Ibn Al-Haitham Journal for Pure and Applied Sciences

Journal homepage: http://jih.uobaghdad.edu.iq/index.php/j/index

Optimization of Process Parameters for Biodiesel Production from Three Indigenous Vegetable Oils

[1]Aliru Olajide Mustapha
aliru.mustapha@kwasu.edu.ng

[2]Kudirat Afolashade Usman
usmankudirat1995@gmail.com

[3]Toheeba Aramide Zakariyah
toheebahzakariyah98@yahoo.com

[4]Oluwaseyi Mojibade Adekoya
adekoyaoluwaseyi656@gmail.com

[5]Joseph Phillip Tosin
joephil20166@gmail.com

[6]Jesutomisin Oladele
oladelejesutomisin@gmail.com

[7]Toluwalase Solomon Amao
amaotoluwalasesolomon@gmail.com

[8]Yemisi Tokunbo Afolabi
ytokunbo.afolabi@gmail.com

[1-8]Department of Chemical, Geological & Physical Sciences, College of Pure and Applied Sciences, Kwara State University Malete, PMB 1530, Ilorin, Kwara State, Nigeria.

Article history: Received 6 February 2022, Accepted 17 April, 2022, Published in July 2022.

Doi: 10.30526/35.3.2810

Abstract

Optimization procedures using a variety of input parameters have gotten a lot of attention, but using three non-edible seed oils of Jatropha (*Jatropha curcas*), Sesame (*Sesamum indicum*), and Sweet Almond (*Prunusamygdalus dulcis*) has a few advantages, including availability and non-food competitiveness. Optimizing a two-stage trans-esterification process using a sodium hydroxide-based catalyst at a fixed catalyst (1.0wt %) and temperature (60 ^oC) while varying molar ratio (1:3, 1:6, 1:12), time (20–60 min), and mixing speed (500–1000 rpm), to produce optimal responses of yields were studied using response surface methodology (RSM). The optimization solution of molar ratio (1:3), time (40.9 min.), and speed (500 rpm) resulted in an 86.9 % for refined jatropha biodiesel (RJB), the optimization for refined sesame biodiesel (RJB) with molar ratio (1:6), time (41.7 min.), and speed (619 rpm) resulted in an 88.5 %, and the optimization for refined sweet almond biodiesel (RSAB) with the molar ratio (1:3), time (49.359 min.), and speed (500 rpm) resulted in an 88.7 % at the conditions. RJO, RJB, and RSAB had predicted biodiesel yields of 86.9 %, 88.5 %, and 88.7 %, with less than 0.2 % variation, respectively. The characteristics of biodiesel were studied, and the results were determined to meet both ASTM D6751 and EN14214 criteria. The effects of molar ratio, and time on biodiesel yield from their respective oils were important parameters that greatly influenced the yields, but speed only changed the yields marginally. This work has addressed important difficulties influencing mass production of biodiesel such as the utilization of low-cost feedstock such as non-edible vegetable oils, boosting production efficiency through variable optimization of process parameters, and lowering catalyst dosages through catalyst regeneration.

Keywords: vegetable-oils; biodiesel, optimization, yield; non-edible .

1. Introduction

Several important variables influence the transesterification reaction. To achieve the largest biodiesel production, these variables must be at their peak since the rate of reaction is influenced by the reaction temperature. A higher reaction temperature can reduce the viscosity of oils, increasing the reaction rate as more energy was supplied for the reaction to occur. The reaction temperature must be lower than the alcohol's boiling point (60–70 oC at normal atmospheric pressure for methanol) to prevent the alcohol from vaporizing. As a result, raising the reaction temperature over its optimum range reduces biodiesel yield since the saponification reaction was quickened, resulting in less biodiesel. Temperatures between 60 and 80 ^{o}C generate the best production, depending on the type of oil [1-2]. The stoichiometric ratio of the trans-esterification reaction in 3 moles of alcohol and 1 mole of triglyceride produced 3 moles of fatty acid ester and 1 mole of glycerol. The forward reaction was more favorable, during trans-esterification, more alcohol was used to ensure that the oils were completely converted to ester [3-5].

