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Research Article

Response Surface Methodology Process Optimization of Biodiesel Production from Castor Seed Oil

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The increasing demand for energy and the depletion of fossil fuel resources has led to the search for an alternative energy source. The search for such alternative fuel sources has oriented biodiesel synthesis as the ultimate alternative energy resource of the future. The purpose of this study was to produce castor oil and optimize biodiesel production using full factorial central composite design (CCD) through the response surface methodology (RSM) approach. The castor oil was extracted using a mechanical press, and free fatty acids were reduced with acid esterification and biodiesel produced through the transesterification process using homogeneous catalysts. The physicochemical properties of extracted castor oil and biodiesel including density, kinematic viscosity, saponification number, free fatty acid, acid number, cetane number, and iodine number were determined. The ideal conditions for producing biodiesel from castor oil are anticipated to be a reaction period of 105 min at a temperature of 50°C, a catalyst loading weight of 1.5%, and a methanol-to-oil ratio of 5 : 1. The biodiesel yield obtained was 95.0%, and the results of the measured parameters of biodiesel were compared with the international standards of the European norms (EN14214) and the American Society Test Material (ASTM) D6751. The weight composition of both fatty acid and methyl ester was determined by gas chromatography-mass spectroscopy (GC-MS). The study concluded that the results of this research are beneficial in optimizing the parameters for making biodiesel from extracted castor oil.

1. Introduction

An increasing number of nations have made commitments and agreements to encourage the development of alternative renewable energy sources, largely due to their concern for the environment and the necessity of minimizing CO_2 emissions to lessen their impact on global warming [1]. Renewable sources of energy are being sought after due to the growing demand for energy and the depletion of fossil fuel supplies [2]. There is a growing interest in introducing new methods of studying alternative and renewable sources of petroleum products [3]. In addition, the limited fossil fuel reserve has drawn the attention of various research trends to discover alternative renewable sources of energy resources that can be used for energy production [4]. Biodiesel synthesis has been identified as the most promising alternative energy source of the future due to the investigation of such alternative fuel sources [5].

Plant and animal fats are renewable sources of the alkyl ester of fatty acids used to make biodiesel [6]. Biodiesel is one of the cheap biofuels to replace fossil fuels as the primary energy source for machinery and vehicles [7]. Biodiesel meets the criteria of sustainability because its production has less environmental impact and is biodegradable and nontoxic [1]. In addition, biodiesel is an environmentally friendly biofuel and is preferred as an alternative energy source due to its low emissions as compared to fossil fuels [3, 8]. Therefore, biodiesel is a promising alternative energy source and useful for reducing greenhouse gas emissions such as sulfur and nitrogen oxides [9, 10].

Biodiesel is produced worldwide from a variety of raw materials, mostly vegetable oils. Vegetable oils can be used as a raw ingredient in the production of biodiesel [8]. Biodiesel produced from vegetable oil is a fuel that is as efficient as fossil fuels [5, 6, 11]. Commercially available nonedible oils include *Moringa oleifera* (Moringa seeds) and *Jatropha curcas* (Ratanjyot), biodiesel's inedible raw materials [10, 12]. For this reason, more importance is attached to inedible seed oils as a source for biodiesel production [5, 9, 13, 14]. As a result, Ethiopia and other developing countries would be more convenient to produce inedible crops that can support renewable fuel. Choosing the appropriate oil as feedstock is a critical factor in the biodiesel production process. So, the most commonly utilized feedstocks for biodiesel production should possess the characteristics of being nonedible, readily available, low-cost, and adaptable to diverse climatic conditions [15]. This study uses castor bean (*R. communis*) seed oil as a potential feedstock for producing biodiesel.

The castor bean (*R. communis*) plant has a strong adaptation to different weather conditions and can grow in marginal soils. This trait is subsidized directly to reduce land use for the production of biofuels and to reserve it for the cultivation of products for human consumption [8]. Castor seed oil, obtained from the castor (*Ricinus communis*) plant, is characterized by its indigestibility, solubility in methanol and methyl esters, high hygroscopicity, and high viscosity [1]. Additionally, there are other ways to make this biofuel, although transesterification is the most popular [8]. Castor oil-based biodiesel is a practical substitute for other types of biodiesel due to its technical and ecological benefits. Castor oil is one of those inedible vegetable oil seeds and useful raw materials for the production of renewable, biodegradable biodiesel [16].

The production of biodiesel through transesterification reactions typically uses homogeneous alkaline catalysts such as KOH and NaOH. These catalysts are widely used in the industry due to their ability to operate at low temperatures, short reaction times, and low alcohol-to-oil ratios [15]. To produce biodiesel, many researchers often use the transesterification process of oil with alcohol in the presence of a catalyst which is commonly used by many researchers [17]. Short-chain alcohol and alkaline catalysts, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), were often used in the transesterification process to achieve the highest biodiesel yield in the shortest possible time [16]. Several factors influence biodiesel production such as the alcohol-to-oil ratio, the catalyst loading, the reaction time, and the temperature [5]. Optimization of the process variables such as catalyst loading, alcohol-to-oil ratio, reaction time, and reaction temperature is required for the investigation and maximum biodiesel yield with quality assessment. However, there is still a clear research gap in the production of biodiesel from castor oil, especially regarding the optimization of important processing parameters, such as transesterification reaction temperature, catalyst dose, reaction time, and methanol-to-oil ratio.

It is a common practice to utilise the response surface methodology (RSM) technique to optimize process variables in order to obtain the required yield with a minimal number of experiments [18]. RSM is a helpful tool for analysing the combined effects of independent factors on the dependent variable in an experiment, and it shows where the model boundaries should be so that treatment combinations can be avoided [19]. The experimental design for biodiesel production can stimulate transesterification conditions with good error estimates. Several researchers implement RSM for the optimization process of biodiesel production using different raw materials [5, 17, 20, 21]. To close the obvious research gap in the production of biodiesel from castor oil, biodiesel production was investigated by using RSM for the optimization process from extracted castor oil.

The novelty of this work is the production of biodiesel oil from castor seed oil by optimization process and identifying influential variables for biodiesel production using the response surface methodology (RSM)-central composite design (RSM-CCD) method. The yield of biodiesel oil depends on the molar ratio of methanol to oil, reaction time, temperature, and catalyst loading. Therefore, the optimal parameters for the transesterification of castor oil such as the molar ratio of methanol to oil, reaction time, temperature, and catalyst loading were investigated to achieve the highest biodiesel yield. Furthermore, the physicochemical characteristics of the produced biodiesel, including density, acid value, kinematic viscosity, heating value, iodine number, saponification value, and cetane number, are studied.

2. Materials and Methods

2.1. Materials. The fresh castor seed was collected from Nefas Silk Lafto subcity, Addis Ababa, Ethiopia. Methanol (CH_3OH, 99.8% purity), sodium hydroxide (NaOH) and hydrochloric acid (HCl), ethanol (C_2H_5OH), phenolphthalein, and chloroform were used. During the biodiesel production and characterization, all chemicals used were analytically pure.

