COMPUTATIONAL OPTIMIZATION OF BIODIESEL SYNTHESIS FROM ALBIZIA LEBBECK SEEDS BY ACCELERATED REACTIVE EXTRACTION

FAISAL SHAHZAD

Institute of Chemistry, The Islamia University of Bahawalpur, Bahawalpur 63100, Pakistan.

SAJIDA NOUREEN

Institute of Chemistry, The Islamia University of Bahawalpur, Bahawalpur 63100, Pakistan.

IJAZ UL HAQ

Institute of Chemistry, The Islamia University of Bahawalpur, Bahawalpur 63100, Pakistan.

MUHAMMAD ARIF IBRAHIM

Department of Agronomy, The Islamia University of Bahawalpur, 63100, Pakistan.

MUHAMMAD ASGHAR SHAHID

Department of Agronomy, The Islamia University of Bahawalpur, 63100, Pakistan.

## MUHAMMAD ARSHAD JAVED

Department of Plant Breeding and Genetics, Faculty of Agricultural Sciences, University of The Punjab, Lahore, Pakistan.

Abstract

Biodiesel is a renewable, alternative green fuel from sources such as vegetable oils and animal fats. In the present study, Albizia lebbeck (Albizia) seeds were used as a, low cost, non-edible feedstock for biodiesel production. This study aimed to employ the Accelerated reactive extraction (solvent-assisted in situ transesterification) approach for biodiesel synthesis, which involves simultaneous solvent extraction and transesterification in single step. In current study, dichloromethane (DCM) was used as a co-solvent to increase the miscibility of oil in methanol because methanol is a poor solvent for triglycerides, leading to an increase in biodiesel yield and faster completion of the reaction. Our results demonstrate that this method is a simple and rapid method for biodiesel production. Therefore, this approach can make biodiesel synthesis economically viable, involving fewer processing steps. A statistical optimization technique based on the Box-Behnken design (BBD) of response surface methodology (RSM) was employed to achieve the maximum product yield by optimizing process parameters such as reaction time, temperature, molar ratio (methanol to seed), and catalyst amount. The results of optimization revealed that 300 minutes, 70 °C, 21:1 and 3.5% wt. of conc. H_2SO_4 were optimized process parameters under which about 98% biodiesel yield could be achieved. The analysis of variance results showed that reaction time, temperature, Catalyst concentration, interaction of temperature-time and time-molar ratio were the most significant parameters. Gas chromatography (GC-FID) results indicated that the major fatty acids in ALO were Linoleic acid (42%) and oleic acid (27%).

Keywords: Albizia lebbeck, Reactive extraction, solvent-assisted, Response surface methodology (RSM), Non edible, Biodiesel
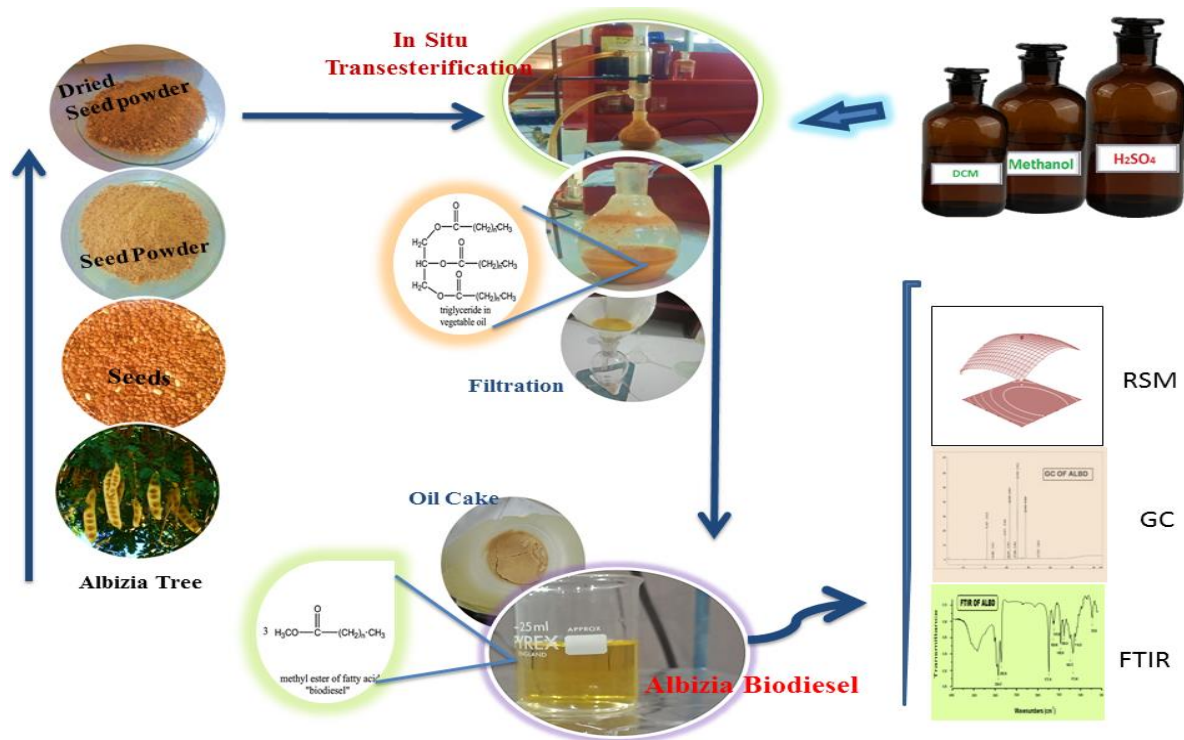
**Figure 1: Schematic representation of current research work**

1. INTRODUCTION

Energy is a fundamental human necessity to power the energy generation sector, facilitate industrial operations, and sustain the transportation system. Over 88% of our energy needs are met using fossil fuel reserves [1]. However, the surging demand for fossil fuels and their limited availability has substantially increased their costs. Furthermore the destructive impact of fossil fuels (often referred as `dirty fuel`) on the environment emphasizes the researcher to probe alternative, eco-friendly, renewable and sustainable fuels which are the only viable and long term solution to ensure future energy security [2-6]. In this context, biomass-derived biofuels are gaining a notable surge of interest as a viable option to replace fossil fuels. Consequently, biodiesel obtained from vegetable oils or animal fats has gained substantial acceptance as a viable alternative to conventional petro-diesel [7-9]. Biodiesel is classified as a liquid biofuel, and is essentially composed of fatty acid methyl esters (FAMEs) which are mono-alkyl esters of long chains of both saturated and unsaturated fatty acids, typically derived from vegetable oils or animal fats (Triglycerides). The physicochemical properties of biodiesel are also closely related to those of traditional petro-diesel; therefore, biodiesel can be used in diesel engines without significant modifications [10-14]. Biodiesel is commonly produced from vegetable oil using the transesterification reaction, which is catalyzed by either homogeneous (Acidic or basic) or heterogeneous (acidic or basic) catalyst and involve the reaction with a monohydric alcohol. After transesterification, triglycerides are converted into glycerol (a by-product) and biodiesel, which presents many striking

characteristics; it originates from biomass, biodegradable, cleaner and eco-friendly, producing less particulates, soot, sulfur oxides, hydro-carbon, and carbon monoxide [15-17].

