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Castor (Ricinuscommunis) And Sesame (Sesamumindicum) Biodiesel Optimization By Alkaline Na+/K+ Catalytic Conversion.

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Castor (Ricinuscommunis) And Sesame (Sesamumindicum) Biodiesel Optimization By Alkaline Na+/K+ Catalytic Conversion.

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Castor (*Ricinuscommunis***) and Sesame (***Sesamumindicum)* **Biodiesel Optimization by Alkaline Na⁺ /K ⁺Catalytic Conversion.**

Authors Names	ABSTRACT
a. AliruOlajide Mustapha	
b. Kamarudeen Seun Akanji	For the optimization of refined castor biodiesel (RCB) and refined sesame
c. Adebola Christianah Akala	biodiesel (RSB), the impacts of specified conditions such as catalyst types
d. Ebenezer Sola Daramola	(NaOH and KOH), catalyst concentration (0.3–1.5 wt. %), speed (500–750)
e. Mercy Mobolaji Ajala	rpm), and time (20–60 min) were investigated. The physicochemical
f. Fatimah Yetunde Ajao	properties of the RCB and RSB were measured using the American Standard
g. Salihu Folorunsho Adisa	for Testing Materials (ASTM) approved protocols for acid, peroxide, iodine,
h. Mubarak Bolakale Abdullahi	and saponification; density, kinematic viscosity, and refractive index value.
Article History	Box-Behnken Design's Response Surface Methodology (RSM) was also used
Received on:15/1/2022	for optimization. The results for four optimization methods were catalyst
Revised on: 17/2/2022	concentration (0.300–0.435 wt. %), speed (500.000–643.242 rpm), and time
Accepted on: 21/2/2022	(20.000–31.386 min). The yield ranged from 81.062 to 102.648 %, with a
Keywords: biodiesel; catalyst; optimization; castor oil; sesame	desirability range of 0.812 to 0.980 %. The projected RSB maximum yield
oil	while using KOH was 102.648 %, whereas the RCB maximum yields when
	using NaOH was 81.062 %, with a variance of 21.586 %. Under these reaction
DOI: https://doi.org/10.29350/	conditions, the optimal yield of RCB (81.062%) using the KOH catalyst was
jops.2022.27.1.1471	lower than that of RCB (92.017%) using the NaOH catalyst, with a variance of
	10.955 %. ANOVA statistics revealed that catalysts were the most critical
	components in biodiesel optimization, based on expected biodiesel yields.
	These yields were higher when compared to ASTM D 6751 and EN 14214
	which required a range of 46–55 % in regular biodiesel production.

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Introduction

Biodiesel is biodegradable, nontoxic, and renewable; it has a high cetane number, built-in oxygen content, and higher combustion without or with reduced sulfur; it contains aromatic components and other regulated emissions; and it completes the carbon cycle. It is easy to install and has a high flash point, so it may be used in current engines with little or no modification [12, 19].Non-edible vegetable oils from castor and sesame were chosen as feedstocks since they are not consumed by humans, the plants can grow in agronomically deficient soils, and their yield is higher than that of other energy crops [18].

Furthermore, castor oil has a high solubility in alcohols, making transesterification a reversible process. The high production of biodiesel generates significant amounts of waste, the majority of which is glycerol. Glycerol is said to make up 10–20 percent of the entire amount of biodiesel generated, according to various sources. Growing biodiesel production will almost certainly result in an overabundance of glycerol, which has been defined as having a low commercial value due to its poor quality [17]. The biodiesel production source is chosen based on what is available in each location or country. Biodiesel can be made from any fatty acid source. Waste vegetable oils and nonedible crude vegetable oils are preferred as prospective low-cost biodiesel sources because the prices of edible vegetable oils, such as soybean oil, are greater than those of diesel fuel.

Biodiesel has been made from low-quality, underutilized feedstocks. This product has a similar composition, engine performance, and emissions, and is expected to be more cost effective. Varied types of vegetable oils have different fatty acid contents [3].The chain length, degree of unsaturation, and existence of various chemical functionalities all varies between fatty acids. More than 350 oilbearing crops have been identified as viable feedstocks for biodiesel synthesis [9, 10]. Despite the diversification of biodiesel research, the cost remains uncompetitive when compared to conventional fuels. Depending on the type of feedstock used, the cost of biodiesel unit pricing is 1.5–3.0 times higher than that of petroleum-derived diesel fuel [1].

