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Optimization of Babassu (Orbignya sp) biodiesel Production from babassu oil by Taguchi Technique and Fuel Characterization

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Abstract

This paper presents transesterification of Babassu oil (BBO) using methanol as alcohol and KOH as a catalyst; it showed a suitable alternative biodiesel with superior yield and lower toxicity. In the present study, signal-to-noise ratios (S/N ratio) with analysis of variance (ANOVA) were employed to optimize the variables and prediction of maximum yield. The various factors influencing the methanolysis of BBO were considered in present work. The temperature of reaction was observed to be most valuable in BBO conversion. In this work it is noted that for low cost purpose, the most efficient conditions for overall process are 50 °C process temperature, 1 wt% of catalyst concentration with 6:1 methanol/oil molar ratio for 1 h. With these optimum combinations up to 99.42% babassu oil methyl esters (BBOME) can be achieved. The major properties such as density, calorific value, viscosity, flash point, pour point and cloud point of the BBOME were observed as 876.2 kg/m^3,40.012 Mj/kg ,4.001 cSt, 126 ^{0}C, 9 ^{0}C and 14 ^{0}C respectively. The BBOME could be used as a highly beneficial substitute for diesel fuel as its properties are within the acceptable limit and comparable to the biodiesel standard.

Keywords- Babassu oil; Transesterification, Taguchi method, Babassu oil methyl ester; Fuel characterization.

1 INTRODUCTION

Esters of BBO can obtained by alkaline catalysis which is green biodieselproduction process, possible with straightforwardreactions and simple equipments. BBO is apparent, faint yellow color oil removed from seeds of the babassu tree, because of higher fatty acid composition; it is categories as non-inedible oil. Arithmetical theories were used by different researchers for optimal transesterification process. Nuclear electromagnetic resonance theory also applied to compute the yield obtained. Transesterification supported with ultrasound indicates better outcomes compared to reaction without ultrasound support. It showed that yields more than 97% can be obtained in 10 min using proper proportion of reaction parameters. [1,2].In view of the millions of acres of forests with a huge quantity of babassu palm plants and the potential of the integral utilization of the coconut, babassu possibly constitutes an appropriate raw material for biodiesel production; however the remaining parts of the plant can be used for different purposes. Additionally, it is more suitable to use non-edible oils like babassu oil, because uses of edible can create food verses price issues.The methyl esters from babassu oil and methanol by adding potassium hydroxide (KOH) as catalyst has been obtained and optimized by using the response surface methodology and factorial design of experiments. The various parameters influencing the alkaline methanolysis of the babassu oil were investigated. Temperature of reaction was found to have the highest significant effect on yield. As per previous study and from an financial point of view, the better conditions for whole reactions are catalyst fraction of 0.95% and an working temperature of 45°C operating with 6:1 methanol/oil molar proportion. With these circumstances the maximum yield obtained was 99.85%. The biodiesel created from babassu oil includes a high level of saturated fatty acids, 91%, majorly composed of lauric acid (51.8%) and myristic fatty acid (22.2%) making it mainly stable regarding oxidation and ensuing in better cold flow properties. The babassu oil biodiesel (BBOME) could be transformed in a mostly promising replacement for conservative fuel probably because of the better low temperature properties, higher oxidative stability shown and fulfilling the Biodiesel Standard EN 14214. These significant properties can make BBOME of a huge interesting fuel babassu kernels (Orbignya phalerata) enclose one or two seeds with a white oily endosperm [3, 4]. The biodiesel creation is promising for diverse and essential parts. The crystallization temperature noted between the fluid and solid portion was decreased by 11 ^{0}C because of the winterization method of BBOME. However, two peaks of the BBOME solid portion were noted in TMDSC results, being the larger one associated with the overlie of transition ascribed to the various saturated esters, it represents dissimilar crystallization temperatures. The association of X-ray diffraction and TMDSC were very effective for observing the BBOME crystallization reaction which permitscategorizing the BBOME according to lower temperature properties [5-9]. An optional fuel to petrodiesel should be scientifically feasible, economical, environmentally clean, and abundantly available. The presentoptional diesel fuel can be termed biodiesel. Biodiesel shows other advantages, like minimization of greenhouse gas emissions and rural developments especially to developing countries. Babassu oil has high potential for biodiesel production due its composition. Even though, in formation of babassu oil is limited, and a more study of its rheological properties is needed because of its predominantly saturated fatty acid composition. Babassu (Orbignya phalerata or Orbignya oleifera) is a palm plant which grows naturally in Colombia and Brazil. The major products are fruits which are small coconuts that hang upin the form ofbunch with about 20 coconut fruits each [10-14]. The cusi oil removed from the seeds of the babassu palms, which are basically consist of saturated fatty acids [15-17]. The kernels are rich in proteins and carbohydrates; it includes 90% to 95% of its total substance. It is generally used as fertilizer and in large quantities as a feed supplement for peccaries. The viscosity of a liquid fuel plays an important role in its pumping and flow inside the engine. So, it is important to test the suitability of converting babassu oil into biodiesel, as earlier studies shown that the physicochemical properties of the oil from babassu seeds are suitable for production of fuel [18-22].