2.2. Extraction of Castor Oil. The castor seeds were washed with distilled water and dried in sun with open air for 24 hours and heated at 60°C for 7 hours until a constant weight was reached. Then, castor seeds were crushed to separate their coats/shells and seed flesh. The mortar was used to grind the castor seeds into cakes to break up the cell walls and release castor oil for extraction. The castor oil was extracted by a mechanical press as described by a previous study [22]. After compression, the crude oil and press cake were separated from one another.

2.3. Esterification. The castor seed oil was subjected to acid esterification to reduce its acid value. After crude castor oil was extracted, gum, high free fatty acid (FFA), moisture, and other undesirable components in the crude castor oil were removed by degumming, neutralization, deodorization, and drying. The extracted castor oil was degummed by adding boiling water to the castor crude oil. The mixture of crude oil and boiled water was then stirred for 2 min. After that, the mixture was moved and put in a funnel for 24 hours to

separate the castor oil-containing lower layer from the unreacted upper layer. Water from the bottom layer was drained in order to isolate the oil. The free fatty acid concentrations of the castor oil were subsequently decreased using an acid esterification pretreatment. The samples were heated to 120°C for 15 min to eliminate the moisture from the crude oil that had been extracted. By using air conditioning, the temperature was dropped to 50°C. For acid esterification, the dried crude oil was next combined with a methanol and H_2SO_4 solution. The amount of refined oil was calculated using the following equation:

$$\text{yield of oil (\%)} = \frac{\text{weight of castor oil}}{\text{weight of castor seed}} \times 100. \quad (1)$$

2.4. Biodiesel Production. The biodiesel was produced from castor oil using the transesterification process. 30 mL of castor oil was measured out and transferred to an Erlenmeyer flask. The castor oil was preheated to 110°C for 60 min to remove the moisture content in the oil. A known amount of methanol and caustic soda was prepared with different proportions of catalysts in the mixture and added to the preheated oil, and the mixture was stirred. The pretreated castor oil and sodium methylate solution were poured into the 250 mL three-round-bottom flask. Before the addition of the sodium methylate solution, preheated oil was cooled to a selected temperature (30, 40, and 60°C), and then sodium methylate was mixed in castor oil at the temperatures. The sodium methylate solution was mixed with the preheated castor oil in a 250 mL Erlenmeyer flask and fixed on a hot plate magnetic stirrer with constant stirring at a selected temperature (30, 40, and 60°C). The reaction mixture was then left to stand for 30, 60, and 90 min reaction times. After the transesterification process, the mixture was transferred into a separatory funnel and kept overnight to undertake to settle and form two layers (thick brown layer of glycerol and yellowish colored fatty acid methyl ester).

The top level with the biodiesel was collected and mixed with distilled water at 40°C to remove residual impurities. The resulting biodiesel was separated from the glycerol by the separatory funnel. The water washing process was used to further purify biodiesel. The remaining water was subsequently evaporated by heating the cleaned biodiesel fraction to 100°C for 60 min.

2.5. Gas Chromatography Analysis. The Agilent gas chromatography/mass spectrometry instruments provide the efficiency, flexibility, and robust performance that modern analytical laboratories required. The profile of the most important fatty acids contained in the castor oil was obtained by gas chromatography with mass spectroscopy (GC-MS). Specific fatty acids were quantified by calculating the relative percentage of total fatty acid esters. The sample analyses were conducted in the JIJE Lab Glass Laboratory, Addis Ababa, Ethiopia, using the method described in [15].

2.6. Characterization of Castor Oil and Biodiesel. The physicochemical parameters considered for refined castor oil and biodiesel during the experimental study were specific gravity, moisture content, viscosity, saponification number, acid number, and iodine number and were compared with the standard specifications of American Society Test Material (ASTM) [2, 15, 23].

2.7. Experimental Design. Full factorial experiment was conducted during the study period with the four selected experimental factors with the three levels as shown in Table 1. The selected three levels of four experimental factors were based on the previous research findings. Researchers showed from their experimental results that the oil-to-methanol ratio increases from 1 : 3 to 1 : 9, the percentage yield of biodiesel production increases from 70% to 95%, and the oil-to-methanol ratio increases from 1 : 9 to 1 : 10, but the percentage yield of biodiesel production decreases up to 80%. They also reported that the increase in reaction time from 15 minutes to 90 minutes increases the biodiesel yield from 65% to 95%, but an increase in reaction time from 90 minutes to 120 minutes decreases the biodiesel yield from 95% to 85%. They conducted the transesterification reaction at various reaction temperatures. By increasing the reaction temperature from 35°C to 60°C, the percentage yield of biodiesel production increases from 37% to 95%, but after passed the temperature more than 60°C, the percentage yield of biodiesel decreases to 85% [24].

The number of experimental runs was minimized to 30 runs using Design Expert 12 from the original formulation of a 3^4, which is predicted to generate 81 runs. The central composite design was used to optimize the experiments which have a quantitative independent variable and a dependent variable. The independent variables selected to optimize the experiment were reaction temperature (X_1), methanol-to-oil molar ratio (X_2), reaction time (X_3), and catalyst loading (X_4). The response selected was the biodiesel yields produced by the transesterification process of castor oil. Then, reaction temperature, methanol-to-oil molar ratio, reaction time, and loading catalyst were designed for the experimental study at a constant mixing speed of 400 revs/min. The optimal yield of biodiesel in percentage was calculated using the following equation:

$$\text{yield of biodiesel (\%)} = \frac{\text{weight of biodiesel obtained from each experiment}}{\text{weight of seed oil used in each sample}} \times 100. \quad (2)$$

Table 1: The full factorial experimental design of biodiesel production with the four factors and three levels.

Factors	Unit	Levels of the factors		
		Low (–1)	Medium (0)	High (+1)
Temperature (°C)	X_1	30	40	60
The ratio of methanol: castor oil	X_2	6 : 1	9 : 1	12 : 1
Time (min)	X_3	30	60	90
Catalyst load (%)	X_4	1	1.5	2

The matrices for the four independent factors were changed at three levels: low (–1), medium (0), and high (1). The batch reactions for each experiment were conducted randomly to minimize systematic error. To validate the biodiesel yield obtained, regression analysis was performed and calculated from the following equation:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1,i<j}^{k=1} \sum_{i=2}^{k} \beta_{ij} X_i X_j, \quad (3)$$

where Y denotes the anticipated production of biodiesel while β_i, β_{ii}, and β_{ij} denote coefficients determined by regression analysis. The regression equation represents the linear, quadratic, and interactions of the independent variable on the response.

3. Results and Discussions

3.1. Castor Oil Extraction. About 11.0 kg of castor seeds was used in this experiment and 69.2%wt oil was extracted. This finding showed that castor oil has a great tendency for the yield of biodiesel production, has an economic benefit, and is feasible for the production of oil. Castor oil seed contains about 30%–50% oil (m/m) [22]. Castor bean is a high-potential feedstock that might provide up to 60% of the nonedible oil required to make biodiesel [8].

