Optimizing Biodiesel Yield and Investigating CI Engine Performance Using Biodiesel Blends of Pongamia, Animal Fat, and Waste Cooking Oils

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Abstract: Biofuel plays a predominant role in the sustainable growth of any developing country due to its renewability and lower toxicity. This led the researcher to work on alternative fuels. In this aspect, Sample 1 consists of a composition comprising 50% by weight of raw karanja (pongamia) oil, 25% by weight of raw animal fat (beef tallow) oil, and 25% by weight of cooked oil. On the other hand, Sample-2 comprises a composition consisting of 60% by weight of crude karanja oil, 20% by weight of raw beef tallow oil, and 20% by weight of cooked oil. Biodiesel blends are investigated for their optimization and performance through diesel engines. Optimization is performed using RSM, with yields of 98.32% and 97.82% obtained for samples 1 and 2, respectively. The optimized conditions were maintained as 8.15:1 methanol-oil ratio, 1.2 wt.% catalyst concentrations, 75 min. of reaction time, 64°C temperature, and 6.74:1 methanol-oil ratio, 1.193 wt.% catalyst concentrations, 74 min of duration, and 64°C temperature for samples 1 and 2, respectively. The sample properties meet the ASTM D6751 standards. Both the samples are blended with the diesel in an appropriate ratio, and the performance tests are conducted in a single-cylinder diesel engine at variable workload conditions. The brake thermal efficiency obtained for sample 2 is higher than that of sample 1. At B100, sample 1 had 27% of BTE, whereas sample 2 had 29%. The BTE of B20 blends in samples 1 and 2 is 31.5% and 33%, respectively. The brake-specific fuel consumption for samples 1 and 2 at B100 blend ratios is 1.45 kg/kWh and 1.4 kg/kWh, respectively.

Keywords: Pongamia; Animal Fat; Waste Cooking Oils; RSM; Biodiesel; CI Engine

1. Introduction

After the invention of the diesel engine by Rudolph Diesel, the locomotive sector boosten up. The energy required for these locomotives has been significantly supplied through conventional sources[1]. The availability of energy sources is the key factor that affects the economic and social status of any country[2]. As per the survey, more than 80% of total world energy consumption is obtained from three sources, such as coal, oil, and natural gasses. These sources are nothing but fossil fuels, which were the main energy-producing sources in previous centuries, use of non-conventional source can replace the fossil fuels[3]. The conventional fuels took millions of years to form. Using these sources in internal combustion engines causes negative effects on the atmosphere, such as increasing carbon emissions on the surface of the earth[4], which leads to global warming which impact on global climatic system[5]. In addition to these, fossil fuels depleting in a faster manner and enhancing the greenhouse gases in the atmosphere drive researchers to work towards green fuels such as biodiesel[6]. Biodiesel is a single-chain mono-alkyl ester. Potential sources for biodiesel production are Jajoba oil[7], cotton seed oil[8], microalgae[9], waste cooking oil[10], soybean oil[11], rocket seed oil[12], etc. Initially, primary-generation feedstocks were used for biodiesel synthesis, but their usability affected the food cycle. Therefore, alternative sources such as animal fats, used cooking oil, inedible seed oils, and similar materials prove to be exceptionally effective for the production of biodiesel[13].

The non-edible seed oils such as Pongamia, jatropha, neem etc., and waste oils such as fried oil[14], chicken fat oil[15], duck fat[16], etc. reduce the biodiesel production cost. The expenditure on raw materials typically constitutes approximately 60 % to 75 % of the overall cost of biodiesel production[6,17,18]. Alkaline catalysts like

NaOH and KOH are commonly preferred for biodiesel manufacturing due to their cost-effectiveness, ample availability, and rapid reaction times in the base transesterification process[19]. Currently, the trend is towards the use of heterogeneous catalysts for several of their benefits, such as reusability, no soap formation, being environmentally compatible and better productivity. But it requires complex methods to prepare it, and it is not economical due to its higher production cost compared to homogeneous catalysts.

The process of biodiesel production entails the transesterification reaction of triglycerides with alcohols such as methanol, facilitated by a catalyst[19]. Presently, for mass production, usually biofuel plants are using NaOH (alkaline) catalysts. After the reaction, triglyceride converts into a single-chain fatty acid, methyl ester. If the reaction is incomplete, then a proportion of the catalyst remains in mono-di-triglycerides[20]. The transesterification depends on many parameters[21-24], including molar ratio (generally considered for three moles of oil to one mole of methanol), catalyst concentration (an alkaline catalyst such as NaOH or KOH, ranging from 0.5% to 1% wt.), reaction temperature (around 60°C to 65°C), and agitation speed (for enhancing the reaction rate around 600 rpm). The various statistical tools, such as ANOVA, ANN, etc., optimize the process variable of transesterification by using response surface methodology[25]. Some of the Numerous studies have indicated that incorporating biodiesel into internal combustion engines results in reduced exhaust emissions and comparable performance to conventional diesel fuel[29-31]. Biodiesel has shown decreased emissions of greenhouse gases, including carbon monoxide (CO), hydrocarbons (HC), and carbon dioxide (CO_2). The study found a substantial reduction in emissions: 57% in carbon monoxide (CO), approximately 40% in unburned hydrocarbons (HC), CO_2 emissions by 13.8%, and about 23% in smoke opacity compared to diesel fuel[32,33]. Despite its positive attributes, biodiesel faces challenges such as lower calorific value, elevated NOx emissions, and suboptimal oxidation stability during extended storage periods, as highlighted in prior research[34]. In the performance evaluation of waste cooking oil shows decrease in brake power of 1.68 %, brake torque of 1.7 %, brake thermal efficiency of 21% and an increase in brake specific fuel consumption(BSFC) of 36%[35].The research on studying mustard oil biodiesel blends in diesel engine indicate that decrease in brake specific energy consumption and a lean increase in brake thermal efficiency[36]. The investigation on distilled biodiesel from WCO blended with diesel improves the engine performance and reduce the emission rate. Also, this study explains the feasibility and cost effectiveness in the usage of WCO for biodiesel production. 10% and 20% samples can be directly used in engine without any engine modification[37]. The biodiesel from waste swine oil demonstrated a higher

