA conversion at constant mole ratio of alcohol to oil, time, temperature, and agitation speed. Mass ratio of catalyst to oil was varied from 2 to 5% (w/w). In esterification reaction, oil is the limiting reactant, methanol is the excess reactant, and sodium hydroxide is the catalyst. According to the standard operating procedure of esterification, mass ratio between catalyst and oil should be between 2 and 5% (w/w). The % FFA conversion initially increased with increase in mass ratio of catalyst to oil from 2 to 4% (w/w). Beyond the mass ratio of 4% (w/w), conversion started to remain constant. This is because catalytic poisoning occurs at higher concentration of catalyst [32].



(b)

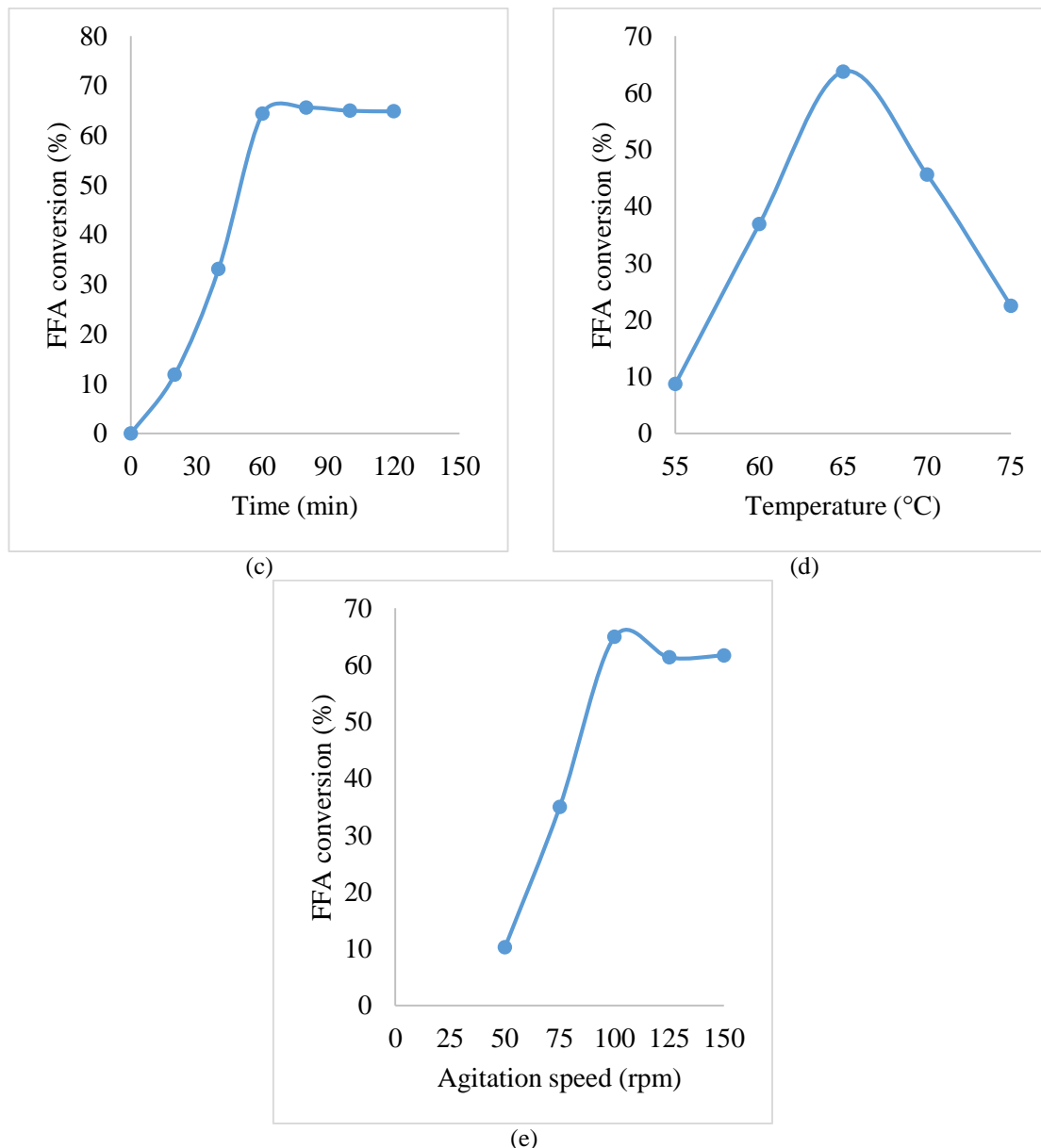


Figure 1. Effect of (a) mole ratio of alcohol to oil; (b) mass ratio of catalyst to oil; (c) reaction time; (d) temperature; and (e) agitation speed on % FFA conversion during esterification of crude pungam oil

Figure 1(c) shows the effect of reaction time on FFA conversion at constant mass ratio of catalyst to oil, mole ratio of alcohol to oil, temperature, and agitation speed. Reaction time varied from 0 to 2 h. In esterification, reaction should be carried out between oil, methanol and acid catalyst for significant reduction in FFA. The % FFA conversion initially increased with increase in time and thereafter remained plateau. It means that equilibrium is not achieved before plateau. The reaction reaches equilibrium from a region of plateau [33]. In the case of pungam oil, an equilibrium conversion of 74% is achieved at 1 h [33,34].