3.2. Physicochemical Properties of Castor Oil. The physicochemical properties of castor oil were determined, and the results obtained are presented in Table 2. Castor oil has qualities that make it a suitable raw material for the production of biodiesel, including high viscosity, high miscibility, low iodine concentration, and low freezing point [8].

At ambient temperature, the extracted oil was yellowish, and examination revealed moisture content of 3.72%, which was higher than the expected range. Moisture content is an impurity that needs to be diminished or eliminated prior to the synthesis of biodiesel by heating crude oil [14]. The moisture content is one indication of the quality measurement of oil for biodiesel production. As the moisture content of the oil is high, the conversion of oil to biodiesel production was reduced. Another researcher indicated that the lower moisture content of oil activates the transesterification process [14]. The finding of the present study showed that the moisture content of castor seed oil was accepted for biodiesel production.

The density of refined castor oil was 0.961 (Table 2). The density showed that the refined castor oil was within the range of the ASTM standard specification. Therefore, the result indicated that the value of the specific gravity of castor oil met the criteria for biodiesel production quality indication. Another study reported that the density of extracted crude oil at 15°C was found to be 0.963 kg/lit [14] which is in agreement with the present result. The study result showed that the kinematic viscosity of castor oils was 124 mm^2/sec. Castor oil's kinetic viscosity was stated to be 196 cSt and ranged from 6.6 to 248.8 cSt in the literature, respectively [14].

The acid number is a significant property, which indicates the free fatty acid value in the fuel. The acid value of castor seed oil (2.4 mL·KOH/g oil) obtained was complying with ASTM specification quality indication standards. A higher acid value can cause severe corrosion to a fuel supply system and an internal combustion engine [5]. Thus, the result showed that castor seed oil is possible to use as feedstock for biodiesel production. The saponification number of castor oil was found at 185.2 mg·KOH/g, which was well within the range of the ASTM specification quality standard. Exactingly speaking, a lower saponification value is preferred for a higher yield of biodiesel [14]. Oil with a high level of saturated fat is not good for producing biodiesel [25]. The present finding revealed that the saponification value was acceptable for the production of biodiesel.

The iodine number was found at 86 gI_2/100 g, which was good and within the range ASTM standard value of 82–88 gI_2/100 g. The amount of double bonds in biodiesel is measured by the iodine number, which establishes how unsaturated it is. Additionally, a greater iodine number may result in the production of oxides in the diesel engine injectors [14].

3.3. Experimental and Predicted Yield of Biodiesel Using RSM. The results of the experimental and predicted yield of biodiesel from castor oil using RSM are shown in Table 3.

3.4. Physicochemical Properties of Biodiesel. The qualities of the fuel and how they affect engines are shown by the fuel attributes of biodiesel. Table 4 details the physical and chemical characteristics of castor oil-based biodiesel.

The findings showed that it complied with both European and American standards, making it suitable for use as an alternative fuel. The density of pure CME (0.89 g/ml) is within the range of the ASTM standard value. Different authors reported the density of COME (0.942) [26] and Jatropha curcas oil (0.932 g/mL) [1], which are greater than that of the present results. The density of CME falls within the range of EN 14214, which was 0.89 g/ml. Biodiesel cleanliness and fatty acid profile propose the density of fuel,

Table 2: Physicochemical properties of refined castor oil.

Parameters	Unit	Refined castor oil	ASTM standard value
Moisture content		3.72	
Specific gravity		0.961	0.957–0.968
Viscosity (at 40°C)	mm^2/sec	12.4	6.3–8.8
Acid number	mL·KOH/g oil	2.4	0.4–4
Saponification number	mg·KOH/g oil	185.2	175–187
Iodine number	—	86	82–88
Cetane number	min	56.4	47
Heating value	MJ/kg	40.6	37.5–42.8

Table 3: Experimental and predicted yield of biodiesel using RSM.

Experimental run	Experimental yield of biodiesel	Predicted yield of biodiesel
1	81.34	77.40
2	78.21	79.21
3	83.46	84.81
4	87.25	88.48
5	84.72	84.95
6	88.54	90.26
7	85.25	82.15
8	89.61	89.32
9	54.00	53.60
10	58.00	58.67
11	71.60	67.44
12	75.29	74.38
13	73.60	69.93
14	80.54	78.51
15	75.26	73.57
16	82.50	84.01
17	64.81	70.94
18	86.20	83.19
19	67.16	68.81
20	80.24	81.71
21	78.91	79.93
22	95.00	97.10
23	83.56	82.90
24	50.00	53.78
25	90.12	89.93
26	90.12	89.93
27	90.12	89.93
28	90.12	89.93
29	90.12	89.93
30	89.00	89.93

Table 4: Physiochemical properties of biodiesel (CME).

Parameter	Unit	CME	ASTM standard
Density at 15°C	g/ml	0.89	0.86–0.9
Kinematic viscosity at 40°C	cSt	5.22	1.9–6
Acid value	mg·KOH/g oil	4.3	4.3
FFA	%	2.15	2.15
Saponification value	mg·KOH/g oil	179	179
Iodine value	mgI_2/100 oil	97.4	80.12–82.88
Heating value	MJ/kg	40.6	37.5–42.8
Cetane number	min	54.8	47 min

which can be determined by the atoms of carbon [9]. The measured value of kinematic viscosity in produced castor biodiesel was 5.22 cSt and found within the range of ASTM standards of 1.9 to 6.0 cSt. The fuel properties of biodiesel presented in Table 4 are within the range of ASTM D6751 and European standard (EN14214) specification quality standard. Kinematic viscosity is an important property that is used to determine the efficiency of biodiesel as a fuel [14]. When a biodiesel having a viscosity in this range is used in a diesel engine, it helps to lubricate parts of the engine [2].

Higher kinematic viscosity causes poor atomization which forms operational problems when introduced into the chamber and leads to deposits in engines [5]. The finding revealed that the biodiesel produced from castor oil can fit with the specification of European standard (EN14214) and it is possible to use instead of petroleum diesel. The findings revealed that castor oil has high viscosity and it requires the next process to reduce its viscosity. High viscosities will result in poor atomization, incomplete combustion of fuel, and deposition of carbon in the injector [17]. The transesterification process was needed to reduce the viscosity of the pure castor oil from 12.4 to 5.22 cSt, which is within the recommended range of 1.9–6.0 by ASTM. Oils with lower viscosities are simpler to pump, atomize, and produce finer droplets [26].

