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Effect of Process Parameters Variation and Optimization of Biodiesel Production from Dehulled Orange Seed Oils Using Acid Modified Clay

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Abstract

This study explores the feasibility of producing biodiesel from dehulled orange seed oil, a non-edible agro-industrial byproduct with significant potential as a renewable energy feedstock. The research aims to enhance biodiesel yield through the optimization of transesterification process parameters using Response Surface Methodology (RSM). Dehulled orange seeds were processed to extract oil, after which transesterification was carried out using methanol. Five key process factors—reaction temperature, reaction time, catalyst concentration, methanol-to-oil molar ratio, and agitation speed—were systematically varied based on a central composite design to assess their individual and interactive effects on biodiesel yield. Statistical analysis indicated that all variables influenced conversion efficiency, with methanol ratio and catalyst concentration exerting particularly strong effects. The quadratic model developed showed high predictive accuracy and statistical significance, confirming its suitability for optimization. The optimal reaction conditions were identified as a temperature of 75 °C, reaction time of 150 minutes, catalyst concentration of 5 wt%, methanol-to-oil molar ratio of 12:1, and agitation speed of 350 rpm. Under these conditions, the biodiesel yield reached 95.23%, demonstrating efficient conversion and validating the optimization strategy. The physico-chemical characteristics of the produced biodiesel further complied with standard fuel specifications, underscoring its suitability as a renewable fuel. Overall, the results affirm that dehulled orange seed oil is a viable and sustainable feedstock for biodiesel production. The optimized process not only achieves high yields but also adds value to agricultural waste streams, contributing to cleaner energy alternatives and supporting circular bioeconomy initiatives. This study highlights the importance of exploring non-edible oils for biodiesel production to reduce competition with food resources and promote environmental sustainability.

Keywords: Dehulled Orange Seed Oil, Transesterification; Response Surface Methodology (RSM), Process Optimization; Renewable Energy, Non-Edible Feedstock, Sustainable Biofuels.

INTRODUCTION

The increasing global demand for energy and the depletion of fossil fuel reserves have heightened the urgency to explore sustainable and renewable energy sources [1, 2]. Fossil fuels remain the dominant source of energy, but their combustion contributes significantly to greenhouse gas emissions, environmental pollution, and climate change [3]. Biodiesel which is a renewable, biodegradable, and clean-burning alternative to petroleum diesel has gained attention as a promising candidate for reducing the carbon footprint of transportation and industrial sectors [4]. Biodiesel is commonly produced through the transesterification of triglycerides found in vegetable oils and animal fats with alcohols (typically methanol or ethanol) in the presence of a catalyst, resulting in fatty acid methyl esters (FAMEs) and glycerol as a by-product [5]. Though biodiesel offers environmental and performance benefits, the sustainability of its production process depends on the choice of feedstock and catalyst [6]. Conventionally, biodiesel feedstocks such as soybean, rapeseed, and palm oils are edible, leading to competition between food and fuel [7]. Therefore, attention has shifted toward non-edible and waste-derived oils that can provide an economically viable and environmentally responsible pathway for biodiesel production [8]. One such promising source is orange seed oil, an agro-industrial by-product obtained from the citrus processing industry. The oil extracted from dehulled orange seeds

