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Statistical Optimization of Biodiesel Production from Non-edible Pongamia Pinnata Oil

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Abstract

Biodiesel production has received considerable attention in the recent past as a renewable fuel to mitigate climate change and for achieving the UN Sustainable Development Goals (SDGs) clean affordable energy and climate action. This research study was based on optimization of the conversion of pongame oil into biodiesel fuel through lab scale batch reactor by acid esterification and alkali transesterification. using Statistical simulation analysis was conducted by Taguchi Orthogonal Array Design, with input factor variables were volume of methanol (20, 25, 30, 40 mL), quantity of catalyst used (0.8, 1.0, 1.8, 2.0 g), volume of acid used (0.2, 0.3, 0.4 0.5 mL), reaction time (55, 60, 90, 120 min) and reaction temperature levels (55, 60, 65, 70 °C). The highest pongamia biodiesel yield was obtained 78.25% experimentally as compared to simulation yield of 84.02% by weight with an error of 5.44%. The highest biodiesel yield was achieved with transesterification carried out for 100 mL pongamia oil under optimized reaction parameters such as methanol amount of 20 mL, KOH amount of 0.8 g, acid amount of 0.2 mL, reaction time 60 min reaction temperature of 55 °C at stirring rate of 700 rpm. The results showed that the properties of the pongamia biodiesel were in compliance with ASTM D 6751 and can be used as blended fuel with petro-diesel in transportation sector, thus reducing environmental pollution causing climate change and global warming.

Keywords

biodiesel, pongamia, transesterification, optimization, alternative fuel

1 Introduction

Biofuels are the most suited alternative fuels to reduce the effects of fossil fuel combustion, which causes climate change and global warming [1–3]. With characteristics similar to petroleum-derived diesel, biodiesel is an alternative, renewable, biodegradable, and ecologically beneficial fuel for transportation that may be used straight in a compression ignition engine without any change [4, 5].

The biodiesel is produced by transesterification reaction that reduces the viscosity of vegetable oil, can be used to turn vegetable oils into biodiesel [6, 7]. Refined vegetable oil triglycerides (long chain fatty acids) combine with alcohol, such as methanol or ethanol, in the presence of a sodium hydroxide or potassium hydroxide catalyst to produce esters, which are used in the synthesis of biodiesel. The byproduct of the reaction, glycerol can also be used in pharmaceutical industry. Tests on diesel engines have shown that they operate well on biodiesel fuel without requiring extensive engine hardware modifications and with lower greenhouse gas emissions [8].

Pongamia pinnata (karanja) is a non-edible legume tree that produces seeds that are high in fatty acids and triglycerides [9]. Unsuitable for human consumption, non-edible oils contain a number of hazardous and unsaponifiable ingredients [10]. Pongamia is quickly turning into the focus of several biodiesel research work due to its high oil yield content (about 40% by weight) and ability to thrive on deficient soils with low levels of nitrogen and high levels of salt. Pongamia has a number of advantages over other crops, including a better oil recovery and quality than other crops, the ability to be cultivated on

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marginal and damaged areas, and the fact that it doesn't directly compete with food crops or existing cropland. The amount of potassium hydroxide needed to neutralize the sample is used to calculate the acid value, which is a measurement of the number of acidic functional groups in a sample. Alkaline transesterification feedstock free fatty acid value must be decreased to less than 4 mg KOH/g because water and higher FFA (free fatty acids) promote soap production, deplete the catalyst, and limit its efficiency, resulting in decreased alkyl ester yield [11].

