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Optimization of Biodiesel Synthesis using Response Surface Methodology

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Abstract: ***Biodiesel was produced through transesterification of neem seed oil with methanol and activated calcium oxide (CaO) derived from waste bone as a catalyst. The influence of process parameters which include catalyst concentration, methanol to oil ratio, reaction time and reaction temperature were determined. These process parameters were optimized using response surface methodology (RSM) and analysis of variance (ANOVA). The significance of the different process parameters and their combined effects on transesterification efficiency were determined through central composite designs (CCD) based on a five level, five variables. The 2-level -5- factor experimental design was employed in this study requiring 32 experiments, consisting of 16 factorial points, 10 axial points and 6 center points. The optimal conditions derived through response surface methodology were: catalyst concentration 4wt%; methanol/oil molar ratio 8:1; reaction temperature 55°C; reaction time 4hr; agitation speed 400rpm; and under these conditions an optimum yield of 94.00% was achieved. The quality of biodiesel produced at these conditions was within the American Society for Testing and Materials (ASTM D6751) and European Union (EN 14214) specification.***

Keywords: Activation; Biodiesel; Central Composite Design; Neem oil; Optimization, Response Surface Methodology; Transesterification

I. INTRODUCTION

The fast depletion of world's petroleum reserves and increasing ecological concerns has created a great demand for environmentally benign renewable energy resources [2]. There is an increasing worldwide concern for environmental protection and for the conservation of non-renewable natural resources. For this reason, the possibility of developing alternative energy source to replace traditional fossil fuels has been receiving a large interest in the last few decades [19]. Biodiesel is well-chosen as a feasible alternate and prospective petroleum for diesel engine because of the forecast scarcity of non-renewable fuels and rise in the cost of fuels [22]. It is biodegradable, renewable, non-toxic, environmentally-friendly, and has high flash point, better lubrication, high cetane number and has quite resemblance in regard with physical and chemical characteristics with that of conventional diesel fuel [7,10,17]. Biodiesel is the mixture of mono alkyl esters that can be continuously derived from vegetable oils or animal fats and therefore it is termed as renewable energy [12]. There are various processes/techniques that have been adopted in production of biodiesel from vegetable oils and animal fats namely: dilution, micro-emulsification, pyrolysis and transesterification process/technique [1,8]. Among these methods, transesterification is the key and foremost important process to produce the cleaner and environmentally safe fuel [3,20]. Biodiesel is produced through a chemical process known as transesterification of different vegetable oil or animal fat with a short chain alcohol, in which one mole of glyceride reacts with three moles of alcohol in the presence of appropriate amount of catalyst to form mono methyl ester and glycerol [4,8,9]. The most common alcohols widely used are methyl alcohol and ethyl alcohol. Methanol, found constant purpose in the commercial uses because of its low cost and its physical and chemical advantages[23]. Another advantage of using methanol is the separation of glycerine, which can be obtained through simple decantation [15,21]. Solid waste based catalyst was used in order to produce cost effective catalysts and biodiesel. Among these solid wastes, animal bone is one of the best solid wastes that are easily and abundantly available all over the world. Although, the waste bone derived catalysts have shown a reasonable performance and constancy in the reaction, however these catalysts are required in high amount, high methanol/oil molar ratio with longer time for the reaction to occur [24]. All these disadvantages make waste bone derived catalysts practically and economically unsuitable. To overcome these difficulties, it would be imperative to impregnate waste animal bones with other catalysts using H_3PO_4 to make waste animal bone derived catalyst more active and to boost the surface chemical properties[24]. Waste animal bones have proved to be highly effective as a catalyst support. The properties of calcined bone make it advantageous for use as catalyst support in transesterification reaction. It contains hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$], that is highly porous and also has a large surface area which allows catalyst to disperse over it largely and effectively. Calcined bones can also be used in high pressure and temperature reaction conditions.