Figure 1(d) shows the effect of temperature on FFA conversion at constant mass ratio of catalyst to oil, mole ratio of alcohol to oil, time, and agitation speed. Reaction temperature was selected based on the boiling point of alcohol. Hence, reaction temperature varied boiling point of methanol (65 ± 10 °C). The % FFA conversion initially increased with increase in feed temperature from 55 to 65 °C. Beyond the temperature of 65 °C,

conversion started to decline. This is because above 65 °C, loss of methanol is more which leads to the decrease in conversion [35].

Figure 1(e) shows the effect of agitation speed on FFA conversion at constant mass ratio of catalyst to oil, mole ratio of alcohol to oil, time, and temperature. Agitation enhances mass transfer between components in the mixture to facilitate maximum reaction conversion. The % FFA conversion initially increased with increase in agitation speed from 50 to 100 rpm. Above the agitation speed of 100 rpm, conversion started to remain constant. This is because mass transfer resistance occurs at higher speed of mixing [36].

From the analysis of OFAT results for esterification, it was found that a maximum FFA conversion of 66.8% was achieved at the optimum process parameters of molar ratio of alcohol to

oil, mass ratio of catalyst (NaOH) to oil, reaction time, temperature, and agitation speed of 9, 4, 1 h, 65 °C and 100 rpm.

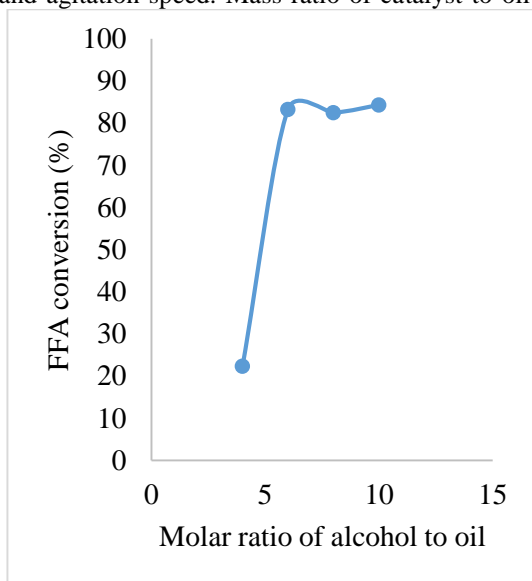
Transesterification of esterified pongam oil

Figure 2(a) shows the effect of molar ratio of alcohol to oil on FFA conversion at constant mass ratio of catalyst to oil, time, temperature, and agitation speed. The % FFA conversion initially increased with increase in molar ratio of alcohol to oil from 4 to 10. Above the molar ratio of 6, conversion started to remain constant.