2. EXPERIMENTAL

2.1 Materials

Refined babassu oil was purchased from Oil supplier industry Paras Perfumers, Delhi (India). It was extracted by hydraulic press (cold pressing), and as oil obtained by cold pressing has higher oxidative stability. All chemical reagents such as KOH, Methanol, distilled water etc. required for reaction were obtained from D.Haridas & Company, Pune (India).

2.2 Characterization of Babassu Oil (BBO)

Physicochemical properties of BBO and BBOME were evaluated by ASTM described methods at Chem Tech Laboratories Pvt Ltd, Which is ISO/IEC 17025:2005 certified and NABL Gov. of India accredited laboratory. The Comparison of Physico-chemical Properties of BBO from different researchers and properties of oil in current study are shown in Table-1

Table 1: Properties of BBO by different researchers and present study

Sr. No.	**Test Description**	**Units**	**References**			**Properties of BBO in present study**	
			[1,2,17,20]	**[3]**	**[4]**	**Results**	**Test Method**
1	Density at 15 ^{0}C	Kg/m^{3}	923	-	920	**918.9**	ASTM D 1298 2012 b
2	Kinematic Viscosity at 40^{0}C	cSt	34.40	38.28		**33.46**	ASTM D445 2012
3	Gross Calorific Value	MJ/kg (Cal/g)	-	-	-	**38.12 (9130)**	ASTM D 240 2009
4	Acid Number	mg KOH/g	0.505 meq/g	0.15	0.59 meq/100g	**1.24**	ASTM D 664 2011 a
5	Molecular wt.	g/mol	709.90			-	-
6	Moisture and sediments	(% v/v)	0.039	0.02		**0.022**	ASTM D 2974 2014
7	Flash Point(COC)	^{0}C	-	-	-	**>288**	ASTM D 92 2012b
8	Fire Point	^{0}C	-	-	-	**>288**	ASTM D 92 2012b
9	Cloud Point	^{0}C	-	-	-	**19**	ASTM D 2500 2011
10	Pour Point	^{0}C	-	-	-	**18**	ASTM D 97 2012

2.3 Catalyst preparation

A catalyst solution was prepared by dissolving KOH in methanol under atmospheric condition magnetic stirring for 20 min or until complete KOH dissolution. The weight percentage of the KOH needed for the process was added in volume percentage of methanol calculated by different molar ratios.