There are several parameters that affect the yield of biodiesel through transesterification of vegetable oils. They are catalyst concentration, methanol to oil ratio molar ratio, reaction time and reaction temperature. The optimization of transesterification reaction which requires a wide number of experiments help to predict the effect of the process parameter of the reaction and their interaction .Response surface methodology in combination with central composite design has been successfully applied to the optimization of biodiesel production from different raw materials and different catalysts .It is a combination of mathematical and statistical techniques which is widely used for designing experiments, building models, determining optimum conditions and evaluating the relative significance of several factors affecting a process.

The experimental work carried out and reported in this paper was aimed at obtaining the optimal production conditions for base catalyzed transesterification of methyl ester from neemseed oil. The experiments were performed based on central composite design (CCD) and response surface methodology (RSM) was further used to analyze the relationship between the parameters and to determine the optimum conditions for optimal production of methyl ester from neemseed oil with methanol in the presence of CaO(acid activated) derived from waste bone .

II. MATERIALS AND METHODS

A. *Materials*

The non-edible neem seed oil was purchased from National Research Institute for Chemical Technology (NARICT) Zaria, Kaduna state, Nigeria and the waste bones was sourced from a local market (Kubwa village market), Kubwa, Abuja. All chemicals such as H_3PO_4, methanol,were of analytical reagent grade and 99% pure.

B. *Catalyst Preparation*

The catalyst (waste animal bones) from goats was sourced from a local market in Kubwa, Abuja. The sourced waste bones was soaked in boiling water for several hours (6-8 hours) at about 75^0C to remove tissues and fats in the bone and then rinsed with distilled water for 3-4 times to remove dust and impurities.The waste bones were dried in the drying oven at 110^0C for 4hours to remove water and moisture before being ground finely to a <2mm particle size powder using a hammer mill. The crushed and powdered catalysts was sieved using various mesh sizes (100-200) to get particle of uniform size of mesh screens. They were stored in a desiccator in the presence of silica and KOH pellets in order to avoid water and CO_2 (reaction with air) contact with the catalysts prior to further usage because the CaO catalyst will be reacted with CO_2 and converted into $CaCO_3$, thus reducing its activity as a catalyst.

C. *Catalyst Activation*

Sample of the crushed/powdered waste bone was impregnated with concentrated phosphoric acid (H_3PO_4) at different ratios of raw material to acid (1:1, 1:2, 1:3, 1; 4, and 1:5 w/v) on weight basis. The impregnated samples were dried in a Memmert oven at 100^OC for 12hours. After the activation, and slow cooling in air at room temperature, the slurry was washed with distilled water several times until P^H 6-7 ,filtered to be free from acid and then dried at a temperature of 110^OC for 4hours and finally ground into a fine powder, sieved and stored in desiccators for further usage.

D. *Procedures*

The transesterification reaction was carried out in a three necked 250 ml round bottom flask fitted with a thermometer, condenser and a mechanical stirrer whose speed was also controlled The neem oil reacts with methanol in the presence of catalyst derived from waste of animal bone to produce methyl esters of fatty acids (biodiesel) and glycerol. The refined neemseed oil (30ml) was quantitatively transferred into a flat bottom flask placed on a hot magnetic stirrer. Then specific amount of catalyst (by weight of refined neemseed oil) dissolved in the required amount of methanol was added. The reaction flask was kept on a hot magnetic stirrer under constant temperature with defined agitation throughout the reaction. At defined time 1-2 hours, sample was taken out, cooled, and the biodiesel (i.e. the methyl ester in the upper layer) was separated from the by-product (i.e. the glycerol in the lower layer) by settlement overnight under ambient condition. The percentage of the biodiesel yield was determined by comparing the weight of layer biodiesel with the weight of refined neemseed oil used.

The percentage conversion of each sample was calculated from the below equation.

$$\text{Yield (\%)} = \frac{\text{weight of methylester}}{\text{weight of oil used}} \text{x} 100 \quad (1)$$

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E. Experimental Design

The experimental design for optimization was done by applying response surface methodology (RSM) with central composite design to investigate the influence process conditions. The 5-level -5- factor experimental design was employed in this study requiring 32 experiments, consisting of 16 factorial points, 10 axial points and 6 center points. The level of each was chosen based on the importance to the experiment. Based on the effect of the process conditions considered, the range and variable investigated are listed in Table 1 below. Central Composite Design (CCD) a suitable design for sequential experiments to obtain appropriate information for testing lack of fit without a large number of design points[13,14], was applied with five design factors; namely, methanol to oil molar ratio, catalyst weight, temperature, reaction time and agitation speed, on the yield of neem seed oil methyl ester using the 2 level-5-factor central composite design (2^5 fractional factoria points).These factors are the major factors that affect biodiesel yield[5].