Previous research study conducted on refined *Pongamia pinnata* (pongame) oil obtained from local source using acid esterification and alkaline transesterification in a batch reactor, was successfully converted into biodiesel yield (97.35%). The optimum reaction conditions were (temperature 60 °C, methanol to oil volume ratio 48%, H_2SO_4 to oil volume ratio 0.2%, KOH to oil volume ratio 1%, stirring speed 600 rpm, and reaction time 240 min) were found by a series of tests. The analysis of the pongame oil biodiesel were in accordance with ASTM D 6751 and EN 14214 international standards revealed that it had superior qualities to those of canola and jatropha oils. In comparison to canola B20 and jatropha B20, the environmental emissions profile of pongame B20 (80% mineral diesel and 20% biodiesel) from a compression ignition engine was found better. It was noted that pongame B20's engine performance was comparable to that of jatropha B20. According to the research's findings, it is theoretically possible to use biodiesel made from the local *Pongamia Pinnata* plant rather than jatropha curcas to meet Pakistan's rising energy needs [12].

According to previous research study Fourier transform infrared (FTIR) analysis, of pongamia (karanja) biodiesel (B100) fuel primarily included esters, whereas the diesel fuel primarily contained alkanes and alkenes. The gas chromatography (GC) analysis of B100 showed that karanja oil could only yield a maximum of 97% methyl ester. The results of the engine experiments revealed that while all biodiesel blends decreased engine emissions of CO, smoke, and noise, they increased NO_x. B100 reduced CO and smoke emissions by 50% and 43% respectively, when compared to diesel fuel, while an increase in NO_x emissions of 15% was observed. Engine noise was recorded 2.5 dB quieter with B100 than diesel fuel [13]. Characteristics of mixed biodiesel fuel emissions when combusted in a four-stroke, one-cylinder compression ignition engine operating at constant speed by investigated in a research study. By using acid and base catalyzed transesterification processes, refined oils from *Jatropha curcas* and *Pongamia pinnata* are converted into biodiesel. The biodiesel made from pongamia and jatropha was mixed equally with mineral diesel fuel. Emission testing was done on diesel fuel, B10, B20, and B40 fuel samples, when compared to other samples, the B20 mixed bio diesel fuel blend produced better results, according to the emission analysis. When using B20 (10% jatropha + 10% pongamia) blend with mineral diesel, there is a 60% and 35% decreased in emissions of carbon monoxide and sulphur dioxide, respectively were measured. According to the test results, biodiesel fuel has 10% greater NO_x emissions than regular diesel fuel. However, (EGR Exhaust Gas Recirculation) technology may be able to lower these emissions. Additionally, current research has shown that B20 blended bio diesel fuel may be utilized in compression ignition (CI) engines without any modifications [14]. In an study conducted by Nandkishore et al. [15], optimization of biodiesel production from pongamia pinnata oil was conducted. The authors used Taguchi's technique for performing statistical optimization. An orthogonal array experimental design was constructed with 9 trials in total for the identification of best or optimum combination of methanol to oil molar ratio, reaction temperature and catalyst concentration.

Response surface methodology (RSM) has been utilized in previous research studies for performing statistical optimization [16–18]. RSM has several advantages over other statistical optimization tools such as model flexibility, continuous variables incorporation, higher resolutions designs, curvature analysis for finding the relationships between independent variables and response variable. RSM also helps in providing various diagnosis methods to explore the performance. Researchers looked into improving the engine performance by optimizing the input parameters of a diesel engine using pongamia biodiesel. The engine load in relation to BTE (brake thermal efficiency), BSFC (brake specific fuel consumption), exhaust gas temperature, and Pmax, as well as fuel injection pressure, timing, and blends of pongamia biodiesel, were the input parameters chosen for optimization analysis. Using the RSM technique, an experimental investigation was carried out. The optimal engine input parameters were found to be 74% of the maximum rated engine load, fuel injection pressure of 226 bar, fuel injection timing of 25 bTDC (before top dead centre), using 40% pongamia biodiesel mixing with mineral diesel. The output responses at optimal input parameters were compared between optimized and experimental findings, and the results were determined to be within the suggested error range [19].

