

Statistical modeling and optimization of biodiesel production from custard apple seed oil and characterization

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Research Article

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Posted Date: April 20th, 2022

DOI: <https://doi.org/10.21203/rs.3.rs-1554808/v1>

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Statistical modeling and optimization of biodiesel production from custard apple seed oil and characterization

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Abstract

The best condition for the biodiesel synthesis process from a non-edible and renewable source, custard apple seed oil, is presented in this paper, as well as its characterization. The seeds were harvested from Ethiopia's Jimma zone, and their average moisture, ash, fiber, protein, and oil contents are 5.7, 3.9, 2.1, 19.8, and 37.2 percent, respectively. Soxhlet and hexane as a solvent were used to extract the oil. Trans-esterification of oil with methanol and sodium hydroxide (NaOH) as a catalyst for biodiesel generation was investigated. The molar ratio of methanol to oil, temperature, catalyst, and reaction time all had a significant ($p < 0.05$) impact on biodiesel yield. The optimum process parameters coupled to obtain the highest biodiesel output were reaction temperature of 72.426°C, catalyst 2.002wt %, methanol: oil molar ratio 12.334, and reaction time of 84.010min, according to the Experimental Design Response Surface Methodology (RSM) and ANOVA analysis. With a desirability of 1, the model anticipated a yield of 89.230±0.28 percent. Acid value, Saponification value, Kinematic viscosity, Density, Heating Value, Fire Point, and Flash Point are all physicochemical parameters of biodiesel made from custard apple seed oil (FP) 1.12 mg KOH/g, 16.75 mg KOH/g, 5.67mm²/s, 894.5kg/m³, 42.072kJ/kg 134oC and 179oC satisfied the ASTM criteria for biodiesel, respectively. In general, this research supports the manufacturing of biodiesel from Custard Apple seeds (*Annona squamosa*) oil as a means of replacing non-renewable energy with renewable energy and increasing the value of custard apple fruit.

Keywords: Biodiesel, Custard apple seed, Trans-esterification, Solvent extraction, RSM

1. Introduction

Energy consumption is constantly increasing all over the world despite the rationalization measures that have been undertaken. Liquid fossil fuels are the main and most frequently used fuels for mobile machinery([Rajeshkumar, et 2018](#)). Because the entire development of mobile

machinery is based on the use of liquid fossil fuel, it is difficult to expect a shift from this trend to mass development and the use of new engine constructions that would be suitable for some other types of fuel. Fossil fuels are the major sources of energy demand since their exploration; these are non-renewable, scarce, and extensive use leads to environmental pollution(Yathish et al. 2013).

During the last decade, biodiesel has become the most common renewable liquid fuel. Custard apple belongs to the family of *Annonaceae* with the botanical name *Annona squamosa*, which is commonly found in deciduous forests, also cultivated in wild in various parts of the world. Residual agricultural products and food-processing by-products or wastes are often considered a problem(Hotti and Hebbal 2015). After processing, a large amount of plant material often remains without any application. The conversion of such material into a valuable product can be a good contribution to the reduction of waste. Biodiesel is one of the renewable energy sources and its alternative to substitute diesel (which is from crude oil). Biodiesel is produced from vegetable oil and different seeds(Sani and Hasnan 2012). One of those seeds is custard apple seed. In Ethiopia, a high quantity of custard apples is planted in the southern region and Jimma zone. But the people used only to eat the internal part and throw the seeds into the environment. It is possible to collect the seeds and convert them into valuable products such as biodiesel and oil.

The studies have been focused on discovering the fuel that would be adaptable to the existing engine constructions and that would meet the criteria regarding renewability, ecology, and reliability of use. These studies aim to study the proximate composition of a custard apple seed, produce biodiesel from custard apple seed oil by the trans-esterification process by using sodium hydroxide as a catalyst, statistical modeling by using RSM software and analyzing the effect of Temperature, Reaction time, and the molar ratio of Methanol: oil. Response surface methodology (RSM) is a way of establishing an empirical relationship between process factors and desired responses or product qualities using a combination of mathematical and statistical analysis of experimental data. It provides a complete experimental design for data exploration, model fitting, as well as process optimization(Bacha 2022). The study also includes the physicochemical properties of biodiesel and compares it with the existing and international standards of biodiesel.