BSFC about 10.2% but lesser BTE than diesel and recorded lower rate of emission except NOx and CO2[38]. The B30 sample of rice bran oil at an 18:1 compression ratio achieves a better brake thermal efficiency of 25% and brake power of 3.5 kW with a reduction in harmful gases. The study suggests that using B30 with diesel at half load is a more optimal choice for a diesel engine[39]. The investigation on mixed Jatropa and castor as four different blend proportions at 20% with diesel is tested in the engine. The test showed higher BSFC, EGT, CO, and NOx emissions but lower BTE, CO_2, HC, and smoke emissions than diesel[40]. This study aims to optimize the composition of two biodiesel samples. In Sample 1, there is a combination of 50% weight raw Karanja oil, 25% volume weight raw beef tallow, and 25% volume weight three times used restaurant culinary oil. In Sample 2, there is a composition of 60% volume weight raw Karanja oil, 20% weight crude beef tallow, and 20% weight three times used culinary oil. Response surface methodology (RSM) is applied to assess optimized condition for biodiesel synthesis. The parameters such as density, viscosity, flash point, fire point, cloud point, and calorific value are assessed according to ASTM standards. The study involves evaluating the performance attributes of a single-cylinder direct-injection diesel engine across diverse load conditions, employing biodiesel blends ranging from B20 to B100.

2. Materials and Methodology

2.1. Extraction of oil from Pongamia/Karanja, Beef tallow and Reused Cooking Oil

Pongamia trees are larger in size and found abundantly in forests, tribal areas, and roadsides. These trees give 500 to1000 kg of seeds per year. The kernel of the seed is extracted and processed in a mechanical expeller to get crude oil. In this experiment, crude pongamia oil is purchased from the T Narsipura oil mill, crude animal fat is brought from a mutton shop, and the reused waste cooking oil (refined sunflower oil) is taken from restaurant after three use. The collected oils are filtered. Thus, the debris presented gets separated. The animal fat from the mutton shop was heated to a mild temperature of 45°C optimized results obtained for different oil samples carried out by various researchers are presented in table 1.

Table 1. The optimized values for biodiesel synthesis from the various oils are listed.

Reference	Oil	Preparation Condition					FAME Yield
		Methanol to oil ratio	Catalyst	Catalyst conc.	Reaction temp.	Reaction time	
26)	Jatropha	5:1	KOH	0.55 %(w/w)	62	28 min	99%
27)	Waste cooking oil	6:1	Calcined scallop shell	5 wt%	66	120 min	86 %
28)	Camelina sativa	6:1	NaOH	1.5%(w/w)	54	64 min	97.4%

To convert into oil. Thus, the obtained crude oils are ready to intermix, as presented in Fig. 1.
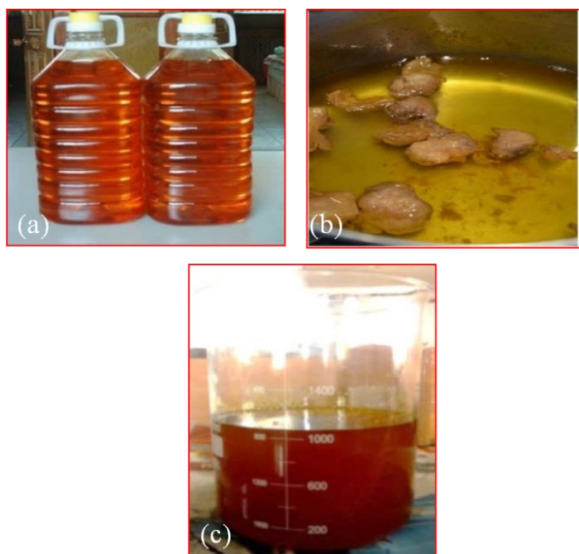

Fig. 1: Crude Karanja(a), Beef tallow(b) and, fried oils(c).

2.2. Biodiesel Preparation Method

2.2.1. Free Fatty Acid Composition

The free fatty acid (FFA) of samples 1 and 2 is determined through a gas chromatography experiment, as illustrated in Table 2. In short, FFA is a measure of the degradation of unsaturated fats. By using the titration method of 0.1N, NaOH solution FFA can be reduced[41].

FFA can be calculated by following equation -1,

$$FFA\ (\%) = \frac{28.2 \times N \times V}{W_{oil}}$$

Where:

- V = Volume of the titrant (NaOH) used in titration (in milliliters).
- N = Normality of the titrant (in equivalents per liter).
- W_{oil} = Weight of the oil sample (in grams).
- 28.2 = molecular weight of oleic acid (282 g/mol).

Table 2. The composition of fatty acids in the oils of Sample-1 and Sample-2

Name of Fatty acids - Carbons	Fatty acids	
	Sample-1	Sample-2
"Caprioc – 6:0"	-	-
"Capric – 10:0"	-	-
"Lauric – 12:0"	0.14	0.19
"Myristic – 14:0"	1.49	1.43
"Palmatic – 16:0"	21.44	22.5
"Stearic – 18:0"	12.1	9.83
"Oleic – 18:1"	42.9	43.01
"Linoleic – 18:2"	11.55	14.1
"Linolenic – 18:2"	2.27	0.97
"Behenic – 22:0"	-	1.89
"Erucic – 22:1"	-	-
"Lignoceric – 24:0"	0.56	0.39
"Heneicosanoic – 21:0"	-	0.12

2.2.2. Esterification Setup and Process

Samples 1 & 2 were prepared with 2% FFA. Thus, a direct transesterification process is performed. The experiment is conducted in a three-neck round-bottomed flask with a reflux condenser placed on the heat jacket. The heat jacket contains a thermostat to regulate the temperature to the required value. The reflexive condenser helps maintain the atmospheric pressure inside the reactor and refluxes the methanol back into the reactor during condensation. The process parameters for sample 1 involve an alcoholysis ratio of 8.15:1, a catalyst wt. % of 1.193, a thermal condition of 64°C, and a processing duration of 60 minutes. Sample 2, on the other hand, utilizes a 6.74:1 molar ratio, a 1.198 wt. % catalyst concentration, an operating temperature of 64°C, and a duration of 75 minutes. Subsequently, the esterified solution is moved to a decantation vessel, where the mixture is left undisturbed for a period of 6 hours to allow settling. During this period, the methyl ester, driven by gravity, settles at the top, while glycerol settles at the bottom, as depicted in Fig. 2.