2.4 Transesterification of BBO and Taguchi method

The preparation process of babassu methyl ester (BBOME) was performed in three necked reactor vessel of volume 1000 ml. A thermometer was inserted in the reactor for observing the process hotness and a condenser was connected to another neck for retaining the steadiness of methanol to oil proportion by avoiding the evaporation losses. The entire investigational method for converting BBO into methyl ester (BBOME) is described in Fig. 1. At starting 500 ml BBO was poured in the reactor and heated up to appropriate temperature. The catalyst KOH dissolved in the proper quantity of methanol depending on methanol to oil ratio (4:1, 5:1 and 6:1). Dissolved solution of KOH and methanol was poured in the reactor containing BBO. The complete mixture was heated to the required hotness and constantly stirred by stirrer at speed of 600 rpm to ensure the homogeneous mixing of oil and Potassium-methoxide.

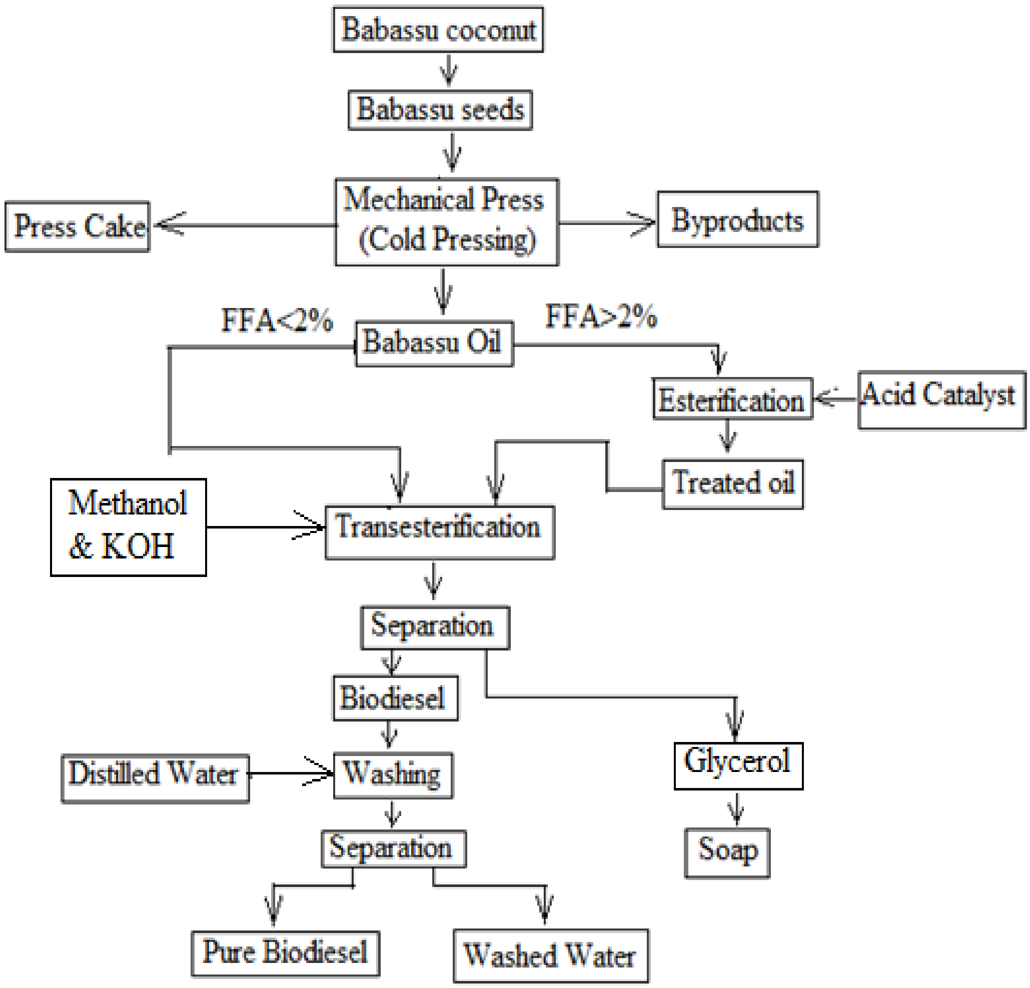

Fig.1 Flow chart of biodiesel production process

After finishing the of the transesterification process, mixture was poured in to conical shaped separating funnel. The low density BBOME part was observed to be floating over the high density glycerol portion. After 1 h glycerol got settled down at bottom. Both the separated layers were taken out and the BBOME was washed systematically four times with hot water to eliminate residual impurities, catalyst and methanol. End product layer was considered as pure biodiesel and used to calculate the percentage yield of biodiesel. Separation of biodiesel and glycerol in first phase and biodiesel and water in second phase is shown in fig. 2