2. Materials and Methods

2.1.Sample preparation

Custard apple (*Annona squamosa*) seed, n-Hexane, Methanol, Sodium hydroxide, Starch, Sulfuric Acid, Potassium Iodide (KI), Hydrochloric Acid (HCl), H₂SO₄, 0.1N Na₂S₂O₃ were among the chemicals and raw materials utilized in the process of oil extraction, transesterification and biodiesel characterization process. All of the chemicals were purchased from Heparin and Volumetric Chemicals in Addis Ababa, Ethiopia, and were analytical grade. Crushers, sieves, ovens, muffle furnaces, fiber analyzers, fat analyzers, Soxhlet setups, electric heaters, digital weight balances, viscometers, refractometers, rotary evaporators, pipettes, biurets, and titration setups were utilized. Custard apple seeds were harvested in Ethiopia's Jimma town and processed to remove undesired contaminants. The samples were also milled to a uniform homogeneous powder to improve the surface-to-volume ratio, which improves solvent extraction. Light was kept away from the samples. Figure 1 shows a simple experimental flow diagram to produce biodiesel through the transesterification process.

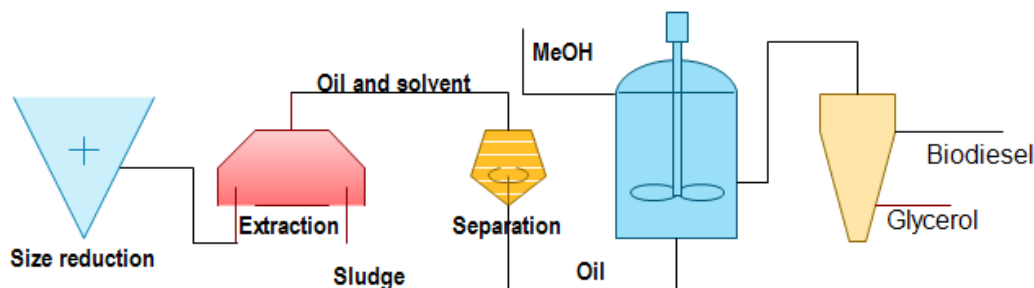


Figure 1 simple flow diagram for biodiesel production

2.2. Proximate composition of a custard apple seed

Proximate analysis of custard apple seeds sample for determining moisture, ash, crude protein, crude fiber, and oil content was carried out by adopting (Al-mentafji 2016) methods.

2.3. Extraction of oil

The oil from the apple seeds was extracted using a Soxhlet equipment. The extraction procedure was carried out with the help of (Shumi and Bacha 2022). The processing parameters and their values have a positive impact on oil yield. The following equation was used to compute the oil yield from the extraction

$$\text{Yield of oil (\%)} = \frac{L_1 - L_2}{L_1} \times 100$$

Where L_1 refers to the mass of the seed powder before extraction and L_2 refers to the mass of the sample after extraction.

2.4. Transesterification process

In the trans-esterification phase, glyceride (oil) and alcohol are combined in the presence of a catalyst, sodium hydroxide (NaOH). Based on the established data of the parameters, the process was carried out according to ([Surface et al. 2022](#)),([Raja et al. 2011](#)). Custard apple seed oil (CASO) (triglycerides) was trans esterified with methanol in the presence of a NaOH catalyst to generate biodiesel (Methyl ester). Response surface approach was used to optimize this process (RSM). A second-order mathematical model was used to analyze the statistical data, and the yield was predicted as a function of the methanol/oil molar ratio, temperature, catalyst and reaction time. A funnel was used to separate the transesterification biodiesel and the co-product glycerin based on their specific gravity.



Figure 2 Biodiesel and glycerol separation

2.5. Experimental Design

RSM is a tried-and-true method for developing models that combines statistical and mathematical approaches. It can also be used to assess the impact of various factors. It is used to find the best condition within a set of parameters. Using RSM, the impact of the specified operating parameters temperature (T), catalyst, reaction time (t), and the molar ratio of methanol to oil on the output variable (biodiesel yield) was investigated ([Raja et al. 2011](#)),([Singh et al. 2021](#)). As shown in the equation below, the output variable, biodiesel yield (percent), can be written as a function of the specified variables ([Shumi and Bacha 2022](#)).

$$yield = p_o + \sum_{i=1}^n b_i X_i + \sum_{i=1}^n b_{ii} X_i^2 + \sum_{i=1}^{n-1} \sum_{j=2}^n b_{ij} X_i X_j + \lambda$$

$X_1; X_2; X_3; \dots X_n$ denotes the input independent factors that can affect the output yield; n denotes the number of parameters; p_o denotes the constant; b_{ii} ($i = 1, 2, \dots, n$) denotes the quadratic term coefficient; b_{ij} ($i = 1, 2, \dots, n; j = 1, 2, \dots, n$) denotes the interaction term coefficient; and λ denotes the random error. Temperature, catalyst, reaction duration, and the molar ratio of methanol: oil were chosen as independent parameters in this investigation, and the response was the transesterification yield of CASO biodiesel.