Fig. 2: Esterification set-up (left side) and gravity separation (right side)

2.2.3. Washing and Drying

The methyl ester settled at the top of the separating flask, leaving small traces of glycerol and methanol. To eradicate these ingredients, water-washing methods are employed. The solution is taken into the washing funnel, which adds warm water (about 45 ˚C) from the top and allows it to settle for 20 to 25 minutes. Soap water collected at the bottom is removed, and the process is repeated until the pH of the outlet water matches the pH of the inlet water, as depicted in Fig. 3. The obtained glycerol-free solution is heated in a methanol recovery setup up to 65 °C. Thus, 70% to 80% of methanol can be recovered. The acquired solution is transferred into the glass vessel and positioned atop the magnetic stirring apparatus, with the magnetic bar submerged within the vessel. Maintain the speed and temperature of 1000 rpm and 100 °C for 1 hour. Thus, all the moisture contents present in the solution evaporate, as demonstrated in Fig. 4. After the moisture evaporation, the neat fatty acid methyl ester (FAME) is obtained, which is shown in Fig. 4 (right side).

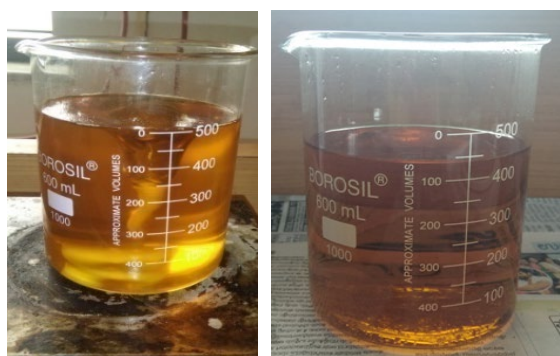**Fig. 3**: Water washing first stage (left side) and final stage (right side).

Fig. 4: Drying process and neat biodiesel.

3. Experimental design technique for optimization using 'Response Surface Method (RSM)'

The statistical analysis is performed to fine-tune the operational variables for transesterification. 5 levels and 4 parameters of experiments are designed using Central Composite Design (CCD). Table 3 shows the factors and experimental ranges of process parameters for samples 1 and 2. The levels and ranges for both samples are the same. It was taken based on a literature review[42-45]. The levels and ranges of various parameters are set based on previous literature. The -1 and + 1 are manually entered in the software, and the remaining -α and +α were generated in the ANOVA software. The -α and +α resemble the least and highest values below and above which the significant biodiesel yield cannot be obtained. Among the 4 factors, molar ratio and catalyst concentration show more significant contributions towards yield. However, all four factors need to be maintained at an optimal level with the aim of improving the FAME percentage. Table 4 describes the effect of operating variables on the FAME yield when they are out of range.

The RSM is used to optimize the process variables of the transesterification reaction, which include methanol to oil ratio, catalyst wt. %, reaction temperature &time. CCD developed the 30 experiment ($= 2^K + 2K + 6$), where K = 4 (no. of variables)[42]. The experiment center point was adopted by eight actual points and six replicates, as illustrated in Table 4. The CCD of RSM involves statistical & mathematical techniques, which are utilized to modeling and analyzing the problems. Through RSM, knowledge about the process to reduce cost & time can be gained[46]. The levels and ranges provided for both samples are the same, including an alcohol-oil ratio of 4.5 to 10.5 ratios, a catalyst wt. % of 0.60 to 1.4, a reaction period of 30 to 90 minutes, and a process temperature of 40° C to 70° C. The least yield obtained from the sodium methoxide-catalyzed process is 74.2%, and the maximum yield is 98.2% for sample 1 and sample 2. The lowest yield is 74.6%, and the highest is 97.7%. The quadratic regression model for sodium methoxide-catalyzed biodiesel synthesis is given in equations 2 and 3.

The transesterification reaction process determines the yield. As can be seen in Tables 5 and 6, analysis of variance was employed to evaluate the suitability and competence of the second order polynomial model for both the samples PO50:WCO25:AFO25 and PO60:WCO20:AFO20. For both the samples PO50:WCO25: AFO25 and PO60:WCO20:AFO20, the model F values are 376.70 and 439.84, respectively. The model accurately forecasts FAME yield at a value of 0.0001 and is highly significant at the 95% confidence level. The relevance of the model term was assessed using a P-value of 0.0001. The values larger than 0.1

indicate that the source is unimportant. Based on the analysis of variance findings, the methanol-oil ratio (A), catalyst weight percentage (B), operating temperature (C), and process duration (D) are the most significant variables impacting the biodiesel yield response for NaOH catalytic techniques. For the prepared biodiesel samples (1 and 2), a strong interaction between the critical variables AC, BD, and CD is evident due to the quadratic influence of A^2, B^2, and C^2. The results of the experimental discoveries are consistent with the model's acceptable accuracy, as shown by the values of R^2 and Adj-R^2. According to the R^2 (0.9969 and 0.997) and Adj-R^2 (0.9940 and 0.9947) values for the biodiesel samples 1 and 2, respectively, the independent factors explained 99.69% and 99.72% of the total variance in the biodiesel output.

Substantial agreement between estimated and measured values is typically evident when the coefficient of determination (R^2) surpasses 0.8. When R^2 nears unity, it signifies a high degree of correspondence between the empirical models and the experimental data. Niken Taufiqurrahmi et al. (2011) noted a similar model in palm oil biodiesel process optimization[47]. Additionally, the analysis procedure is in line with the literature findings[48,45]. According to Figs. 5, the plot of actual values vs. anticipated values is very similar to a curve of slope 1 at 45°, with points related to null error. This is true for biodiesel yield using both samples of oils PO50:WCO25:AFO25 and PO60:WCO20:AFO20. This demonstrates that the model is still accurate. The appropriate precision values for biodiesel when PO50:WCO25:AFO25 and PO60:WCO20:AFO20 are used as oil samples are 64.6358 and 72.594, respectively. The low coefficients of variation (CV), which were 0.5905 and 0.5068 percent for Sample 1 & 2 respectively, demonstrated the great accuracy and consistency between the actual and predicted values. The model is unremarkable when compared to the pure error, as indicated by the absence of a fit F-value of 2.48 and 0.6954 for biodiesel with both samples of oils PO50:WCO25:AFO25 and PO60:WCO20:AFO20 as presented in Table 6.