A larger alcohol-to-triglyceride ratio can also lead to faster ester conversion. The molar ratio is strongly influenced by the type of catalyst used. In base-catalyzed biodiesel production, a molar ratio of 5:1 or 6:1 of methanol to oil is sufficient to convert Jatropha oil to biodiesel if free fatty acids after pretreatment are less than 1% [6-9]. If the fraction of free fatty acids in oils was large, a molar ratio of 20:1 or 24:1 was necessary when using acid-catalyzed trans-esterification [10-13]. The amount of catalyst used can alter the yield of biodiesel produced as basic catalysts are often favored over acid catalysts due to their better reactivity and reduced process temperature requirements [14]. Freedman *et al.* and Ifeoluwa *et al.* [15-16] discovered that sodium methoxide was more effective than sodium hydroxide due to the lesser amount of water produced when mixing sodium hydroxide with methanol. As the catalyst concentration was increased, the conversion of triglycerides and the generation of biodiesel both increased. It has been demonstrated that a concentration of NaOH in the range of 1.0–1.4 percent (w/w) converts jatropha oil to methyl ester by 90–98%. [17-19]. About 95–99 % of jatropha biodiesel has been obtained with KOH concentrations ranging from 0.55 to 2.0 percent (w/w) [20-21]. However, if the alkali catalysts were used at higher concentrations than their optimum, the generation of biodiesel was reduced because more soap was generated [22]

According to the literature, as the reaction time reduces, the conversion rate increases. Because the alcohol was blended and spread into the oil, the reaction was first delayed. After a period, the reaction picks up pace until it reaches its maximum yield. For base-catalyzed trans-esterification, the output of biodiesel peaks at 120 minutes or less [1]. Acid catalyzed trans-esterification takes much longer than base catalyzed trans-esterification because base catalysts are frequently more reactive than acid catalysts [14]. According to previous studies [12-13], the reaction time needed to convert triglycerides to biodiesel might be anywhere from 18 to 24 hours. On the other hand, excessive reaction time would limit the product yield due to the reverse reaction of trans-esterification, which causes more fatty acids to be produced in the form of soaps [20]. Before biodiesel can be developed and optimized on a large scale, many factors and difficulties must be resolved. The key issues include the utilization of low-cost feedstock such as non-edible vegetable oils, increasing production efficiency through optimization of process parameters, lowering catalyst costs through catalyst regeneration, and the optimization of process parameters to maximize the biodiesel yield. This work seeks to address these issues.

2. Materials and Procedures

The three seeds of Jatropha (Jatropha curcas), sesame (Sesamum indicum) and sweet almond (Prunusamygdalus dulcis), and were collected in Ilorin markets in Kwara State, Nigeria. Sigma Aldrich provided the chemicals and equipment (Gillingham, Dorset, UK). Cold oil extraction was used to extract refined Jatropha oil (RJO), Sweet Almond oil (RSAO), and sesame oil (CSO) from crude Jatropha oil (CJO), Sweet Almond oil (CSAO), and sesame oil (CSO) (RSO). Following refinement, the oils were transesterified to obtain refined jatropha biodiesel (RJB), Sweet Almond biodiesel (RSAB), and sesame biodiesel (RSB) using the two-step method recommended by the American Standard for Testing Materials, the Association of Official Analytical Chemists and Mustapha et al. [22-26].

2.1The Response Surface Method for Optimization of Biodiesel

The Response Surface Method (RSM) is a technique for calculating the number of parameters it takes for optimal response. Correlations between independent and response variables are established using the RSM approach. Although Box and Wilson [27] were the first to create a model or optimal response using experimental data, various techniques to process optimization have expanded its practical use. The p-value for each of the models can be determined using ANOVA. When the values were less than 0.05, the 0.05 p-value for most process variables was favorable, indicating that model terms were significant. Design Expert II was chosen as the statistical tool because it includes the three minimal categories of input and response variables, as well as anticipated and experimental values, which are required for the adequacy assessment.