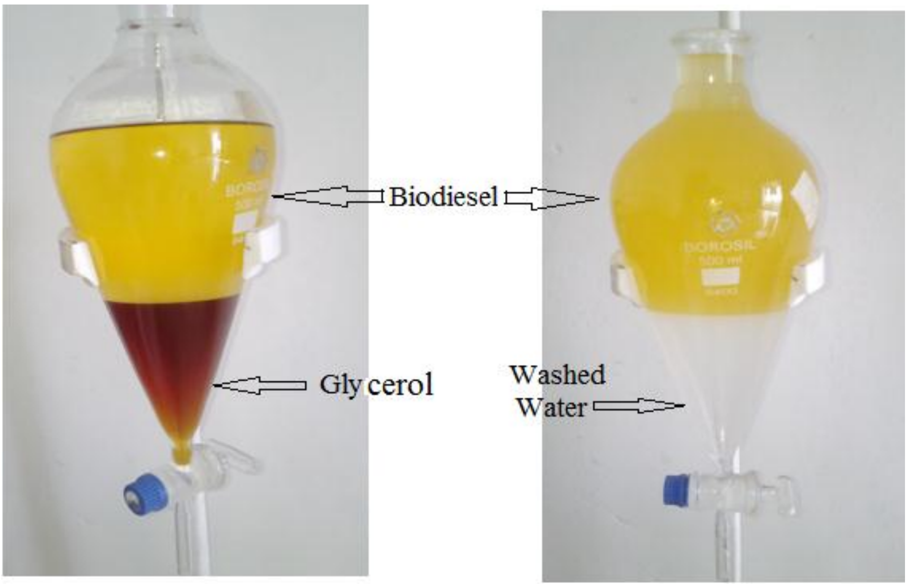

Fig. 2 Layer separation of Biodiesel, glycerol and washed water

The most important parameters taken in account for optimization of the transesterification reaction are reaction temperature in ^{0}C, methanol to oil ratio, catalyst concentration in percentage of weight and reaction time in minutes. Table2 shows the major reaction parameters with their experimentation levels.

Table 2: Transesterification reaction parameters and their levels

Symbols	**Transesterification Parameters**	**Levels**		
		1	**2**	**3**
A	Temp of reaction (°C)	30	40	50
B	Molar Ratio (Methanol/Oil)	4: 1	5: 1	6: 1
C	Catalyst Concentration (wt %)	0.8	1.0	1.2
D	Reaction Time (min)	30	45	60

The choice of the orthogonal array for the experiment needs calculation of the total degree of Freedom (DOF). DOF is related with interface among two reaction factors and calculated by the product of DOF for two reactionfactors. In the current investigation, the interaction among reaction factors was neglected. So that, there were eight DOF due to presence four reaction parameters. After calculating the necessary DOF, the next part is to choose a proper orthogonal array to get the particular intention. DOF for an orthogonal array must be more than or equal tothe DOF for the reactionfactors. In presentwork, an L9 orthogonal array was selected. It has eight DOF and it can be used for three-level reaction factor. Each reaction factor is allocated in separate column, nine reaction-factor grouping are accessible. Thus, total nine runs are necessary to investigate the all parameter space using taguchi method. The experimental arrangement for the four reaction factors is shown in Table 3.