Table 3. Levels and ranges of process parameters for design of experiments.

Process parameters (Units)	Coding	Levels and ranges				
		–α	–1	0	+1	+α
Molar ratio(mol/mol)	A	4.5	6	7.5	9	10.5
Catalyst conc.(wt.%)	B	0.6	0.8	1	1.2	1.4
Reaction temperature(°C)	C	30	45	60	75	90
Reaction Time(min)	D	40	48	56	64	72

Table 4. Effect of operating variables on FAME yield, when they are out of range[40].

Process parameters	Molar ratio	Catalyst concentration	Temperature	Time
High [> +α]	High-cost consumption during recovery	i) Cause reverse transesterification which leads to saponification ii) higher cost	Chances of oil get fried. Thus, it largely effects on fuel property	Backward reaction occurs result in soap formation
Low [< -α]	Inadequate methanol causes incomplete reaction. Result in failing of complete conversion from triglyceride to monoglyceride	A smaller number of activation sites cause incomplete reaction. Which have negative effect on yield	Too less temperature does not expose the surface of the catalyst. Results in less activation sites available for the reaction	Conversion from triglycerides to monoglycerides requires specific time. If reaction time is less, then incomplete process occurs.

Table 5. Design based on CCD of the experimental test and result obtained for PO50:WCO25:AFO25 (sample-1) & PO60:WCO20:AFO20 (sample-2).

Trials	Methanol to oil ratio	Catalyst Conc. (wt %)	Reaction Time (min)	Reaction temperature (°C)	(%) Biodiesel Yield for S-1		(%) Biodiesel Yield for S-2	
					Obtained	Predicted	Obtained	Predicted
1	7.5	1.4	60	56	96.6	96.9	95.9	96.3
2	7.5	1	60	56	91.9	92.6	85.4	87.0
3	7.5	1	60	56	91.1	91.6	86.5	87.0
4	6	1.2	45	64	89.9	90.8	92.5	92.5
5	9	0.8	45	48	82.6	83.3	84.8	85.9
6	9	1.2	45	48	92.8	91.1	85.2	85.9
7	6	1.2	45	48	91.8	92.4	87.4	87.5
8	7.5	0.6	60	56	75.5	76.7	79.3	78.0
9	7.5	1	60	56	91.3	91.6	85.8	87.1
10	6	0.8	75	64	76.5	74.8	80.1	80.0
11	9	1.2	75	64	98.1	98.5	91.3	91.9
12	7.5	1	60	56	89.8	91.6	86.9	87.0
13	9	1.2	45	64	92.7	93.1	90.9	92.3
14	7.5	1	90	56	91.0	91.1	94.9	94.9
15	6	0.8	45	64	82.3	82.2	80.3	78.9
16	9	0.8	75	64	83.6	83.9	84.4	84.4
17	6	0.8	75	48	82.2	82.1	81.4	81.3
18	7.5	1	60	56	92.0	91.0	85.9	85.8
19	10.5	1	60	56	81.9	82.1	79.1	77.9
20	7.5	1	60	56	91.4	91.0	87.1	87.0
21	6	1.2	75	64	89.9	90.1	97.7	97.6
22	4.5	1	60	56	75.2	73.6	73.6	75.0
23	9	1.2	75	48	97.5	97.9	88.9	89.3
24	6	0.8	45	48	83.6	83.8	76.8	77.2
25	6	1.2	75	48	94.9	94.6	95.0	94.7
26	9	0.8	75	48	84.8	84.7	85.8	85.9
27	7.5	1	60	40	98.2	98.5	89.7	89.3
28	9	0.8	45	64	85.3	85.3	87.3	87.7
29	7.5	1	60	72	96.7	96.5	93.8	93.9
30	7.5	1	30	56	87.9	87.8	92.0	91.5

Table 6. Sequential Model Sums (SMS) of Squares for PO50:WCO25:AFO25and PO60: WCO20: AFO20.

Source	Sequential Model Sum of Squares for PO50:WCO25:AFO25					Sequential Model Sum of Squares for PO60:WCO20:AFO20				
	Sum of the Squares	Df	Mean Square	F Value	p-value Prob> F	Sum of the Squares	Df	Mean Square	F Value	p-value Prob> F
Mean	2.346E+05	1	2.346E+05			2.278E+05	1	2.278E+05		
Linear	801.39	4	200.35	9.88	< 0.0001	555.43	4	138.86	6.90	0.0007
2FI	91.82	6	15.30	0.7005	0.6527	157.28	6	26.21	1.44	0.2516
Quadratic	410.95	4	102.74	376.70	< 0.0001 Suggested	343.16	4	85.79	439.84	< 0.0001 Suggested
Cubic	2.78	8	0.3473	1.85	0.2157 Aliased	1.51	8	0.1888	0.9340	0.5425 Aliased
Residual	1.31	7	0.1875			1.42	7	0.2022		
Total	2.359E+05	30	7863.94			2.288E+05	30	7628.32		

Table 7. ANOVA for second-order polynomial models for PO50:WCO25: AFO25 (sample-1) and PO60:WCO20: AFO20 (smaple-2).