A quadratic polynomial equation by central composite design was developed to predict the response as a function of independent variables and their interaction [16]. A mathematical model, following a second –order polynomial which includes interaction terms was used to calculate the predicted response. The response for the quadratic polynomials is described below[13]:

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

Where Y is % methyl ester yield, xi and xj are the independent study factors(coded variables), and β_0 ,β_i,β_{ii} and β_{ij} are constant co-efficient, regression co-efficient of the linear terms, regression co-efficient of quadratic terms, and regression co-efficient of the interaction terms, respectively, and k is the number of factors studied and optimized in the experiment(number of independent variables). The Design– Expert 10.0.6.0 software package was used for regression analysis and analysis of variance (ANOVA)

Table 1: Experimental range and levels of the independent variable

Independent variable	Units	Low level (-1)	High level (+1)	-α	+α	0 level
Catalyst conc. (A)	Wt%	3(-1)	5(+1)	2(-2)	6(+2)	4
Methanol, (B)	Mol/mol	6(-1)	10(+1)	4(-2)	12(+2)	8
Temperature, (C)	°C	45(-1)	65(+1)	35(-2)	75(+2)	55
Reaction time (D)	Hours	3(-1)	5(+1)	2(-2)	6(+2)	4
Agitation speed (E)	Rpm	300(-1)	500(+1)	200(-2)	600(+2)	400

III. RESULTS AND DISCUSSION

A. Development of Regression Model

Central Composite design model was used to optimize transesterification parameters. The statistical combination of the independent variables in coded and actual values along with the predicted and experimental response is represented in Table 2 From the table, it can be seen that the biodiesel yield obtained was in the range of 33 to 94% and experiment 12 and 29 gave the minimum and maximum yield respectively. The center point (run 27-32) was used to determine the experimental error (pure error) and the reproducibility of the data. The regression equation as a function of the selected variables for FAME yield (%) is given by equation (2)

Design expert 10.0.6.0 was used to calculate the effects of each factor and its interactions. The model expressed by equation 2 represents neem seed oil methyl ester yield (y) as a function of catalyst concentration (A), methanol to oil ratio(B), reaction temperature (C), reaction time (D) and agitation spped (E).

$$Y=+90.34-0.083*A+2.50*B+2.25*C-2.00*D-3.00*E+0.63*AB-0.73*AC-4.62*AD+6.75*AE+1.88*BC-3.12*BD+1.00*BE+5.63*CD-3.50*CE-1.00*DE-8.09* A^2-8.97* B^2-6.84* C^2-3.97* D^2-7.59* E^2 \quad (3)$$

Where "Y" is the response, that is the conversion to biodiesel, and A, B, C D and E shows the values of the test variables, catalyst concentration, methanol to oil molar ratio, temperature, reaction time and agitation speed, respectively. The below equation represents the quantitative effect of the factors (A, B, C, D, and E) upon the response (Y). Positive sign in front of the terms indicates synergistic effect in increase FAME yield,while negative sign indicates antagonistic effect of the factor [19].

Statistical analysis obtained from the analysis of variance (ANOVA) for response surface reduced quadratic model is shown in Table 3. The coefficient of determination (R2) and the parameters F value (Fisher F-test) and P value were used to judge the adequacy of the model. The model F-value of 94.83 with a very low probability value {(Pmodel >F) =0.0001} implies a very high significance for the regression model.