The conventional form of transesterification is a multistep technique, involving oil extraction, purification, alcohlysis reaction, and phase separation (separation of glycerol from biodiesel). Due to many processing steps, the cost of biodiesel increases significantly and yield decreases. Therefore, there is a need for the development of an innovative biodiesel production method that is simple, compact, cost-effective, and consumes less energy [18]. Reactive extraction, also known as in situ transesterification, offers an alternative approach to biodiesel production. The initial investigation of this concept was carried out by Harrington and D'arcy-Evans [19]. It involves the direct reaction of alcohol with oil-containing material such as plant seeds, instead of reacting with pre-extracted oil [20, 21]. In essence, oil extraction and transesterification take place in situ in a single step, with alcohol serving as both the extraction solvent and transesterification reagent [22, 21]. It offers many advantages, such as minimizing the quantity of solvent used and reducing processing steps, time and cost [20].

Currently, more than 90% of biodiesel is produced from feedstock from edible plant seeds such as sunflower oil, rapeseed oil, soybean oil and groundnut oil [23]. In fact, using edible oil for biodiesel production may raise controversy about food security for human consumption. In this context, recent research has focused on producing biodiesel from non-edible feedstock such as Jatropha curcas, Azadiracta indica, and Pongamia pinnata [24-27] and novel non-edible feedstock option are still being explored. In the present study, we used novel Albizia lebbeck seeds for biodiesel production, and our results revealed that it could be a promising source for biodiesel. In Pakistan (Punjab), it is commonly called Shreen and is cultivated as an ornamental plant due to its pleasant appearance and medicinal value [28]. Despite the many advantages of biodiesel, the production of biodiesel from edible oil is not economically viable because the cost of edible oil constitute more than 70% of overall cost of biodiesel [29]. Therefore using inexpensive non-edible feedstock and simplifying the production process can significantly reduce the cost of biodiesel. Only a few researchers have reported biodiesel production using the in situ transesterification technique [30]. The current study reports the computational optimization using response surface methodology (RSM) of solvent-assisted acid-catalyzed in situ transesterification of Albizia lebbeck seeds. Dichloromethane (DCM) was employed as a co-solvent. The role of co-solvent is very important, as it tends to improve the miscibility of triglycerides in the alcohol, reducing the mass transfer limitations and enabling better interaction between reactants, resulting in more product formation with rapid completion of reaction. The optimization of accelerated reactive extraction parameters was carried out using response surface methodology (RSM) with a Box-Behnken Design (BBD) to attain highest possible biodiesel yield. The results of solvent-assisted in situ transesterification were compared with those of in situ transesterification using the optimum operating conditions of solvent-assisted approach [31]. To the best of our knowledge, no previous work has been conducted on biodiesel synthesis from Albizia lebbeck seeds using accelerated reactive extraction method.
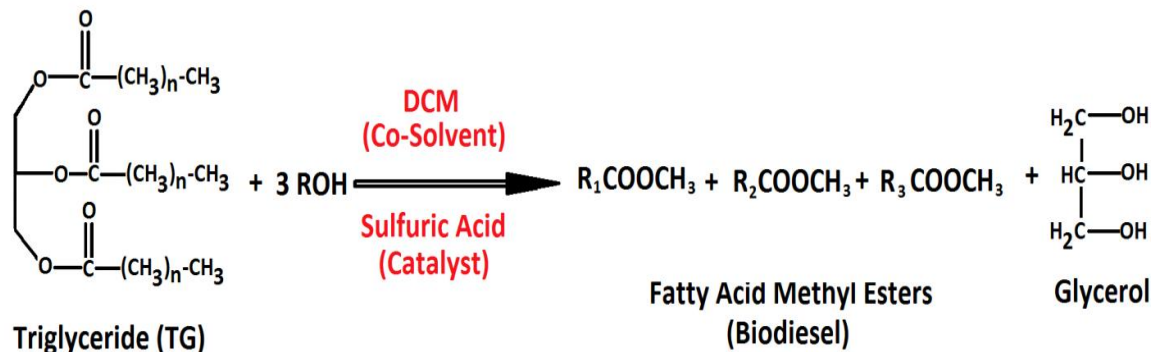
Figure 2: Solvent-assisted Acid-catalyzed Transesterification Reaction

2. EXPERIMENTAL SECTION

2.1. Materials

Analytical grade methanol (99.8%), Sulfuric acid and Dichloromethane, were purchased from Merck while acetic acid, diethyl ether and petroleum ether were purchased from sigma Aldrich. All other chemicals employed were also analytical reagent grade and were used as received without further purification. The mature seed pods of Albizia were collected from the rural areas in Bahawalpur, Pakistan and employed as the feedstock for biodiesel synthesis. The seeds were then separated from their pods. To eliminate dust, the seeds were washed with distilled water and subsequently dried in an oven at 110 °C for 6 hours to remove moisture content. The thoroughly dried seeds were finely ground into powder using a commercial grinder to increase the surface area of oil-containing cells. The powder was then sieved and dried in oven at 110°C for an hour before using for in situ transesterification **Fig. 3**.
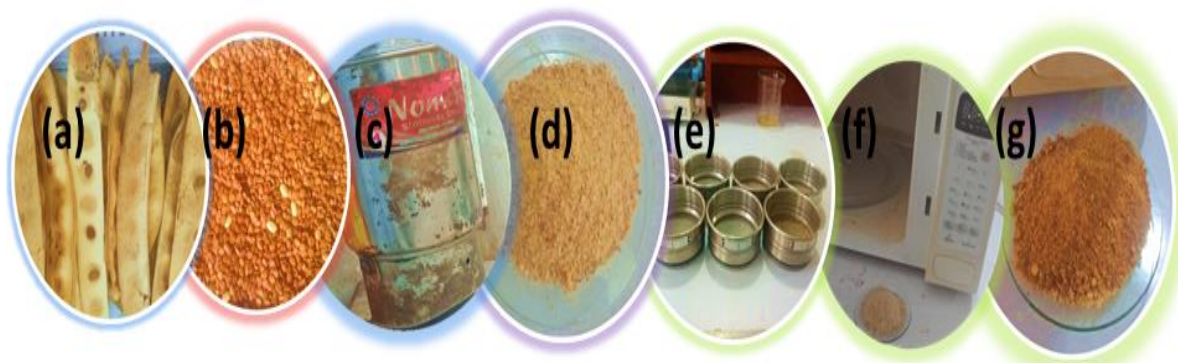
Figure 3: Collection and preparation of feedstock: (a) Albizia seed pods, (b) Albizia seeds, (c) Grinding of seeds, (d) Powdered seeds, (e) Sieving of seeds powder, (f) Drying in oven, (g) Dried seeds powder.