has a favorable lipid profile and low free fatty acid (FFA) content, making it suitable for transesterification [1]. The efficiency and yield of biodiesel production are profoundly influenced by the catalyst used. Homogeneous catalysts such as sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4) have been widely utilized for their high catalytic activity and short reaction times. However, they have several drawbacks, including soap formation, difficulty in product separation, and environmental concerns due to wastewater generation [10]. Among heterogeneous catalysts, natural clays such as bentonite and kaolinite have shown potential due to their abundance, low cost, large surface area, and tunable acidity or basicity [11]. The catalytic activity of clays can be further improved through chemical modification using alkaline, brine, thermal or acid treatments [20]. Such modifications alter the clay's surface characteristics—enhancing pore size distribution, cation exchange capacity, and the density of active sites [12]. For instance, acid treatment increases Lewis acidity, alkaline activation generates basic active centers favorable for transesterification, and brine treatment enhances structural stability and ionic exchangeability [10-14], investigated the use of palm oil biodiesel for production under several conditions, verifying the relationship between production varieties to optimize biofuel production using RSM. The biodiesel was produced through a transesterification process by the methyl route and alkali catalyst (NaOH) 1% (m/m). The analyzed variables were: temperatures (45, 52 *and* 60 ℃), molar ratios (3:1, 4:1, 6:1, and 8:1), and reaction times (40, 60, and 80 mins.) which gave a yield of 93 % with optimal values of 3:1, 52 ℃ and 60 mins [15], studied the effect of acid feedstock before transesterification when combined with other vegetable oils. A homogeneous catalyst of potassium hydroxide (KOH) was used with methanol as another raw material. There was a decrease in the acid value of *Calophyllum inophyllum* oil from 54 mg KOH/g oil to about 2.15 mg KOH/g oil after two steps of esterification. The yield of biodiesel from the multi-feedstock was 87.926 % at optimal values of temperature of 60 ℃ and a methanol-to-oil ratio of 6:1 using a catalyst of 1 wt% [16], evaluated the optimization and microwave-assisted transesterification process using CaO and KOH catalysts from waste cotton-seed cooking oil. While using CaO catalyst, the methanol to oil ratio (9.6:1), catalyst loading (1.33 w/w%), and reaction time (9.7 minutes), respectively, with a predicted model yield of 89.94 % were utilized. For the KOH catalyst, the optimum values of methanol to oil ratio (7:1), catalyst loading (0.65 w/w%) and reaction time (9.6 minutes), respectively were used, which gave a predicted model yield of 96.44 % [17], investigated the effect of five process parameters catalyst types, methanol: oil molar ratio, catalyst concentration, reaction temperature and reaction time on the transesterification of stone fruit (*Prunus Armeniaca L.*) oil. The stone fruit oil physiochemical properties of the derived biodiesel were characterized and found to satisfy both the ASTM-D D6751 and EN14214 standards. Transesterification optimum process parameter of stone fruit kernel oil at an agitated speed of 600 rpm were: KOH catalyst concentration 0.5% (oil weight), reaction temperature of 55℃, methanol: oil ratio of 6:1 and reaction time of 60min. The biodiesel yield was 95.8% showing that the stone fruit kernel oil will serve as low-cost feedstock for second generation biodiesel production and can be used in diesel engine without modification [19], investigated the extraction of oil from *Jatropha curcas* seed. The biodiesel production was carried out using transesterification method, the variables considered were methanol-to-oil ratio 1:1, 2:1, 3:1, 4:1, 5:1 and 6:1, reaction time of 30, 60, 90, 120 and 180min, at a reaction temperature of 60 ° C. The study revealed that the maximum biodiesel yield was 86wt% at methanol-to-oil ratio of 6:1, at reaction time of 180min. An energy input of ~1.4MJ/kg was estimated to produce a unit biodiesel from *Jatropha curca* [20], investigated the production of biodiesel from orange seed oil and raw and thermal clay as catalyst using response surface methodology to study the process parameters. The design was a full fractional factorial design which identified the various design points as being numerical and discrete. The process optimization was performed by varying five factors, each at two different levels. The process parameters: methanol to oil molal ratio (mol/mol), catalyst concentration (weight %), reaction time (minutes), temperature (°C) and agitation speed (revolution per minutes, rpm) were the independent variables (input), while the biodiesel yield (vol/vol) was the dependent variable (response) in the optimization process. The experimental/actual maximum optimal biodiesel yield for the biodiesel production from the orange seed oil using raw and thermal clay as catalyst was 79.53 and 94.58% v/v while the predicted biodiesel yield was 79.55 and 92.98% v/v under these optimal conditions time of 150 minutes, temperature 65 °C, methanol /sample molal ratio of 12:1, catalyst concentration of 3.0 wt. % and agitation speed at 300 rpm respectively.