The result revealed that the acid number of CME obtained was 4.3 mg·KOH/g oil which met the ASTM standard. The acid number was used to calculate the percentage of free fatty acids, specifically those derived from ricinoleic acid, which is found in greater quantity, according to the fatty acid composition profile [1]. The acid number is one of the paramount indicators of oil quality [2]. As a result, an excellent predictor of vegetable oil which is potentially useful for refining is a low acid value. Some recorded castor oil acid readings range from 0.14 to 1.97 mg/g oil. [27]. The free fatty acid obtained in this study was 2.15% which is within the range of ASTM standards. The composition of FFA determines the fuel properties of any biodiesel. The castor oil contains saturated as well as unsaturated FFAs. Its characteristics, properties, and composition were studied to examine its viability for partial replacement of petrodiesel in internal combustion engines [1, 14].

The saponification value of castor oil obtained was 179 mg·KOH/g. The average range of saponification values reported in castor oil is 165.50 to 187 mg·KOH/g oil [27]. A lower saponification value is preferred for a higher yield of biodiesel because a higher saponification value decreases yield. The iodine number of castor biodiesel produced was 97.4 mgI_2/g, which is greater than the range of ASTM 80.12–82.88 mgI_2/g. Based on the European standard (EN14214), the maximum value of iodine number was 120 mgI_2/g. The iodine number measures the double bonds present in biodiesel which determines the degree of unsaturated free fatty acids present in biodiesel. A higher iodine number may lead to deposition in diesel engine injectors [14]. A higher iodine value implies a higher level of unsaturation and a lower value also indicates a low unsaturation level. Iodine content in castor oil typically ranges from 83 to 93 g I_2 per 100 g oil [27].

The cetane number (CN) of CME (54.8 min) was greater than the standard values of both ASTM (47 min) and EN 14214 (51 min). Cetane number is considered as one of the most important properties of fuel affecting the quality of combustion and ignition delay [14]. Indicators of biodiesel ignition quality, such as the cetane number, decline as the chain length, branching, and unsaturation increase. Shorter delay between fuel injection and ignition is indicated by a higher cetane number [26]. A higher calorific value is preferred because it releases higher heat, ultimately improving the performance of the engine [14].

3.5. Fatty Acid Profile of Castor Oil. Table 5 displays the outcome of the gas chromatography analysis used to identify the castor oil fatty acid profile used throughout the experiment. The dominant fatty acid in CME was 84.1% of methyl ricinoleate (9,12-octadecadienoic acid (Z, Z)-methyl ester, 4.9% of 9-octadecenoic acid (Z)-methyl ester, 2.6% of 9-octadecenoic acid, 12-hydroxy-ethyl ester, 1.5% of methyl stearate, 1.3% of hexadecanoic acid, methyl ester, and 0.5% of 13-octadecenoic acid-methyl ester. The fatty acid methyl esters (FAME) that were produced as a byproduct of the transesterification of castor oil into biodiesel were always a combination with the majority composition being methyl ricinoleate (85%) and methyl stearate (1%) [28] which is in agreement with the present findings. The primary fatty acid in castor oil, ricinoleic acid (12-hydroxy-9-octadecenoic acid), accounts for 86% to 92% of the oil's total fatty acids [1].

Castor oil is an attractive option since it may be used in a variety of processes, especially those involving polymers and fuels. [1]. The results obtained are very much consistent with those reported in other research findings. Ricinoleic acid, a monounsaturated fatty acid, is the most prevalent among them, making up roughly 75 to 90% of the total oil composition [27].

3.6. Effect of Variables

3.6.1. Methanol-to-Oil Molar Ratio. For the purpose of applying NaOH catalysts to the transesterification of castor oil, a study was conducted to determine the amounts for each independent variable. A range of 6 : 1 to 12 : 1 was chosen for the methanol-to-oil molar ratio. According to the study's findings, reversible reactions were possible when the molar ratio was kept at less than or equal to 3 : 1. The cost of methanol increased with a molar ratio increase of more than 12 : 1, but the percentage production of biodiesel remained the same. According to another set of findings, separation of the transesterification products was made possible by raising the methanol-to-oil molar ratio from 6 : 1 to 9 : 1, which increased the methyl ester yield to 95% [24]. Then, as the molar ratio rises to 24 : 1, the yield falls from 95% to 45%, which is caused by the accumulation of methanol and the fluid's viscosity [26].

3.6.2. Catalyst Loading. When the catalyst loading was less than 1% or greater than 2%, soap production was seen during washing. The chosen range for the catalyst loading was 1% to 2%. Similar findings were reported by other researchers, who found that by raising the catalyst concentration to 1%, the COME yield rose to 95% [24]. This behaviour is caused by a strong alkaline catalyst that dominates the saponification reaction, which produces soap in the presence of fatty acids, causing an emulsion to form between soap molecules and water molecules [26].

3.6.3. Reaction Time. The production of biodiesel depends critically on reaction time, which also affects the process's costs. The reaction period ranged from 30 to 90 min since

Table 5: The composition of free fatty acids (FFAs) castor biodiesel.

Name	Formula	%
Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	1.36
Hexadecanoic acid, ethyl ester	$C_{18}H_{36}O_2$	0.03
Heptadecanoic acid, methyl ester	$C_{18}H_{36}O_2$	0.032
13-Hexyloxacyclotridec-10-en-2-one	$C_{18}H_{32}O_2$	0.21
9,12-Octadecadienoic acid (Z, Z)-, methyl ester	$C_{19}H_{34}O_2$	4.91
9-Octadecenoic acid (Z)-, methyl ester	$C_{19}H_{36}O_2$	3.73
13-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	0.50
Methyl stearate	$C_{19}H_{38}O_2$	1.50
Linoleic acid ethyl ester	$C_{20}H_{36}O_2$	0.13
Ethyl oleate	$C_{20}H_{38}O_2$	0.07
Methyl ricinoleate	$C_{19}H_{36}O_3$	84.10
Ricinoleic acid	$C_{18}H_{34}O_3$	0.37
9-Octadecenoic acid, 12-hydroxy-, ethyl ester, [R-(Z)]-	$C_{20}H_{38}O_3$	2.59
Octadecanoic acid, 9,10-dihydroxy-, methyl ester	$C_{19}H_{38}O_4$	0.47

less than 30 min of reaction time resulted in an incomplete reaction, which produced a lower yield, and more than 90 min did not significantly enhance the biodiesel yield. It is evident that the conversion gradually rises with reaction time. The instability of castor oil biodiesel at higher temperatures, which results in a thermal breakdown, may be the cause of this activity.

3.6.4. Reaction Temperature. The production of biodiesel will be impacted by a number of significant factors, including the reaction temperature. The chosen reaction temperature was in the range of 30°C to 60°C; as the temperature at which the reaction occurred was raised, the rate of reaction was discovered to increase up to a specific point. Because methanol has a boiling point of 65°C, the pressure within the vessel was raised as a result of the higher temperature. In accordance with another study, the biodiesel production rises as the reaction temperature rises, reaching an ideal level at 60°C and a biodiesel yield of 96.7%. Beyond this point, the yield rapidly dropped to 68.3% at 70°C. The maximum yield produced by transesterifying castor oil with KOH is 95.8% [7].