2.2. Oil Extraction and characterization of Albizia oil

Albizia seeds oil was extracted using the Soxhlet extraction system consisting of reflux condenser, round bottom flask (1L), and Soxhlet extractor. 50g of dried seed powder was packed in a filter paper and placed in Soxhlet extractor. Solvent n-hexane (450 mL) was employed as an extraction solvent and oil was extracted at 75 °C for 5h. After oil extraction oil was separated by using rotary evaporator at 65 °C. The weight of oil was taken and oil content was determined by using **equation (1)**, and physicochemical properties were measured according to the standard methods reported in **Table S2**.

$$\text{Oil yield (wt \%)} = \frac{\text{mass of oil extracted (g)}}{\text{mass of seeds (g)}} \times 100 \qquad (1)$$

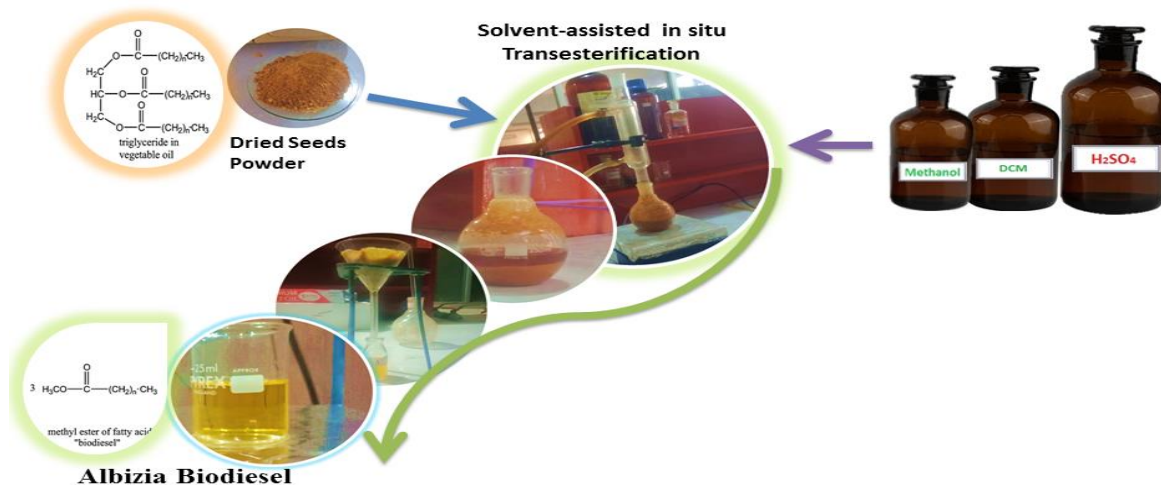## 2.3. Solvent-assisted In Situ Transesterification

The solvent-assisted in situ transesterification (Accelerated reactive extraction) was carried out in a 250 mL round bottom flask fitted with a reflux condenser with constant stirring on a magnetic hot plate (**Fig. 4**). The reaction mixture consisted of Albizia seeds powder (15g), methanol, catalyst (concentrated H_2SO_4) and 3% (by weight of seed powder) DCM (co-solvent). The influence of parameters such as the molar ratio of methanol to oil (from 6:1 to 36:1), reaction temperature (from 40 °C to 70 °C), reaction time (from 30 min to 300 min), and catalyst amount (from 1 to 6% based on seed weight) on biodiesel yield was studied (The experimental design is presented in **Table 2**.). After the completion of reaction, the reaction mixture was filtered by using Whatmann No. 1 filter paper to separate seeds oil-cake from biodiesel. Because all the glycerol formed was adsorbed onto oil-cake, there was no need for additional glycerol separation. Finally crude biodiesel was washed with hot water to remove catalyst and beads of Sodium Sulphate were added to remove moisture from biodiesel. Biodiesel yield was calculated based on **Equation (2).** Physicochemical properties were measured as per standard methods reported in **Table S2**

$$Biodiesel\ Yield\ (wt\%) = \frac{\text{Mass of biodiesel produced (g)}}{\text{Mass of seeds taken (g)}} \times 100\% \qquad (2)$$

2.4. In Situ Transesterification

In situ transesterification of Albizia seeds was conducted at the optimum conditions of solvent-assisted in situ transesterification in the same experimental setup (**Fig. 4**). Yield was calculated by using **equation 2** and results were compared (**Figure 11**).

Figure 4: Solvent-assisted acid-catalyzed in situ transesterification or accelerated reactive-extraction

2.5. Statistical design of experiment and data collection

Optimization of Albizia biodiesel production via solvent-assisted in situ transesterification approach and to investigate the effect of various independent variables (temperature, time, catalyst concentration, and methanol to seed mole ratio) on biodiesel yield, response surface methodology (Design Expert 11) based on Box-Behnken design was used. For optimization, twenty nine random experiments were conducted (**Table 2**) using 3-level and 4-factor Box-Behnken design (**Table 1**), i.e., the effect of four independent variables with minimum and maximum ranges.

Table 1: experimental range and levels of the independent variable

Factors	Symbols	Range and levels		
		Low	Medium	High
Temperature (°C)	A	40	55	70
Time (min)	B	30	165	300
Catalyst Concentration (wt. %)	C	1	3.5	6
Molar ratio (methanol to seed)	D	1:6	1:21	1:36

Table 2: The Box-Behnken design for optimizing solvent-assisted acid-catalyzed in situ transesterification

Run	Factors				Response values %		
	A (Temperature of reaction)	*B (Time of reaction)*	*C (Catalyst concentration)*	*D (Molar ratio)*	*Experimental*	*Predicted*	*Residual*
1	55	165	3.5	221 21	97	96.6	0.38
2	55	165	1	6	95.5	94.95	0.54
3	55	165	3.5	21	96.7	96.62	0.08
4	55	165	6	6	96.7	96.05	0.64
5	40	165	6	21	91.5	91.82	-0.32
6	40	165	3.5	6	92	92.51	-0.50
7	55	300	6	21	96	96.41	-0.40
8	55	165	6	36	94.5	94.00	0.49
9	55	30	3.5	36	93.5	94.14	-0.63
10	55	300	3.5	36	93.5	93.27	0.22
11	70	165	1	21	93.5	93.74	-0.23
12	70	165	6	21	94.5	94.94	-0.43
13	70	300	3.5	21	98	97.90	0.09
14	55	30	3.5	6	92	92.79	-0.78
15	55	165	3.5	21	97	96.62	0.38
16	40	300	3.5	21	90	89.49	0.51
17	55	300	1	21	95	95.51	-0.50
18	55	300	3.5	6	97.5	97.42	0.07
19	70	165	3.5	36	93.5	93.47	0.05
20	70	165	3.5	6	94	93.97	0.02
21	55	30	6	21	94.1	94.08	0.02
22	70	30	3.5	21	90.5	89.97	0.52
23	40	165	1	21	92	92.12	-0.12
24	55	165	1	36	94.6	94.20	0.39
25	55	30	1	21	94	94.08	-0.07
26	55	165	3.5	21	96.2	96.62	-0.42
27	40	165	3.5	36	89.7	90.21	-0.50
28	55	165	3.5	21	96.2	96.62	-0.42
29	40	30	3.5	21	94.6	93.65	0.94

