Journal of Engineering Science and Technology
Vol. 13, No. 9 (2018) 2670 - 2684

PROCESS OPTIMISATION OF PILOT SCALE BIODIESEL PRODUCTION FROM PONGAMIA AND WASTE COOKING OIL FEEDSTOCK

SANTHOSH POOJARY, C. VAMAN RAO*,
SANDESH K., VINAYAKA B. SHET

Department of Biotechnology Engineering, NMAM Institute of Technology,
(VTU Belagavi), Nitte, 574 110, Karnataka, India
*Corresponding Author: vamanrao@nitte.edu.in

Abstract

Biodiesel is the most promising renewable alternative fuel for fossil diesel fuel, which can be produced even in rural settings of an economically backward sector of developing countries. Biodiesel can be produced from non-edible seed oils or used edible oil sources by a transesterification reaction. Biodiesel feedstock differ according to geographic location, therefore, it is essential to optimise the biodiesel production process at least in pilot scale for each source to obtain highest biodiesel yield to be used for domestic purpose. In the present study, biodiesel was produced from non-edible Pongamia seed oil and Waste Cooking Oil separately using the 50 L capacity pilot scale reactor. The crucial reaction parameters such as catalyst (NaOH) concentration, alcohol (Methanol) volume and reaction time were optimised through Box-Behnken Design (BBD) approach to maximise the biodiesel yield. In the optimised pilot study, the biodiesel yield of 89.1% from Pongamia oil and 96% from Waste Cooking Oil sources were obtained. Also, the biodiesels produced by pilot scale met the requisite quality standards of ASTM, EU and India.

Keywords: Biodiesel, Pilot-scale, Pongamia seed oil, Process optimisation, Waste cooking oil.

1. Introduction

In recent years, biodiesel is gaining attention as a promising substitute for petrodiesel in the global fossil fuel supply chain. Even though any triglyceride could be a source for the production of biodiesel through simple transesterification process and utilisation of edible oil feedstock leads to food versus fuel issues [1, 2].

As such there is ever increasing demand for edible oil due to mounting human population and using edible oils for biodiesel production will be an unrealistic approach. In recent decades, many non-edible oilseed feedstocks have been investigated for biodiesel production [3-5]. There are many well-known non-edible seed crops in India, which have been tried for biodiesel production, such as, *Jatropha curcas*, *Pongamia pinnata*, *Calophyllum inophyllum*, *Azadirachta indica*,*Ricinus communis*, etc. From these feedstocks, Jatropha and Pongamia oil are the most extensively studied for biodiesel production [6-9]. The techno-economical feasibility of these sources is under continuous evaluation from lab scale to pilot scale.

The *Pongamia pinnata* trees are found in tropical Asia, Indian Ocean Islands, Australia and predominantly distributed in most of the Indian states where annual rainfall is scanty [10]. It can grow in drought condition and is also capable of fixing atmospheric nitrogen. After 4-6 years of maturity Pongamia trees can yield 900-9000 kg seeds/hectare and at present, due to the ready availability of seeds, it is recommended as the best source for biodiesel production [11]. The Pongamia biodiesel produced through conventional transesterification method from a glass reactor (100 ml) to bench scale set-up (6 L) was found to have excellent fuel properties [12, 13]. The compression ignition engine performance of Pongamia biodiesel blends up to 100% was also reported to be satisfactory in all aspects [14]. Hence indigenous, readily available underutilised, biofuel feedstock could be exploited for sustainable fuel production along with the routine cultivation of energy crops in wastelands [15, 16]. Millions of gallons of Waste Cooking Oil per day is generated all over the world and in many underdeveloped countries, it is being reused for cooking purposes by roadside eateries, which will attract penalties because it is hazardous to health [17]. Used, waste edible oils can be utilised for biodiesel production since the human consumption of such lipids creates physical illness [18]. It is also cost-effective to use Waste Cooking Oil for biodiesel production and it performs satisfactorily in commercial diesel engines [19].

Statistical approach by response surface optimisation, Box-Behnken Design (BBD) was found to be better than central composite designs (CCD) and much superior to 3 level full factorial design [20]. It is also known to be an indispensable tool for predicting near optimum values of experimental parameters along with a limited number of experiments[21]. The best performance for Jatropha biodiesel yield of 73.7% was achieved through BBD method [22]. Also, BBD method was employed for the optimisation of Canola oil-based biodiesel production and the optimal combination of process variables produced greater than 98% biodiesel yield [23]. In another study, BBD design was applied for optimisation of biodiesel production from the waste groundnut, soybean and waste palm kernel oil sources, which yielded 97-98% biodiesel [24].

The demonstration of entire biofuel value chain and successive implementation at the community level with strong government policy can definitely achieve bio-energy projects success as reported in a few case studies carried out in South Pacific [25]. Creating a synergy in renewable energy investments in remote areas is also

necessary for sustainable development [26]. In the Karnataka state of India, A decade ago, The Karnataka State Bio-energy Development Board (KSBDB) started one such biofuel programme to produce biodiesel in order to help rural communities by using locally available non-edible oilseeds. Bioenergy Research, Information and Demonstration Centres (BRIDC) in every district are promoting the biodiesel production activity in the entire state. Each centre is has a 50L capacity biodiesel production reactor plant with other accessories and a laboratory setup for lab scale biodiesel production along with fuel property testing equipment.