Table 3: Chart of experimentation outline using L_9 Orthogonal array

Transesterification Parameters			
Temp of reaction (ºC)	Molar Ratio (Methanol/Oil)	Catalyst (wt %)	Time (min)
1	1	1	1
1	2	2	2
1	3	3	3
2	1	2	3
2	2	3	1
2	3	1	2
3	1	3	2
3	2	1	3
3	3	2	1

The following chronological steps were implemented to estimate the best possiblecombinations of the reaction yieldparameters:

1. Experimental matrix was formed for Taguchi method.
2. S/N ratios determined from the experimental records.
3. Most effective parameters were detected.
4. Maximum possible yield were predicted
5. Optimal levels of Transesterification process parameters were obtained.
6. By ANOVA mathematical model was set for optimum process.

3. RESULTS AND DISCUSSION

3.1 Configuration of Experimental design matrix for Taguchi method

The BBOME yield was calculated by the proportion of weight of BBOME produced to the weight of BBO used in reaction.

$$\text{BBOME Yield}(\%) = \frac{\text{Weight of BBOME produced}}{\text{Weight of BBO used for reaction}} \times 100$$

The yields of Babassu oil methyl ester, as biodiesel, arranged under 9 runs of estimated conditions are shown in Table 4. All nine reactions were performed with three experiments, with similar reaction parameters.The mean values of these results were taken as BBOMEyield percentage.

Table 4: Yield of Babassu Oil Methyl Ester

Expt. No.	% Yield of BBOME			
	Trial-I	**Trial-II**	**Trial-III**	**Mean**
1	81.13	80.27	81.12	80.84
2	86.75	86.41	86.52	86.56
3	88.20	89.40	88.41	88.67
4	90.70	88.92	88.64	89.42
5	91.10	92.38	91.20	91.56
6	92.70	91.65	93.33	92.56
7	91.13	91.93	92.73	91.93
8	94.91	95.86	95.58	95.45
9	98.12	98.87	98.67	98.56
			Average	90.61

From the yield percentage observed in nine set of experimentation, an investigational matrix planned by Taguchi method with BBOME yield percentage as response is arranged.

Table 5: Tabulation of data for Taguchi method with BBOME yield

Set No.	Reaction temp (^{0}C)	Molar ratio	Catalyst (wt%)	Reaction time (Min)	BBOME Yield(%)	S/N Ratio (η)
1	30	4:1	0.8	30	80.84	38.7463
2	30	5:1	1	45	86.56	38.9555
3	30	6:1	1.2	60	88.67	39.0287
4	40	4:1	1	60	89.42	39.2341
5	40	5:1	1.2	30	91.56	39.3285
6	40	6:1	0.8	45	92.56	39.2691
7	50	4:1	1.2	45	91.93	39.5955
8	50	5:1	0.8	60	95.45	39.8740
9	50	6:1	1	30	98.56	38.7463

Signal to noise (S/N)ratio

The S/N ratio for each factor was determined by the Taguchi method. Taguchi provides a straightforward and valuable path for investigating the influence of parameters on outcomes as well as the investigational plan. In this, the S/N ratio was used to express the outputs behaviour, and the maximum value of the S / N ratio required. This is an effective tool for selecting the optimal parameters with levels and prediction of outcomes. Three kinds of S/N ratio are avilable,lesser is the best, the higher is the best and the nominal is the best. The present work needed maximum output yield so higher is the best condition was utilized. The S/N ratio with higher is best is presented as,

$$\eta_{ij} = -10\log\left\{\frac{1}{n}\sum_{j=0}^{n}\frac{1}{y_{ij}^2})\right\} \qquad (1)$$

Where, y_{ij} is the i^{th}output of j^{th}experimentationand n is total number of tests

3.2 Main effects plot

The means of SN ratios viz. parameters temperature, Molar ratio, Catalyst concentration and reaction time is plotted by considering lager is better condition. Fig. 3 shows main effects plot for SN ratios vs. parameters.