2.6. Instrumental characterization

After separation and purification of Albizia biodiesel the extent of transformation from triglycerides to methyl esters was analyzed by using gas chromatography (GC-FID). The EN14103-standard procedure was employed to quantify desired product (fatty acid methyl esters), which was found to be 96.9% (**Fig. S5**). Fourier transform infrared spectroscopy (FTIR) was used to detect the principal functional groups and change in functional groups that occur during transesterification reaction. The infrared spectra of both Albizia oil and its corresponding biodiesel were measured from 400 to 4000 cm⁻¹ by using a Bruker alpha FTIR spectrophotometer. The fatty acid composition of Albizia biodiesel was determined by using a gas chromatograph system manufactured by Agilent Technologies 7890A model, equipped with a flame ionization detector (FID) and capillary column (30 m, ID 0.3 m and 0.25 μm). Nitrogen was used as a carrier gas with a flow rate of 1.5mL / min while oven temperature was kept at 230 °C (5 °C / min). Albizia biodiesel

sample was diluted with n-hexane solvent and its 1 µL was injected into GC-FID for analysis.

2.7. Thin-Layer Chromatography

The Thin layer chromatography (TLC) technique was used for qualitative analysis of Product (fatty acid methyl esters), after the completion of reaction. TLC analysis was conducted by using a silica gel 60 F_{254} plate and solvent system consisting of petroleum ether: diethyl ether: acetic acid (8:2.5:0.5). After elution TLC plate was dried at 100 °C and then kept in iodine chamber to color the component spots.

3. RESULTS AND DISCUSSION

3.1. Determination and validation of statistical model

The experimental data was subjected to Statistical analysis using the Design-Expert software indicated a quadratic response surface model (regression model). The regression model generated from this analysis is presented as the coded second-order polynomial **equation (3)**, which was used to investigate correlation between four independent variables and the output response (Biodiesel yield) and to predict values of output response in term of biodiesel yield.

$$\text{Yield} = 96.62 + 1.18A + 0.9417B + 0.2250C - 0.7000D + 3.03AB + 0.3750AC + 0.4500AD$$

$$+ 0.2250BC - 1.37BD - 0.3250CD - 2.86A^2 - 1.007B^2 - 0.6017C^2 - 1.21D^2 \qquad (3)$$

Yield represents biodiesel yield in percent (%) A is the temperature, B is the reaction time, C is the catalyst concentration, and D is the methanol to oil molar ratio. Each positive term in **equation (3)** denotes a beneficial combined effect that enhances biodiesel yield; conversely, negative terms within the equation indicate an opposing effect that lowers the biodiesel yield.

3.2. Analysis of Variance (ANOVA)

An analysis of variance (ANOVA) was carried out for the comparison of experimental data with the predicted model, is presented in **Table 3**, which shows that the probability value for the model is less than 0.0001 and a higher F-value (24.46) while F-value for lack of fit is 3.06 that is insignificant (p-value = 0.1459), which confirmed the best fitness of the model to the experimental data and therefore the model is statistically quite significant. Analysis of variance shows that out of four independent variables, temperature (A), reaction time (B), and mole ratio (D) have most significant effect on biodiesel yield, as indicated by the lower p- value, which is less than 0.05. The coefficient of determination (R2) value is 0.9607, which was very close to unity.

The predicted R^2 is 0.7928, which is very close to the adjusted R^2 0.9214; i.e., the difference is less than 0.2. This data supports the suggestion that experimental data fit well in predicted model. The proposed model's accuracy was checked using parameters P-value, F-value, R^2 and lack of fit the experimental data. The significance of each coefficient and the interaction of each independent variable were checked by using p-

values (**Table 3**). The smaller P-value (>0.05) indicates the significant of model term. For model validation, the lack of fit must not be significant. In addition, **Fig. 5** shows the plot of experimental and predicted biodiesel yield and shows that all data points are very close to the line of best fit, which means there is significant correlation between the predicted and experimentally calculated biodiesel yield. ANOVA (**Table 3**) reveals that temperature (A), reaction time (B), and molar ratio (D) are the most significant process parameters that control biodiesel yield .While the interaction effects of temperature with methanol to oil mole ratio (AD) and reaction time with molar ratio (BD) and the quadratic terms A^2, B^2, C^2 and D^2 are also significant (p-value <0.05).

Table 3: Analysis of variance (ANOVA) for experimental results of the Box-Behnken design

. Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	137.27	14	9.80	24.46	< 0.0001	significant
A-Temperature	16.80	1	16.80	41.92	< 0.0001	significant
B-Time	10.64	1	10.64	26.54	0.0001	significant
C-Catalyst Conc.	0.6075	1	0.6075	1.52	0.2386	not significant
D-Mole Ratio	5.88	1	5.88	14.67	0.0018	significant
AB	36.60	1	36.60	91.31	< 0.0001	significant
AC	0.5625	1	0.5625	1.40	0.2559	not significant
AD	0.8100	1	0.8100	2.02	0.1771	not significant
BC	0.2025	1	0.2025	0.5052	0.4889	not significant
BD	7.56	1	7.56	18.87	0.0007	significant
CD	0.4225	1	0.4225	1.05	0.3220	not significant
A²	53.21	1	53.21	132.74	< 0.0001	significant
B²	6.51	1	6.51	16.24	0.0012	significant
C²	2.35	1	2.35	5.86	0.0297	significant
D²	9.56	1	9.56	23.85	0.0002	significant
Residual	5.61	14	0.4009			
Lack of Fit	4.96	10	0.4964	3.06	0.1459	not significant
Pure Error	0.6480	4	0.1620			
Cor Total	142.88	28				
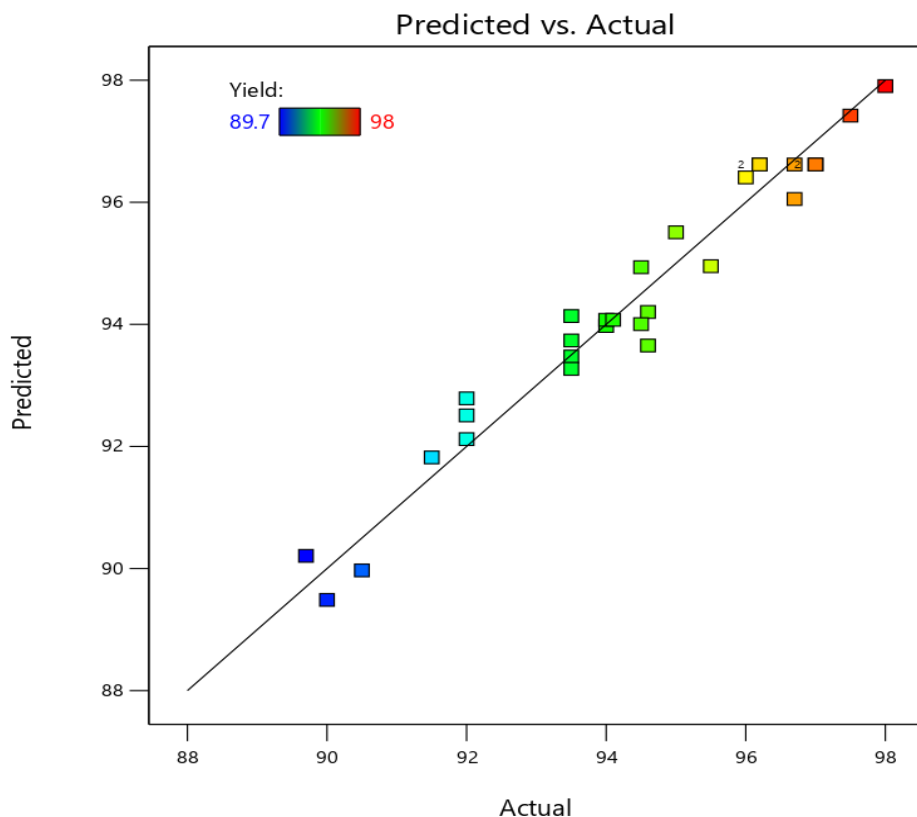
Figure 5: Predicted yields of biodiesel versus the actual yields of biodiesel.