2. MATERIALS AND METHODS

The materials utilized for the study include the following:

Five (5) kg of dehulled orange seed oil, beaker, heater, thermometer, retort stand, round bottom flask, reflux condenser, separating funnel, cotton material, weigh balance, litmus paper, oven, filter paper, hydrochloric (Hcl) acid, distilled water, magnetic capsule, soxhlet extractor, n-hexane, clay, carbonizer and design expert (version 12.0)

2.1. Sample Collection and Preparation

Dehulled orange seeds obtained from the parent fruits were air dried, sorted to remove impurities and grinded using industrial blender. About 0.1 kg of the grounded seeds were weighed into a semi-permeable cotton material and placed into the timble of a 0.5 kg Soxhlet extractor as presented in Fig. 1. About 0.4 kg of n-hexane was measured into a 0.5 kg flat bottom round

flask and the Soxhlet with the extraction timble containing the sample was connected with the condenser which was fitted to the flat bottom round flask containing n-hexane. The Soxhlet extraction system was heated on a hot plate at 60ºC for 60 minutes while water was allowed to circulate at the outer jacket of the condenser. The extraction was discontinued when oil was completely extracted from the sample. The de-fatted sample in the semi permeable membrane was discarded, while the oil and n-hexane mixture in the flat bottom flask was separated by distillation.

2.2. Clay Collection, Preparation and Modification

About 5 kg of clay was collected from local water bore hole drillers in 22 Mamman Vatsa Estate, Basin Authority, 8miles, Calabar. The clay was allowed to dry in hot air oven for 24 hours at 60ºC. After drying, it was milled to powdery form using an industrial ball mill. The milled clay was sieved using a 500 µm pore size sieve. The sieved clay was submerged in in distilled water and allowed to stand for an hour after which. This is to separate the clay (lower portion) from impurities (upper portion which floats on water). The overlaying water portion was decanted to remove all impurities in the clay sample. The clay was placed in hot air oven at 60ºC to dry. Thus, the acid clay modification was performed: About 1.5 kg clay sample was weighed into a 2 kg, flat bottom, round flask and 1 kg of 5% hydrochloric acid solution (50 g of HCl made up to 1 kg using distilled water) was added to the flask and refluxed. The mixture was allowed to cool and filtered using qualitative filter paper (240 mm). The residue was washed with hot water until pH of filtrate is neutral. The acid modified clay was dried in hot air oven at 60ºC and subsequently pulverized. The sample was stored in air tight container.

2.3. Biodiesel Production and Characterization

2.3.1. Transesterification

The transesterification reactions to produce methyl ester was carried out in a 500g round bottom, glass, spherical, three neck reactor provided with a thermometer, sampling outlet, and condensation system. The heating system was an electromagnetic hot plate which heated the reactor and rotated the metal knob in the reactor through an electromagnetic field. The reactor was preheated to 75 ºC to eliminate moisture and then 50g of the used DOSO was added for each of the experiment. When the reactor reached the temperature established for the reaction, the methanol and the catalyst were added in the amount required for the experiment. The stirring system afterwards was switched on at the desired speed, taking this moment as time zero of the reaction. Each mixture was vigorously stirred for 10 minutes using the magnetic stirrer and refluxed for the required reaction time. After methanolysis reaction had finished, the transesterification product was allowed to stand for 24 hours in a separating funnel (Fig. 1) for glycerol separation. The crude glycerol was removed through the funnel tap leaving the methyl ester (biodiesel) behind. The biodiesel was washed with hot water and dispensed into a 250 g beaker. It was heated at 100ºC to remove water molecules from the biodiesel and was allowed to cool.