The process was carried out for predefined configurations of process parameters, such as temperature, catalyst concentration, reaction time, and molar methanol-to-oil ratio. The maximum, lowest, and average values of selected independent parameters taken for this experiment are shown in [Table 1](#). Using Design-Expert Software, the trials were planned using a Box-Behnken design (BBD). For the combination of parameters, BBD provides a predetermined experimental design. Twenty-nine (29) transesterification experiments were carried out using BBD.

Table 1 Levels of chosen parameters for Experimental Design

Factors	Units	Minimum (-1)	Average (0)	Maximum (+1)
Temperature	°C	65	70	75
Reaction Time	min	45	67.5	90
Methanol: Oil ratio	v/v	7	10	13
Catalyst	Wt. %	2	3	4

Based on these arrangements, in each cycle, the amount of biodiesel was determined by separating the byproduct glycerin using a separator funnel. Further, the produced biodiesel was stored in a bottle in the refrigerator for further analysis.

2.6. Physicochemical properties of CASO Biodiesel

The acid value, saponification value, and specific gravity of the biodiesel produced were determined according to ([AOAC 2000](#)).

The specific gravity of oil was assessed using a pycnometer bottle by adopting ([Abdulmumin et al. 2017](#)). the relationship is given in Equation (2).

$$\text{Specific gravity}(SG) = \frac{W_o - W}{W_1 - W}$$

Where W= Weight of blank bottles, W_o= weight of the blank bottle and the oil, W₁= weight of the bottle and distilled water content.

The dynamic viscosity of CASO was assessed using a vibro-viscometer (TA Instruments, ARES-G2). Then, the kinematic viscometer was calculated using the relationship in equation (4).

$$\text{Kinematic viscosity} = \frac{\text{dynamic viscosity of CASO}}{\text{density of CASO}}$$

2.7.Determination of Heating Value, Fire point, and flashpoint

When a hydrocarbon (fuel) undergoes complete combustion with oxygen under standard conditions, the energy generated in the form of heat is known as the calorific value. A bomb calorimeter is used to measure it. A hydrocarbon reacts with oxygen to produce carbon dioxide, water, and heat in a chemical reaction. Biodiesel's flashpoint, fire point, and viscosity were established using methods adopted from (Sivaramakrishnan and Ravikumar 2011), (YungChee et al 2013).

3. Result and Discussion

3.1.Proximate composition of a custard apple seed

The average value of composition of the Custard apple seed exhibit was determined and revealed in table 2. The result is also incomparable with the Jatropha seed.

Table 2 Proximate composition of custard apple seed and comparison

Composition (%)	Custard apple seed	Jatropha seed	References
Moisture content	5.7 ±0.1	6.20	(Raja et al. 2011)
Protein content	19.8 ±0.3	18.00	(Raja et al. 2011)
Ash content	3.9 ±0.05	4.72	(Lee et al. 2015)

Fiber content	7.1 $\pm$ 0.03	15.50	(Raja et al. 2011)
Oil content	37.2 $\pm$ 0.21	25-30	(Raja et al. 2011)

3.2.Experimental design and biodiesel yield analysis

The studied factors, temperature (T), catalyst, methanol molar ratio, and reaction time (t), were grouped into multiple combinations to improve the transesterification process, as shown in [Table 3](#). The process was repeated twice, and the mean biodiesel yield from the experiments, as well as the predicted values for the corresponding combinations, are presented below.

Table 3 BBD of constraints and the actual and predicted values

Run	Reaction temperature (°C)	Catalyst (wt.%)	Methanol: oil molar ratio(mol/mol)	Reaction time	Actual Yield (%)	Predicted value (%)
1	65	3	10	90	77	76.83
2	65	3	7	67.5	68	66.38
3	70	2	7	67.5	72	71.36
4	70	4	7	67.5	78	79.78
5	70	2	13	67.5	82	80.81
6	70	3	10	67.5	74.7	75.24
7	65	3	13	67.5	70.5	69.03
8	70	3	10	67.5	76.1	75.24
9	75	3	10	90	70.2	69.73
10	70	3	10	67.5	76.4	75.24
11	70	3	10	67.5	74.7	75.24
12	70	4	10	90	84.7	85.80
13	70	3	7	45	60.1	59.80
14	65	3	10	45	50.2	51.26
15	70	3	10	67.5	74.3	75.24
16	70	3	13	45	59.5	59.60
17	70	3	7	90	81.5	80.01
18	65	4	10	67.5	79.2	79.61
19	70	4	13	67.5	76.8	78.03
20	75	2	10	67.5	72.8	71.00
21	70	2	10	45	59	58.71
22	75	3	13	67.5	62	64.43
23	70	3	13	90	89	87.91
24	75	3	10	45	46	46.76
25	70	4	10	45	66	64.68
26	70	2	10	90	84	86.13
27	65	2	10	67.5	58	59.80

28	75	3	7	67.5	57.1	59.38
29	75	4	10	67.5	60	56.81

3.3. RSM Model for the Transesterification process

The choice of the model's equation is critical in providing sufficient knowledge for process modeling. Furthermore, determining the statistical significance of model variables is critical for prospective biodiesel yield maximization optimization. Equation models were developed based on the CASO yield (fed as a response) experimental design and the evaluated ANOVA analysis. For several types of models, Table 4 shows the observed values of standard deviation, R-square, R-square (predicted), R-square (adjusted), and their significance.