3.7. Optimization of Biodiesel Production. The quadratic model is acceptable for describing the experimental findings, the predictive value is significant with these data, and the "lack of fit" is also significant, according to the ANOVA results presented in Table 6. The model for the percentage yield of biodiesel (Y) in terms of the coded independent process variables is given by equation (4).

It was shown that the quadratic equation gave the best fit with a regression coefficient (R^2) of 0.9611. The model adequacy can also be checked using the factors given in Table 7.

$$\begin{aligned} Y = {} & 89.93 + 3.06X_1 + 3.23X_2 + 4.29X_3\text{-}7.28X_4 \\ & + 0.47X_1X_2 + 0.88X_1X_3 + 0.81X_1X_4\text{-}2.55X_2X_3 \\ & + 1.61X_2X_4 + 2.20X_3X_4\text{-}3.22X_1^2\text{-}3.67X_2^2\text{-}0.35X_3^2\text{-}5.40X_4^2. \end{aligned} \quad (4)$$

F-values and P values were used to assess each coefficient's significance, as shown in Table 6. For a model to be considered statistically significant, each relevant independent parameter must have a P value of less than 0.05. For the "lack of fit" in the model to be considered insignificant, the P value must be higher than 0.05. The analysis in Table 6 showed that the linear terms X_1, X_2, X_3, and X_4, the quadratic terms X_1^2, X_2^2, and X_4^2, and the interaction term X_2X_3 are significant model terms. Therefore, the model was reduced to equation (5) after eliminating the insignificant coefficients.

$$\begin{aligned} Y = {} & 89.93 + 3.06X_1 + 3.23X_2 + 4.29X_3\text{-}7.28X_4 \\ & \text{-}2.55X_2X_3\text{-}3.22X_1^2\text{-}3.67X_2^2\text{-}5.40\,X_4^2. \end{aligned} \quad (5)$$

Based on the statistical significance of the independent variable, the nonsignificant independent variables were removed from equation (4), and then the proposed equation (5) was a selected model. The findings of the analysis of variance (ANOVA) revealed that the quadratic form of the model in terms of independent parameters was an appropriate empirical model for describing the generation of biodiesel from castor oil. The findings showed that the yield of biodiesel was linearly increased with an increase in temperature, the ratio of methanol to oil, and reaction time, and decreased loading catalyst but started to decrease after reaching the optimum value. The model's applicability was further supported by the high coefficient of determination (R^2) value of 0.9611 and the adj R^2 value of 0.9248. These statistical factors account for the fact that 96.11% of the experimental data was within 2% of the percent yields that the model anticipated. The present finding was agreed with the results of other researchers [16, 17].

The "Pred R-Squared" of 0.7772 is in reasonable agreement with the "Adj R-Squared" of 0.9248 because their difference is 0.1476 which is less than 0.2. From this finding, it concluded that the developed model can be matched with actual response data. The ratio of 19.670 indicated that an adequate signal can be used to navigate the design space. The R-square value of 0.9611 indicates that 96.11% of the total variation in the yield of biodiesel was described by the developed model.

Table 6: Analysis of variance for the yield of biodiesel from castor seed oil.

Source	Sum of square	Degree of freedom	Mean square	F value	Prob > F	Remark
Model	3625.72	14	258.98	26.48	<0.0001	Significant
X_1	225.03	1	225.03	23.01	0.0002	
X_2	249.81	1	249.81	25.54	0.0001	
X_3	442.47	1	442.47	45.23	<0.0001	
X_4	1271.82	1	1271.82	130.02	<0.0001	
X_1X_2	3.47	1	3.47	0.35	0.5604	
X_1X_3	12.27	1	12.27	1.25	0.2804	
X_1X_4	10.61	1	10.61	1.08	0.3141	
X_2X_3	104.19	1	104.19	10.65	0.0052	
X_2X_4	41.44	1	41.44	4.24	0.0574	
X_3X_4	77.22	1	77.22	7.89	0.0132	
X_1^2	283.93	1	283.93	29.03	<0.0001	
X_2^2	369.16	1	369.16	37.74	<0.0001	
X_3^2	3.45	1	3.45	0.35	0.5612	
X_4^2	799.42	1	799.42	81.72	<0.0001	
Residual	146.73	15	9.78			
Lack of fit	145.68	10	14.57	69.68	<0.0001	Significant
Pure error	1.05	5	0.21			
Correlation total	3772.45	29				

Table 7: Model adequacy (validity) measures.

Std. dev.	3.13
Mean	79.82
C.V.	3.92
PRESS	840.64
R^2	0.9611
Adj R^2	0.9248
Pred R^2	0.7772
Adeq precision	19.670

A model is considered to be well if the data obtained from expert design model actual versus predicted points are along the straight line which concludes zero error. The results obtained observed that the variable having the most significance was catalyst load. It is also shown that the F values and P values of interactions showed that interactive effects of molar ratio of methanol to oil versus time and time versus catalyst load are significant on the yield of biodiesel. In comparison with the squared terms of temperature, time, and the molar ratio of methanol to oil, the linear impacts of these variables are more important and have a favourable impact on the output of biodiesel. There was also a good agreement in actual and modeled yield as shown in Figure 1. The result showed effectiveness in capturing the correlation between the four transesterification process variable yields of biodiesel.

3.7.1. Time versus the Molar Ratio of Methanol to Oil. The interaction between reaction time versus the molar ratio of methanol to oil had a linear significant effect on the yield of biodiesel with temperature and loading catalyst kept constant at 50°C and 1.5%, respectively, at the center of response surface methodology (Figure 2). The yield of biodiesel was increased until the time and the molar ratio of biodiesel reached the optimal point, but the yield of biodiesel was decreased after the time and the molar ratio of methanol to oil passed the optimal point. The findings showed that if the time and the molar ratio of methanol to oil increased after optimum value, it became difficult to separate the biodiesel production from glycerol during the process, causing soap development.

The effective interaction between reaction time and methanol-to-oil molar ratio on the yield of biodiesel is shown in 3D in Figure 3. The maximum yield of biodiesel was obtained when the methanol-to-oil ratio was 9 : 1 and the time required was 75 min for completion of the process. As the reaction time increased from 60 min, the biodiesel yield started to increase and the maximum yield of biodiesel obtained was 90.89% at 90 min and a molar ratio close to 9.0. If the molar ratio of methanol to oil became high during the process, soap was produced instead of the biodiesel. Therefore, the time and molar ratio had a significant effect on the yield of biodiesel.

3.7.2. Time versus Catalyst Load. The interaction of time versus loading catalyst had a linear relationship between them with the temperature and molar ratio of methanol to oil kept constant at 50°C and 9 : 1, respectively, at the optimum point as shown in Figure 4. As the time and catalyst load were increased, the yield of biodiesel also increased. Also, after the optimum value was obtained, the yield of biodiesel also increased, but this was not feasible due to high consumption time and cost. Therefore, the time versus loading catalyst had a significant effect on the yield of biodiesel.