This is desirable as it indicates that the terms in the model have a significant effect on the response. The value of P<0.0001 implies that there is only a 0.01% chance that a "model F-value" this large could occur due to noise. Generally P-values lower than 0.01 indicate that the model is considered to be statistically significant at the 99% confidence level, that is (p-value <0.05). Values greater than 0.1000 indicate the model terms are not significant ,that is (p-value >0.05). The lack of fit F-value of 0.081 and p-value of 0.9959 (p-value > 0.05 is not significant) implied the lack of fit is not significant relative to the pure error and the model is satisfactorily fitted to experimental data. There is a 99.59% chance that a lack of fit F-value "this large could occur due to noise''. Insignificant lack of fit is most needed because significant lack of fit indicates that there might be contributions in the regressor-response relationship that is not accounted for by the model. The smaller the P-value for a parameter the more significant the parameter, hence neglecting the relative importance of the term attached to that parameter (Pradhan et al., 2012). In this case,B(methanol to oil molar ratio),C(temperature),D(time),E(agitation speed),AD,AE,BC,BD,CD,CE,A^2,B^2,C2,D^2,and E^2 are significant model terms. However, A,AB,AC, BE, and DE, have less effect (Prob>F more than 0.05) on the biodiesel yield from acid activated waste bone methyl ester on neem seed oil. The value of regression coefficient R^2 for the model is 0.9942,which means 99.42% of the total variation in the biodiesel yield was attributed to the experimental variables studied indicating the good fitness of the model. High values of predicted R^2 (0.9804) and adjusted coefficient of determination (R^2Adj:0.9837) and low value of coefficient of variation (C.V:3.34%), are an indication of precision of fitted model . The relationship between predicted and experimental biodiesel yield is shown in figure 2. It can be seen that there is high correlation (R^2= 0.9942) between the predicted and experimental biodiesel yield. The predicted values and experimental values were in reasonable agreement(R^2 value close to unity), which means that the data fit well with the model.

Table 2: Design matrix of experiments and their respective experimental yield and predicted yield.

Run order	Catalyst conc. (wt %) A		Methanol/Oil molar ratio(mol) B		Temperature (oC) C		Time (Hours) D		Agitation Speed (Rpm) E		Yield (%)	Yield (%)
	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Exptal.	Pred.
1	-1	3.0	-1	6:1	-1	45.0	-1	3.0	+1	500.0	50.00	45.97
2	+1	5.0	-1	6:1	-1	45.0	-1	3.0	-1	300.0	60.00	53.55
3	-1	3.0	+1	10:1	-1	45.0	-1	3.0	-1	300.0	70.00	62.72
4	+1	5.0	+1	10:1	-1	45.0	-1	3.0	+1	500.0	86.00	78.80
5	-1	3.0	-1	6:1	+1	65.0	-1	3.0	-1	300.0	63.00	55.72
6	+1	5.0	-1	6:1	+1	65.0	-1	3.0	+1	500.0	57.00	49.80
7	-1	3.0	+1	10:1	+1	65.0	-1	3.0	+1	500.0	53.00	44.97
8	+1	5.0	+1	10:1	+1	65.0	-1	3.0	-1	300.0	73.00	63.55
9	-1	3.0	-1	6:1	-1	45.0	+1	5.0	-1	300.0	60.00	60.72
10	+1	5.0	-1	6:1	-1	45.0	+1	5.0	+1	500.0	47.00	47.80
11	-1	3.0	+1	10:1	-1	45.0	+1	5.0	+1	500.0	40.00	39.97
12	+1	5.0	+1	10:1	-1	45.0	+1	5.0	-1	300.0	33.00	31.55
13	-1	3.0	-1	6:1	+1	65.0	+1	5.0	+1	500.0	50.00	49.97
14	+1	5.0	-1	6:1	+1	65.0	+1	5.0	-1	300.0	57.00	55.55
15	-1	3.0	+1	10:1	+1	65.0	+1	5.0	-1	300.0	83.00	79.72
16	+1	5.0	+1	10:1	+1	65.0	+1	5.0	+1	500.0	60.00	57.80
17	-2	2.0	0	8:1	0	55.0	0	4.0	0	400.0	53.00	58.14
18	-1	6.0	0	8:1	0	55.0	0	4.0	0	400.0	50.00	57.81
19	0	4.0	0	4:1	0	55.0	0	4.0	0	400.0	47.00	49.48
20	0	4.0	+2	12:1	0	55.0	0	4.0	0	400.0	50.00	59.48
21	0	4.0	0	8:1	-2	35.0	0	4.0	0	400.0	56.00	58.48
22	0	4.0	0	8:1	+2	75.0	0	4.0	0	400.0	58.00	67.48
23	0	4.0	0	8:1	0	55.0	-2	2.0	0	400	60.00	78.48
24	0	4.0	0	8:1	0	55.0	+2	6.0	0	400	77.00	70.48
25	0	4.0	0	8:1	0	55.0	0	4.0	-2	200	57.00	65.98
26	0	4.0	0	8:1	0	55.0	0	4.0	+2	600	50.00	53.98
27	0	4.0	0	8:1	0	55.0	0	4.0	0	400	86.00	90.34
28	0	4.0	0	8:1	0	55.0	0	4.0	0	400	88.00	90.34
29	0	4.0	0	8:1	0	55.0	0	4.0	0	400	94.00	90.34
30	0	4.0	0	8:1	0	55.0	0	4.0	0	400	93.00	90.34
31	0	4.0	0	8:1	0	55.0	0	4.0	0	400	91.00	90.34
32	0	4.0	0	8:1	0	55.0	0	4.0	0	400	91.00	90.34