A study using sodium methoxide (CH_3NaO), an alkaline catalyst (NaOH), was done on the production of biodiesel from composite oil of *Pongamia pinnata*, animal fat, and waste cooking oil. The yields of the two distinct sample oils obtained by using methanol and catalyst as a reaction mixture were compared. RSM was used in the design of the experiments to determine the ideal process conditions and to obtain the highest possible yield. Two oil samples were obtained: sample 01 has 50% pongamia, 25% of each type of cooking waste, and 25% of animal fat oil; sample 2 has 60% pongamia and 20% by volume of each type of cooking waste and animal fat oil. The maximum yield of 98.29% was obtained with 100 mL 01 oil, a 7.5 Molar ratio of methanol to oil (7.5:1), 1 wt% catalyst, a 60 min reaction time, a 40 °C reaction temperature, and a 500 rpm stirring rate. While, for 100 mL of sample 02 oil, 6 M ratio, 1.2 wt% of catalyst, 75 min reaction time at a reaction temperature of 64 °C and stirring speed of 500 rpm resulted in maximum yield of 97.76% [20].

Kumar et al. [21] used various machine learning approaches such as type 1 fuzzy logic system (T1FLS), RSM, adaptive neuro-fuzzy inference system (ANFIS), and type 2 fuzzy inference logic system (T2FLS) for statistical optimization of biodiesel from waste cooking oil catalyzed by ostrich-eggshell derived CaO. The authors identified that RSM is one of the most feasible techniques for performing statistical optimization. In present study Design Expert version 13.0.5.0 software [22] was used to carry out process optimization for the RSM to optimize the conversion of pongamia oil into biodiesel at a batch scale setup using quadratic model with varying dependent variables methanol to oil ratio, catalyst concentration, acid concentration and reaction temperature carried out. The analysis of variance (ANOVA) was used to investigate the factor significance and model validations and the model with the highest R^2 was obtained for best fitted model.

2 Materials and methods

Pongamia pinnata vegetable oil was obtained from Sigma Energy (Pvt) Limited, Karachi for the production of biodiesel and the experimental work was carried out in the Biodiesel Laboratory of the Department of Environmental Engineering, NED University at a room temperature of 30 ± 1 °C and relative humidity 45%.

2.1 Pongamia oil conversion into biodiesel

Due to the higher content of free fatty acids in *Pongamia pinnata* oil, found to be 5.5 mg KOH/g, with standard titration method. The two stage reactions were involved in the synthesis of biodiesel from *Pongamia pinnata* oil, i.e., acid esterification and alkaline transesterification. Reducing the amount of free fatty acids in oil requires the acid esterification stage using H_2SO_4, requires at least 1 h reaction time. Each sample of *Pongamia pinnata* oil (100 mL) was used in the experiments. The yield of biodiesel (%) produced was evaluated by varying the methanol to oil molar ratio, KOH catalyst amount, reaction, temperature, and reaction time as per the previous literature [12]. The schematic diagram of *Pongamia pinnata* biodiesel production process is depicted in Fig. 1.

2.2 Characterization of *Pongamia pinnata* biodiesel

The highest yield of *Pongamia pinnata* biodiesel produced from optimization was evaluated for the subsequent physical and chemical characteristics as per ASTM standards.

2.3 Statistical optimization of *Pongamia pinnata* biodiesel production

RSM is used for statistical optimization based on design of experiment (DOE). The process optimization was carried out using 3 factors Box-Behnken Design (BBD), performed by Design Expert software version 13.0.5.0. Three independent variables i.e, volume of methanol (A), amount of KOH catalyst (B) and reaction temperature (C) were selected for optimization study and listed in Table 1. Pongamia biodiesel yield (%) was selected as response variable. The design of experiments based on BBD ad its corresponding results are shown in Table 2.