Table 4 Statistical Summary of the Models

Source	Std. Dev.	R ²	Adjusted R ²	Predicted R ²	
Linear	7.36	0.5985	0.5315	0.3897	
2FI	7.26	0.7066	0.5436	0.1120	
Quadratic	2.00	0.9828	0.9655	0.9053	Suggested
Cubic	1.25	0.9971	0.9864	0.7374	Aliased

The quadratic model appeared to be more significant based on the results. In addition, the quadratic model revealed that lack of fitness had no bearing. As a result, the quadratic model can be used to describe the relationship between biodiesel yield and independent parameters. The coefficient of determination (R²) and adjusted coefficient of determination (R²adj) are both 0.9828 and 0.9655, indicating that the computed model adequately matches the experimental data. It means that the experimental factors analyzed are responsible for 98.28% of the overall variation in yield percentage. R² should be at least 0.85 for a model to fit well, according to (Leivisk 2013), (Shumi and Bacha 2022). The R² for these response variables was greater than 0.85, indicating that the regression models correctly predicted the outcome. In terms of the quadratic model, the process model for transesterification process is provided in the Equation.

$$\text{Biodiesel yield} = +75.24 - 2.90A + 1.41B + 1.92C + 12.13D - 8.50AB + 0.6000AC - 0.6500AD - 2.80BC - 1.57BD + 2.02CD - 10.56A^2 + 2.13B^2 + 0.1258C^2 - 3.54D^2$$

The normal plot of residuals and experimental against projected oil yield is shown in Figure 3. A linear distribution can be seen in the predicted versus actual plot, indicating that the model is well-fitting. The predicted oil yield values were very similar to the actual yield values.

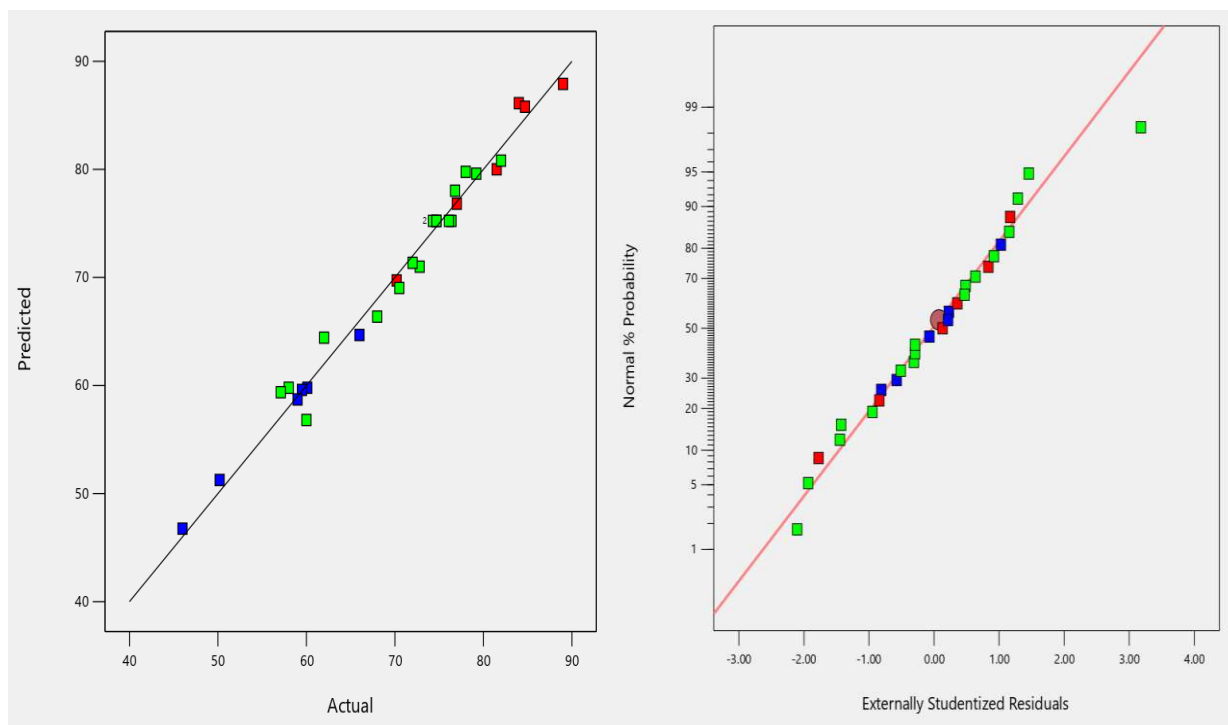


Figure 3 Analytical plots for acceptability of proposed model a) Actual Vs Expected plot b) Normal plot of residual