2.2. Design of Experiments

To produce reliable ANOVA models, the RSM must create a design of experiments (DoE) using the smallest amount of data possible. Because Box–Behnken Design (BBD) designs do not contain axial points, all design points must fall between operating restrictions, and a design matrix (inputs) must be constructed using a BBD. It necessitates a decrease in the number of treatment options. The input components (molar ratio, time and speed) in fixed catalyst and temperature were chosen in a variety of combinations to give yield as an output. A fixed sodium hydoxide dose of 1.0 wt. %, a molar ratio of 1:3, 1:6, 1:12, and a temperature of 60 ºC were randomly tuned with variable time (20, 40, 60 min), and speed (500, 750, 1000 rpm) [26].

Table1. Design levels with multiple independent variables.

Independent factors to production	
Molar ratio	1:3, 1:6, 1:12
NaOH (%)	1
Speed (rpm)	500, 750, 1000
Temperature (ºC)	60
Time (min)	20, 40, 60

3. Biodiesel optimization test matrices were developed using a fixed sodium hydroxide dose and time.

3.1. Biodiesel derived from refined jatropha biodiesel (RJB)

Table 2. Experimental matrix with a variety of molar ratios, times, and speeds

	Factor 1	Factor 2	Factor 3	Response	
Run	A:Molar ratio	B:Time	C:Speed	Yield	(%)
		s	rpm	Actual	Predicted
1	7.5	45	750	80.00	80.00
2	7.5	45	750	80.00	80.00
3	12	60	750	73.30	75.64
4	7.5	60	500	86.67	82.34
5	12	45	1000	96.00	89.32
6	7.5	45	750	80.00	80.00
7	7.5	30	1000	66.70	71.03
8	3	60	750	80.00	77.66
9	3	45	500	82.67	89.35
10	12	45	500	85.30	87.30
11	7.5	45	750	80.00	80.00
12	12	30	750	73.30	75.65
13	3	30	750	66.70	64.36
14	7.5	60	1000	73.30	77.64
15	7.5	45	750	80.00	80.00
16	7.5	30	500	80.00	75.66
17	3	45	1000	80.00	78.00

Based on the three levels of inputs, the Design Expert program generated the most number of runs possible. **Figure 1** depicts the link between the actual values acquired experimentally (**Table 2**) and the yield values predicted by various models.

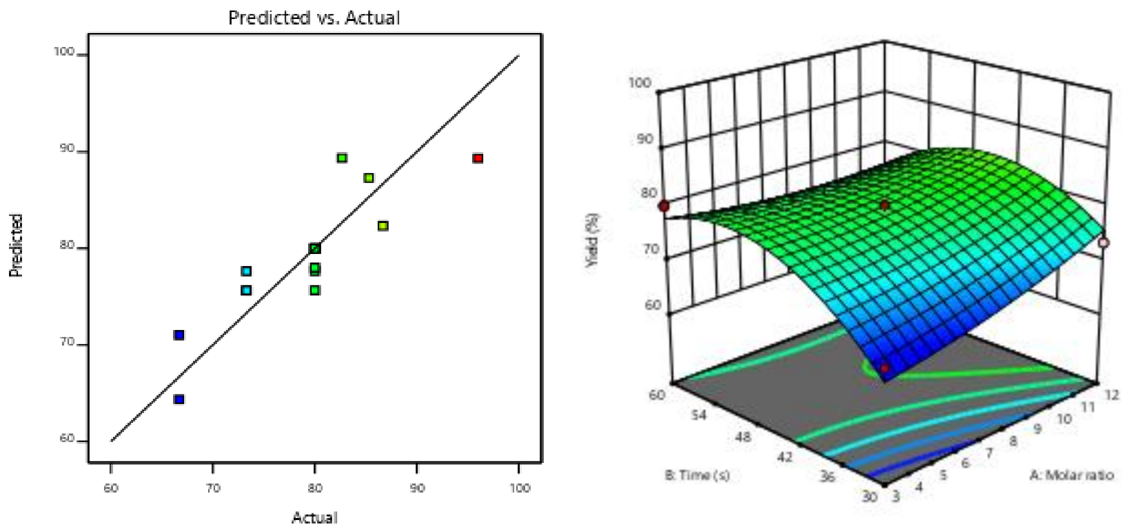

Figure 1. shows a scatter diagram with the 3D surfaces that correspond to it.