In the present study, *Pongamia pinnata* Seed Oil (PSO) and Waste Cooking Oil (WCO) were used for pilot scale biodiesel production and critical reaction parameters such as catalyst concentration, alcohol concentration and reaction time were optimised through Box-Behnken Design (BBD) approach.

2. Materials and Methods

The mature Pongamia seeds were procured from the Northern region of Karnataka state. The Waste Cooking Oil was procured from local hotels, restaurants and food industries. For pilot-scale biodiesel production, industrial grade alcohol (methanol) and catalysts (sodium hydroxide, sulphuric acid) were used. All the chemicals used for testing the properties of biodiesel were of analytical grade and procured from E. Merck. The process adopted in this study is represented in Fig. 1.

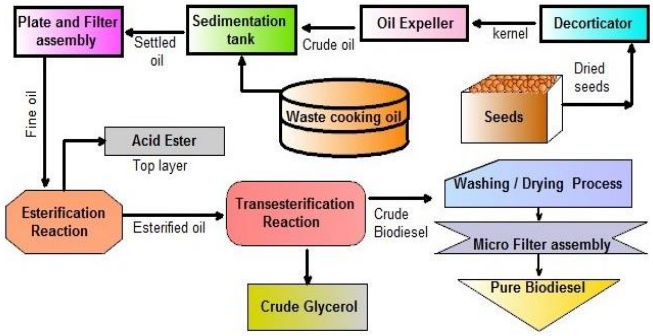


Fig. 1. Flow diagram of process adopted for biodiesel production.

2.1. Processing of raw material

The dried Pongamia seeds with the outer coat are decorticated to obtain inner kernel. Then the kernels were crushed in an electrically driven mechanical oil expeller, which is able to crush 25-30 kg seeds/hour. The expelled oil contains suspended solid particles, which is allowed to settle in a sedimentation tank for 2-3 days for sedimentation of particles. Waste Cooking Oil was also settled for a few days and both these oils were filtered in Plate and Frame filter assembly. The fine filtered oil is directly utilised in the pilot reactor. Free Fatty Acid (FFA) content of the oil is estimated and if it is more than 4%, the esterification using methanol in the presence of an acid catalyst (sulphuric acid) was carried out as pre-treatment step as recommended. The optimisation study was carried only for the oil containing FFA levels of 1-2% and 0.5-1% in Pongamia and Waste Cooking Oils respectively.

2.2. Biodiesel production

Forpilot-scale optimisation study, the batch reactor of 50L biodiesel production capacity (Malnad Oil Extraction Industries-Shimoga, India) was used. The esterification and transesterification reactions were carried out in these reactors and an open vessel with heater coil was used for washing and drying of biodiesel.In the present reactor setup since there is no provision for change of mixing rate and temperature of the reaction mixture, these parameters were kept constant throughout the experiments. The schematic layout of the production setup is shown in Fig. 2.

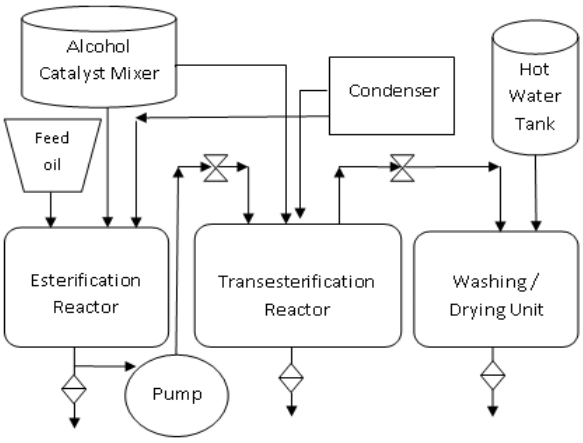


Fig. 2. Schematic outline of pilot-scale biodiesel reactor setup.

The percentage conversion of oil to biodiesel in the process was calculated using the Eq. (1).

$$\text{Biodiesel yield } Y\ (\%) = \frac{\text{Volume of the biodiesel produced (L)}}{\text{Volume of the oil used (L)}} \times 100 \qquad (1)$$

2.3. Biodiesel characterisation

BothPongamia Seed Oil and Waste Cooking Oil biodiesels were characterised for major fuel properties such as density, kinematic viscosity, flash point, acid value, copper strip corrosion test, etc. The tested fuel properties were compared with Indian IS-15607, European EN-14214 and American ASTM-D6751 biodiesel standards.

2.4. Design of experiments

The reaction conditions for biodiesel production were optimised by Box-Behnken response surface experimental design (BBD) with three factors, namely, catalyst concentration (*A*), alcohol concentration (*B*) and reaction time (*C*). Based on the preliminary experiments conducted adopting One Variable at a Time (OVAT) method, the levels and centre points were fixed for each variable [27]. The process parameters and their levels are listed in Table 1.

Table 1. Process variables and their coded levels.