Source	PO50:WCO25:AFO25					PO60:WCO20:AFO20				
	Sum of Squares	df	Mean Square	F Value	p-value Prob> F	Sum of Squares	df	Mean Square	F Value	p-value Prob> F
Model	1304.15	14	93.15	341.56	< 0.0001 Significant	1055.87	14	75.42	386.67	< 0.0001 Significant
A-Molar ratio	79.42	1	79.42	291.22	< 0.0001	16.90	1	16.90	86.65	< 0.0001
B-Catalyst Amount (wt.%)	708.51	1	708.51	2597.86	< 0.0001	484.74	1	484.74	2485.23	< 0.0001
C-Reaction Time	7.77	1	7.77	28.51	< 0.0001	22.78	1	22.78	116.77	< 0.0001
D-Reaction Temperature	5.68	1	5.68	20.84	0.0004	31.01	1	31.01	158.98	< 0.0001
AB	0.0576	1	0.0576	0.2112	0.6524	121.00	1	121.00	620.36	< 0.0001
AC	20.43	1	20.43	74.91	< 0.0001	10.60	1	10.60	54.32	< 0.0001
AD	30.75	1	30.75	112.74	< 0.0001	0.0090	1	0.0090	0.0463	0.8326
BC	28.41	1	28.41	104.17	< 0.0001	9.92	1	9.92	50.87	< 0.0001
BD	1.05	1	1.05	3.85	0.0685	10.89	1	10.89	55.83	< 0.0001
CD	11.12	1	11.12	40.78	< 0.0001	4.86	1	4.86	24.93	0.0002
A^2	259.88	1	259.88	952.90	< 0.0001	177.51	1	177.51	910.08	< 0.0001
B^2	27.12	1	27.12	99.44	< 0.0001	0.1064	1	0.1064	0.5457	0.4715
C^2	4.40	1	4.40	16.14	0.0011	76.82	1	76.82	393.85	< 0.0001
D^2	81.56	1	81.56	299.05	< 0.0001	40.14	1	40.14	205.82	< 0.0001
Residual	4.09	15	0.2727			2.93	15	0.1950		
Lack of Fit	3.41	10	0.3405	2.48	0.1638 Not Significant	1.70	10	0.1702	0.6954	0.7083 Not Significant
Pure Error	0.6859	5	0.1372			1.22	5	0.2447		
Cor Total	1308.24	29				1058.79	29			
R^2 – 0.9969; Adj. R^2 – 0.9940; Pred. R^2 – 0.9843; Adequate Precision – 64.6358; C.V. % – 0.5905						R^2 – 0.9972; Adj. R^2 – 0.9947; Pred. R^2 – 0.9891; Adequate Precision – 72.5944; C.V. % – 0.5068				

(a)

(b)

Fig. 5: Plot of predicted v/s actual values for sample-1 (a) and sample-2 (b).

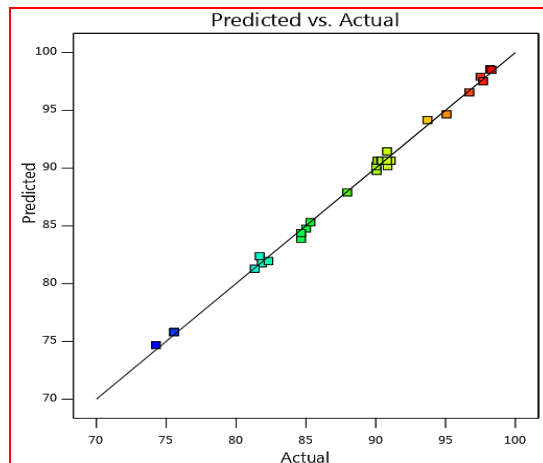### 3.1. Response Surface Plots for Sample-1 and Sample-2

A second-order polynomial equation generated by the software is successfully fitted with the results of the experiments. The polynomial equation considers linear, quadratic, and interacting variables. The polynomial equation 4 is given below.

$$Y = b_0 + \sum_{i=1}^{n} b_i X_i + \sum_{j=1}^{n} b_{ij} X_i^2 + \sum_{i=1}^{j-1} \sum_{j=2}^{n} b_{ij} X_i X_j \ \dots \dots 4$$
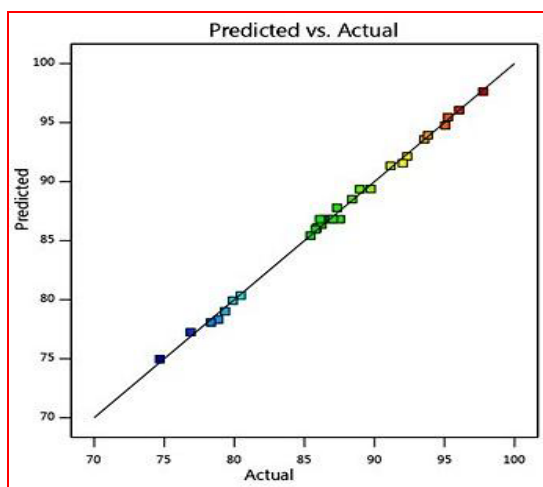
Where,

- Y represents the response factor.
- b_o stands for the intercept.
- b_{ii} denotes the quadratic coefficient of the factor i.
- b_i represents the linear order coefficient of the model.
- b_{ij} denotes the coefficient representing the interaction between factors i and j in the linear model.
- n equals 4, indicating the number of factors analyzed and optimized in the experiment.
- xi represents the independent variable.

The precision of the model is ascertained by the R^2 coefficient. 3-dimensional graphs were generated by plotting polynomial equations derived from regression analysis. The 3-dimensional graph was constructed by holding two variables constant while plotting the remaining two variables against the yield.
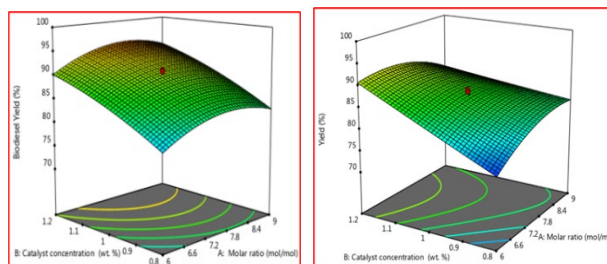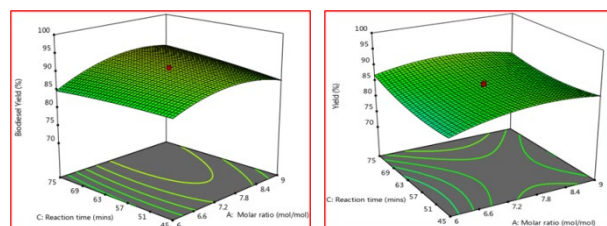

(a)