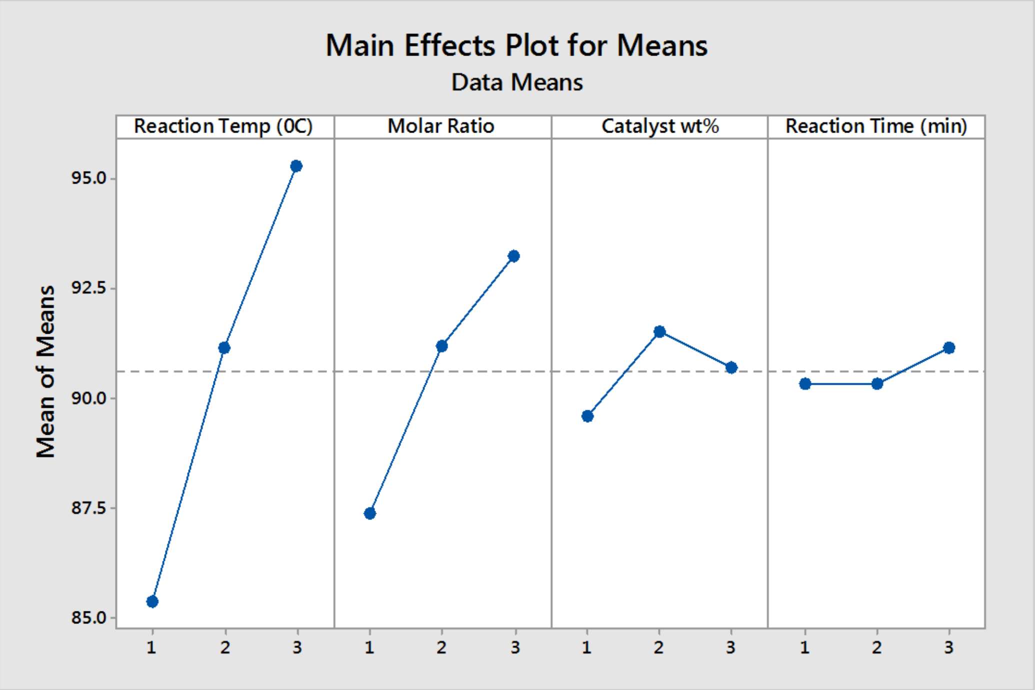

Fig. 3 Main effect plot for SN ratio

From the main effects plot, it has been revealed that yield of BBOME increased with increase in temperature range from 30ºC to 50ºC. It shows that yield of BBOME also increases with increasing molar ratio from 4:1 to 6:1.Yield of BBOME increases with increase of catalyst concentration from 0.8% to 1% for further increase in catalyst, yield was decreases. It shows that yield increases with increase in reaction time from 30 min to 60 min. The main effects plot clearly shows that temperature of reaction has most influence on the yield followed by molar ratio and catalyst concentration. It shows that reaction time has minimum effect on the percentage of yield. The plot indicates that condition for maximum yield of BBOME is temperature of level 3, Molar ratio of level 3, catalyst of level 2 and reaction time at level 3. By using these levels as optimum levels, 99.42% yield is predicted by taguchi method. The role of every reaction parameter on BBOME yield is shown in Table 6

Table 6: Parameter contributions in BBOME yield.

Parameter	**Contribution factor (%)**
Reaction temp (^{0}C)	71.45
Molar ratio(Methanol:oil)	25.27
Catalyst(wt%)	2.59
Reaction time(Min)	0.67

3.3. ANOVA Model

In the current work regression analysis was used to find out the influence of only most significant parameters of the process on the BBOME yield, as the contribution of reaction time is very less so it was neglected with confidence level of more than 95%. From the regression analysis ANOVA of the model acquired which is shown in Table 7. P-value of model was found 0.001, which shows model is acceptable. The values of every coefficients got for every factor is represented with statistical formula to calculate the BBOME percentage yield. The equation (2) used for calculation of output at specified levels of reaction parameters.

$$Y = 90.61 - 5.26 \times A1 + 0.56 \times A2 - 3.22 \times B1 + 0.57 \times B2 - 1 \times C1 + 0.90 \times C2 \qquad (2)$$

Table 7: ANOVA model and reaction parameters.