Furthermore, the high production of biodiesel generates significant amounts of waste [17], and when waste is thrown without proper treatment, it causes social and environmental problems. A lot of research is being done on converting glycerol into high-value and useful products in order to improve biodiesel's economics by cutting its manufacturing costs. Glycerol's flexibility is well known, given its applications in practically every sphere of human endeavor [8]. Glycerol, on the other hand, must be pure in order to be employed in the food, pharmaceutical, cosmetics, and other personal care industries.

Unfortunately, the purification process, which includes filtering, chemical treatment, and vacuum distillation, is costly, particularly for small and medium-sized manufacturing facilities, and hence does not correspond to its present low market value [13]. As a result, it's not unexpected that the use of the optimum optimization conditions in the manufacture of higher-value products has recently received a lot of attention (biodiesel and blends).This is intended to encourage the use of integrated biodiesel in chemical production and optimization, a concept dubbed "bio-refinery."The goal of this study is to find the optimized conditions for producing biodiesel from sesame and castor seed oils utilizing NaOH/KOH catalysts and dosages.

2. Materials and Method

2.1 Materials

The crude castor oil (CCO) and sesame (CSO) oil were obtained in Ilorin markets, Kwara State, Nigeria. The Sigma Aldrich company provided the equipment and chemicals (Gillingham Dorset, UK). Two liters of each oil were extracted using cold oil extraction, followed by a transesterification reaction and purified to refined castor oil (RCO) and refined sesame oil (RSO) to preserve the properties of the refined castor biodiesel (RCB) and refined (RSB) obtained, as recommended by the American Standard for Testing Materials [5], the Association of Official Analytical Chemists [6, 7] and Mustapha *et al*. [4, 14, 15].

2.2 Pre-treatment, refinement (CSAO and CJO) and trans-esterification processes

The levels of the free fatty acids (FFAs) of the two feedstocks were carried out to ensure the percentages of FFAs were <0.5%. Refinement of the CCO and CSO was an important phase in biodiesel production for removing impurities such as phosphorus compounds prior for efficient transesterification. The crude oils refining processes: degumming, alkaline and bleaching treatment were carried and were followed by the trans-esterification processes using NaOH and KOH as catalysts at different dosages (wt. %) for the final product of biodiesels were obtained as a clear, light yellow liquids (biodiesels) [4].

2.3 Using a Response Surface Method (RSM) for Biodiesel Optimization

The RSM approach was used to establish correlations between independent and response variables. Box and Wilson [11] were the first to develop a model or optimal response for experimental data, but other ways to optimize processes have boosted its practical application. The p -value can be calculated using ANOVA for each of the models. When the values were less than 0.0500, the p-value of 0.05 for

4 AliruOlajide Mustapha*,* Kamarudeen Seun Akanji*,* Adebola Christianah Akala. Ebenezer Sola Daramola*,* Mercy Mobolaji Ajala*,*Fatimah Yetunde Ajao*,* Salihu Folorunsho Adisa*,* Mubarak Bolakale Abdullahi, Al-Qadisiyah Journal of Pure Science **27 , 1 (2022)) Chem.pp. 1-16**

most process variables was favorable, indicating that model terms were significant. The statistical program utilized was the Design Expert II version, which was chosen because it includes the three minimal categories of input and response variables required for the adequacy check, as well as the projected and experimental values.

2.4 Experimental Design

To construct correct ANOVA models, the RSM must create a design of experiments (DoE) using the smallest amount of data. Many approaches have been proposed, but a design matrix (inputs) must be developed using a Box–Behnken Design (BBD) because BBD designs do not have axial points, hence all design points must fall between operating limits. The input factors (catalyst, speed, and time) were chosen in a variety of combinations to produce yield as output. Catalyst doses/concentration (0.3, 0.9, 1.5 wt.%), speed (500, 750, 1000 rpm), and time (20, 40, 60 min) were randomized ranges of experimental parameters based on the range of biodiesel production in the literatures [2, 16, 20].