3.4. Analysis of variance (ANOVA) for Quadratic model

Analysis of variance (ANOVA) for the process model is given in [Table 4](#). The table shows the significance of different terms.

Table 5 Analysis of variance for transesterification process

Source	Sum of Squares	Mean Square	F-value	p-value	
Model	3178.85	227.06	57.03	< 0.0001	significant
A-Temperature	100.92	100.92	25.35	0.0002	
B-Catalyst	23.80	23.80	5.98	0.0283	
C-Methanol: Oil molar ratio	44.47	44.47	11.17	0.0048	
D-Reaction time	1766.61	1766.61	443.70	< 0.0001	
AB	289.00	289.00	72.59	< 0.0001	
AC	1.44	1.44	0.3617	0.5572	

AD	1.69	1.69	0.4245	0.5253	
BC	31.36	31.36	7.88	0.0140	
BD	9.92	9.92	2.49	0.1367	
CD	16.40	16.40	4.12	0.0618	
A²	723.56	723.56	181.73	< 0.0001	
B²	29.31	29.31	7.36	0.0168	
C²	0.1027	0.1027	0.0258	0.8747	
D²	81.13	81.13	20.38	0.0005	
Residual	55.74	3.98			
Lack of Fit	52.19	5.22	5.88	0.0513	not significant
Pure Error	3.55	0.8880			
Cor Total	3234.59				

The F-value of 57.03 for the model indicates that it is significant. An F-value of this magnitude has a 0.01 percent chance of occurring due to noise. Model terms with P-values less than 0.0500 are significant (Bullo and Bayisa 2022). A, B, C, D, AB, BC, A², B², D² are significant model terms in this situation. The model terms are not significant if the value is bigger than 0.1000. Model reduction may improve your model if there are many inconsequential model terms (not including those required to support hierarchy). With a Lack of Fit F-value of 5.88, there's a 5.13 percent possibility that a significant Lack of Fit F-value is caused by noise. It's not good to desire the model to fit. This low chance (less than 10%) is concerning. As a result, it is obvious that the resulting equation provides excellent model fit.

3.5.Effect of individual parameters

3.5.1. Temperature influence on transesterification process

The temperature in the transesterification process had a big impact on the conversion of triglycerides to biodiesel. The temperature is the most important factor that influences biodiesel yield. Figure 4A shows how the reaction was carried out at various temperatures ranging from 65 to 75 degrees Celsius. As the temperature rises, the boiling temperature of methanol rises, and the yield of biodiesel rises with it. The conversion of triglyceride to methyl ester is lowered at the greatest temperature.(Shehu and Clarke 2020). The yield of biodiesel was achieved at 46% at 89%.

Conversely, the highest conversion is achieved at the mid-temperature of 72°C, Hence, the temperature has a more extensive cause.

3.5.2. Effect of catalyst on transesterification process

The reaction was carried out by using Sodium hydroxide catalyst with different weight percentage. Depending on the predetermined experimental design and the selected sodium hydroxide weight was from 2 to 4wt%. [figure 4B](#) shows effect of catalyst on the yield of biodiesel. High yield of methyl ester was 89.230 which obtained at 4wt% and the optimized point was 2.002wt%. The increment in yield is due to forward reaction is being favorable at high catalyst concentrations and the formation of methyl ester is high.

3.5.3. Effect of Methanol: oil molar ratio

One of the important factors affecting the conversion of custard apple seed oil to biodiesel, as well as the overall processing cost of biodiesel, is the mole ratio of methanol to oil. More methanol is needed to complete the transesterification reaction([Dukare et al 2010](#)). In contrast, in order to strengthen the reaction towards complete conversion, the methanol to oil mole ratios have to be greater than the stoichiometric relative quantity ([Okullo et al 2010](#)). The mole ratio of methanol to oil was investigated in this study, which ranged from 7 to 13:1. [Figure 4C](#) depicts the effect of the methanol mole ratio on biodiesel conversion; it was discovered that when the mole ratio increases, the CASO to biodiesel conversion increases. The best conversion of 89 percent was attained at a ratio of 12.334:1. It was also discovered that due to the mass transfer constraint, supplemental increases in mole relative amount of methanol reduced the pace of biodiesel conversion([Kasirajan 2021](#)).