Table 3: Analysis of variance (ANOVA) for model regression

Source	Coefficient estimate	Degree of freedom	Sum of square	Mean squares	F-value	P-value (Prob >F)
Model	90.34	20	8608.08	430.40	94.83	< 0.0001 significant
A	-0.083	1	0.17	0.17	0.037	0.8515
B	2.50	1	150.00	150.00	33.05	0.0001
C	2.25	1	121.50	121.50	26.77	0.0003
D	-2.00	1	96.00	96.00	21.15	0.0008
E	-3.00	1	216.00	216.00	47.59	< 0.0001
AB	0.63	1	6.25	6.25	1.38	0.2654
AC	-0.37	1	2.25	2.25	0.50	0.4960
AD	-4.62	1	342.25	342.25	75.41	< 0.0001
AE	6.75	1	729.00	729.00	160.62	< 0.0001
BC	1.88	1	56.25	56.25	12.39	0.0048
BD	-3.12	1	156.25	156.25	34.43	0.0001
BE	1.00	1	16.00	16.00	3.53	0.0872
CD	5.63	1	506.25	506.25	111.54	< 0.0001
CE	-3.50	1	196.00	196.00	43.19	< 0.0001
DE	-1.00	1	16.00	16.00	3.53	0.0872
A^2	-8.09	1	1920.24	1920.24	423.09	< 0.0001
B^2	-8.97	1	2358.03	2358.03	519.55	< 0.0001
C^2	-6.84	1	1372.74	1372.74	302.46	< 0.0001
D^2	-3.97	1	461.37	461.37	101.65	< 0.0001
E^2	-7.59	1	1690.24	1690.24	372.42	< 0.0001
Residual		11	49.92	4.54		
Lack of fit		6	4.42	0.74	0.081	0.9959 Not significant
Pure error		5	45.50	9.10		
Cor Total		31	8658.00			

Std Dev. = 2.13, Mean = 63.75, C.V. % = 3.34, Press = 169.76 , R^2=0.9942, Adj R^2 =0.9837 ,Pred R^2 = 0.9804, Adeq precision =34.0

B. Validation of the Optimization Result

A transesterification reaction under the obtained optimum operating conditions was carried out in order to evaluate the precision of the quadratic model.Further experiments were conducted to verify the accuracy of the predicted model and the experiment at the selected optimal conditions was performed to confirm the experimental result.The predicted conversion value of 95.6% was observed. Therefore, the experimental value (obtained) showed acceptable agreement with the predicted value, with relative small percentage error (1.6%).This indicated that the proposed statistical model was suitable for prediction of optimized biodiesel yield and for optimization of transesterification process. Therefore it can be concluded that the generated model showed reasonable predictability and sufficient accuracy for the biodiesel yield in the experimental condition.

Table 4 :Results of the model validation at optimum conditions.