In table 1, NaOH and KOH catalyst doses, mixing speed, reaction temperature at a set methanol to oil molar ratio, and duration were all variables in the manufacture of biodiesels from refined oils (RCO and RSO) to biodiesels (RCB and RSB).

3. Results and Discussions

3.1. Design of an experimental matrix for RCB and RSB optimization using NaOH/KOH.

3.1.1. Effects of different NaOH dosages on the output of refined castor biodiesel (RCB).

At a fixed oil to molar ratio (6:1), temperature (60 $^{\circ}$ C), but with varied NaOH loading (0.3–1.5 wt.%), speed (500–1000 rpm), and time (20–60 min), the experimental impacts of NaOH catalyst dosages on sweet almond-based biodiesel were explored. The biodiesel production, specific gravity, and density experimental findings were subjected to analyses of variance (ANOVA) utilizing the Box–Behnken DoE design.

Table 2: Design experimental matrix at different NaOH concentration (wt. %) speed and time

Based on the three levels of inputs, the design expert program created the most number of runs. Figure 1 depicts the link between the yield values acquired experimentally and the yield values projected by several models (Table 2).

Figure 1: (A) Scatter diagram for yield (B) with the corresponding 3D surface

6 AliruOlajide Mustapha*,* Kamarudeen Seun Akanji*,* Adebola Christianah Akala. Ebenezer Sola Daramola*,* Mercy Mobolaji Ajala*,*Fatimah Yetunde Ajao*,* Salihu Folorunsho Adisa*,* Mubarak Bolakale Abdullahi, Al-Qadisiyah Journal of Pure Science **27 , 1 (2022)) Chem.pp. 1-16**

Analysis of Variance (ANOVA)

The equation shows the second polynomial functions in term of actual factors that were obtained to model yield, specific gravity and density.

Final Equation in Terms of Actual Factors:

Yield =+73.89553-22.79965Catalyst+0.002700Speed+0.201011 Time (1)

During the optimization, the input variables of NaOH concentration (0.435 wt. %), speed (643.242 rpm), and duration (31. 386 min) were computed. The yield of the biodiesel produced was 92.017 % percent, with an overall desirability of 0.812. As illustrated by the scenario, the best catalyst dose has an impact on biodiesel optimization.

Table 5: Optimization solutions found according to the biodiesel optimization scenario.

3.1.2. Effects of different KOH dosages on the output of refined castor biodiesel (RCB).

At a constant oil-to-molar ratio (6:1), temperature (60 $^{\circ}$ C), but with varied KOH catalyst loading (0.3–1.5 wt. %), speed (500–1000 rpm), and time (20–60 min), the experimental impacts of catalyst dosages on sweet almondbased biodiesel were investigated. The biodiesel yield, specific gravity, and density experimental findings were analyzed using the Box–Behnken DoE design.

	Factor 1	Factor 2 Factor 3		Response 1	
Run	A:Catalyst B:Speed C:Time			Yield %	
	g	rpm	min	Actual	Predicted
1	0.9	750	40	60.00	60.00
$\overline{2}$	0.9	1000	60	60.00	62.91
3	0.9	500	20	75.00	72.09
$\overline{4}$	0.3	500	40	59.00	70.46
5	0.9	750	40	60.00	60.00
6	1.5	500	40	65.00	61.04
7	0.9	750	40	60.00	60.00
8	0.9	750	40	60.00	60.00
9	1.5	750	20	67.50	74.38
10	0.3	750	20	89.20	80.65
11	1.5	750	60	60.00	68.55
12	0.3	1000	40	60.00	63.96
13	0.9	1000	20	57.00	61.59
14	0.3	750	60	90.00	83.13
15	1.5	1000	40	64.00	52.54
16	0.9	750	40	60.00	60.00
17	0.9	500	60	72.00	67.41

Table 6: Design experimental matrix at different KOH catalyst concentration (wt. %), speed and time