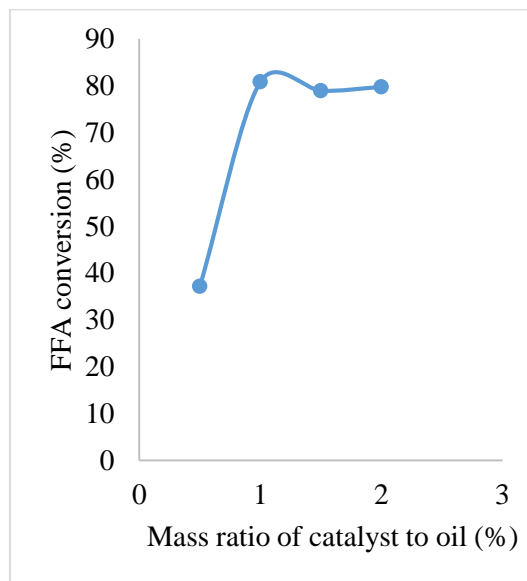
Figure 2(b) shows the effect of mass ratio of catalyst to oil on FFA conversion at constant mole ratio of alcohol to oil, time, temperature, and agitation speed. Mass ratio of catalyst to oil

was varied from 0.5 to 2% (w/w). The % FFA conversion initially increased with increase in mass ratio of catalyst to oil from 0.5 to 1% (w/w). Beyond the mass ratio of 1% (w/w), conversion started to remain constant.

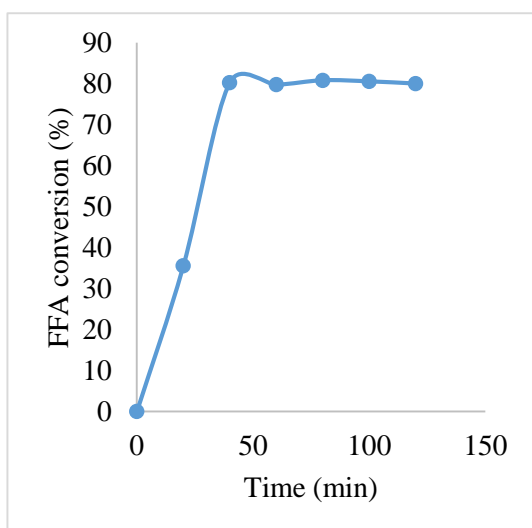
Figure 2(c) shows the effect of reaction time on FFA conversion at constant mass ratio of catalyst to oil, mole ratio of alcohol to oil, temperature, and agitation speed. The % FFA conversion initially increased with increase in time and thereafter remained plateau. It means that equilibrium is not achieved before plateau. The reaction reaches equilibrium from a region of plateau [33]. In the case of pongam oil, an equilibrium conversion of 90% is achieved at 40 min.



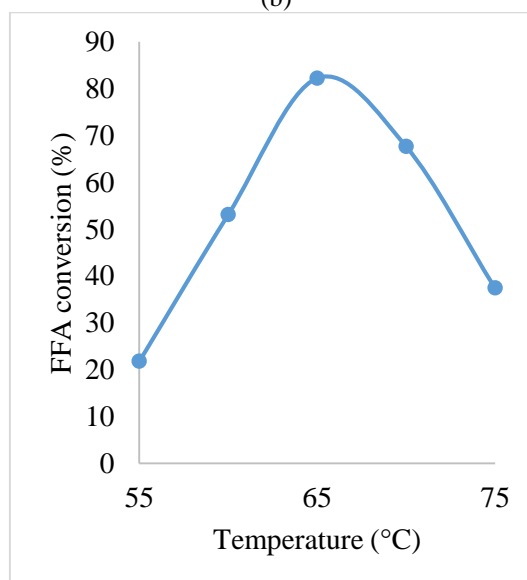
(a)