3.3. Response surface plots for the biodiesel yield

On the basis of **equation (3),** reaction systems can be visualized in term of 2D contour plots and 3D surface plots (**Fig. 6 and 7**). These three dimensional response surfaces illustrate the main effect of independent process parameters and combined effect of two process variables on the biodiesel yield.
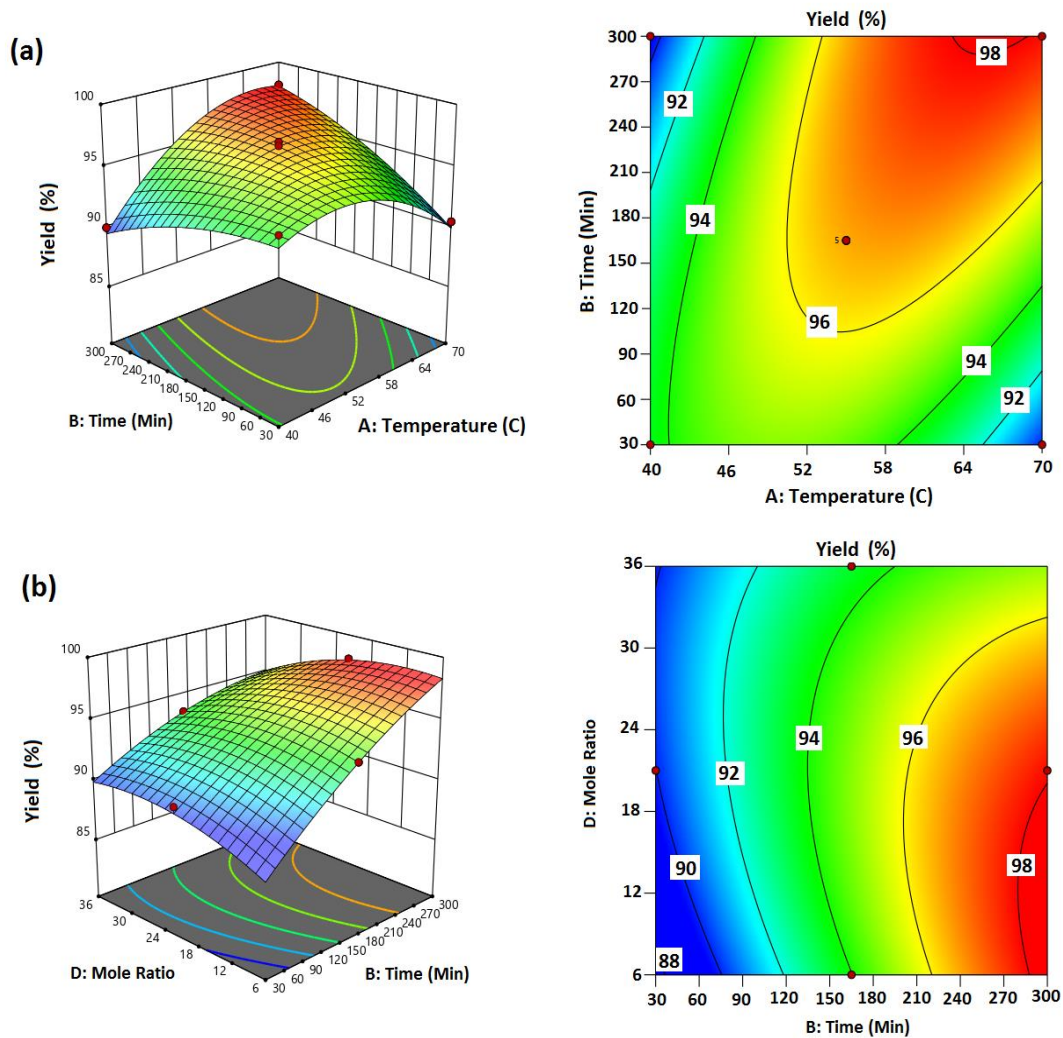
1. Figure 6a explains the interaction effect of temperature and reaction time (AB) on biodiesel yield at constant catalyst concentration and alcohol to seed mole ration. It shows that biodiesel yield is increased with the increase of reaction temperature and highest biodiesel yield (red colored zone) can be achieved at temperature 75°C and methanol to oil mole ratio at 11:1. While after 75°C temperature and mole ratio 11:1 yield began to decreases.

2. Figure 6b represents the combine effect of reaction time and mole ratio (BD) on product yield at constant temperature and catalyst concentration.

3. Figure 7a represents the combine impact of two variables such as reaction temperature and catalyst concentration (AC) on biodiesel yield when other two parameters are kept constant.

4. Figure 7b presents the collective impact reaction temperature and mole ratio (AD) on output response at constant time and catalyst concentration.

5. Figure 7c depicts joint influence of time and catalyst concentration (BC) at constant temperature and mole ratio.

6. Figure 7d shows combined effect of catalyst concentration and mole ratio (CD) on output response at constant temperature and time.

3.4. Significant interaction effect of variables on biodiesel yield

The ANOVA results presented in **Table 3** indicate that the interaction effect terms temperature-reaction time (AB) and reaction time-methanol to seed ratio (BD) are statistically significant (with p-values <0.05). This implies that these variable pairs have simultaneous impact on the biodiesel yield. 3D Surface plots with contour plots in **Figure 6a and 6b** illustrate simultaneous impact of temperature-reaction time (AB) and reaction time-methanol to seed ratio (BD) respectively on biodiesel yield. **Figure 6a** presents that at constant catalyst concentration (3.5%) and methanol to seed mole ratio (21:1) yield tends to increase with the rise of both parameters, maximum yield could be achieved at 70 °C temperatures with maximum reaction time (300 min) this is because acid catalyzed reaction is relatively slow reaction which tends to complete at higher temperature and prolong time. This increase in yield is due to the endothermic nature of transesterification reaction [32] higher reaction temperature increase or speed up the reaction rates similar results are reported in published work [33]. Moreover increase in temperature generally reduces the viscosity of oil, increasing solubility which enhances oil-methanol contact [30].