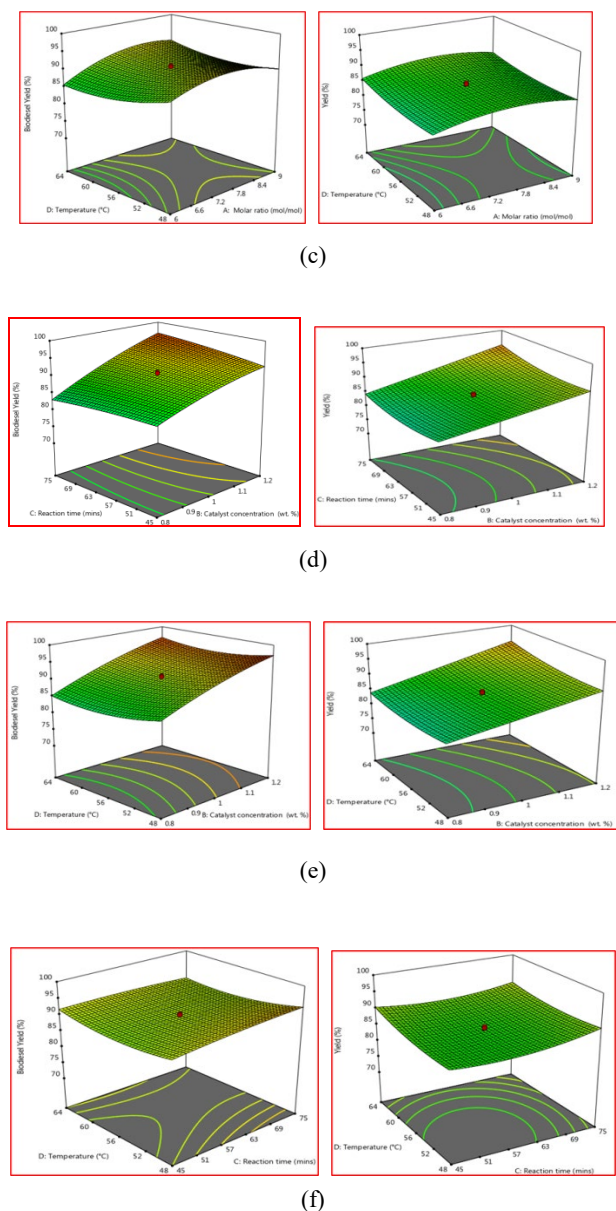

(b)

(c)

(d)

(e)

(f)

Fig. 6: Presents Response Surface Methodology (RSM) plots for both Sample-1 and Sample-2, where the left-side graphs depict A) Alcohol ratio v/s Catalyst concentration, B) Reaction time v/s Molar ratio, and C) Temperature v/s Molar ratio for Sample-1. Conversely, the right-side graphs illustrate D) Reaction time v/s Catalyst concentration, E) reaction temp. v/s Catalyst concentration, and F) reaction temp. v/s Time for Sample-2.

The above plots describe the fatty acid methyl ester (FAME) yield for various combinations of process parameters. Figure 6(A) shows the significance of methanol and catalyst on the yield percentage. The various graphs show the 3-D plots of samples 1 and 2. Figure 6(A) shows the effect of molar ratio and catalyst wt. percentage against the yield value. Sample 1 shows a highest yield of 98% and a minimum of 74%, and sample 2 shows a highest yield of 95% and the lowest yield of 70%. This value signifies that the effects of methanol to oil ratio and catalyst wt. % are dominant in the yield of

FAME. Excessive catalysts lead to an increase in raw costs and also cause saponification. Lesser catalyst concentration and methanol lead to improper chemical reactions. Figure 6(B) shows the 3-D contour of FAME yield vs. reaction time vs. molar ratio. Sufficient time should be given to carry out the chemical reaction; excessive time leads to unfavorable chemical reactions, such as when monoglyceride undergoes a chemical reaction and causes saponification[41]. Molar ratio's having a higher significance effect compared to reaction time. Figure 6(C) displays the effect of molar ratio and reaction temperature on the FAME yield. A higher curvature variation in the graph is obtained whenever the molar ratios are significantly changed by keeping another parameter constant. Excessive temperature leads to the inactivation of the active sites of the catalyst[49], which results in lower chemical reaction efficiency. Figure 6(F) shows the effect of temperature and reaction time on the yield. The graph is almost flat for both samples. This resembles too much variation in reaction time and reaction temperature, which can affect the FAME yield considerably; otherwise, their effects are cherry-picked.

4. Results Interpretation

4.1. Fuel Characterization

Table 8 demonstrates the physico-chemical properties of crude samples and biodiesel samples. The kinematic viscosity of FAME depends on monoenoic fat[50]. In this study, both sample 1 and sample 2 met the ASTM D6751[51] biodiesel standards. Samples 1 and 2 of FAME exhibit elevated levels of viscosity, density, and molecular configuration compared to traditional diesel. The molecular arrangement influences fuel characteristics such as volatility and viscosity. Furthermore, it has an impact on the chemical properties of the fuel, which play a crucial role in governing the combustion processes[52]. Thus, the biodiesel properties intertwined with the injection process lead to fuel atomization, improper burning, and carbon deposition[53]. Viscosity is an important fuel property that predominantly affects the fuel system line and fuel dispersion. The kinematic viscosity of sample 1 is 5.25 mm²/s, and sample 2 is 5.18 mm²/s, which are within the zone of ASTM D-6751 standards (1.9 mm²/s to 6 mm²/s). Both sample 1 and sample 2 have a higher cetane number (i.e., 51 and 53, respectively) compared to diesel (CN = 49.7). Hence, FAME has a lower ignition delay, which is an added advantage to the combustion process. In general, FAME has lower volatility. Thus, the flash and fire points of biodiesel are incomparable with those of diesel. Both the samples are in the range of around 200°C, with flashpoint values that are four times higher than the neat diesel. Biodiesel is also called fatty acid methyl ester; the name itself implies there is a fatty acid composition, even after performing the FFA test and base esterification. It is inevitable to proceed with these

compositions. Hence, the acid values of biofuel are higher than those of neat diesel. Sample 1 and sample 2 have 0.13 and 0.15 mg/g, respectively. Whereas diesel have 0.051 mg KOH/g. The larger fuel droplet formation under intensified spray causes impingement of the fuel on the cold wall region. This leads to incomplete combustion and carbon deposition[44]. But the presence of excess oxygen content can promote the combustion reaction[54]. Biodiesel has a higher oxygen content compared to traditional fuel. The key properties of B20 fuel in both sample-1 and sample-2, such as density, viscosity, heating value, and cetane number, are very similar to those of conventional diesel.