2.4 Fatty acid composition of pongamia oil

The fatty acid analysis of pongamia oil was tested at the International Centre for Chemical and Biological Sciences

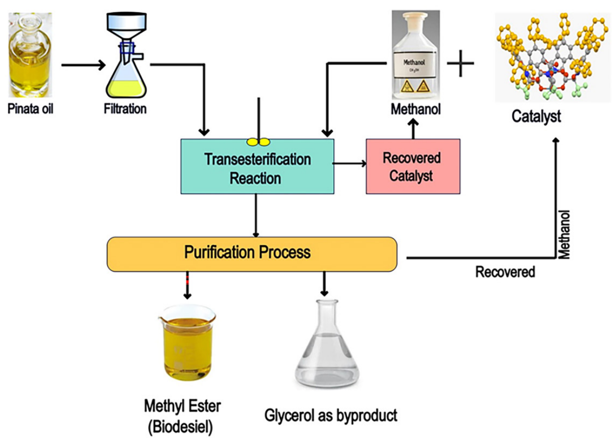

Fig. 1 Schematic diagram of *Pongamia pinnata* biodiesel production process

Table 1 The independent variables level for RSM of biodiesel yield

Independent Variables	Parameters	Low Level	High Level
A	Volume of methanol (mL)	20	40
B	Amount of KOH Catalyst (g)	0.8	2.0
C	Reaction temperature (°C)	55	70

Table 2 Experimental design for pongamia biodiesel yield (%) and corresponding experimental results

Run	Volume of methanol (mL)	Amount of KOH catalyst (g)	Reaction temperature (°C)	Pongamia biodiesel yield (%)
1	20	0.8	55	78.25
2	20	1.0	60	70.45
3	20	1.8	65	74.87
4	20	2.0	70	60.35
5	25	0.8	70	70.45
6	25	1.0	65	74.87
7	25	1.8	60	75.58
8	25	2.0	55	60.35
9	30	0.8	60	74.45
10	30	1.0	55	75.55
11	30	1.8	70	66.14
12	30	2.0	65	60.50
13	40	0.8	65	64.00
14	40	1.0	70	58.16
15	40	1.8	55	52.70
16	40	2.0	60	53.00

(ICCBS), University of Karachi, Pakistan as the literature [23]. A GC analyzer (7890B, GC systems, Aligent Technologies Inc., USA) fitted with a flame ionization detector was used to analyze the pongamia oil in order to ascertain the fatty acid profile. The methyl esters of the fatty acids were prepared by transesterification, which involved the addition of sodium chloride and sodium methoxide. 1 µL of the methyl ester extract was injected in split less mode onto a Rtx-wax (crossbonded PEG) column (30 m long, 9 0.25 mm i.d., 9 0.25 µm df (film thickness)), at an injector temperature of 250 °C, an initial oven temperature of 180 °C, and a final oven temperature of 240 °C (programmed at 5 °C min^{-1}), helium was used as a carrier gas 100 kPa (pressure of), total flow of 30 mL /min, oven equilibration time of 1.01 min, and interface temperature of 250 °C.

3 Results and discussion

3.1 Biodiesel optimized yield with respect to statistical analysis

The optimized number of results was taken out using RSM technique. The 15-number of experiments were conducted to carry out the optimization conversion of biodiesel and its balance green chemistry. In the study, three independent variables: volume of methanol (mL), amount of KOH (g), reaction temperature (°C) and one dependent variable i.e. pongamia biodiesel yield (%) were studied. The response of the conducted study is shown in the Table 2. A normal plot of residuals was plotted to analyze the experimental data and system response is shown in Fig. 2. The plot features that all number of experiments are closer to the regression line, predicting a successful run of experiments between variables.