Process variable	Coded levels					
	Pongamia oil			Waste Cooking Oil		
	-1	**0**	**+1**	**-1**	**0**	**+1**
Catalyst concentration *A* (wt/v %)	0.4	0.6	0.8	0.4	0.5	0.6
Alcohol concentration *B*(v/v %)	25	30	35	15	20	25
Reaction time, *C* (min)	75	90	105	60	75	90

In the study, individual and interactive effects of these process variables on the response (biodiesel yield-*Y*) were analysed. The experiments were conducted in a randomised order to prevent biasing and regression analysis of the experimental data was done to develop a mathematical model with second order polynomial expression as seen in Eq. (2).

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X^2{}_i + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{ij} X_i \, X_j \quad (2)$$

Where Y is the response, β_0, β_i, β_{ii} and β_{ij} are the intercept, linear, quadratic and interaction constant coefficients respectively; and X_i, X_j are the independent variables [22]. This equation predicts the relationship between response and process variables.

The Design-Expert® 9.0.0 (State-Ease Inc., USA) statistical software was used for optimisation study. The significance of the model developed and linear as well as the quadratic interaction was analysed by performing The Analysis of Variance (ANOVA). The validation of the model was performed under an optimal condition in triplicate and the average values of the experiments were compared with the predicted values of the developed model equations.

3. Results and Discussion

3.1. Statistical analysis

The effect of three process variables, namely, catalyst concentration, alcohol concentration and reaction time on the pilot scale production of PSO and WCO biodiesel were statistically evaluated using BBD. For both feedstock source, 15 designed experiments were conducted with three centre points and data was analysed by multiple regression method. The experimental data was evaluated against the four models namely linear, interactive (2FI), quadratic and cubic, to select the better fitting model. The aliased cubic model along with linear and interactive models of high R-squared and p-values was discarded [28]. The quadratic model was found to be the best model for the current study.

3.2. PSO biodiesel production optimisation

The PSO utilised for the biodiesel production had the FFA level of approximately 8% and hence it was pre-treated with 1% (v/v) sulphuric acid in the presence of 15% (v/v) methanol to reduce its FFA, nearly to 1-2% in the esterification reactor. The reaction was carried out at a reaction temperature of 60-65 ^{0}C for 90-120min. After the reaction, the two phases separated, where, an upper layer consisting of acid esters and lower phase of esterified oil. The esterified oil layer was used for optimisation studies. The transesterification reaction was performed at 63±2 ^{0}C in the presence of NaOH catalyst,

and mixing rate of 2800 rpm was maintained.After transesterification, the upper layer with crude methyl esters (biodiesel) and a lower layer of crude glycerol formed were separated. The crude biodiesel was purified by warm water wash until all the impurities were removed and the washed water was clear. Washed biodiesel was dried at 110 ^{0}C to remove moisture content and final biodiesel yield was calculated. The design matrix of process parameters and response values in terms of the percentage of biodiesel yield for PSO biodiesel is given in Table 2.

Table 2. Experimental design matrix for PSO biodiesel production.

Catalyst concentration *A*(wt/v %)	Alcohol concentration *B*(v/v %)	Reaction time *C* (min)	Biodiesel yield *Y*(%)
0.4	30	75	88.1
0.6	35	75	89.6
0.6	25	75	88.2
0.6	30	90	91.8
0.8	30	105	87.3
0.6	30	90	92.4
0.8	35	90	88.8
0.4	25	90	87.8
0.8	30	75	87.8
0.4	35	90	89.3
0.6	25	105	89.1
0.8	25	90	86.5
0.6	30	90	92.1
0.4	30	105	89.7
0.6	35	105	91.2

The Analysis of Variance (ANOVA) for regression models of optimisation indicates the fitness of the models in describing the relationship between the biodiesel yield and the process variables [24]. The significance of each term in the ANOVA table was determined by the p-value, which indicates the probability of error at a 95% confidence interval (p=0.05). The results of ANOVA and fitness of the quadratic model developed for PSO biodiesel is summarised in Table 3.

Table 3. ANOVA for PSO biodiesel production optimisation.

Source	Sum of squares	DF	Mean square	*F*-value	*p*-value
Model	46.30	9	5.14	47.86	0.0003
***A*-Catalyst**	2.53	1	2.53	23.55	0.0047
***B*-Alcohol**	6.66	1	6.66	61.97	0.0005
***C*-Reaction time**	1.62	1	1.62	15.07	0.0116
AB	0.16	1	0.16	1.49	0.2769
AC	1.10	1	1.10	10.26	0.0239
BC	0.12	1	0.12	1.14	0.3346
A^2	25.93	1	25.93	241.20	< 0.0001
B^2	6.73	1	6.73	62.60	0.0005
C^2	5.54	1	5.54	51.54	0.0008
Residual	0.54	5	0.11	-	-
Lack of fit	0.36	3	0.12	1.32	*0.*4576

The results from the ANOVA showed that the p-value developed for the model was found to be 0.0003, indicating that the model is very adequate and highly

significant at 95% confidence interval ($p<0.05$) and insignificant lack of fit value ($p>0.05$) approves the model for validation [29]. The high F-value (47.86) of the model also reveals the model is significant. It can be confirmed that the proposed regression model for biodiesel yield is acceptable with high R-Squared (0.9885) and Adj. R-Squared (0.9679) values, which exhibited a close conformity between the experimental and the predicted values by polynomial model [28]. The p-values of A, B, C, AC, A^2, B^2, C^2 are lesser than 0.05 indicating that they are the significant model terms [30]. By applying multiple regression analysis on the experimental data, the following second-order polynomial mathematical expression (coded form) arrives in Eq. (3).