4.2. Engine performance characteristics

A 4-stroke, single-cylinder, water cooled, eddy current dynamometer, direct-injection at variable load diesel engine is employed to assess the performance of different mixtures of biodiesel samples, namely sample-1 and sample-2. Various biodiesel blend ratios, including B20, B40, B60, B80, and B100, are formulated for experimentation. The engine operates under varying loads while maintaining a consistent injection pressure of 200 bars at speed of 1500 rpm and compression ratio of 19:1 at injection angle of 23°.

BTE is one of the key parameters which express the capacity of the engine. Diesel exhibits superior brake thermal efficiency (BTE) compared to biodiesel blends, with diesel achieving the highest BTE at 37%. In contrast, B100 biodiesel in sample 1 and sample 2 records 27% and 29% BTE, respectively, indicating that sample 1has lower efficiency compared to sample 2. Sample 2 outperforms sample 1 in BTE, with the B20 blend in sample 1 at 31.5%, while sample 2 achieves 33%. The remaining blend samples in Figure 7 fall below these BTE values. Notably, as the load increases, there is a proportional rise in brake power, contributing to an overall increase in BTE. BSFC is an indicator of the fuel efficiency in the engine as it quantified the proposition of fuel used to produce unit power. Figure 8 illustrates that with increasing load, brake-specific fuel consumption (BSFC) decreases, highlighting a more efficient fuel consumption pattern. In the idle range, BSFC is higher due to lower engine heat and inadequate fuel combustion, while transitioning to cruising and power ranges sees a reduction in BSFC due to increasing engine heat. Consistently, biodiesel exhibits higher BSFC values than diesel, impacting overall engine efficiency. The graph for sample 1 reveals that an elevated blend ratio correlates with an increase in BSFC, primarily attributed to poor combustion resulting from improper atomization caused by higher viscosity. This emphasizes the critical role of fuel composition in influencing both BSFC and BTE in internal combustion engines. The blend sample B100 shows 1.45 kg/kWh of fuel consumption at no load condition. Whereas, neat diesel has 1.32 kg/kWh of specific fuel consumption at

idle conditions. The remaining samples fall within the zone of 1.45 kg/kWh to 1.32 kg/kWh in an order of B80, B60, B40, and B20, respectively, at zero load conditions. As the load increases, brake-specific fuel consumption gets reduced due to improved engine combustion at a higher temperature as shown in Fig. 8. At higher temperatures, fine atomization of fuel takes place, which results in improving combustion efficiency. Similarly, for sample 2, the BSFC at no load condition for the B 100 sample is 1.4 kg/kWh, and the remaining samples, such as B80, B60, B40, B20, and D100, fall below the value of 1.4 kg/kWh. Compared to sample 1, the fuel consumption of sample 2 is slightly superior. This is due to the variation in fuel properties between the two samples.

Table 8. Physico-chemical properties of Sample 1 and 2 crude oils and their FAME

| Property Specification-Testing Protocol	ASTM Biodiesel Stand. 6751-02	Crude Sample-1 Oil	Biodiesel Sample-1 Methyl Ester	Diesel	Crude Sample-2 Oil	Biodiesel Sample-2 Methyl Ester
"Density at 15°C – D1298"	880 kg/m³	926	851	840	935	855
"Kinematic viscosity at 40°C – D445"	1.9 mm²/s - 6.0 mm²/s	44	5.25	3.72	43	5.18
"Calorific value – D240"	(MJ/kg)	37415	37850	42500	38130	38800
"Flash point – D93"	Min. 130 °C	245	210	50	223	189
"Cloud point – D2500"	-3°C to -12°C	15	6	-7	15	6
"Pour point – D97"	-15°C to 10°C	8	4	-15	8	4
"Cetane number – D613"	47 min.	29	51	49.7	31	53
"Acid value – D664"	Max.0.80 mg NaOH/g	11	0.132	0.052	12	0.15
"Carbon residue – D4530"	Max. 0.05 (% mass)	0.510	0.11	0.821	0.562	0.15
"Sulphur content – D5453"	Max.0.05 (% m/m)	0.015	0.04	0.08	0.017	0.06
"Ash content – D0482"	Max. 0.05 (% mass)	0.0625	0.012	0.08	0.06	0.015
"Distillation temperature 90% - D1160"	Max. 360 °C at 95% distilled	-	330	370	-	310
"Copper strip test – D130"	Max. No.3	1a	1a	1	1a	1a
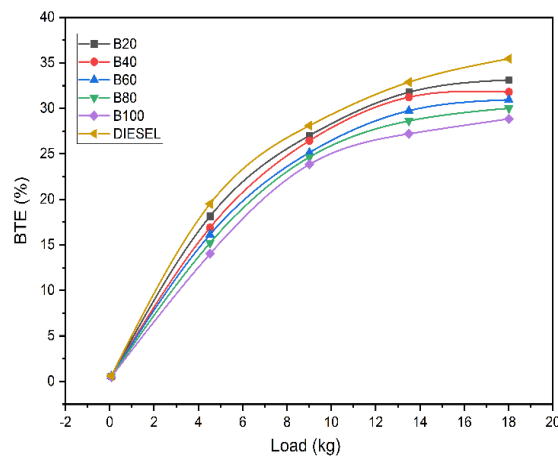
(a)
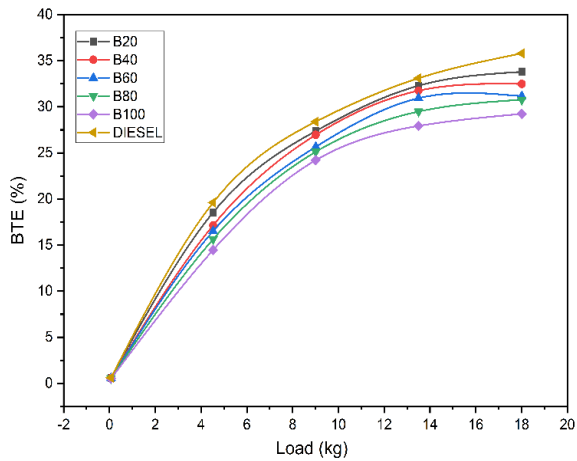
(b)