The yield of biodiesel increased as the loading catalyst increased from 1 to 1.5%wt with the temperature and molar ratio of methanol to oil kept constant at 50°C and 9 : 1, respectively, at the middle of the Design Expert software as shown in Figure 5. At time versus loading catalyst 75 min and 1.5%wt, respectively, 94% maximum biodiesel yield was achieved. Therefore, the loading catalyst and time had a significant effect on the yield of biodiesel.

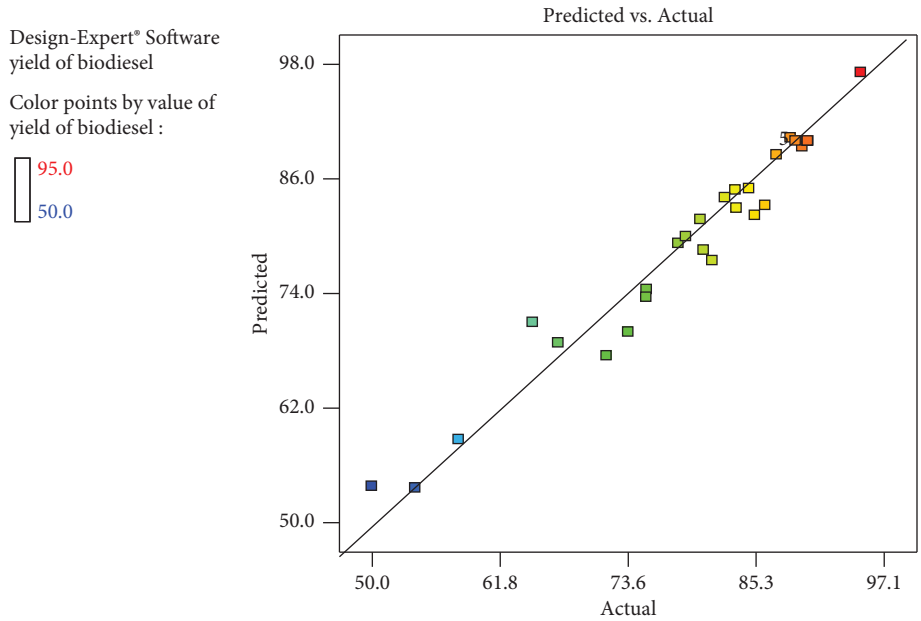

Figure 1: Predicted versus actual percent yield of biodiesel from castor oil.

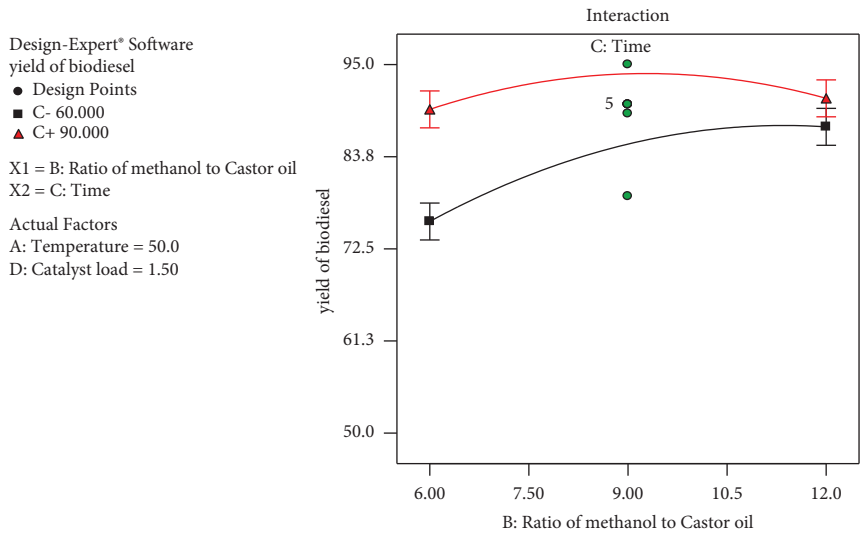

Figure 2: The effect of the interaction between the ratio of methanol to oil and time.

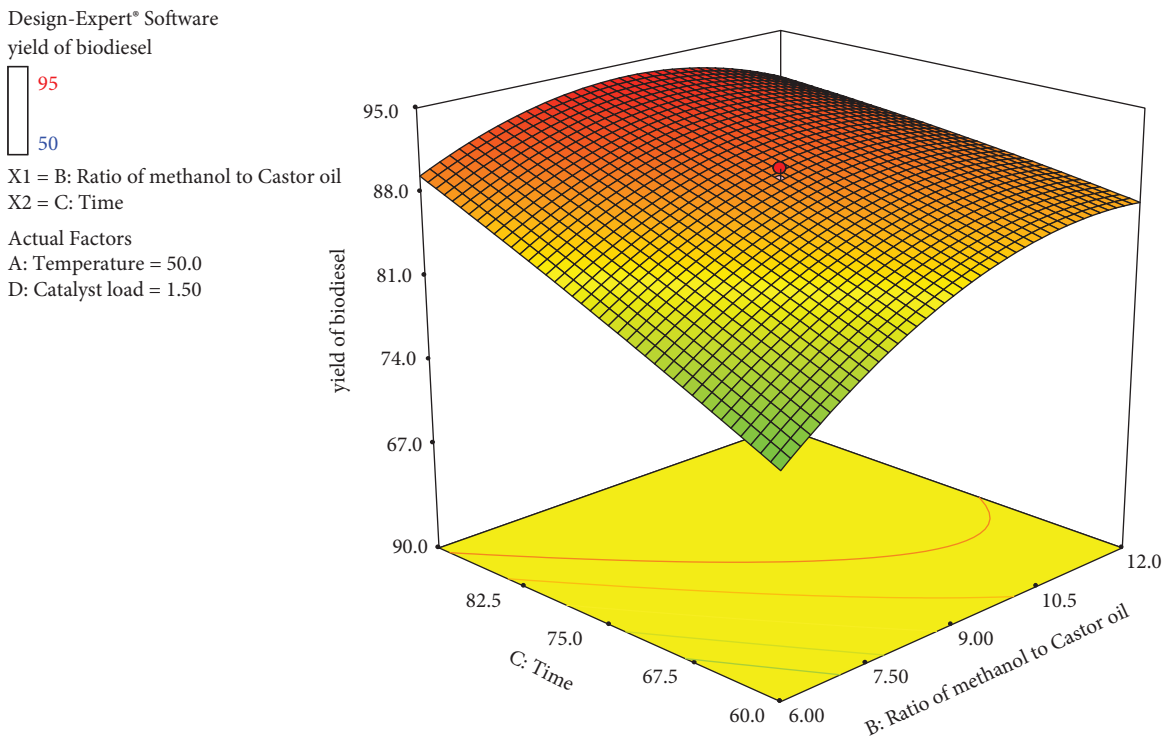

Figure 3: The effect of the interaction between the ratio of methanol to oil and time on the yield of biodiesel using 3D plots.