$$Y(\%) = 92.10 - 0.56A + 0.91B + 0.45C + 0.20AB - 0.53AC + 0.18BC - 2.65A^2 - 1.35B^2 + 1.23C^2 \quad (3)$$

The graphical plots for process parameter interactions of PSO biodiesel production optimisation are shown in Fig. 3. The curvature in the 3D-plots as seen in Figs. 3(a), (b) and (c), indicates that the process parameters are having significant interactions. The base catalyst concentration is the important process parameter in the transesterification reaction of triglycerides [31, 32]. The effect of catalyst concentration for PSO biodiesel production is described in Figs. 3(a) and (b). As the catalyst concentration increases the biodiesel yield increases and with a higher amount of catalyst, biodiesel yield decreases. This can be attributed to the fact that, PSO consists of elevated FFA and hence soap formation occurs during the reaction leading to less product recovery [31].

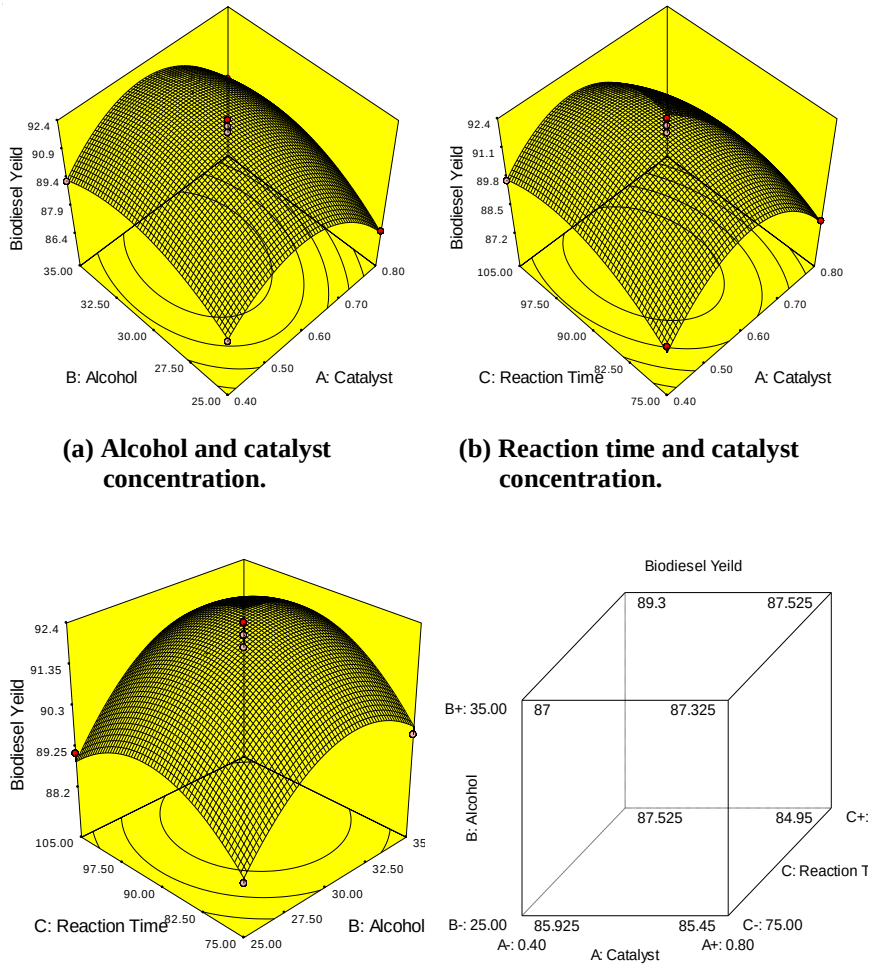
In the biodiesel production process, the molar ratio of alcohol to oil is also a critical reaction parameter. As seen in Figs. 3(a) and (c), increase in alcohol concentration increases the biodiesel yield. But the higher concentration of alcohol leads to decreases in yield because the glycerol solubilises in excess alcohol and reverses the equilibrium of the reaction [32]. The reaction time of transesterification decides the extent of biodiesel conversion. As observed in Figs. 3(b) and (c), PSO biodiesel yield increases as the time increases. Interaction of catalyst-alcohol (AB) at a lower concentration of both components, the biodiesel yield is minimal and at optimum concentrations yield higher. In the plots, the interaction of alcohol with reaction time (BC) was found to be stronger with steep curvature. It signifies that longer reaction time with more amount of alcohol will decrease the biodiesel yield. With regards to the interaction of catalyst with reaction time (AC), the effect of the catalyst is greater than reaction time on biodiesel yield. The cube diagram (Fig. 3 (d)) represents the variation of biodiesel yield by changing the process parameter from lower to higher levels.