Based on the three levels of inputs, the design expert program created the most number of runs. Figure 1 depicts the link between the actual yield values acquired experimentally (Table 6) and the yield values projected by several models (Figure 2):

8 AliruOlajide Mustapha*,* Kamarudeen Seun Akanji*,* Adebola Christianah Akala. Ebenezer Sola Daramola*,* Mercy Mobolaji Ajala*,*Fatimah Yetunde Ajao*,* Salihu Folorunsho Adisa*,* Mubarak Bolakale Abdullahi, Al-Qadisiyah Journal of Pure Science **27 , 1 (2022)) Chem.pp. 1-16**

The equation shows the second polynomial functions in term of actual factors that were obtained to

model yield, specific gravity and density.

Final Equation in Terms of Actual Factors:

Yield=+97.84063-30.95833Catalyst+0.080100Speed-2.17875Time-0.003333Catalyst*Speed-0.172917Catalyst * Time+0.000300Speed * Time+17.60417Catalyst²-0.000069Speed²+0.025844Time² (2)

Source	Sum of Squares df Mean Square F-value p-value					
Model	1061.88 9		117.99			1.39 0.0394 significant
A-Catalyst	217.36 1		217.36		2.56 0.1535	
B-Speed	112.50 1		112.50		1.33 0.2874	
C-Time	5.61 1		5.61		0.0661 0.8045	
AB	1.0000	$\overline{1}$	1.0000		0.0118 0.9166	
AC	17.22 1			17.22 0.2030 0.6660		
BC	9.00	$\mathbf{1}$	9.00	0.1061	0.7542	
A^2	169.11	$\overline{1}$	169.11	1.99	0.2009	
B ²	79.22 1		79.22	0.9336 0.3661		
C ²	449.95 1		449.95		5.30 0.0548	
Residual	593.97 7		84.85			
Lack of Fit	593.97 3		197.99			
Pure Error	0.00004		0.0000			
Cor Total	1655.85 16					

Table 7: ANOVA Table for the "Yield" Quadratic model

During the optimization, the input variables of KOH concentration (0.300 wt. %), speed (500.002 rpm), and duration (20 min) were computed. The yield of the biodiesel produced was 81.062 %, with an overall desirability of 0.877. As illustrated by the scenario, the best catalyst dose has an impact on biodiesel optimization.

Table 9: Optimization solutions found according to the biodiesel optimization scenario

3.1.3. Effects of different NaOH dosages on the output of refined sesame biodiesel (RSB).

At a fixed oil to molar ratio (6:1), temperature (60 $^{\circ}$ C), but various NaOH catalyst loading (0.3–1.5 wt. %), speed (500–1000 rpm), and time (20–60 min), the experimental impacts of catalyst dosages on sweet almond-based biodiesel were explored. The experimental findings for biodiesel yield, specific gravity, and density were analyzed using the Box–Behnken DoE design.

Table 10: Design experimental matrix at different NaOH catalyst concentration (wt. %), speed and time

	Factor 1 Factor 2 Factor 3 Response 1							
	Run A:Catalyst B:Speed C:Time			Yield %				
	g	rpm	min	Actual	Predicted			
	0.9	750	40	90.00	90.00			
2	0.9	1000	60	90.00	90.00			
3	0.9	500	20	83.30	84.56			
4	0.3	500	40	90.00	90.42			
5	0.9	750	40	80.00	79.14			
6	15	500	40	90.00	90.00			

10 AliruOlajide Mustapha*,* Kamarudeen Seun Akanji*,* Adebola Christianah Akala. Ebenezer Sola Daramola*,* Mercy Mobolaji Ajala*,*Fatimah Yetunde Ajao*,* Salihu Folorunsho Adisa*,* Mubarak Bolakale Abdullahi, Al-Qadisiyah Journal of Pure Science **27 , 1 (2022)) Chem.pp. 1-16**