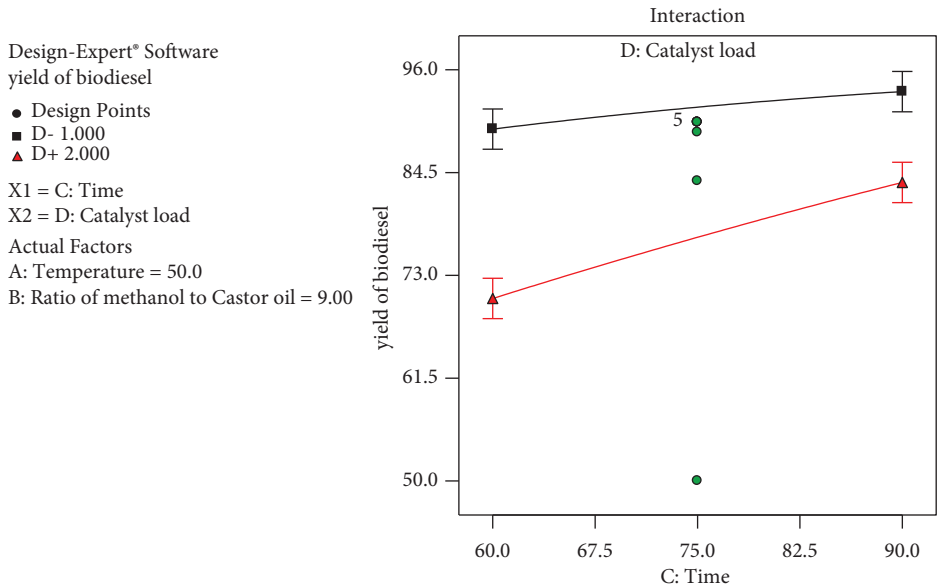

Figure 4: The effect of the interaction between the ratio of loading catalyst and time on the yield of biodiesel.

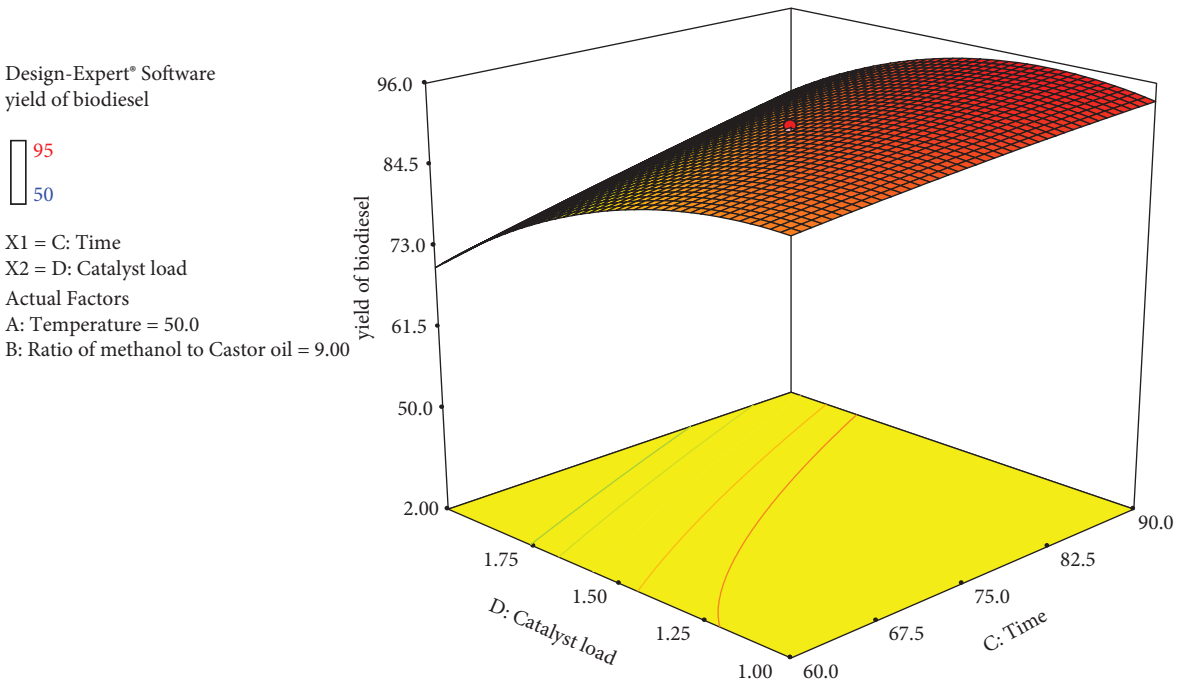

Figure 5: The effect of the interaction of loading catalyst versus time using 3D plots.

4. Conclusion

With the help of response surface methodology's central composite design, the synthesis of biodiesel from castor oil, which is obtained from castor seeds and treated with a NaOH catalyst, was successfully optimised. The study was mainly based on biodiesel transesterification optimization derived from novel nonedible castor seed oil. In this study, different independent factors were investigated for the optimization of the yield of biodiesel. To attain the best operating conditions, response surface methodology was used to adjust the temperature, time, catalyst loading percentage, and methanol-to-oil ratio. According to the findings, the best operating conditions for transesterifying castor oil were a 9 : 1 molar ratio of methanol to oil, a 1.5% catalyst loading percentage, a temperature of 50°C, and a reaction duration of 105 min with a 95.0% yield. The findings showed a significant increase in fuel properties of castor methyl esters (CME) such as kinematic viscosity (5.22 mm^2/s), acid number (4.3 mg KOH/g), cetane number (54.9 min), saponification value (179 mg·KOH/g oil), iodine number (97.4 mg I_2/100g oil), FFF (2.15%), and density (890 kg/m^3which fall in the range of ASTM and EN14214. The study concluded that biodiesel produced from castor oil is within the recommended standards for biodiesel fuel. In addition to highlighting its potential for large-scale manufacturing, the castor oil biodiesel that complies with ASTM criteria helps to further sustain development targets. It can be concluded that the biodiesel produced from castor oil can be used as fuel.

Data Availability

All the data used in this study are included in the manuscript.

Disclosure

The research was performed as part of the Addis Ababa Science and Technology University.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Authors' Contributions

KA and ET contributed to experimental design, experimental supervision, statistical analysis, and manuscript writing and editing. TG and SK contributed to editing the manuscript. Finally, data were collected by ET.

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References

[1] G. Torrentes-espinoza, B. Miranda, J. Vega-Baudrit, J. Matasegreda, and S. Rica, "Castor oil (Ricinus communis) supercritical methanolysis," *Energy*, vol. 140, pp. 426–435, 2017.

[2] S. B. L. Ngomade, R. D. T. Tchuifon, R. F. T. Tagne et al., "Optimization by response surface methodology of biodiesel production from podocarpus falcatus oil as a Cameroonian novel nonedible feedstock," *Journal of Chemistry*, vol. 2022, Article ID 3786602, 14 pages, 2022.