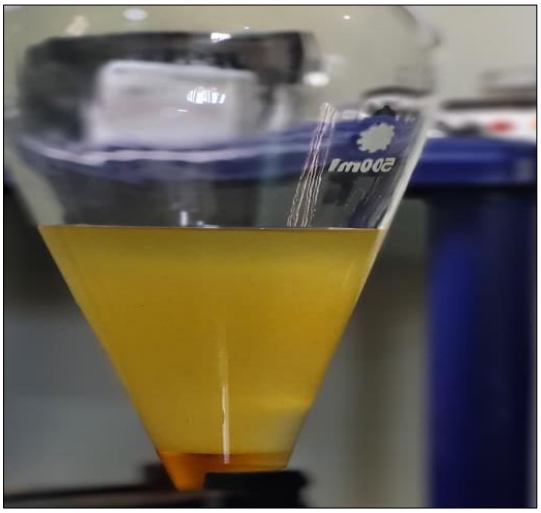

Fig. 1: Biodiesel production (transesterification)

3.0 RESULTS AND DISCUSSION

3.1 Experimental Matrix Using the Fractional Factorial Design

3.1.1 Analysis of Variance (ANOVA) for Quadratic Model

Presented in Table 1 is the analysis of variance (ANOVA) result obtained using the quadratic model where the factors were coded. The ANOVA was applied to estimate the significance of the model at 5% significant level. A model is considered statistically significant if the p-value (significance probability value) is less than 0.05 (typically ≤0.05).

From the table, the model F-value of 4879.37 implies that the model was significant. There was only a 0.01% chance that an F-value as large of that could occur

due to noise. The P-value less than 0.0500 indicated that the model terms were significant. In this case, the linear terms: A, B, C, D and E, the interaction terms: A, B,C, D, E, AB, AC, BC, BD, BE, CD, CE, DE and the quadratic terms: A^2, B^2, C^2, D^2 and E^2 were statistically significant models, while the values greater than 0.1000 indicated that the model terms were not significant.

Table 1: ANOVA for quadratic model (Response 1: Biodiesel yield)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	5101.30	20	255.07	4879.37	< 0.0001	significant
A-Catalyst conc.	98.08	1	98.08	1876.36	< 0.0001	
B-Methanol/oil	182.03	1	182.03	3482.15	< 0.0001	
C-Temperature	526.30	1	526.30	10068.07	< 0.0001	
D-Reaction time	1314.77	1	1314.77	25151.50	< 0.0001	
E-Agitation speed	256.46	1	256.46	4906.05	< 0.0001	
AB	7.85	1	7.85	150.12	< 0.0001	
AC	4.29	1	4.29	82.00	< 0.0001	
AD	0.2047	1	0.2047	3.92	0.0734	
AE	0.0842	1	0.0842	1.61	0.2307	
BC	1.13	1	1.13	21.55	0.0007	
BD	16.39	1	16.39	313.50	< 0.0001	
BE	30.77	1	30.77	588.57	< 0.0001	
CD	372.22	1	372.22	7120.58	< 0.0001	
CE	124.45	1	124.45	2380.66	< 0.0001	
DE	26.45	1	26.45	506.06	< 0.0001	
A^2	4.19	1	4.19	80.18	< 0.0001	
B^2	88.21	1	88.21	1687.38	< 0.0001	
C^2	100.80	1	100.80	1928.27	< 0.0001	
D^2	130.78	1	130.78	2501.74	< 0.0001	
E^2	1.56	1	1.56	29.90	0.0002	
Residual	0.5750	11	0.0523			
Lack of Fit	0.0750	5	0.0150	0.1800	0.9602	not significant
Pure Error	0.5000	6	0.0833			
Cor Total	5101.88	31				

However, the lack of fit (F-value) of 0.18 implies that it was not significant relative to the pure error. Thus, there was a 96.02% chance that a Lack of Fit F-value this large could occur due to noise. Therefore, a non-significant Lack of Fit is good as the anticipated outcome is that the model fits. As seen in Table 3, that amongst the five variables investigated, exposure time (D) had the largest effect on biodiesel yield as it has the highest F-test value of 25151.50 for the single effect, which the least was the reaction temperature (C) that showed the lowest F-test value of 1876.36.