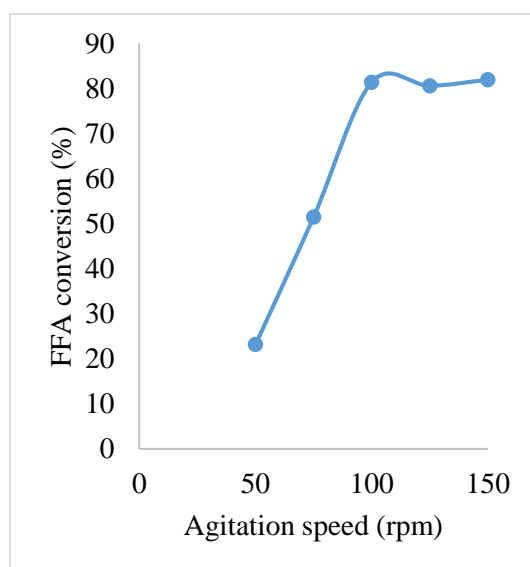
(b)



(c)



(d)



(e)

Figure 2. Effect of (a) mole ratio of alcohol to oil; (b) mass ratio of catalyst to oil; (c) reaction time; (d) temperature; and (e) agitation speed on % FFA conversion during transesterification of esterified pungam oil

Flash point (°C)	223	209	72
Fire point (°C)	251	232	74
Cloud point (°C)	13	8	-12
Pour point (°C)	14	3	-26
Free fatty acid (%)	18	0.73	Not detected

Figure 2(d) shows the effect of temperature on FFA conversion at constant mass ratio of catalyst to oil, mole ratio of alcohol to oil, time, and agitation speed. The % FFA conversion initially increased with increase in feed temperature from 55 to 65 °C. Beyond the temperature of 65 °C, conversion started to decline.

Figure 2(e) shows the effect of agitation speed on FFA conversion at constant mass ratio of catalyst to oil, mole ratio of alcohol to oil, time, and temperature. The % FFA conversion initially increased with increase in agitation speed from 50 to 100 rpm. Above the agitation speed of 100 rpm, conversion started to remain constant.

From the analysis of OFAT results for transesterification, it was found that a maximum FFA conversion of 83.3% was achieved at the optimum process parameters of molar ratio of alcohol to oil, mass ratio of catalyst (NaOH) to oil, reaction time, temperature, and agitation speed of 6, 1, 40 min, 65 °C and 100 rpm.

Table 1 shows the properties of various samples such as crude pungam oil, pungam oil methyl ester (MOME), petrodiesel. The values given in the table further validate that crude pungam oil could be utilized as a potential substrate to produce biodiesel.

Property	Crude pungam oil	Pungam oil methyl ester	Petrodiesel
Density (g/mL)	0.952	0.924	0.839
Kinematic viscosity (mm ² /s)	24	4.1	2.3

CONCLUSION

This work aimed to study the applicability of crude pungam oil to produce biodiesel using fractional factorial design based one-factor-at-a-time (OFAT) approach. The optimal values show that a maximum FFA conversion of 66.8% was achieved at the optimum process parameters of molar ratio of alcohol to oil, mass ratio of catalyst (NaOH) to oil, reaction time, temperature, and agitation speed of 9, 4, 1 h, 65 °C and 100 rpm for esterification, and 83.3% was obtained at the optimum process parameters of molar ratio of alcohol to oil, mass ratio of catalyst (NaOH) to oil, reaction time, temperature, and agitation speed of 6, 1, 40 min, 65 °C and 100 rpm for transesterification. A total conversion of 95.94% was attained from esterification followed by transesterification of crude pungam oil. Thus, the results showed that crude pungam oil could be utilized as a potential substrate to produce biodiesel.

ACKNOWLEDGEMENT

We would like to acknowledge the management of Vels Institute of Science, Technology & Advanced Studies and Kumaraguru College of Technology for their kind cooperation and support in completing the research.

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