Figure 6b illustrate 3D response plots with its corresponding 2D contour plots sowing the combined effect of reaction time with methanol to seed mole ratio (BD) at constant temperature (70 °C) and catalyst concentration (3.5%). According to which with the increase in mole ratio from 6:1 to 21:1 yield tends to increase which is due to in situ transesterification reaction which required more solvent but with further increase in mole ratio yield would not increase which is might be due to the hindrance with added co-solvent (DCM) while due to slow rate of reaction of acid-catalyzed transesterification maximum yield was achieved at maximum time (300 min). Due to substantial dilution of reactants and presence of polar hydroxyl group might instigate reverse reaction causing decrease in biodiesel yield [30].

Figure 6: 3D response surface plots with 2D contour plots indicating the interaction effect of most significant reaction parameters on biodiesel yields
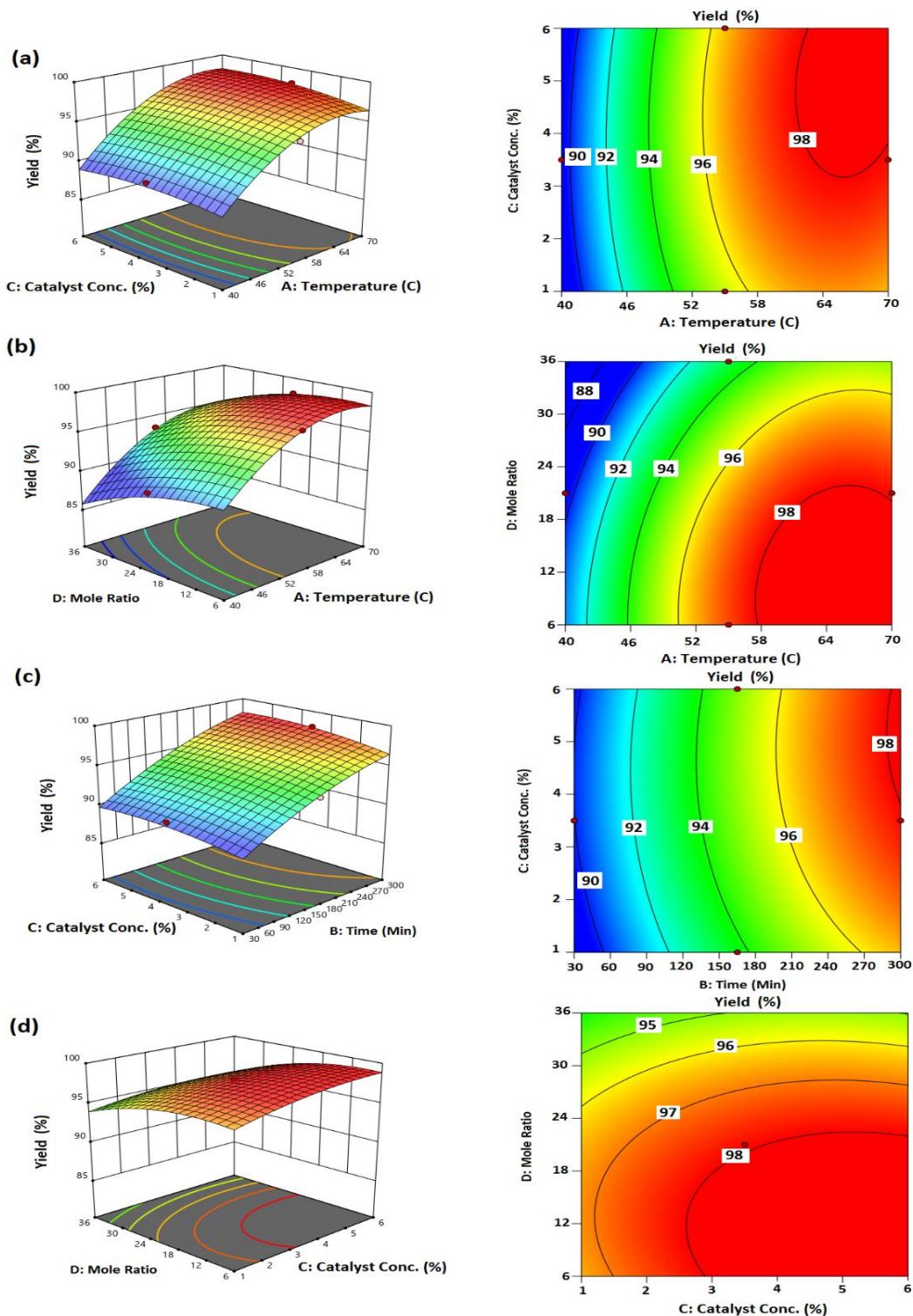
Figure 7: 3-dimensional surface plots with 2D contour plots of non-significant parameter

3.5. Optimization process using response surface methodology (RSM)

To attain the highest possible yield, the optimal values of each process variable were predicted by using **Eq. (3)**, that is relies on a regression polynomial model, derived from experimental design (BBD) of response surface methodology (RSM). According to the model the optimal values for all scrutinized variables were determined as follows: reaction temperature of 70°C, reaction time of 300 min, methanol to seed mole ratio is of 21:1, and catalyst concentration of 3.5%. Based on the regression model (**Table 2.Run 13**) the highest biodiesel yield (97.90%) can be achieved when all four process variables are at their optimal values. This predicted value was experimentally confirmed to be 98% which means there is a close agreement between the predicted values and experimental results.