3.1.2 Fit Statistics

As seen in the statistical analysis in Table 2, the coefficient of determinant (R^2) is 0.9999), the predicted R^2 is 0.9990 are in reasonable agreement with the adjusted R^2 value of 0.9997, which implied a difference of less than 0.2. The coded equations as presented were useful for identifying the relative impact of the factors by comparing the factor coefficients. Also, since the adequate precision measures the signal to noise ratio, the value of 268.3513 indicates an adequate signal where model could be used to navigate the design space, as a ratio greater than 4 is desirable. Overall, other test results from the statistical analysis perspective are as presented in the Table 3.

Table 2: Fit statistics

Parameters	Values
Std. Dev	0.2286
Mean	59.28
C.V. %	0.3857
R^2	0.9999
Adjusted R^2	0.9997
Predicted R^2	0.9990
Adequate Precision	268.3513

3.1.3 Coefficient Estimate

i. The coefficient estimate column represents the direction and magnitude of the effect of each factor on the biodiesel yield. Positive coefficients (eg., A + 2.52) indicates an increase in biodiesel yield as the factor increases, while negative coefficients (e.g., AB = -0.9672) indicates diminishing returns or curvature.

ii. The standard error (SE) measures the variability of the coefficient estimate. Smaller standard errors indicate more precise estimates. Methanol/oil molar ratio (B) has a relatively low SE (0.0547), this shows that the model estimates this coefficient reliably.

iii. The degree of freedom as seen in Table 3 is one (1), because each term represents a single predictor and thus is used to compute the F-values and P-values in RSM regression.

iv. The 95% confidence limits define the range within which the true value of the coefficient is expected to lie with 95% confidence. A coefficient is statistically significant if the confidence interval does not contain zero (eg., Catalyst concentration (A) has a 95%Cl from 2.39 to 2.63 and this does not include zero (0), meaning it is significant while AB has a 95%Cl of -1.14 to -0.7934 and since it includes zero (0), it is not significant. From Table 3, we have 13 (thirteen) significant interactions and 7 (seven) not significant interactions.

v. The variance inflation factor (VIF) indicate the degree of multicollinearity among predictors. VIF < 5 is acceptable multicollinearity, VIF 5 to 10 is high but acceptable and VIF > 10 is termed problematic. As seen in Table 3, the VIF's is less than 1.7, this shows that no harmful multicollinearity is present and the model is stable.

Table 3: Coefficients in terms of coded factors

Factor	Coefficient Estimate	df	Standard Error	95%CI Low	95%CI High	VIF
Intercept	67.55	1	0.1168	67.29	67.81	
A-Catalyst conc.	2.52	1	0.0582	2.39	2.65	1.29
B-Methanol/oil	3.23	1	0.0547	3.11	3.35	1.15
C-Temperature	5.83	1	0.0581	5.70	5.96	1.19
D-Reaction time	9.23	1	0.0582	9.11	9.36	1.25
E-Agitation speed	4.04	1	0.0576	3.91	4.16	1.27
AB	-0.9672	1	0.0789	-1.14	-0.7934	1.46
AC	-0.6676	1	0.0737	-0.8299	-0.5053	1.28
AD	0.1620	1	0.0818	-0.0182	0.3421	1.61
AE	0.0981	1	0.0773	-0.0721	0.2682	1.55
BC	0.3412	1	0.0735	0.1795	0.5030	1.33
BD	1.21	1	0.0683	1.06	1.36	1.24
BE	1.83	1	0.0755	1.66	2.00	1.44
CD	6.25	1	0.0741	6.09	6.42	1.31
CE	3.63	1	0.0743	3.46	3.79	1.27
DE	-1.80	1	0.0799	-1.97	-1.62	1.46
A^2	0.9726	1	0.1086	0.7335	1.21	1.35
B^2	-4.20	1	0.1023	-4.43	-3.98	1.30
C^2	-4.86	1	0.1107	-5.11	-4.62	1.39
D^2	-5.42	1	0.1083	-5.66	-5.18	1.51
E^2	-0.5264	1	0.0963	-0.7383	-0.3145	1.20