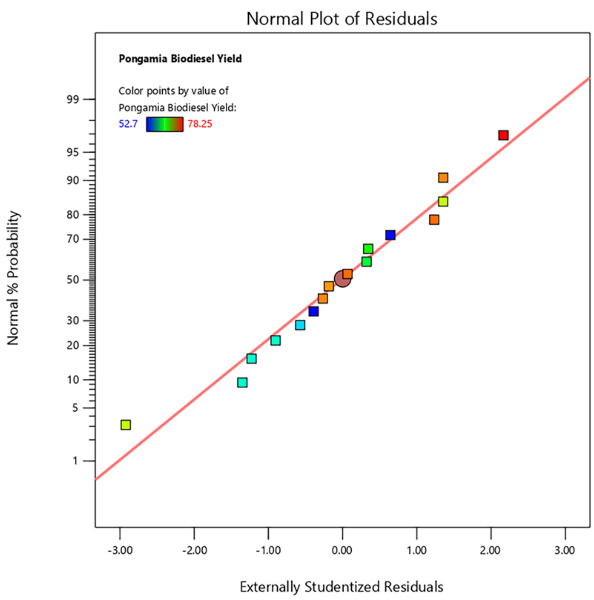

Fig. 2 Normal plot of residuals

The effect of methanol, KOH concentration and reaction temperature is shown in Fig. 3. The perturbation plot assists to find the impact of each process parameters while fixing all other parameters at constant. The behavior of curvature presents the sensitivity of the variables. The curve with steeper slope shows great impact on yield of biodesiel from pongamia pinnata as compared to the curve with flatter slope. Thus, A (volume of methanol) and B (amount of KOH) shows a good correlation or high effect on the yield of biodiesel from pongamia pinnata.

The experimental design of *Pongamia pinata* showed that a maximum amount of 78.25% biodiesel yield is obtained with the study of 20 mL of methanol with 0.8 g KOH at 55 °C.

To build a statistically successful regression model, the *p*-values were evaluated to check the significance of regression coefficients. The coefficient was estimated to be insignificant if their values exceed the limit of 0.05, and were removed from the regression model. The analysis in Table 3 shows that the model is based on linear terms such as A, B, C; quadratic terms, A^2, B^2, C^2 and the interaction terms of AB, AC and BC.

The ANOVA indicated that the polynomial model was significant as *p*-value is < 0.0334 for the model. This *p*-value indicate that the system is adequate to represent the actual relationship between transesterification and model significant variables. The adequacy and significance were further elaborated from the established model showing high values of R^2 (Coefficient of determination) 0.8787 and adjusted R^2 0.6968. The adequate relationship between predicted values and experimental data values of independent variables further showed acceptability of the model.

Table 3 Pongamia Biodiesel Yield ANOVA for response surface quadratic model of

Source model	Sum of squares	D_f (degree of freedom)	Mean square	*F* value	*P*-value Prob>*F*
Model	962.37	9	106.93	4.83	0.0334
A-volume of methanol	264.18	1	264.18	11.93	0.0136
B-amount of KOH	236.76	1	236.76	10.70	0.0170
C-reaction temperature	19.03	1	19.03	0.86	0.3896
AB	6.92	1	6.92	0.31	0.5963
AC	0.51	1	0.51	0.023	0.8844
BC	20.32	1	20.32	0.92	0.3750
A^2	73.72	1	73.72	3.33	0.1178
B^2	45.46	1	45.46	2.05	0.2018
C^2	41.51	1	41.51	1.87	0.2199
Residual	132.82	6	22.14		
Cor Total	1095.19	15			

R^2 = 0.8787, adjusted R^2 = 0.6968, Prob (probability)

The interactive effects were studied for the process variables of biodiesel yield by plotting 3D surface curve model against two independent variables. The other variables are kept at central level (0) during the study. Figs. (4a–4f) shows the 3D response curves of pongamia biodiesel yield featuring the interaction between the variables. To find the optimum level of each variable, the response surfaces were developed to understand the interaction of different variables. The curves with elliptical shapes indicate a good interaction of variables. The extracted curves from the current study showed the significant interaction between all variables. Optimum conditions were also obtained from the surfaces of response plot. The central point or stationary point shows a zero slope of contour in all directions. The optimum value of respective variables was achieved from the coordinates of stationary points that shows the highest contour levels in each plot. The optimum value of the variables such as temperature, volume of methanol and catalyst were 55, 20 mL and 0.8 g respectively. The response from these values were 78.25%.