The Variance Analysis (ANOVA)

The equation represents the second polynomial functions in terms of the actual components used to describe yield

In terms of actual factors, the following is the final equation:

$$\text{Yield} = +46.78389 - 0.478426\,\text{Molar ratio} + 3.79436\,\text{Time} - 0.143413\,\text{Speed} - 0.049259\,\text{Molar ratio} * \text{Time} + 0.002971\,\text{Molar ratio} * \text{Speed} - 4.66667E\text{-}06\,\text{Time} * \text{Speed} + 0.065432\,\text{Molar ratio}^2 - 0.035556\,\text{Time}^2 + 0.000075\,\text{Speed}^2 \quad (1)$$

Table 3.ANOVA Quadratic model "RJB Yield"

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	614.62	9	68.29	2.46	0.1243 not significant
A-Molar ratio	42.92	1	42.92	1.55	0.2538
B-Time	88.25	1	88.25	3.18	0.1178
C-Speed	43.43	1	43.43	1.56	0.2512
AB	44.22	1	44.22	1.59	0.2474
AC	44.69	1	44.69	1.61	0.2451
BC	0.0012	1	0.0012	0.0000	0.9949
A^2	7.39	1	7.39	0.2662	0.6218
B^2	269.47	1	269.47	9.71	0.0170
C^2	91.73	1	91.73	3.30	0.1120
Residual	194.36	7	27.77		
Lack of Fit	194.36	3	64.79		
Pure Error	0.0000	4	0.0000		
Cor Total	808.99	16			

Table 4: Constraints for RJB biodiesel optimization

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Molar ratio	minimize	3	12	1	1	3
B:Time	minimize	30	60	1	1	3
C:Speed	minimize	500	1000	1	1	3
Yield	maximize	66.7	96	1	1	3

Table 5. Results discovered based on the RSAB biodiesel optimization scenario

Number	Molar ratio	Time	Speed	Yield	Desirability	
1	**3.000**	**40.910**	**500.000**	**86.937**	**0.843**	**Selected**
2	3.000	41.024	500.000	87.021	0.843	
3	3.000	41.102	500.000	87.077	0.843	
4	3.000	40.609	500.000	86.712	0.843	
5	3.000	40.372	500.000	86.531	0.843	

Tables 2–4 show desirability functions for three different criteria using varied input components (molar ratio, time and speed) for constant NaOH, temperature, and the combination of processes that were examined. The optimization strategies identified based on the biodiesel optimization scenario is shown in **Table 5**. Using a fixed catalyst of 1.0 wt. %, temperature 60 ^oC and a molar ratio (1:3, 1:6, 1:12), the optimization solution with the molar ratio (1:3), time (40.910) and speed (500.00 rpm) yielded biodiesel (RJB) of 86.937 %, with the stipulated overall desirability of 0.843. Molar ratio, time, and speed were all important variables in biodiesel synthesis, according to the results of the analysis of variance (ANOVA).

3.1.2 Biodiesel derived from refined sesame biodiesel (RSB)

Table 6. Experimental matrix with a variety of molar ratios, times, and speeds

	Factor 1	Factor 2	Factor 3	Response	
Run	A:Molar ratio	B:Time	C:Speed	Yield (%)	
		s	rpm	Actual	Predicted
1	7.5	45	750	90.00	90.00
2	7.5	45	750	90.00	90.00
3	12	60	750	83.30	84.56
4	7.5	60	500	90.00	90.42
5	12	45	1000	80.00	79.14
6	7.5	45	750	90.00	90.00
7	7.5	30	1000	86.67	86.25
8	3	60	750	86.67	85.40
9	3	45	500	78.30	79.16
10	12	45	500	90.00	88.32
11	7.5	45	750	90.00	90.00
12	12	30	750	83.30	84.57
13	3	30	750	81.67	80.41
14	7.5	60	1000	83.30	82.89
15	7.5	45	750	90.00	90.00
16	7.5	30	500	81.67	82.08
17	3	45	1000	83.30	84.98

Based on the three levels of inputs, the Design Expert program generated the most number of runs possible. Figure 2 depicts the link between the actual values acquired experimentally (Table 6) and the yield values predicted by various models.