		τ	0.9	750	40	86.67	86.25			
		8	0.9	750	40	86.67	85.40			
		9	1.5	750	20	78.30	79.16			
		10	0.3	750	20	90.00	88.32			
		11	1.5	750	60	90.00	90.00			
		12	0.3	1000	40	83.30	84.57			
		13	0.9	1000	20	81.67	80.41			
		14	0.3	750	60	83.30	82.89			
		15	1.5	1000	40	90.00	90.00			
		16	0.9	750	40	81.67	82.08			
Based the on	three	17	0.9	500	60	83.30	84.98	levels of inputs,		the
Design Expert program								created	the	most

number of runs. Figure 1 depicts the link between the actual yield values acquired experimentally (Table 10) and the yield values projected by several models (Figure 3)

Figure 3: (A) Scatter diagram for yield (B) with the corresponding 3D surface

Analysis of Variance (ANOVA)

The equation shows the second polynomial functions in term of actual factors that were obtained to model yield, specific gravity and density.

 $Yield = +69.46118$ (3)

(2022)) Chem.pp. 1-16

Table 12: Constraints

During the optimization, the input variables of NaOH concentration (0.318 wt. %), speed (500 rpm), and time (20 minutes) were computed. The yield of the biodiesel produced was 89.461%, with an overall desirability of 0.900. As illustrated by the scenario, the best catalyst dose has an impact on biodiesel optimization.

Table 13:Optimization solutions found according to the biodiesel optimization scenario

			Number Catalyst Speed Time Yield Desirability
	0.318 500.000 20.000 89.461		0.900 Selected
	0.323 500.001 20.000 89.461		0.900
3	0.313 500.000 20.000 89.461		0.900
4	0.308 500.001 20.000 89.461		0.900
5	0.330 500.003 20.001 89.461		0.800

3.1.4 Effects of different KOH dosages on the output of refined sesame biodiesel (RSB).

At a constant oil-to-molar ratio (6:1), temperature (60 $^{\circ}$ C), but with varied KOH catalyst loading (0.3– 1.5 wt. %), speed (500–1000 rpm), and time (20–60 min), the experimental impacts of catalyst dosages on sweet almond-based biodiesel were investigated. The biodiesel yield, specific gravity, and density experimental findings were analyzed using the Box–Behnken DoE design.

Table 14: Design experimental matrix at different KOH catalyst doses (wt. %), speed and time

	Factor 1 Factor 2 Factor 3 Response 1								
	Run A:Catalyst B:Speed C:Time			Yield					
	g	rpm	m ₁ n	Actual	Predicted				
	0.9	750	40	54.60	67.98				
2	0.9	1000	60	98.80	93.48				
3	09	500	20	54.21	82.48				
	03	500	40	74.70	77.35				

12 AliruOlajide Mustapha*,* Kamarudeen Seun Akanji*,* Adebola Christianah Akala. Ebenezer Sola Daramola*,* Mercy Mobolaji Ajala*,*Fatimah Yetunde Ajao*,* Salihu Folorunsho Adisa*,* Mubarak Bolakale Abdullahi, Al-Qadisiyah Journal of Pure Science **27 , 1**

acquired experimentally (Table 14) and the yield values projected by several models (Figure 4).

 (A) (B) Figure 4. (A) Scatter diagram for yield (B) with the corresponding 3D surfaces

Analysis of Variance (ANOVA)

The equation shows the second polynomial functions in term of actual factors that were obtained to model yield, specific gravity and density.

Final Equation in Terms of Actual Factors:

Yield=+138.89827-41.80625Catalyst-0.017170Speed-0.510437Time (1)

Source Sum of Squares df Mean Square F-value p-value Model 6014.71 3 2004.90 6.79 0.0054 significant A-Catalyst 5033.56 1 5033.56 17.04 0.0012 B-Speed 147.40 1 147.40 0.4989 0.4924 C-Time 833.75 1 833.75 2.82 0.1168 **Residual** 3840.96 13 295.46 Lack of Fit 3840.96 9 426.77 Pure Error 0.0000 4 0.0000 **Cor Total** 9855.67 16

Table 15: ANOVA Table for the "Yield" Quadratic model

During the optimization, the input variables of KOH concentration (0.300 wt. %), speed (500 rpm), and duration (29.627 minutes) were computed. The yield of the biodiesel produced was 102.649 %, with an overall desirability of 0.980. As suggested by the scenario, this revealed the impact of the best catalyst dose in biodiesel optimization.