3.5.4. Influence of reaction time on transesterification method

To complete the transesterification reaction, you'll need a long enough contact time. The impacts of reaction time were investigated throughout a range of time periods from 45 to 90 minutes. The cause of reaction time on the conversion of oil to biodiesel is shown in [Figure 4D](#). It was observed that as the transesterification reaction time increased, the conversion of biodiesel increased. At 84 min of reaction time, the maximal conversion of triglyceride to biodiesel was attained. As the reaction time is increased, no significant changes in the conversion of triglyceride to biodiesel are detected. As a result of the backward reaction of transesterification, which results in a thrashing of

esters as well as producing more fatty acids to form soaps, an excess reaction period in time will result in a drop in product yield([Kasirajan 2017](#)).

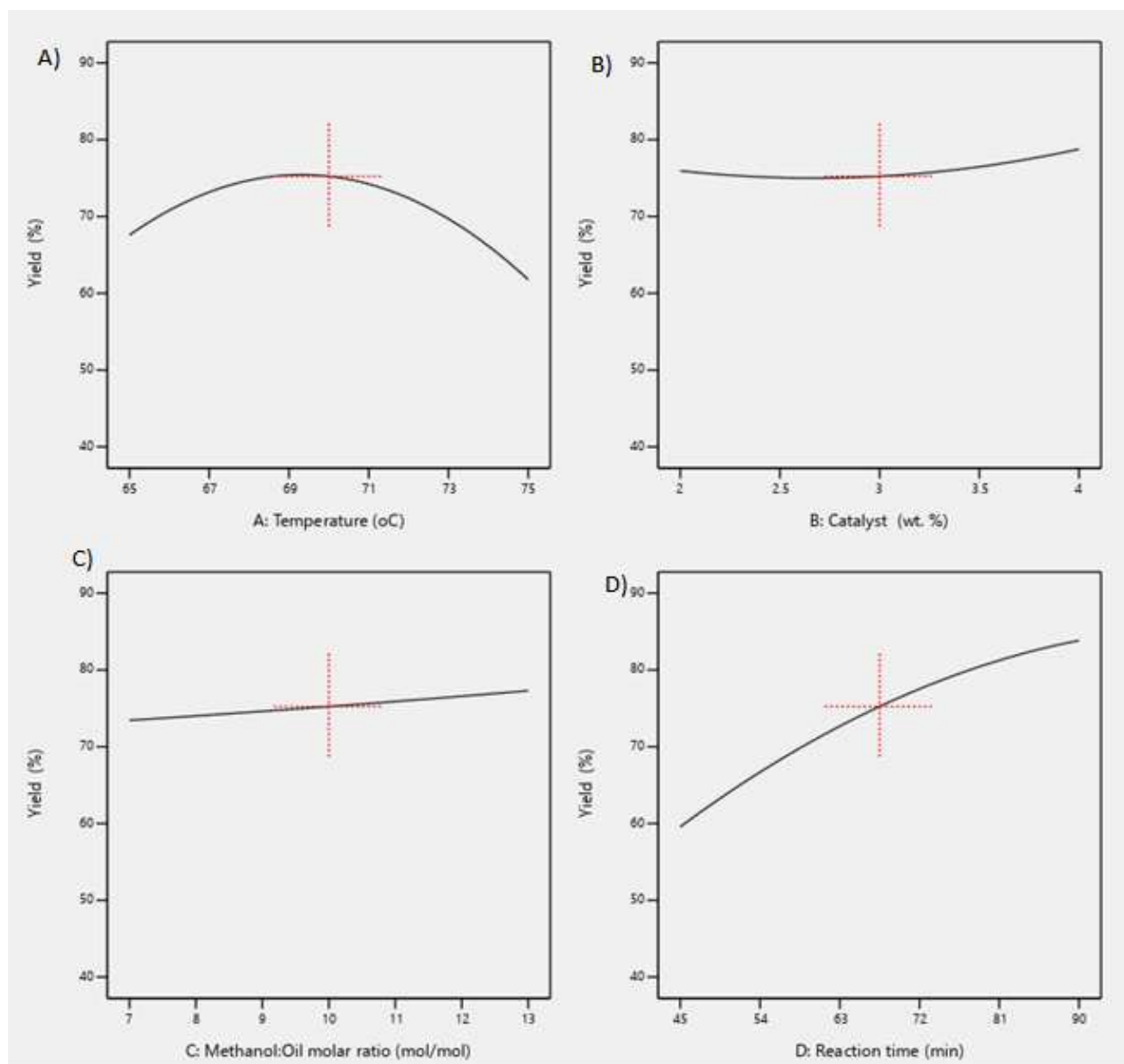


Figure 4 Individual effect of parameters A) Temperature(°C), B) Catalyst(wt%) C) Methanol: oil molar ratio(mol/mol) and D) Reaction time(min) on transesterification process