[3] A. M. Rabie, E. A. Mohammed, and N. A. Negm, "Feasibility of modi fi ed bentonite as acidic heterogeneous catalyst in low temperature catalytic cracking process of biofuel production from nonedible vegetable oils," *Journal of Molecular Liquids*, vol. 254, pp. 260–266, 2018.

[4] S. Kumar, S. Jain, and H. Kumar, "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," *American Chemical Society Omega*, vol. 5, no. 28, pp. 17033–17041, 2020.

[5] K. Hasni, Z. Ilham, S. Dharma, and M. Varman, "Optimization of biodiesel production from Brucea javanica seeds oil as novel non-edible feedstock using response surface methodology," *Energy Conversion and Management*, vol. 149, pp. 392–400, 2017.

[6] R. Foroutan, H. Esmaeili, and G. Ff, "Optimization of biodiesel production from waste cooking oil by alkaline catalysts," *Journal of Food Technology and Food Chemistry Research*, vol. 1, no. 41, pp. 1–7, 2018.

[7] S. Ismail, A. S. Ahmed, R. Anr, and S. Hamdan, "Biodiesel production from Castor oil by using calcium oxide derived from mud clam shell," *Journal of Renewable Energy*, vol. 2016, Article ID 5274917, 8 pages, 2016.

[8] C. S. Osorio-gonz, G. Natali, and F. Sandoval-salas, "Production of biodiesel from Castor oil: a review," *Energies*, vol. 13, no. 10, pp. 1–22, 2020.

[9] D. Beyene, M. Abdulkadir, and A. Befekadu, "Production of biodiesel from mixed Castor seed and microalgal oils: characterization and optimization studies (Ca)," *Research Square*, 2019.

[10] A. A. Siyanbola, P. A. Obasa, and I. Shaba, "Extraction and optimization of azardirachta indica seed oil for biodiesel," *International Journal of Research Publications*, vol. 44, no. 1, 2020.

[11] O. U. Dairo, T. M. Olayanju, E. S. A. Ajisegiri, and O. J. Alamu, "Optimization of in-situ biodiesel production from raw Castor oil-bean seed," *Journal of Energy Technologies and Policy*, vol. 3, no. 13, pp. 14–20, 2013.

[12] S. Niju, G. Vishnupriya, and M. Balajii, "Process optimization of Calophyllum inophyllum-waste cooking oil mixture for biodiesel production using Donax deltoides shells as heterogeneous catalyst," *Sustainable Environment Research*, vol. 29, no. 1, pp. 18–12, 2019.

[13] D. O. Onukwuli, L. N. Emembolu, C. N. Ude, S. O. Aliozo, and M. C. Menkiti, "Optimization of biodiesel production from refined cotton seed oil and its characterization," *Egyptian Journal of Petroleum*, vol. 26, no. 1, pp. 103–110, 2017.

[14] M. M. Tunio, S. R. Samo, Z. M. Ali, A. Q. Jakhrani, and K. C. Mukwana, "Production and characterization of biodiesel from indigenous Castor seeds," *International Journal of Engineering and Applied Sciences*, vol. 3, no. 7, pp. 28–33, 2016.

[15] N. Mohammadi, N. Ostovar, R. Niromand, and F. Absalan, "Advancing biodiesel production from Pyrus glabra seed oil: kinetic study and RSM optimization via microwave-assisted transesterification with biocompatible hydroxyapatite catalyst," *Sustainable Chemistry and Pharmacy*, vol. 36, Article ID 101272, 2023.

[16] K. S. Obayomi, J. O. Bello, T. A. Ogundipe, and O. Olawale, "Extraction of castor oil from castor seed for optimization of biodiesel production," *Institute of Physics Conference Series: Earth and Environmental Science*, vol. 445, no. 1, Article ID 12055, 2020.

[17] T. Ahmad, M. Danish, P. Kale et al., "Optimization of process variables for biodiesel production by transesterification of flaxseed oil and produced biodiesel characterizations," *Renewable Energy*, vol. 139, pp. 1272–1280, 2019.

[18] A. Garba, M. M. Abarshi, M. B. Shuaib, and R. Sulaiman, "Optimization of biodiesel production from castor oil by response surface methodology," *Nigerian Journal of Biotechnology*, vol. 33, no. 1, pp. 66–71, 2017.

[19] K. N. Krishnamurthy, S. N. Sridhara, and C. S. Ananda Kumar, "Synthesis and optimization of Hydnocarpus wightiana and dairy waste scum as feed stock for biodiesel production by using response surface methodology," *Energy*, vol. 153, pp. 1073–1086, 2018.

[20] M. G. Nayak and A. P. Vyas, "Optimization of microwave-assisted biodiesel production from Papaya oil using response surface methodology," *Renewable Energy*, vol. 138, pp. 18–28, 2019.

[21] S. M. Safieddin Ardebili, T. T. Hashjin, B. Ghobadian, G. Najafi, S. Mantegna, and G. Cravotto, "Optimization of biodiesel synthesis under simultaneous ultrasound-microwave irradiation using response surface methodology (RSM)," *Green Processing and Synthesis*, vol. 4, no. 4, pp. 259–267, 2015.

[22] V. R. Patel, G. G. Dumancas, L. C. K. Viswanath, R. Maples, and B. J. J. Subong, "Castor oil: properties, uses, and optimization of processing parameters in commercial production," *Lipid Insights*, vol. 9, pp. 1–12, 2016.

[23] T. Tamoradi, A. R. Kiasat, H. Veisi, V. Nobakht, and B. Karmakar, "RSM process optimization of biodiesel production from rapeseed oil and waste corn oil in the presence of green and novel catalyst," *Scientific Reports*, vol. 12, no. 1, pp. 19652–19715, 2022.

[24] M. Mahatale, S. Kapoor, and D. Patil, "Optimization study of biodiesel production from used sunflower oil," *International Journal of Research and Innovation in Applied Science (IJRIAS)*, vol. 2, no. 7, pp. 1–4, 2017.

[25] A. Banerjee, D. Varshney, S. Kumar, P. Chaudhary, and V. K. Gupta, "Biodiesel production from castor oil: ANN modeling and kinetic parameter estimation," *International Journal of Integrated Care*, vol. 8, no. 3, pp. 253–262, 2017.

[26] S. T. Keera, S. M. El Sabagh, and A. R. Taman, "Castor oil biodiesel production and optimization," *Egyptian Journal of Petroleum*, vol. 27, no. 4, pp. 979–984, 2018.

[27] X. Yeboah, A. Ying, S. Lu et al., "Castor oil (Ricinus communis): a review on the chemical composition and physicochemical properties," *Food Science and Technology*, vol. 41, no. 2, 2020.

[28] S. Sembiring, N. Bangun, J. Kaban, and J. Bangun, "Purification of ricinoleic acid methyl ester using mesoporous calcium silicate (CaSiO 3) adsorben," *Journal of Physics: Conference Series*, vol. 1116, no. 4, Article ID 42031, 2018.