3.1.4 Predicted and Experimental or Actual Values

As seen in Fig. 2, the predicted values versus the actual values shows the yield plot for biodiesel production from orange seed oil using raw clay as catalyst. The plot is used to check if the points will follow a straight line to ascertain if the residuals follow a normal distribution. Fig. 2 shows that the points were closely distributed to the straight line which confirms a good relationship between the actual (experimental) values and the predicted values of the response. The plots also prove that the selected model was adequate in predicting the response variables in the experimental values. The predicted versus the actual plot showed upward progression of the biodiesel yield from 43.45 to 95.23%, indicating that the model effectively captured the improvement in conversion efficiency across the experimental design space. The close correlation between the predicted and actual confirms that the model reliably describes the process variables on biodiesel yield.

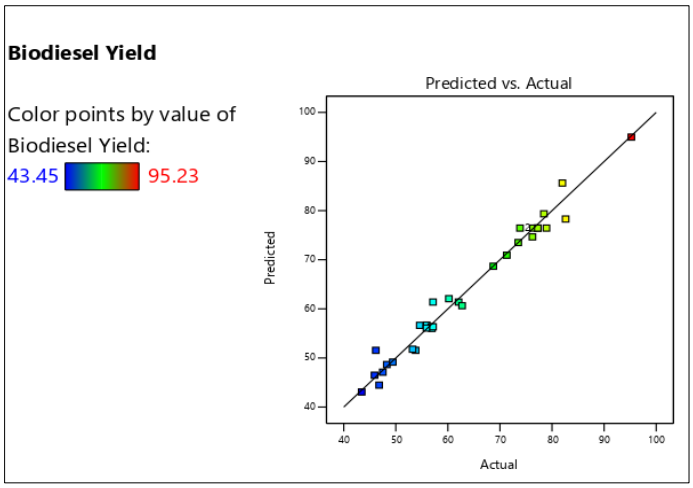

Fig. 2: Predicted versus actual plot

3.1.5 Contour and 3-Dimensional (3D) Plots Interactions of the Process Parameters

i. Interactions between Catalyst Concentration and Temperature

Fig. 3 and 4 Fig. depicts the interaction effect between catalyst concentration and temperature under contour and 3D plots respectively. They indicated that the biodiesel yield increases with increase in temperature 45 to 75ºC for the brine modified catalyst and the catalyst concentration from 1 to 5wt % respectively. It was observed the yield was improved when the rapid reaction occurred at higher temperature and also at a higher catalyst concentration. Higher temperatures above 75 ºC accelerates reaction but excessive heat leads to methanol vapour loss and thus causes a reverse reaction [20].