From the Fig. 4 (contour plot), it is evident that the *Pongamia pinnata* biodiesel yield increases with respect to increasing the catalyst concentration. This could be because adding too much catalyst causes the triglycerides

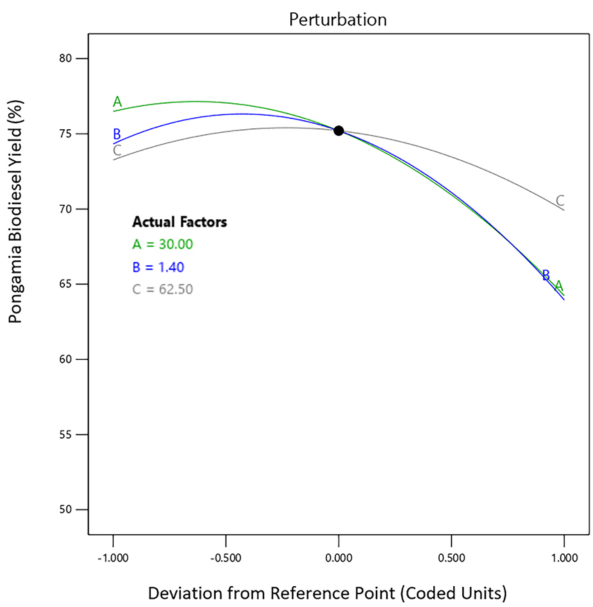

Fig. 3 Perturbation curve for pongamia biodiesel yield

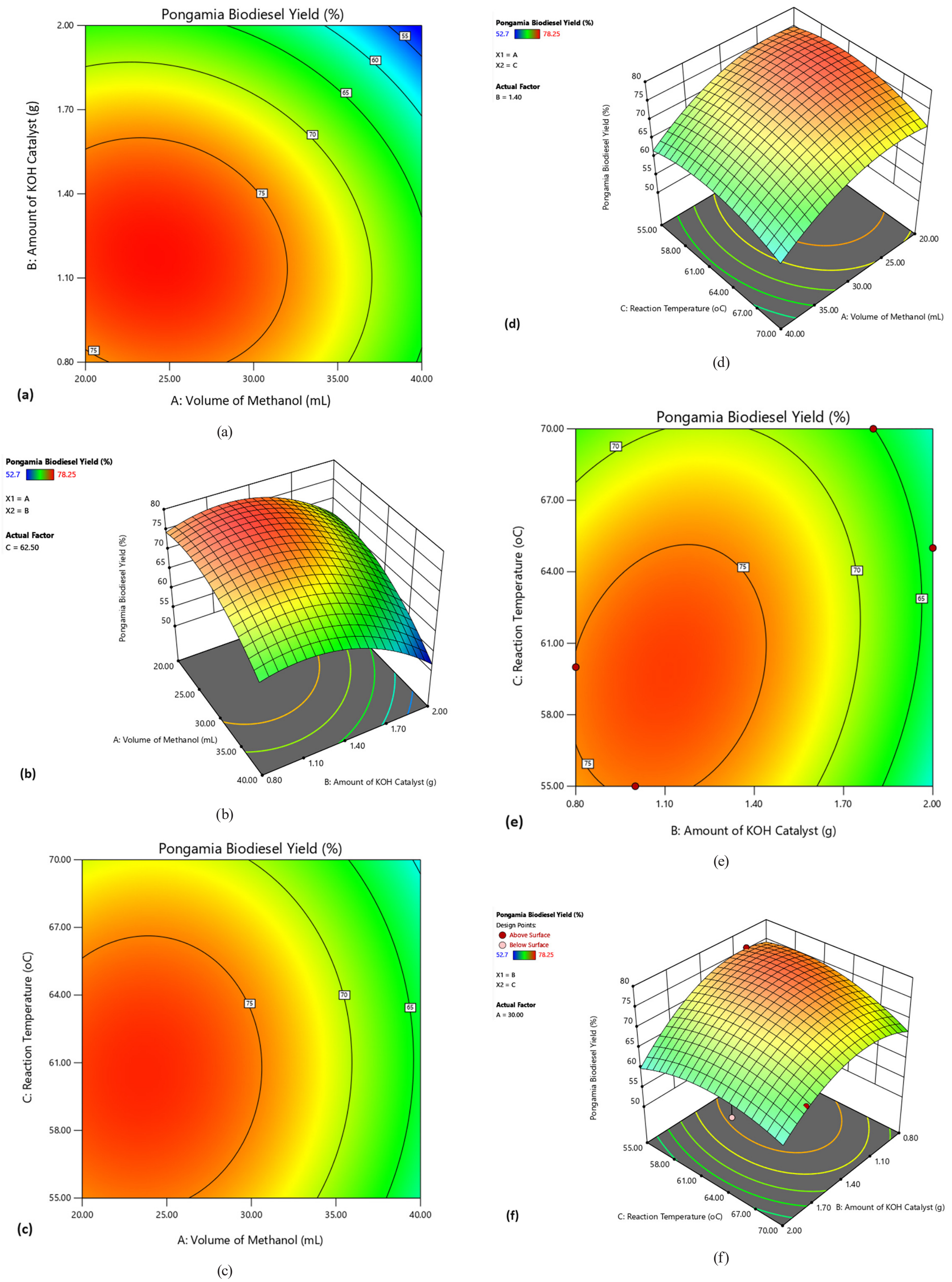

Fig. 4 Contour and 3 D surfaces (a-f)

to react more quickly, increasing the production of biodiesel. Pongamia biodiesel yield first increases with temperature and then shows decreasing trend with increasing temperature. Moreover, the production of pongamia biodiesel decreases as the methanol to oil ratio is increased; as the transesterification step is reversible in nature, therefore, additional methanol for the conversion of triglycerides into biodiesel having reduced impact on the yield. Thus, when the quantity of methanol is increased does not influence higher biodiesel yield.

3.2 Biodiesel yield and its characterization

The pongamia biodiesel produced were having highest experimental yield of 78.25% by weight as compared to simulation yield 84.02%, with a difference of 5.77% by weight. Biodiesel are characterized by their viscosity, density, cetane number, Cloud point, pour point, distillation range, Flash point, and higher heating value (HHV). In the current research study, the pongamia biodiesel physical and chemical properties were found in accordance with International Biodiesel ASTM D 6751 (see Table 4).

3.3 Fatty acid analysis of pongamia biodiesel produced