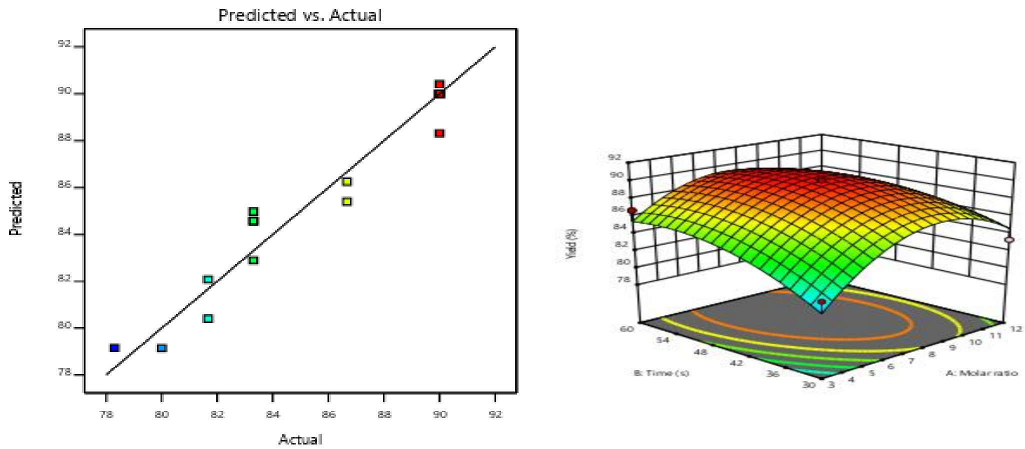

Figure 2. shows a scatter diagram with the 3D surfaces that correspond to it**.**

The Variance Analysis (ANOVA)

The equation represents the second polynomial functions in terms of the actual components used to describe yield

In terms of actual factors, the following is the final equation:

$$Yield=-17.43250+6.76833\,\text{Molar ratio}+1.55789\,\text{Time}+0.121850\,\text{Speed}-0.018519\,\text{Molar ratio} * \text{Time}-0.003333\,\text{Molar ratio} * \text{Speed}-0.000780\ \text{Time} * \text{Speed}-0.216667\,\text{Molar ratio}^2-0.008344\,\text{Time}^2-0.000043\,\text{Speed}^2 \quad (2)$$

Table 7. ANOVA Quadratic model "RSB Yield"

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	259.77	9	28.86	14.21	0.0010 significant
A-Molar ratio	5.54	1	5.54	2.73	0.1424
B-Time	12.40	1	12.40	6.11	0.0428
C-Speed	5.61	1	5.61	2.76	0.1404
AB	6.25	1	6.25	3.08	0.1228
AC	56.25	1	56.25	27.70	0.0012
BC	34.22	1	34.22	16.85	0.0045
A^2	81.05	1	81.05	39.92	0.0004
B^2	14.84	1	14.84	7.31	0.0305
C^2	30.98	1	30.98	15.26	0.0059
Residual	14.21	7	2.03		
Lack of Fit	14.21	3	4.74		
Pure Error	0.0000	4	0.0000		
Cor Total	273.99	16			

Table 8. Constraints for RSB biodiesel optimization

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Molar ratio	minimize	3	12	1	1	3
B:Time	minimize	30	60	1	1	3
C:Speed	minimize	500	1000	1	1	3
Yield	maximize	78.3	90	1	1	3

Table 9. Results discovered based on the RSB biodiesel optimization scenario

Number	Molar ratio	Time	Speed	Yield	Desirability	
1	**6.930**	**41.734**	**619.262**	**88.545**	**0.967**	**Selected**
2	6.920	41.738	621.375	88.562	0.967	

Tables 6–8 show desirability functions for three different criteria using varied input components (molar ratio, time and speed) for constant NaOH, temperature, and the combination of processes that were examined. The optimization strategies identified based on the biodiesel optimization scenario are shown in Table 6. Using a fixed catalyst of 1.0 wt. %, temperature 60 ^{o}C and a molar ratio (1:3, 1:6, 1:12), the optimization solution with the molar ratio (1:6), time (41.734) and speed (619.262 rpm) yielded biodiesel (RSB) of 88.545 %, with the stipulated overall desirability of 0.967. Molar ratio, time, and speed were all important variables in biodiesel synthesis, according to the results of the analysis of variance (ANOVA).