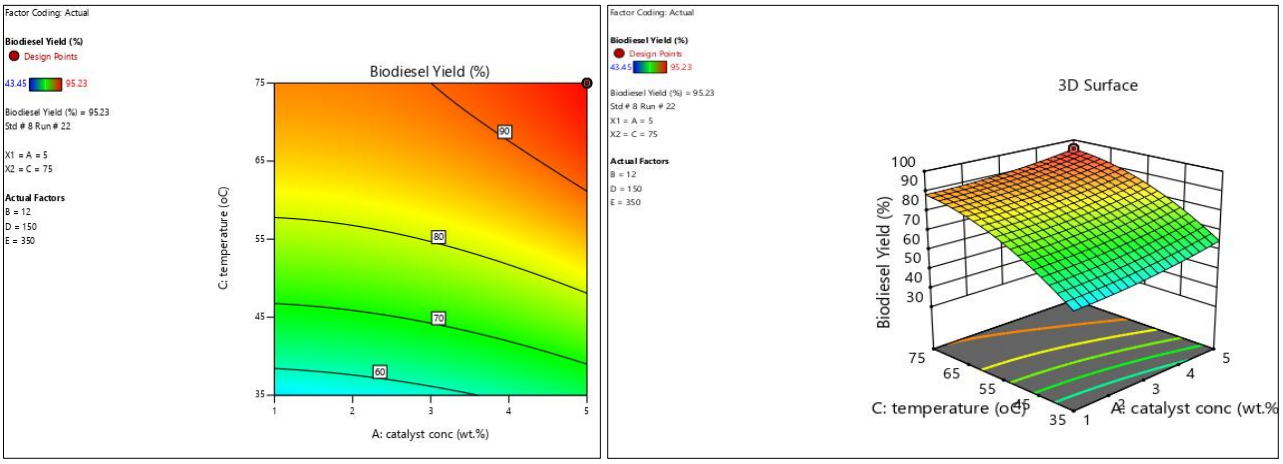

Fig. 3 and 4: Contour and 3D plots interaction for catalyst concentration and temperature

ii. Interactions between Reaction Time and Temperature on Biodiesel Yield

Fig. 5 and 6 depicts the interaction effect between methanol/oil molal ratio and temperature on contour and 3D plots respectively. It indicated that the biodiesel yield increases with increase in methanol/oil molar ratio and temperature from 45 to 75 ºC and from 4:1 to 12:1 respectively. The methanol and temperature surface revealed an elliptical optimum zone, demonstrating their combined influence on equilibrium and reaction kinetics. Above the optimal values, they will be a decline in biodiesel yield because excessive methanol dilutes reactants and high temperature cause evaporation and equilibrium shift [20].

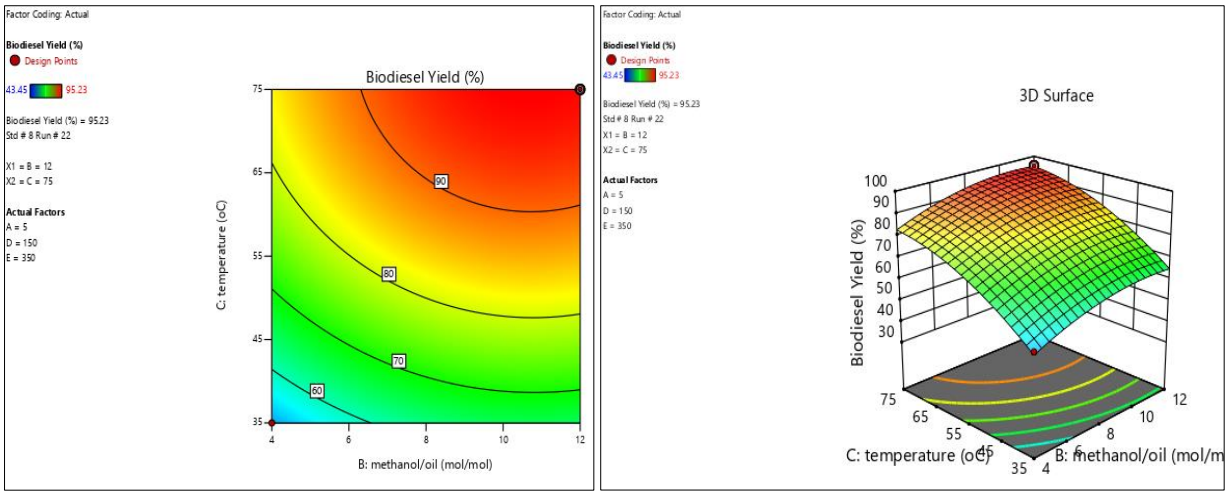

Fig. 5 and 6: Contour and 3D plots interaction for methanol/oil molar ratio and reaction temperature