3.6. Instrumental characterization

3.6.1. Fourier Transform Infrared Spectroscopy (FT-IR) analysis of biodiesel

Figure 8 presents the FTIR spectra of Albizia oil (a) and its corresponding biodiesel (b). Both spectra show remarkable similarity due to the presence of identical functional groups in Albizia oil and its biodiesel. However, minor disparities in band intensities and vibration frequencies are observed. These variations are likely a result of subtle distinction in properties of Albizia oil and its biodiesel. The most prominent peaks in oil sample (Triglycerides) are observed at 2932 cm^{-1} (sp^2 C-H stretching), 2853 cm^{-1}(sp^3 C-H stretching), and 1735 cm^{-1} (C=O stretching), 1382 cm^{-1} (C-O stretching), 1175 cm^{-1} (O-CH_3 stretching), 720 cm^{-1} (C-H bending rocking) [34]. While biodiesel spectra between 4000-2000 cm^{-1} contain almost identical peaks as in oil spectra, however certain characteristics peaks were detected between 1700-1000 cm^{-1} (at 1743 cm^{-1} , 1639 cm^{-1}, 1615 cm^{-1},and 1459 cm^{-1}). The peak at 1459 cm^{-1} indicate the presence of CH_3 group of FAME [35]. In the context of biodiesel, a prominent and sharp peak was detected at 1743 cm^{-1} which was attributed to the C=O stretching within methoxycarbonyl group. Conversely in Albizia oil spectra, a quite substantial peak at 1635 cm^{-1} was resolved into two distinct peaks at 1639 cm^{-1} and 1615 $cm^{-1.}$ This clear separation provides compelling supports for the conclusion that biodiesel has been formed [36].
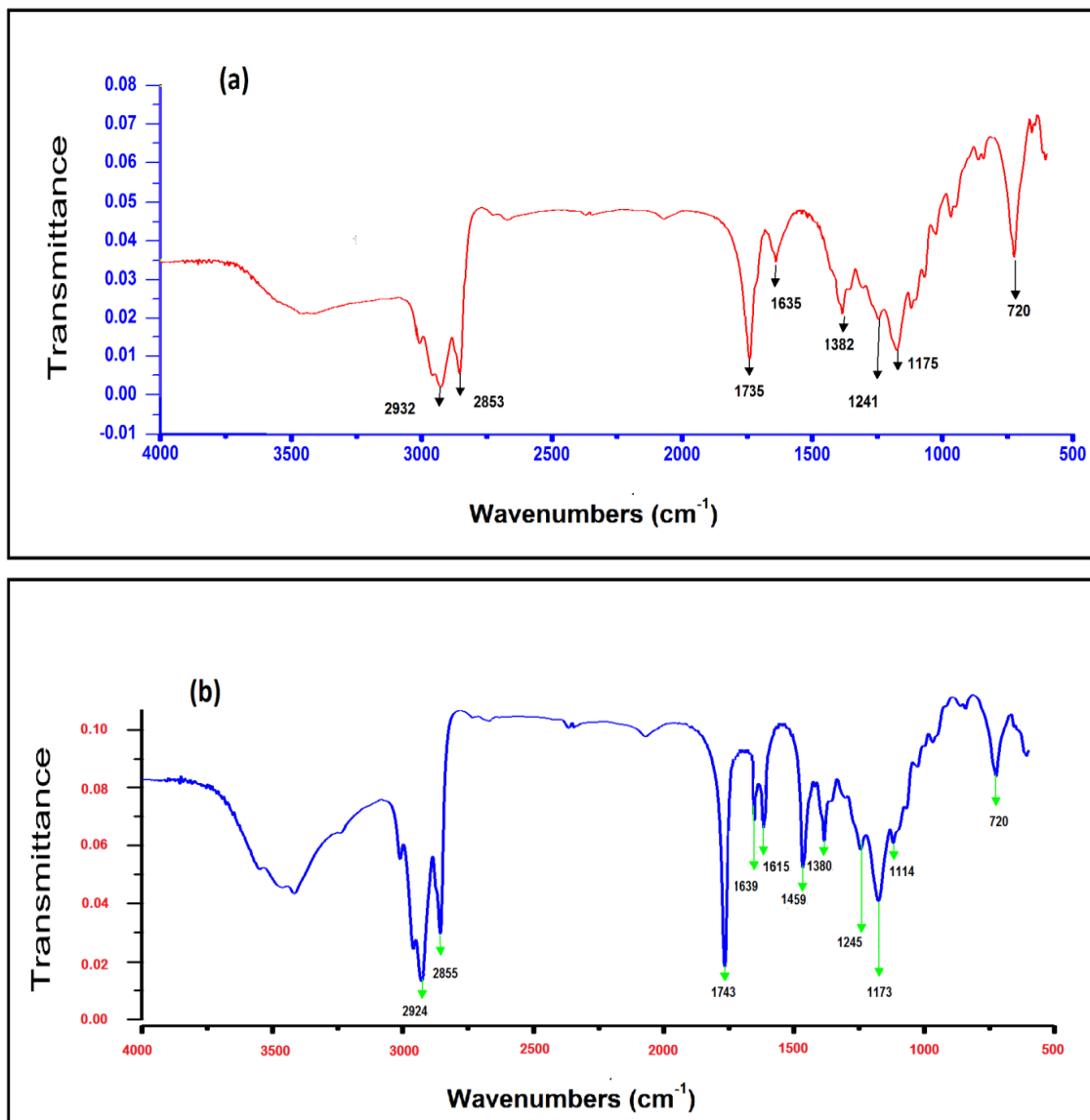
**Figure 8: FTIR of Albizia lebbeck oil (a) and Albizia biodiesel (b)**

3.6.2. Gas chromatography analysis of Albizia lebbeck seeds oil

Figure 9 present fatty acid profile of Albizia oil, it revealed that C18:2 is most abundant fatty acid (42%), which is consistent with the previously reported value. In Albizia oil the notable fatty acids were oleic acid (27%), linoleic acid (13%), Palmitic acid (7%), and stearic acid (6%), (**Table 4**). Their total content was detected to be around 95%. Despite the presence of substantial amount of unsaturated fatty acids our results revealed its promising suitability as a biodiesel feedstock. 75% of total content was comprised of

unsaturated fatty acids and 24% was saturated fatty acids and our findings are in good agreement with previous reported work [37].

Table 4: Main fatty acids identified in the oil w/w. %

Fatty Acids	Systematic name	Structure	Mass%	Type of fatty acid
Linoleic acid	Cis-9, Cis-12 Octadecadienoic	C18:2	42	unsaturated
Oleic acid	Cis-9-Octadecanoic	C18:1	27	unsaturated
Palmitic acid	Hexadecanoic	C16:0	7	saturated
Linolenic acid		C18:3	13	unsaturated
Stearic acid	Octadecanoic	C18:0	6	saturated
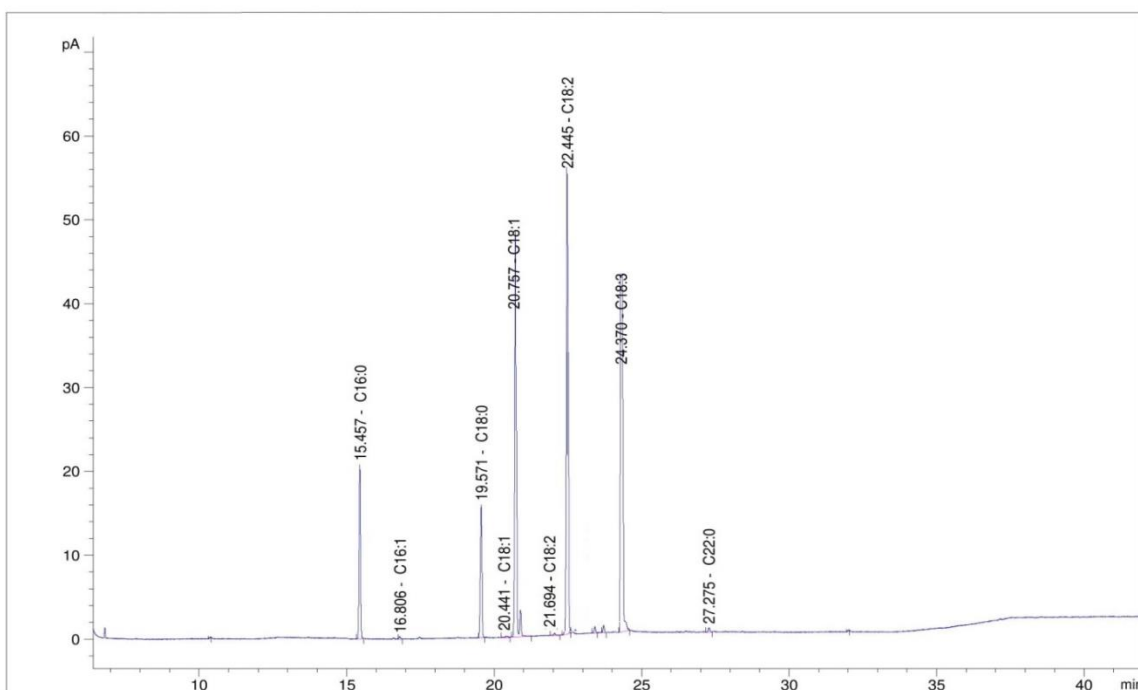
Figure 9: Gas chromatogram of Albizia lebbeck biodiesel.