The GC chromatogram (Fig. 5) showed the major fatty acids presence of myristic acid (2.035% by weight), palmitic acid (55.82%), palmitoleic acid (1.327%), stearic acid (9.993%), oleic acid (14.420%), linoleic acid (1.420%), linolenic acid (0.889%) and eicosanoic Acid (0.986%). Previous research analysis by Anwar et al [24] showed the major categories of determined fatty acids in pongamia oil were comprising of palmitic acid (C16:0), stearic acid (C18:0), behenic acid (C22:0) and lignoceric acid (C24:0), oleic acid (C18:1) and linoleic acid (C18:2), linolenic acid (C18:3) and eicosenoic acid (C20:1).

Table 4 Physicochemical properties of pongamia biodiesel

Parameters	Pongamia biodiesel	Biodiesel Standard ASTM 6751
Density (g/mL) @ 15 °C	0.962	0.870–0.890
Specific gravity	0.967	-
Kinematic viscosity @ 40 °C (mm^2/sec)	5.47	1.9–6.0
Flash point (°C)	182	130 min
Cloud point (°C)	19	-
Pour point (°C)	12	-
Cold filter plugging point (°C)	15	-
Calorific value (MJ/kg)	42.50	35

4 Implications of this study

The study aims to show two great impacts that the society can get with generation of biodiesel from *pongamia pinnata* oil. Firstly, the dependence on traditional fossil fuel can potentially be reduced by the generation of a sustainable biodiesel. Secondly, It is more environment friendly fuel as it produces a fewer gas (greenhouse gas) emission and its optimization can help to create production of biodiesel from *pongamia pinnata* oil which has a good possible market solution and serves as an alternative sustainable renewable source of energy.