3.1.3 Biodiesel derived from refined sweet almond biodiesel (RSAB)

Table 10. Experimental matrix with a variety of molar ratios, times, and speeds

Factor 1		Factor 2	Factor 3	Response	
Run	A:Molar ratio	B:Time	C:Speed	Yield (%)	
		s	rpm	Actual	Predicted
1	7.5	45	750	81.40	82.63
2	7.5	45	750	81.40	82.63
3	12	60	750	90.00	79.04
4	7.5	60	500	89.50	86.13
5	12	45	1000	85.80	88.67
6	7.5	45	750	81.40	82.63
7	7.5	30	1000	88.50	90.28
8	3	60	750	85.70	83.64
9	3	45	500	82.80	86.69
10	12	45	500	64.20	76.99
11	7.5	45	750	81.40	82.63
12	12	30	750	92.80	86.62
13	3	30	750	78.50	81.22
14	7.5	60	1000	70.00	76.55
15	7.5	45	750	81.40	82.63
16	7.5	30	500	85.70	77.55
17	3	45	1000	84.20	78.17

Based on the three levels of inputs, the Design Expert program generated the most number of runs possible. **Figure 3** depicts the link between the actual values acquired experimentally (**Table 10**) and the yield values predicted by various models.

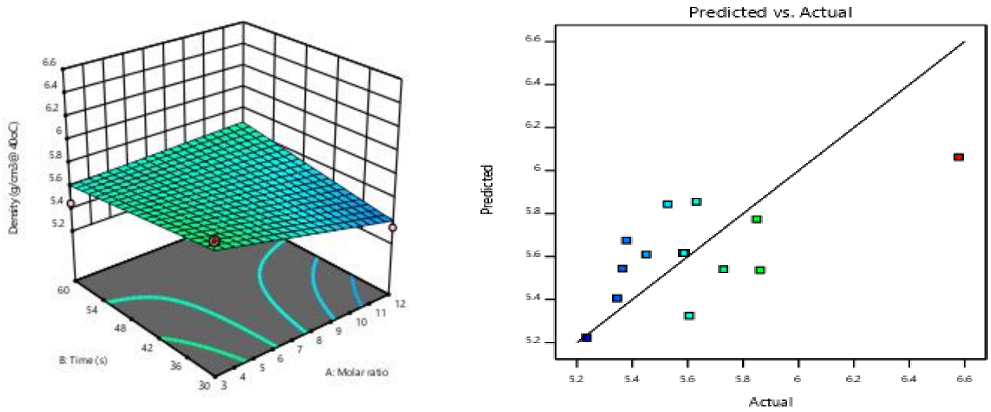

Figure 3. shows a scatter diagram of with the 3D surfaces that correspond to it

The Variance Analysis (ANOVA)

The equation represents the second polynomial function in terms of the actual components used to describe the yield

In terms of actual factors, the following is the final equation:

Yield=+46.37108-1.65556Molar ratio+1.30694Time+0.036383Speed-0.037037Molar ratio * Time +0.004489Molar ratio * Speed-0.001487Time * Speed (3)

Table 11. ANOVA Linear Model "RSAB Yield"

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	269.87	6	44.98	0.8572	0.5561	not significant
A-Molar ratio	0.3200	1	0.3200	0.0061	0.9393	
B-Time	13.26	1	13.26	0.2527	0.6260	
C-Speed	4.96	1	4.96	0.0946	0.7648	
AB	25.00	1	25.00	0.4764	0.5057	
AC	102.01	1	102.01	1.94	0.1934	
BC	124.32	1	124.32	2.37	0.1548	
Residual	524.72	10	52.47			
Lack of Fit	524.72	6	87.45			
Pure Error	0.0000	4	0.0000			
Cor Total	794.60	16				

Table 12. Constraints for RSAB biodiesel optimization

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Molar ratio	minimize	3	12	1	1	3
B:Time	minimize	30	60	1	1	3

C:Speed	minimize	500	1000	1	1	3
Yield	maximize	64.2	92.8	1	1	3

Table 13. Results discovered based on the RSAB biodiesel optimization scenario

Number	Molar ratio	Time	Speed	Yield	Desirability	
1	**3.000**	**49.359**	**500.000**	**88.664**	**0.892**	**Selected**
2	3.000	49.461	500.000	88.710	0.892	
3	3.000	49.186	500.001	88.586	0.892	
4	3.000	49.629	500.001	88.786	0.892	
5	3.000	48.729	500.000	88.379	0.892	