3.7. Thin-Layer Chromatographic analysis

Figure 10a illustrate the results of thin layer chromatography of Albizia oil, reveal four separate spots corresponding to monoglycerides (MGs), diglycerides (DGs), free fatty acids (FFAs) and triglycerides (TGs). Due to the elevated concentration of TGs, the spot corresponding to them appeared larger and darker. **Figure 10b** presents the results of Albizia biodiesel revealed a single prominent spot, serving as a clear indicator of presence of fatty acid methyl esters, which was due to the completion of reaction and purity of sample [38].
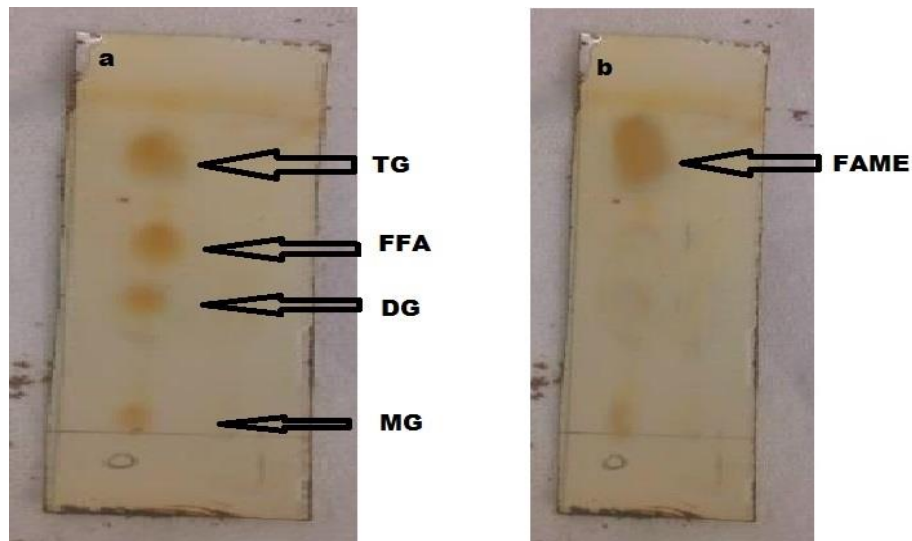
Figure 10: TLC of Albizia lebbeck oil (a) and its biodiesel (b)

3.8. Physicochemical characterization

Physicochemical properties for ALO such as kinematic viscosity, density, free fatty acid content and iodine value were determined and are shown in Table 1
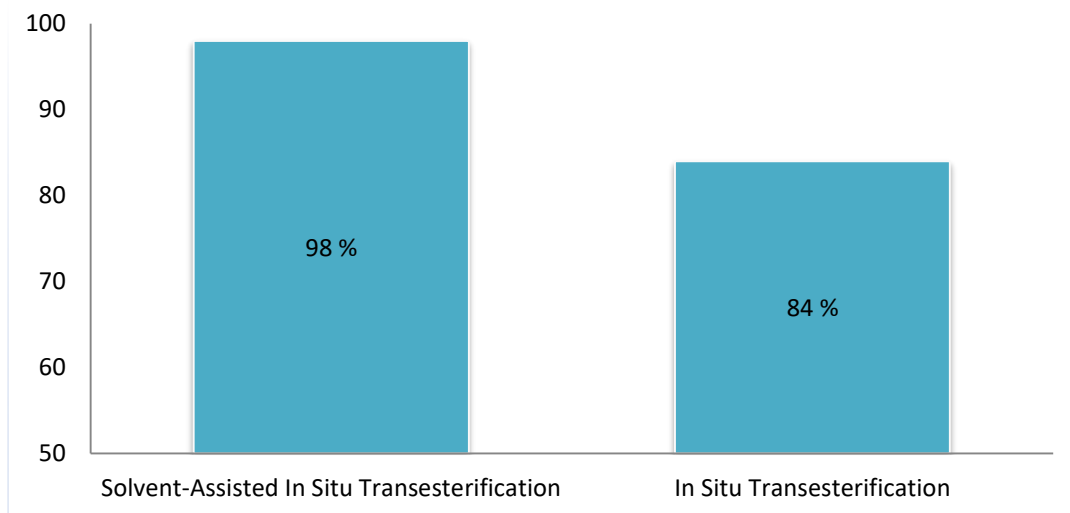

Figure 11: Comparison of biodiesel yield on solvent-assisted In situ transesterification and In situ transesterification

CONCLUSION

In this study, acid-catalyzed solvent-assisted in situ transesterification of novel Albizia lebbeck seeds were carried out and to attain maximum yield of biodiesel, process parameters such as temperature, time, catalyst concentration, and methanol to seed mole ratio were optimized by using RSM based Box-Behnken design of experiment. The RSM

established the optimal process parameters were 70°C of temperature, 300 min of time, 21:1 of molar ratio and 3.5% of catalyst concentration. Analysis of variance presented that temperature, time and mole ratio were most significant process parameters. Results of our study revealed that it could a promising approach for the production of biodiesel that can minimize processing steps and cost with excellent yield (98%). Moreover in situ transesterification was carried out at the optimum conditions of solvent-assisted in situ transesterification and result were compared (Figure 11) which shows without solvent in situ transesterification gives lower yield (84%) hence solvent-assisted approach is superior technique because solvent (DCM) can improve miscibility of triglycerides in methanol and overcome mass transfer limitation which lead to substantial increase in yield. The fatty acid composition of oil was determined by GC-FID, and according to which the unsaturated fatty acid content was 75%.

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