3.3. WCO biodiesel production optimisation

Waste Cooking Oil (WCO) procured from hotel, restaurants and food industries contains suspended food particles and thus need to be finely filtered before subjecting it for transesterification reaction. WCO was subjected for sedimentation to settle larger solids and the remaining fine solids were removed using plate and frame filter assembly. As a precautionary measure, the oil was heated above 1000 C to remove moisture. Most of the WCO collected was found to have FFA levels below 2% and for the present optimisation study, the oil with FFA 0.5-1% was utilised. Because of low levels of FFA content, the direct single step

transesterification was adopted for biodiesel production in the pilot reactor. The reaction temperature was kept at 63 ± 2^{0}C during transesterification reaction and mixing rate of 2800 rpm were maintained. The design matrix of process parameters and response values in terms of the percentage of biodiesel yield for WCO biodiesel is given in Table 4.

The WCO biodiesel yield is considerably better than non-edible PSO biodiesel because of a better quality of the WCO. Since WCO is already refined in many respects there were fewer amounts of FFA and gummy substances, hence the biodiesel quality was better than PSO biodiesel. Since the Pongamia seed oil was not refined to that extent, hence the feedstock quality is slightly inferior to WCO.



(a) Alcohol and catalyst concentration.

(b) Reaction time and catalyst concentration.

(c) Reaction time and alcohol concentration.

(d) Cubical representation of all three parameters interactions.

Fig. 3. Graphical plots for process parameter interactions of PSO biodiesel production optimisation.

Table 4. Experimental design matrix for WCO biodiesel production.

Catalyst concentration *A* (wt/v%)	Alcohol concentration *B*(v/v%)	Reaction time *C*(min)	Biodiesel yield *Y*(%)
0.6	25	75	91.4
0.5	25	60	93.1
0.4	20	60	94.6
0.5	20	75	97.6
0.6	20	90	94.7
0.4	15	75	90.9
0.5	20	75	97.8
0.4	20	90	96.4
0.4	25	75	94.8
0.6	15	75	90.2
0.5	20	75	97.6
0.6	20	60	93.1
0.5	15	60	90.6
0.5	15	90	93.7
0.5	25	90	95.9

The results of Analysis of Variance (ANOVA) of experimental study and fitness of the model of WCO biodiesel production process is summarised in Table 5. The p-value developed for the model was 0.0026, which indicates that the model is satisfactory and significant at a 95% confidence interval (p<0.05) [29]. As seen in optimisation PSO biodiesel, the high F-value and insignificant lack of fit value for optimisation of WCO biodiesel also confirmed that the model is realistic and further it can be validated by experiments. The p-values of *A*, *B*, *C*, A^2, B^2terms in the ANOVA table are <0.05, indicating that they are the significant model terms [28]. The proposed regression model for biodiesel yield is acceptable with high R-Squared (0.9704) and Adj. R-Squared (0.9170) values, which exhibited a close match between the experimental and the predicted values by the polynomial model [26].

Table 5. ANOVA for WCO biodiesel production optimisation.

Source	Sum of squares	DF	Mean square	*F*-value	*p*-value
Model	83.64	9	9.29	18.19	0.0026
***A*-Catalyst**	7.80	1	7.80	15.27	0.0113
***B*-Alcohol**	6.48	1	6.48	12.69	0.0162
***C*-Reaction time**	10.81	1	10.81	21.16	0.0058
AB	2.72	1	2.72	5.33	0.0690
AC	0.01	1	0.01	0.02	0.8942
BC	0.02	1	0.02	0.04	0.8421
A^2	15.39	1	15.39	30.13	0.0027
B^2	43.10	1	43.10	84.38	0.0003
C^2	2.94	1	2.94	5.75	0.0618
Residual	2.55	5	0.51	-	-
Lack of fit	1.67	3	0.56	1.25	0.4725

By applying multiple regression analysis on the experimental data, the following second-order polynomial mathematical expression (coded form) is obtained by the software and it is represented in the Eq. (4).

$$Y(\%) = 97.63 - 0.99A + 0.9B + 1.16C - 0.82AB - 0.05AC - 0.075BC - 2.04A^2 - 3.42B^2 - 0.89C^2 \quad (4)$$

The graphical plots for process parameter interactions of WCO biodiesel production optimisation were shown in Fig. 4.

Similar to optimized PSO biodiesel model, the curvature in the 3D-response surface plots of WCO biodiesel production model indicates that the process parameters have explicated interactions. The effect of catalyst concentration on biodiesel yield depicted in Figs. 4(a) and (b). Increasing catalyst concentration increases the biodiesel yield, but does not decrease the yield substantially as seen in PSO biodiesel optimisation model. This may be because of the lower value of FFA in WCO and less soap formation. In the in Figs. 4(a) and (c) the positive effect of increasing alcohol to the biodiesel yield was realised. Usually 6:1, alcohol to oil molar ratio is suggested for better biodiesel yield, which is approximately 30% alcohol on the dry volume of oil. Adding more amount of alcohol will lead to the reversible reaction as well as the settling of glycerol [32].