Tables 10–12 show desirability functions for three different criteria using varied input components (molar ratio, time and speed) for constant NaOH, temperature, and the combination of processes that were examined. The optimization strategies identified based on the biodiesel optimization scenario is shown in Table 5. Using a fixed catalyst of 1.0 wt.%, temperature 60 ºC and a molar ratio (1:3, 1:6, 1:12), the optimization solution with the molar ratio (1:3), time (49.359) and speed (500.00 rpm) yielded biodiesel (RSAB) of 88.664 %, with the stipulated overall desirability of 0.892. Molar ratio, time, and speed, were all important variables in biodiesel synthesis, according to the results of the analysis of variance (ANOVA).

Table 14. Optimization solutions for the three biodiesel optimizations (RJB, RSB and RSAB)

Number	Molar ratio	Time	Speed	Yield	Desirability	
RJB	3.000	40.910	500.000	86.937	0.995	Selected
RSB	6.930	41.734	619.262	88.545	0.931	Selected
RSAB	3.000	49.359	500.000	88.664	0.892	Selected

4.Conclusions

The optimal parameters for biodiesel were studied in this study using the Surface Response Methodology of Box-Behnken Design. It demonstrated and compared the desirability package's ability to combine production factors to produce three optimal biodiesel productions with a fixed catalyst, temperature and under diverse molar ratio, time, and speed for the optimization scenarios. The correctness of the projected technique was tested using the biodiesel data obtained from the three sets of combination variable testing. Biodiesel yields of 86.937 %, 88.545 %, and 88.664 % were predicted by RJO, RJB, and RSAB, respectively, with less than 0.2 % variation. Biodiesel properties were investigated, and the results were found to meet both ASTM D6751 and EN14214 standards. The optimal yield outputs for each of these biodiesels were obtained, and the effects of molar ratio, and time on biodiesel yield from the RJO, RSO, and RSAO were major parameters that greatly influenced the yield, although speed altered only a little. Finally, the use of low-cost feedstock, such as non-edible vegetable oils, increasing production efficiency through process parameters and variable optimization, and lowering catalyst prices through catalyst regeneration are all major issues affecting mass production that this work addressed.