3.6.Interaction effect of parameters

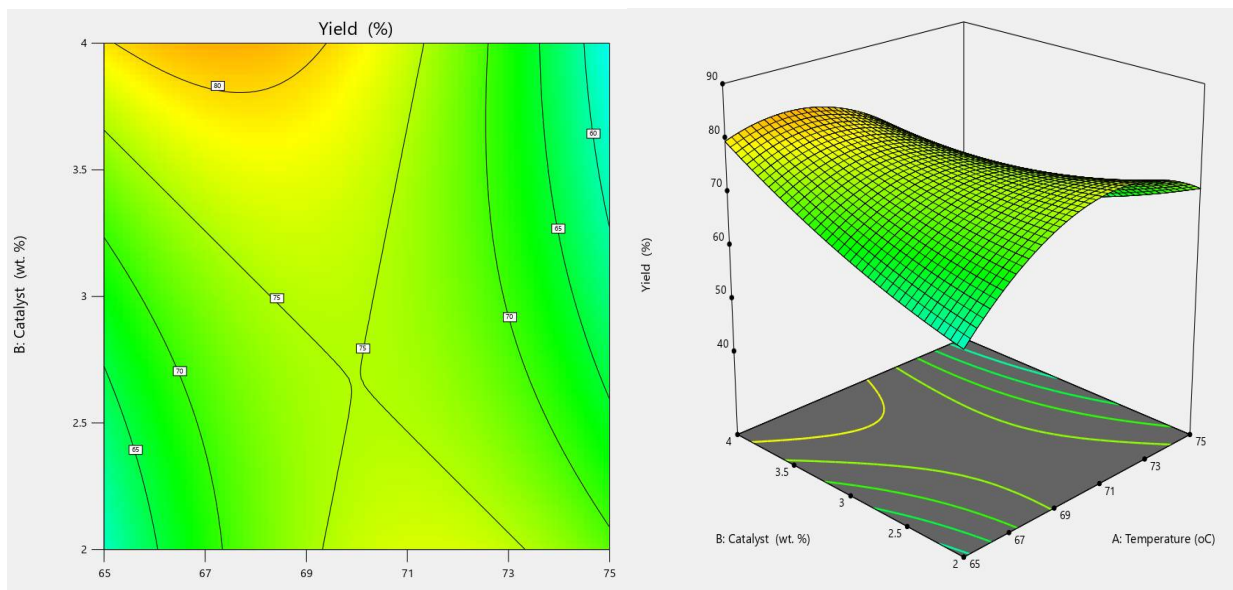


Figure 5 Interaction influence of temperature and catalyst on CASO biodiesel yield, A) Contour plots (B) 3D surfaces plot