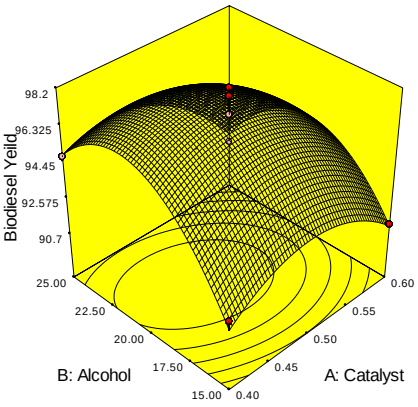
The effect of increasing reaction time on the increment of biodiesel yield depicted in Figs. 4(b) and (c). If the reaction is carried for a long duration with higher alcohol concentration, there is a negative effect on biodiesel yield because of a reversible reaction. As the interaction effect is concerned, almost similar to PSO biodiesel same trend was observed for WCO biodiesel but the catalyst-alcohol (*AB*) interaction is more significant than the interaction of catalyst-reaction time (*AC*) or alcohol-reaction time (*BC*) for WCO biodiesel production. The cube diagram Fig. 4 (d) interprets the difference in biodiesel yield at the extreme values of process variables.

From the optimisation study of both PSO and WCO biodiesel production process, it can be summarised that identifying the effect of important process parameters are very much essential to achieve maximum yield for the pilot scale production setup. In the current study, the target for the optimisation was kept to minimise catalyst and alcohol consumption, thereby reducing production cost. Reaction time was maintained in the range because minimising the reaction time may lead to an incomplete reaction, which would affect the quality of biodiesel.

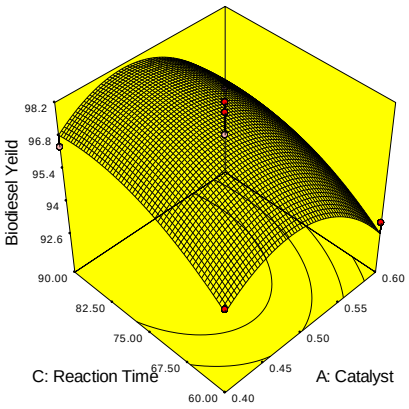
3.4. Model validation experiments

The validation of the quadratic model response prediction was cross confirmed by conducting triplicate experiments in the pilot reactor and response under the optimum condition is tabulated in Table 6. The biodiesel yield obtained from PSO and WCO feedstocks were almost close to the equation predicted values depicting the suitability of the model for the practical application.

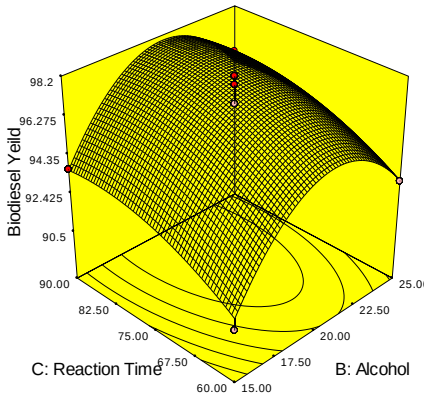
Earlier production methods, without process optimisation and maintaining general conditions of transesterification reaction achieved approximately 80% and 90% biodiesel yields for PSO and WCO biodiesel respectively.



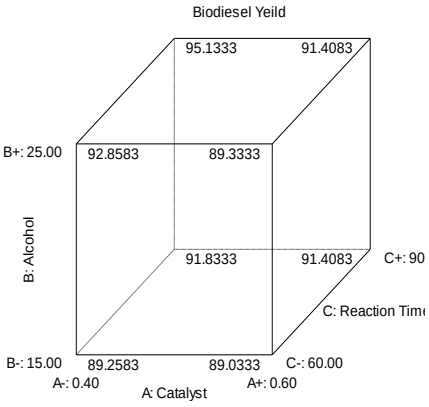
(a) Alcohol and catalyst concentration.



(b) Reaction time and catalyst concentration.



(c) Reaction time and alcohol concentration.



(d) Cubical representation of all three parameters interactions.

Fig. 4. Graphical plots for process parameter interactions of WCO biodiesel production optimisation.

Table 6. Predicted and experimental response under optimum conditions.

	Optimum values			Biodiesel yield(%)	
	Catalyst (wt/v %)	Alcohol (v/v %)	Reaction time (min)	Predicted	Experimental
PSO biodiesel	0.48	26.93	94.08	90.52	89.1± 0.8
WCO biodiesel	0.41	18.16	85.39	96.22	96± 0.1

3.5. Biodiesel fuel properties

Biodiesel fuel properties such as density, kinematic viscosity, flash point, acid value and the copper corrosion test are the most important physicochemical properties to be tested before the biodiesel is used in the vehicle. All the properties were tested with apparatus available at Bioenergy, Research Information and Demonstration Centre (BRIDC). The biodiesel produced at pilot reactor has minor variation in properties because the quality of feedstock oil for the study was kept almost invariable for all batches. The range of property values is given in Table 7, which are arrived at based on the upper and lower values obtained during testing.

Table 7. Physico-chemical properties of PSO and WCO biodiesel.

Fuel properties	Biodiesel standards			Present study values	
	IS 15607	EN 14214	ASTM D6751	PSO Biodiesel	WCO Biodiesel
Density at 15 ^{0}C (kg/m^3)	860-900	860-900	860-900	886-894	872-889
Viscosity at 40 ^{0}C (mm^2/s)	2.5-6.0	3.5-5.0	1.9-6.0	4.8-5.9	4.6-5.8
Flash point (^{0}C)	Min 120	Min 101	Min 93	165-189	154-166
Acid value (mg KOH/g)	Max 0.5	Max 0.5	Max 0.5	0.42-0.46	0.29-0.41
Copper corrosion test (rating)	Max No.1	Max No.1	Max No. 3	No.1	No.1

On an average, the density, kinematic viscosity, flash point and acid value were slightly higher for PSO biodiesel compared to WCO biodiesel. There was no major difference observed in the quality aspect of both the biodiesels even though the resources were different.