The *Pongamia pinnata* can be used in many ways efficiently. Its exploration shows great solution for the utilization of non-edible feedstock in form of biodiesel. Hence can potentially reducing the competition with agricultural crops. Economic feasibility of biodiesel from *Pongamia pinnata* can be increased by implying the methods for optimization of production processes, making it competitive and contributes greatly to energy security. The current study aims to highlight technical advancements from extraction till conversion, in the production processes of biodiesel. This advancement could be worthy for the future industry and research applications.

In addition to this, the findings from the current study also suggests to support and promote the production and the utilization of biodiesel from *pongamia pinnata*, providing insights to policy makers for the implications of biodiesel related policies. For the future perspective, the studies which involves optimization can greatly contribute to the friendly and sustainable environment. The green chemistry principle focuses on the environmental impact and draw attention to improve the efficiency in chemical processes.

5 Conclusions

In the current study, an optimized transesterification process was used to convert *Pongamia pinnata* seed oil into pongamia oil methyl esters and biodiesel following an acid pre-treatment. The results of several factors on the biodiesel yield were evaluated. Optimized combination of acid pre-treatment (H_2SO_4) and methanol containing transesterification an ideal pongamia biodiesel yield 78.25% as compared to simulation yield of 84.02% by weight. GC-MS fatty acid analysis showed presence of fatty acids favourable for biodiesel production. The physical and chemical properties of pongamia biodiesel similar to ASTM ASTM D 6751 guidelines and endorsed this biofuel's practicality. Overall, this study's findings show that pongamia biodiesel are explorable as a substitute to petroleum diesel.

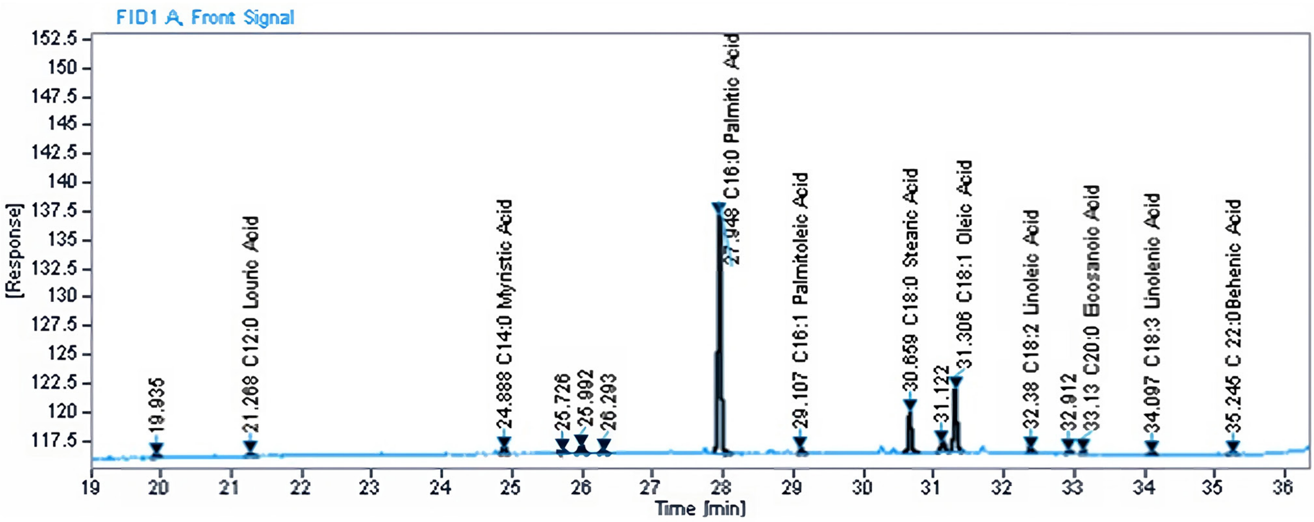

Fig. 5 Gas chromatograph m of fatty acid methyl ester of pongamia oil

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