References

1. Sahoo, P.K.; Das, L.M. Process optimization for biodiesel production from Jatropha, Karanja and Polanga oils, *Fuel.* **2009**, *88*, 94-1588.
2. Mitrović PM, Stamenković OS, Banković-Ilić I, Djalović IG, Nježić ZB, Farooq M, et al. White mustard (Sinapis alba L.) oil in biodiesel production: A review. *Front Plant Sci* **2020**;11:299.
3. Leung, D.Y.C.; WuX; Leung, M.K.H.A. (2010). Review on biodiesel production using catalyzed transesterification. *Applied Energy.* **2010**, *87*, 95-1083.
4. Chitra, P.; Venkatachalam, P.; Sampathrajan, A. Optimisation of experimental conditions for biodiesel production from alkali-catalysedtransesterification of Jatropha curcas oil. *Energy for Sustainable Development.***2005**, *9*, 8-13.
5. Kumar, S.;Jain, S.; Kumar, H. Experimental study on biodiesel production parameter optimization of Jatropha-algae oil mixtures and performance and emission analysis of a diesel engine coupled with a generator fueled with diesel/biodiesel blends. *ACS Omega* **2020**,*5(28):*17033–41.
6. Lu, H.; Liu, Y.; Zhou, H.; Yang, Y.;Chen, M.; Liang, B. Production of biodiesel from Jatropha curcas L. oil. *Computers and Chemical Engineering*, **2009**,*33*, 6-1091.
7. Jafarihaghighi F, Ardjmand M, Salar Hassani M, Mirzajanzadeh M, Bahrami H. Effect of fatty acid profiles and molecular structures of nine new source of biodiesel on combustion and emission. *ACS Omega* **2020**,*5(26)*,16053–63.
8. Tiwari, A.K.; Kumar, A.; Raheman, H. Biodiesel production from jatropha oil (Jatropha curcas) with high free fatty acids: an optimized process. Biomass and Bioenergy, **2007**,*31*,75-569.
9. Mnam, Y.; Nwm Z, Nl S. Sustainability of Palm Biodiesel in Transportation: a Review on Biofuel Standard, Policy and International Collaboration Between Malaysia and Colombia. *Bioenergy Res* pp. **2020**, 1–18.
10. Miao, X.; Li, R.; Yao, H. Effective acid-catalyzed transesterification for biodiesel production. *Energy Conversion and Management*, **2009**,*50*, 4-2680–4.
11. Ishola, F.; Adelekan, D.; Mamudu, A.; Abodunrin, T.; Aworinde, A.; Olatunji, O. et al. Biodiesel production from palm olein: A sustainable bioresource for Nigeria. *Heliyon* **2020**,*6(4):*e03725.
12. Soriano, Jr, N. U.; Venditti, R.; Argyropoulos, D.S. Biodiesel synthesis via homogeneous Lewis acid-catalyzed transesterification. *Fuel*, **2009**, *88*, 5-560.
13. Gade, AL, Hauschild, MZ, Laurent, A. Globally differentiated effect factors for characterizing terrestrial acidification in life cycle impact assesment. *Sci Total Environ*. **2021**,761.
14. Georgogianni, K.G.; Katsoulidis, A.K.; Pomonis, P.J.; Manos, G.; Kontominas, M.G. (2009). Transesterification of rapeseed oil for the production of biodiesel using homogeneous and heterogeneous catalysis. *Fuel Processing Technology*. **2009**, *90*,22-1016.
15. Freedman, B.; Pryde, E.H.; Mounts, T.L. Variables affecting the yields of fatty esters from transesterified vegetable oils. *Journal of the American Oil Chemists Society*, 184, 61, 43-1638.
16. Ifeoluwa, A.; Bamgbose, TA. Ecotoxicity of three plant-based biodiesels and diesel using, Eisenia fetida. *Environmental Pollution*. **2020**,260.

17. Berchmans, H.J.; Hirata, S. Biodiesel production from crude Jatropha curcas L. seed oil with a high content of free fatty acids. *Bioresource Technology*. **2008**, *99*, 21-1716
18. Chouhan APS, Sarma AK. Biodiesel production from Jatropha curcas L. oil using Lemna perpusilla Torrey ash as heterogeneous catalyst. *Biomass Bioenergy* **2013**,*55*:386–9.
19. Cruz, JM, Montagnolli RN, Bidoia ED. Biodegradation of soybean biodiesel generates toxic metabolites in soil. *Water Air Soil Pollut* **2020**,*231(8*).
20. Organisation for Economic Cooperation and Development OECD. OECD Guideline for Testing Chemicals: 301 Ready biodegradability; Adopted by the Council on 17 July 1992-Ready; OECD: Paris, France, **2021**, 1–62.
21. Patil, P.D.; Deng, S. Optimization of biodiesel production from edible and non-edible vegetable oils. Fuel, **2009**, *88*, 6-1302.
22. ASTM, C. Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)", Annual Book of ASTM Standards, Concrete and Mineral Aggregates. American Society for Testing and Materials, Philadelphia, USA, **2003**, 227-03.
23. EN14214. Automotive fuels-fatty acid methyl esters (FAME) for diesel engines requirements and test methods, Berling, Germany: Beuth-Verlag, **2003**.
24. AOAC, AOCS Official Method Cd 8-53 Peroxides in fats and oils. American Oil Chemists' Society. Champaign, Illinois, USA: American Oil Chemists' Society, **2006**
25. AOAC. Official methods of analysis, Washington D.C., USA: Association of official analytical chemist 19th edition, **2012**.
26. Mustapha, A.O.: Adepoju, R.A.: Afolabi, Y.T. Optimization of vegetable oil-based biodiesels by multi-response surface methodology (MRS) using desirability functions, *Journal of the Chemical Society of Nigeria, JCSN*, **2020**, *45,5*, 917 – 924
27. Box, G.E.: Behnken, D.W. Some new three level designs for the study of quantitative variables, *Technometrics*, **1950**, *2*, 455-475