The fuel properties were almost similar for laboratory and pilot scale studies. Quantitatively in terms of biodiesel yield, the pilot scale reactor produced 89-96% biodiesel; it was slightly inferior to lab scale biodiesel yield of 92-98%, due to the scale-up limitation of reaction conditions.

4. Conclusions

Optimisation of pilot-scale biodiesel production process for Pongamia Seed Oil (PSO) and Waste Cooking Oil (WCO) through Box-Behnken Design (BBD) approach was successfully achieved with the empirical quadratic model. The interaction effects of three very important reaction parameters, specifically, catalyst concentration, alcohol concentration and reaction time were studied from the fitted model. The predicted optimum process parameter values were practically validated and reproduced, resulting in highest biodiesel yield of 89.1% and 96% for Pongamia and Waste Cooking Oil respectively. The major fuel property values tested from both biodiesel were found to be within the Indian, American and European biodiesel standard limits. It can be concluded that, by optimising the process parameters of the biodiesel production process, renewable fuel can be generated and utilised for domestic purpose with less expenditure on production cost.

Acknowledgement

The authors are thankful to the Karnataka State Bio-energy Development Board (KSBDB) for funding the present research work and also to Dr. Ujwal P., Co-ordinator and Mr. Manjunath, Mr. Vinay- staff of Udupi District Bioenergy Research, Information and Demonstration Centre (BRIDC) for their kind support and Department of Biotechnology Engineering, NMAM Institute of Technology, Nitte for providing necessary facilities.

Nomenclatures

A	Catalyst concentration, wt/v%
B	Alcohol concentration, v/v%
C	Reaction time, min
Y	Biodiesel yield, %

Abbreviations

ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
BBD	Box-Behnken Design
DF	Degree of Freedom
FFA	Free Fatty Acid
PSO	*Pongamia pinnata* Seed Oil
WCO	Waste Cooking Oil

References

1. Thompson, P.B. (2012).The agricultural ethics of biofuels: The food vs. fuel debate. *Agriculture*, 2(4), 339-358.
2. Tenenbaum, D.J. (2008). Food vs. fuel: Diversion of crops could cause more hunger. *Environmental Health Perspectives*, 116(6), A254-257.
3. Atabani, A.E.; Silitonga, A.S.; Ong, H.C.; Mahlia, T.M.I.; Masjuki, H.H.; Badruddin, I.A.; and Fayaz, H. (2013). Non-edible vegetable oils: A critical evaluation of oil extraction, fatty acid compositions, biodiesel production, characteristics, engine performance and emissions production.*Renewable andSustainable Energy Reviews*, 18, 211-245.
4. Silitonga, A.S.; Masjuki, H.H.; Mahlia, T.M.I.; Ong, H.C.; Chong, W.T.; and Boosroh, M.H. (2013). Overview properties of biodiesel diesel blends from edible and non-edible feedstock. *Renewable and Sustainable Energy Reviews*, 22, 346-360.
5. Murugesan, A.; Umarani, C.; Chinnusamy, T.R.; Krishnan, M.; Subramanian, R.; and Neduzchezhain, N. (2009). Production and analysis of bio-diesel fromnon-edible oils-A review. *Renewable and Sustainable Energy Reviews*, 13(4), 825-834.
6. Tiwari, A.K.; Kumar, A.; andRaheman, H. (2007). Biodiesel production from jatropha oil (Jatropha curcas) with high free fatty acids: An optimized process. *Biomass and Bioenergy*, 31(8), 569-575.