Table 17: Optimization solutions found according to the biodiesel optimization scenario

			Number Catalyst Speed Time Yield Desirability	
		0.300 500.000 29.627 102.649	0.980	Selected
		0.300 500.001 29.478 102.725	0.900	
3		0.300 500.001 29.285 102.823	0.00	
4		0.301 500.000 30.054 102.390	0.899	
5		0.300 500.000 31.237 101.816	0.899	

Table 18: Experimental outputs based on the four biodiesel optimization scenarios

14 AliruOlajide Mustapha*,* Kamarudeen Seun Akanji*,* Adebola Christianah Akala. Ebenezer Sola Daramola*,* Mercy Mobolaji Ajala*,*Fatimah Yetunde Ajao*,* Salihu Folorunsho Adisa*,* Mubarak Bolakale Abdullahi, Al-Qadisiyah Journal of Pure Science **27 , 1 (2022)) Chem.pp. 1-16**

RCB	KOH	0.318	500,000 20,000 89.461	0.900 Selected
RSB	KOH	0.300	500,000 29.627 102.649	0.980 Selected

Table 18 show the optimization results obtained from the Tables 5, 9, 13 and 17 using the NaOH and KOH catalysts and the combination of other process variables that were studied, showing corresponding desirability functions. In the four different criteria starting with the RCB, NaOH concentration (0.435 wt. %), speed (643 rpm) and time (31.386 min) were computed during the biodiesel optimization for 92 % yield with desirability of 0.812, whereas for the RSB, using NaOH concentration (0.300 wt. %), speed (500 rpm) and time (20.000 min) were computed for 81.062 % yield with desirability of 0.877.

Similarly, for the RCB; KOH concentration (0.318 wt. %), speed (500 rpm) and time (20.000 min) were computed during the biodiesel optimization for 89.461 % yieldwith desirability of 0.900, whereas for the RSB, using KOH concentration (0.300 wt. %), speed (500 rpm) and time (29.627 min) were computed for 102.649 % yieldwith desirability of 0.980.

The overall results showed the average input variables such catalyst concentration (0.353 wt. %), speed (535.811 rpm) and time (25.253 min) were computed during the biodiesel optimization for the fourdifferent processes. When using KOH, the expected RSB maximum yield was 102.648 %, whereas when using NaOH, the RCB maximum yield was 81.062 %, with variation of 21.586 %. The optimal yield of RCB (81.062%) using the KOH catalyst was lower than that of RCB (92.017 %) using the NaOH with variation of 10.955 % under these reaction conditions. The anticipated biodiesel yields, according to ANOVA statistics, showed catalysts were the most important factors in biodiesel optimization

These values were higher when compared with the requirements of ASTM D 6751 [5] and EN 14214 [10], which both stipulated a range of 46-55 % in normal biodiesel production. In this work, the optimal optimization conditions were employed for effective combination of process variables using the response surface methodology of Box–Behnken Design.

Conclusions

Using the response surface approach of the Box–Behnken design, the impacts of the catalysts and their nature on biodiesel optimization were investigated. The findings of the ANOVArevealed that concentration levels, speed, and time were the important factors in biodiesel optimization. Furthermore, the current work showed catalyst's type was the most crucial component in increasing

yield. The best catalyst conditions were found in RSB, which had a yield of 102.649 % when using KOH, compared to RCB, which had a yield of 92.017 % when using NaOH. In addition, the yield of RCB using the KOH catalyst (89.461 %) was higher than the yield of RSB using NaOH (81.062 %). The nature of the catalyst was therefore shown to be the most critical aspect in the current study's overall optimization outcomes.

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16 AliruOlajide Mustapha*,* Kamarudeen Seun Akanji*,* Adebola Christianah Akala. Ebenezer Sola Daramola*,* Mercy Mobolaji Ajala*,*Fatimah Yetunde Ajao*,* Salihu Folorunsho Adisa*,* Mubarak Bolakale Abdullahi, Al-Qadisiyah Journal of Pure Science **27 , 1 (2022)) Chem.pp. 1-16**

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