Fig. 7: The variation of the BTE at different loads for Sample 1(a) and Sample -2(b) tested in single cylinder 4-s CI engine
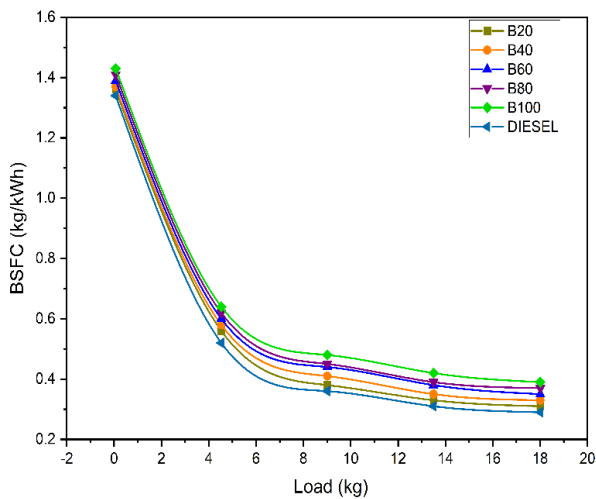
(a)
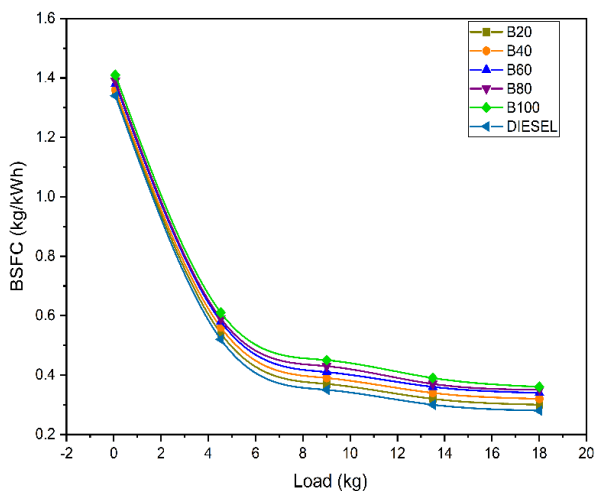
(b)

Fig 8: The variation of the BSFC at different loads for Sample 1(a) and Sample 2(b) tested in single cylinder 4-s CI engine.

5. Conclusion

The present work aims towards the preparation of biodiesel from crude samples 1 and 2. Both the samples have FFA levels less than 2%. Hence, direct base transesterification is performed. The optimization process is performed using RSM. The central composite design of 5 levels and 4 factors of 30 trial experiment tables is generated in ANOVA software, and those trials of experiments are conducted using the transesterification setup. For sample 1, the optimal transesterification parameters include a 6:1 molar ratio, 1.2 wt.% catalyst loading, 75 minutes of duration, and a reaction temp. of 64°C. Likewise, sample 2 demonstrates optimal parameters with a 7.5:1 methanol-oil ratio, 1 wt.% catalyst weight percentage, 60 minutes of duration, and an optimal temp. of 40°C. The resulting yields from these optimum conditions for samples 1 and 2 are 97.7% and 98.2%, respectively. The physico-chemical properties of the crude oil and fatty acid methyl ester are then calculated, encompassing density, viscosity, flash point, fire point, cloud point, copper strip corrosion, acid value, sulfur content, carbon residue, and ash content. The fatty acid methyl ester derived from samples 1 and 2 complies with ASTM D6751 standards. The kinematic viscosities for samples 1 and 2 are recorded as 5.25 and 5.18 mm²/s, respectively. When compared to diesel (CN = 49.7), samples 1 and 2 both had greater cetane numbers (i.e., 51 and 53, respectively). FAME is often less volatile. As a result, diesel and biodiesel have different flash and fire points. Both samples have flashpoint values four times greater than plain diesel, and they are both in the 200°C range. Compared to clean diesel, biofuel has greater acid values, which are about 0.13 and 0.15 mg/g in samples 1 and 2, respectively. In comparison to diesel, biodiesel has more oxygen content due to presence of alcoholic group in its chemical structure. Furthermore, for enhance the fuel properties; biodiesel was blended with the diesel in an appropriate proportion, such as 20%, 40%, 60%, 80% and, 100% of biofuel correspond to diesel. The blended biodiesel properties closely match the properties of the diesel. The prepared samples are tested in a single-cylinder, 4-S CI engine at different load conditions with a constant injection pressure of 200 bars. The value for BTE is 37% for diesel, whereas B100 has 27% and 29% for samples 1 and 2, respectively. Sample 2's brake thermal efficiency is greater than Sample 1's. The BTE for B20 blended sample 2 is 33%, but B20 blended sample 1 displays 31.5%. The blend sample, B100's BSFC, indicates a fuel consumption of 1.45 kg/kWh when there is no load. On the other hand, neat diesel's specific fuel consumption at idle is 1.32 kg/kWh. Between 1.45 kg/kWh and 1.32 kg/kWh is where the remaining samples fell. Because of improved engine combustion brought on by a higher

temperature, brake-specific fuel consumption decreases as the load increases. Higher temperatures cause fine fuel atomization, which raises the efficiency of the combustion engine. The BSFC for B100 sample-2 is 1.4 kg/kWh at no load. The B20 blend closely resembles diesel fuel, requiring no engine modifications. Experimental results show B20 has a higher cetane number and meets ASTM standards. Also, the emissions from B20 can serve as an alternative to conventional diesel. Future work includes studying long-term engine exposure to biodiesel, evaluating large-scale production costs with diverse raw materials, and conducting advanced optimization studies (e.g., ANN, Taguchi) in transesterification for stable mass production conditions.

Acknowledgements

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