7. Portugal-Pereira, J.; Nakatani, J.; Kurisu, K.; and Hanaki, K. (2016). Life cycle assessment of conventional and optimised jatropha biodiesel fuels. *Renewable Energy*, 86(C), 585-593.
8. Meher, L.C.; Dharmagadda, V.S.; and Naik, S.N. (2006). Optimization of alkali-catalyzed transesterification of pongamiapinnata oil for production of biodiesel. *Bioresource Technology*, 97(12), 1392-1397.
9. Anjana, P.A.; Niju, S.; Begum, K.M.M.S.; Anantharaman, N.; Anand, R.; and Babu, D. (2016). Studies on biodiesel production from pongamia oil using heterogeneous catalyst and its effect on diesel engine performance and emission characteristics. *Biofuels*, 7(4), 377-387.
10. Arote, S.R.; and Yeole, P.G. (2010). Pongamia pinnata L: A comprehensive review. *International Journal of Pharm Tech Research*, 2(4), 2283-2290.
11. Bobade, S.N.; and Khyade, V.B. (2012). Detail study on the properties of pongamia pinnata (karanja) for the production of biofuel. *Research Journal of Chemical Sciences*, 2(7), 16-20.
12. Vivek; and Gupta, A.K. (2004). Biodiesel production from karanja oil. *Journal of Scientific and Industrial Research*, 63(1), 39-47.
13. Mamilla, V.R.; Mallikarjun, M.V.; and Rao, G.L.N. (2011). Preparation of biodiesel from karanja oil. *International Journal of Energy Engineering*, 1(2), 94-100.
14. Gopal, K.N.; and Karupparaj, R.T. (2015). Effect of pongamia biodiesel on emission and combustion characteristics of DI compression ignition engine. *Ain Shams Engineering Journal*, 6(1), 297-305.
15. Santhosh, P.; Vaman, R.C.; and Venkatesh, K.H. (2014). Biofuel production from borassusflabellifer. *Journal of Biofuels*, 5(1), 9-15.
16. Poojary, S.; Rao, C.V.; and Venkatesh, K.H. (2017). Scleropyrum pentandrum (dennst.) mabb-oil as a feedstock for biodiesel production engine performance and emission studies. *International Journal of Green Energy*, 14(3), 279-288.
17. Patil, P.D.; Gude, V.G.; Reddy, H.K.; Muppaneni, T.; and Deng, S. (2012). Biodiesel production from waste cooking oil using sulfuric acid and microwave irradiation processes. *Journal of Environmental Protection*, 3(1), 107-113.
18. Jaarin, K.; and Kamisah, Y. (2012). *Lipid peroxidation*. Repeatedly heated vegetable oils and lipid peroxidation. Intech Open.
19. Kulkarni, M.G.; and Dalai, A.K. (2006). Waste cooking oil - An economical source for biodiesel: A review. *Industrial & Engineering Chemistry Research*, 45(9), 2901-2913.
20. Ferreira, S.L.; Bruns, R.E.; Ferreira, H.S.; Matos, G.D.; David, J.M.; Brandao, G.C.; Da Silva, E.G.; Portugal, L.A.; dos Reis, P.S.; Souza, A.S.; and Santos, W.N. (2007). Box-behnken design: An alternative for the optimization of analytical methods. *Analytica Chimica Acta*, 597(2), 179-186.
21. Maran, J.P.; Sivakumar, V.; Thirugnanasambandham, K.; and Sridhar, R. (2014). Microwave assisted extraction of pectin from waste citrulluslanatus fruit rinds. *Carbohydrate Polymers*, 101, 786-791.
22. Supardan, M.D.; Fahrizal, R.M.; Moulana, R.; Safrida, D.; Satriana; and Mustapha, W.A.W. (2017). Optimisation of process parameter conditions for

biodiesel production by reactive extraction of jatropha seeds. *Journal of Engineering Science and Technology* (*JESTEC*), 12(3), 847-859.

23. Paintsil, A.; Armah, F.A.; and Yanful, E.K. (2017). Assessment of the transesterification stage of biodiesel production II: Optimisation of process variables using a box-behnken design. *Waste and Biomass Valorization*, 9(8), 1399-1405.
24. Ayoola, A.A.; Hymore, K.F.; and Omonhinmin, C.A. (2016). Optimization of biodiesel production from selected waste oils using response surface methodology. *Biotechnology*, 16(1), 1-9.
25. Woods, J.; Hemstock, S.L.; and Burnyeat, W. (2006). Bio-energy systems at the community level in the South Pacific: Impacts & monitoring. *Mitigation and Adaptation Strategies for Global Change*, 11(2), 461-492.
26. Magnoni, S.; and Bassi, A.M. (2009). Creating synergies from renewable energy investments, a community success story from Lolland, Denmark. *Energies*, 2(4), 1151-1169.
27. Poojary, S.; Rao, C.V.; and Ujwal P. (2016). The study of pilot-scale biodiesel production process from pongamia and waste cooking oil. *Proceedings of the 12th Annual Students Chemical Engineering Congress (SCHEMCON* 2016*).* Hyderabad, India, 1- 6.
28. Maran, J.P.; Priya, B.; and Manikandan, S. (2014). Modeling and optimization of supercritical fluid extraction of anthocyanin and phenolic compounds from syzygiumcumini fruit pulp. *Journal of Food Science and Technology*, 51(9), 1938-1946.
29. Amenaghawon, A.N.; Henrietta, I.; Agbonghae, E.; Ogbeide, S.E.; and Okieimen, C. (2014). Modelling and optimisation of dilute acid hydrolysis of corn stover using box-behnken design. *Journal of Engineering Science and Technology* (*JESTEC*), 9(4), 443-454.
30. Shazana, A.R.; Masturah, M.; Badlishah, S.B.; Rashidi, O.; and Russly, A. (2016). Optimisation of supercritical fluid extraction of astaxanthin from penaeus monodon waste using ethanol-modified carbon dioxide. *Journal of Engineering Science and Technology* (*JESTEC*), 11(5), 722-736.
31. Singh, A.; He, B.; Thompson, J.; and Van Gerpen, J. (2006). Process optimization of biodiesel production using alkaline catalysts. *Applied Engineering in Agriculture*, 22(4), 597-600.
32. Meher, L.C.; Sagar, D.V.; and Naik, S.N. (2006). Technical aspects of biodiesel production by transesterification-A review. *Renewable and Sustainable Energy Reviews*, 10(3), 248-268.