Source	DF	Sum of squares	Mean Square	F-value	p-value
Regression Model	3	202.155	67.385	42.36	0.001
A-Reaction Temp (^{0}C)	1	148.703	148.703	93.47	0.000
B-Molar ratio (Methanol:oil)	1	51.627	51.627	32.45	0.002
C- Catalyst (wt%)	1	1.826	1.826	1.15	0.333
Error	5	7.955	1.591		
Total	8	210.110			

3.4 Residual Plot

The residual plot for percentage yield of the BBOME is shown in Fig 3. It is helpful to decide whether the model satisfies the hypothesis of the investigation. The residuals are the variation of the experimental values from the predicted values. Analysis of residuals is important to estimate the model satisfactoriness. All residuals in present work were observed to be sprinkled without any specific pattern which indicates the satisfactoriness of the model.

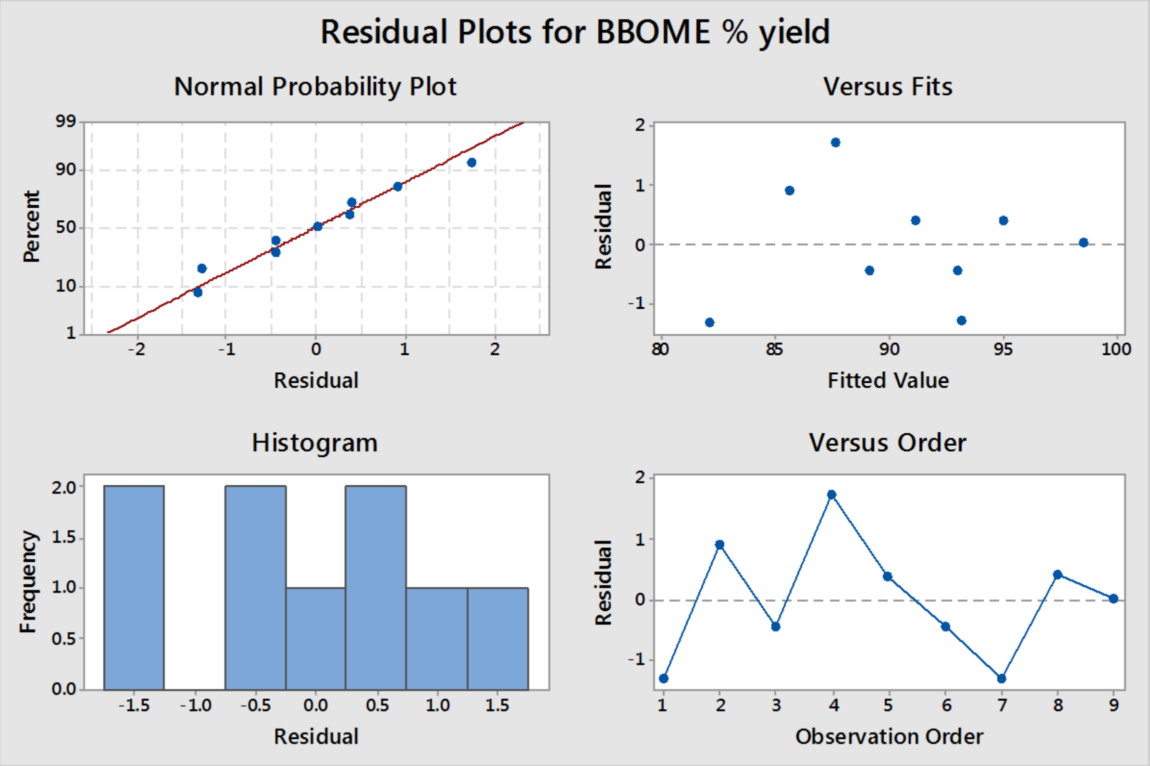

Fig. 4 Residual plot for BBOME yield

The normal probability plot shows that observed data were normally distributed. The residuals versus fitted plot shown steady variance and there were non-linear relationship among residuals and fitted values. Histogram shows frequency of residual values, It indicates that data were not off-centred. Residuals versus order of the observation shows there was logical influence in the data due to data collection order or time.

3.5 Experimental validation

The model obtainedusing regression method was validated by executing the trial at calculated optimalsetting. The validating tests were performed three times and outcomes werecompared relative with the predicted value of yield percentage. The experimental BBOME percentage yield observed from the verifying trials was closer to the predicted BBOME yield. Therefore from the outcomesof the experimentation it can be understood that build up model isuseful in predicting the yield percentage and can beeffectivelyimplemented for theoptimization of BBO to BBOME transmission reaction. Table 8 shows comparison of predicted yield and experimental yieldat optimum conditions.

Table 8: Experimental validation of model

Sr.no.	Reaction temp (^{0}C)	Molar ratio	Catalyst (wt%)	Reaction time (Min)	BBOME Yield (%)	
					Actual	Predicted
1	50	6:1	1	60	99.13	99.42
2	50	6:1	1	60	99.43	99.42