iii. Interactions between Temperature and Reaction Time

Fig. 7 and 8 depict the interaction effect between temperature and reaction time on contour and 3D plots respectively. It indicated that the biodiesel yield increases with increase in reaction time of 30 to 150 minutes and temperature of 35 to 75 ºC respectively. It was observed the yield was improved when the rapid reaction occurred at higher reaction time and also at a higher temperature. However, at higher temperature above the optimal value 75 ºC, a noticeable decrease in biodiesel yield was observed [21]. Higher temperature speeds up the reaction, but prolonged heating leads to methanol loss and product degradation.

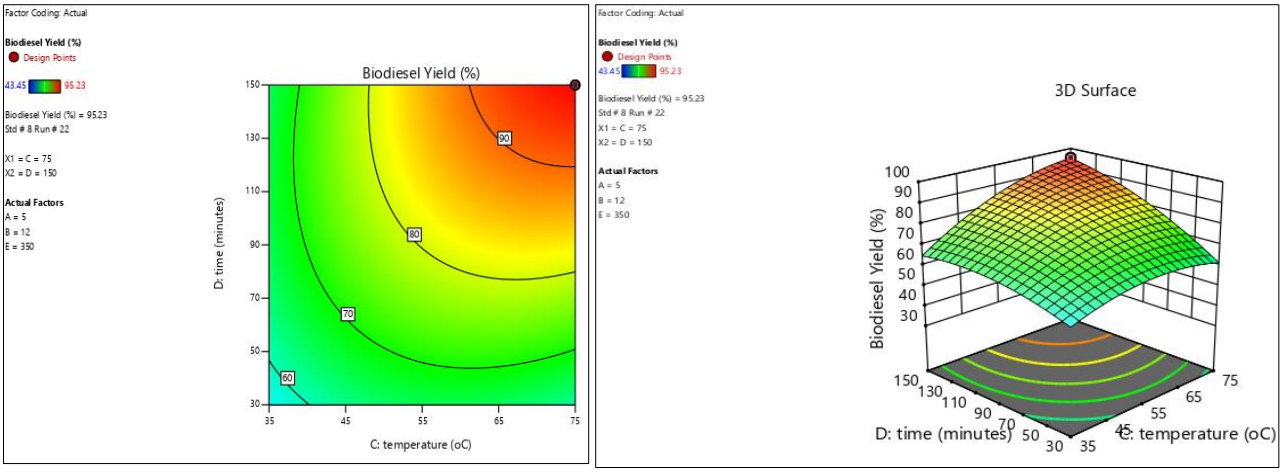

Fig. 7 and 8: Contour and 3D plots interaction for temperature and reaction time

4. CONCLUSION

This study demonstrated the successful production and optimization of biodiesel from dehulled orange seed oil using acid based modified clay as catalyst, confirming its potential as a sustainable non-edible feedstock. Using Response Surface Methodology, optimal transesterification conditions were identified at a reaction temperature of 75 °C, reaction time of 150 minutes, catalyst concentration of 5 wt%, methanol-to-oil molar ratio of 12:1, and agitation speed of 350 rpm. Under these optimal conditions, a high biodiesel yield of 95.23% was achieved, highlighting the feasibility and efficiency of converting dehulled orange seed oil into biodiesel.

The high yield, combined with the reliable model predictions, demonstrates that orange seed oil possesses favorable physicochemical properties suitable for biodiesel production. The optimization results also indicate that the selected reaction parameters exert significant influence on conversion efficiency, and their synergistic effects contribute to achieving near-complete transesterification. Overall, the findings validate dehulled orange seed oil as a promising alternative renewable feedstock capable of supporting biodiesel production, reducing dependence on conventional edible oils, and contributing to environmentally sustainable energy solutions.

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