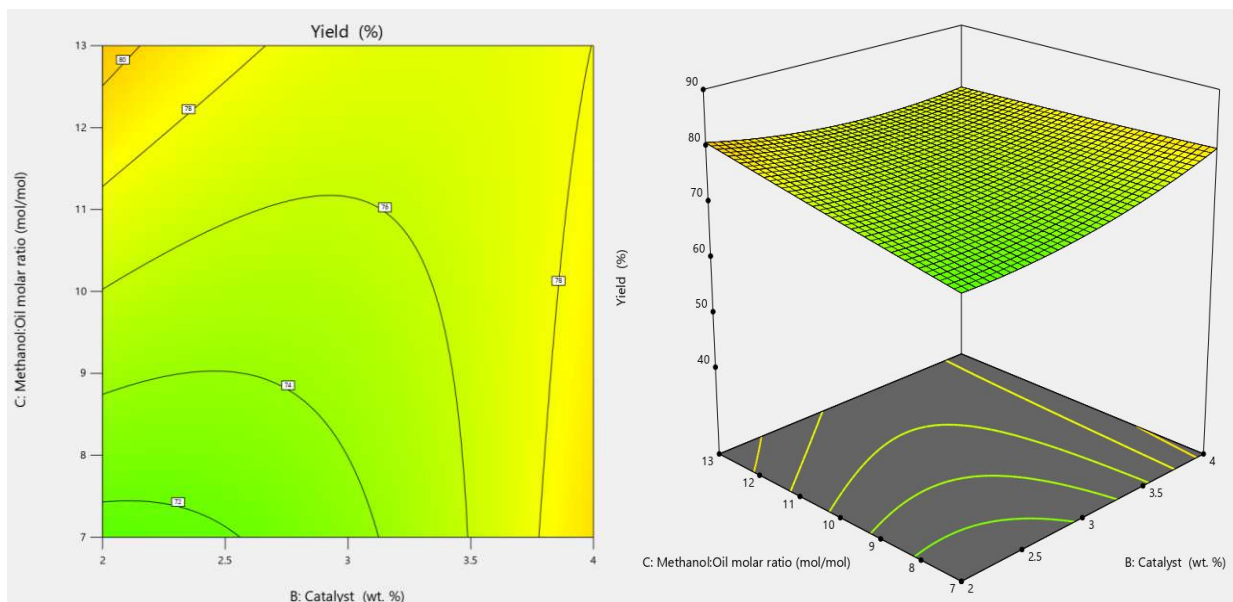


Figure 6 Interaction influence of Methanol: oil molar ratio and catalyst on CASO biodiesel yield, A) Contour plots (B) 3D surfaces plot

The biodiesel yield varies greatly depending on temperature, methanol molar ratio, and catalyst, as shown in the [Figures 5](#) and [6](#). The findings on the link between these parameters revealed that as the temperature rose, so did the oil yield. The interaction was found to be fairly steep, with a substantial mutual relationship between temperature and catalyst, as well as methanol molar ratio

and catalyst on biodiesel yield. According to the interaction study, determining the best combination of parameter conditions will improve the transesterification process greatly. The numerical solution was attempted through the software in order to optimize the selected parameter for maximizing the biodiesel yield.

From the RSM model, the optimized parameters were presented in the following [Table 6](#).

Table 6 The mathematical solution for maximizing CASO yield

Variables	Optimal condition
Temperature (°C)	72.426
Catalyst(wt%)	2.002
Methanol: oil ratio	12.334
Reaction time(min)	84.010
Expected oil yield (%)	89.230±0.28 %
Desirability level	1

1.1.Characterization of acquired biodiesel

Custard apple seed oil biodiesel was produced at optimum conditions and the physicochemical properties were analyzed. [Table](#) shows the comparison of the current studies with the previous result of biodiesel produced from sorghum and Jatropha([Raja et al. 2011](#)), ([Ved and Padam 2013](#)) ([Barabas and Todoru 2011](#))([Surface et al. 2022](#)).

Table 7 Summary of physicochemical properties of obtained biodiesel comparison

Biodiesel properties	This study	Jatropha biodiesel	Sorghum	ASTM
Acid value (Mg (NaOH/g))	1.12±0.002	13.76 ± 0.02	0.31	0.00 – 0.80
Saponification value (Mg (NaOH/g))	16.75±0.012	212.67± 0.01	165	189.00 – 198.00
Iodine value (gI2/100g)	133±.04	86.88± 0.04	101	≥ 130.00

Kinematic viscosity (mm ² /s)	5.67±0.031	22.8±0.03	3.237	35.00
Free fatty acid (mgKOH/g)	21±0.10	4.76±0.12	----	25.00
Specific gravity	0.879±0.023	0.88±0.02	0.871	0.86 – 0.90
Heating value (MJ/Kg)	42.072±0.4	----	----	>35
Flash point (°C)	179±0.34	155.00±0.02	155	130.00 – 170.00

The values are average ± standard deviation for the triplicate experiment

Conclusion

In this project biodiesel production from custard apple seeds, oil by the trans-esterification method was studied. Some of the physicochemical properties of biodiesel such as density, viscosity, Flashpoint, and heating value biodiesel were analyzed and compared with the standard biodiesel properties. The temperature, reaction time, catalyst, and molar ratio of the transesterification process were all tuned to maximize biodiesel yield. The combination of the following critical parameters was shown to be the optimum process condition for the maximum biodiesel yield, according to ANOVA analysis. The reaction temperature was 72.426°C, the catalyst was 2.002wt%, the molar ratio of Methanol to Oil was 12.334 and the reaction duration was 84.010 minutes. Under this condition, the triplicate model predicted the biodiesel yield was 89.230±0.28 %. The physicochemical characteristics of biodiesel synthesized from custard apple seed oil under optimal conditions were examined. The qualities meet ASTM standards, making it a viable source of energy for mixing with other petroleum fuels.

Acknowledgment

The authors appreciatively acknowledged the support of Jimma University Institute of Technology for covering financial support for this study.

Authors' contributions

All Authors conceived, designed, and performed the experiments and wrote this paper, and also participated in experiment design and research supervision. The authors read and approved the final manuscript.

Funding Statement

This researcher did not receive any specific grant from finance agencies in the public, commercial, or non-profit organizations.

Data availability statement

Data incorporated in the article/supplementary material/referenced in the article.

Conflict of Interest:

The authors declare that there is no conflict of interest in connection with submitted manuscripts.

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