3	50	6:1	1	60	98.89	99.42

3.5 Characterization of BBOME

Physicochemical properties of biodiesel were determined by ASTM described test methods at Chem Tech Laboratories Pvt Ltd, Pune. The Comparison of Physico-chemical Properties of BBOME from different researchers and properties of BBOME in present work are shown in Table 9

Table 9: Properties of BBOME by different researchers and present biodiesel

Sr. No.	**Test Description**	**Units**	**References**			**Properties of biodiesel obtained in present study**	
			[2,3]	[5,6]	[15,20]	**Results**	**Test Method**
1	Density at 15 ^{0}C	Kg/m^3	887.2	870	870	876.2	ASTM D 1298 2012 b
2	Viscosity at 40 ^{0}C	cSt	2.98	3.6	4.2	4.001	ASTM D445 2012
3	Gross Calorific Value	MJ/kg (Cal/g)	-	-	-	40.012 (9563)	ASTM D 240 2009
4	Cetane number		-	63	63.7	-	ASTM D7668 2014
5	Flash point	^{0}C	-	120	112	126	ASTM D 93 2013
6	Fire Point	^{0}C	-	-	-	131	ASTM D 92 2012b
7	Moisture	wt%	0.02	-	-	0.021	ASTM D 2974 2014
8	Pour point	^{0}C	-6	2	-	9	ASTM D 97 2012
9	Cloud point	^{0}C	-4	11	-	14	ASTM D 2500 2011

4. CONCLUSIONS

Based on observation of ester recovery, it was found that Taguchi method (L9 Parameter design) given the optimize process parameter of Babassu biodiesel production. The properties of BBOME measured in this experimental investigation were found within the limit of ASTM and EN standards. Investigation of the factors influencing the yield percentage of biodiesel shows that, among the investigational range considered, the most significant factor is the reaction temperature followed by the molar ratio. At optimal condition, and from financial standpoint, the highest yield of biodiesel 99.42% can be achieved. The biodiesel showed 40.012 Mj/Kg of lower heating value and, 4.001 cSt kinematic viscosity and offers physical-chemical properties appropriate for use as diesel fuel. It has been observed that BBOME produced by transesterification of BBO using methanol can improve biodiesel suitability in cold weather conditions. Hence it is concluded that babassu biodiesel could be considered as potential alternative fuels for diesel engine application.

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