Catalyst conc. A	Methanol/oil molar ratio B	Temperature (^oC) C	Time (Hour) D	Agitation speed(rpm) E	Exptal. Yield (%)	Predicted yield (%)	Percentage error (%)
4	8	55	4	400	94.0	95.6	1.6

C. Model Diagnostic Plot

The analysis was further examined using the normal probability plot of the residuals as shown in figure 1 and residual of the predicted plot as shown in figure 2. The normal probability plot of the residuals indicates that the errors are distributed normally in a straight line and are insignificant [11].The residual of the predicted plot indicates that the model does not show any violation of the independence or constant variance assumption hence confirming the literature by [11].

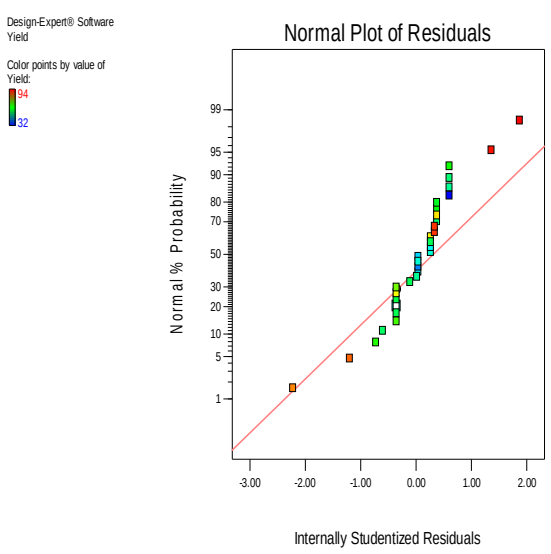

Fig.1.Normal Probability (%) plot of residuals

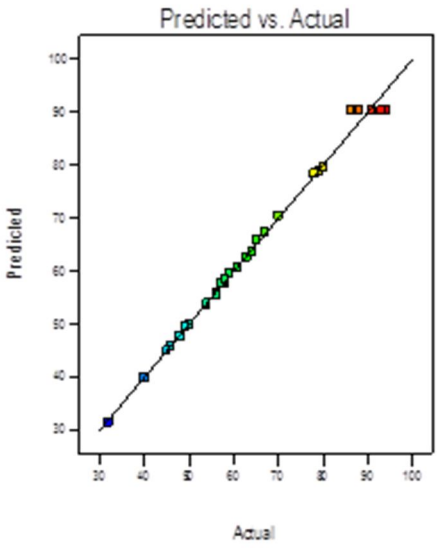

Fig.2 Predicted versus actual FAME yield

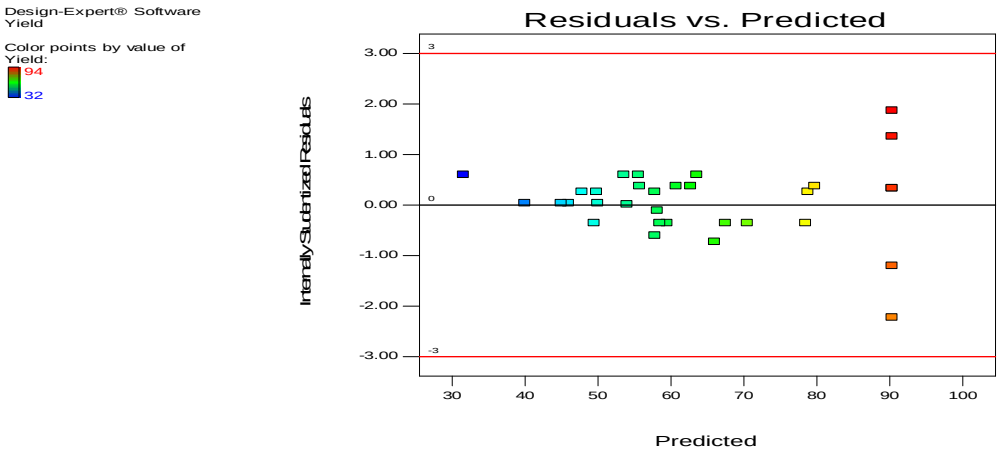

Fig.3 Residuals plot of predicted values

D. Three Dimensional (3D) Response Surface Plots

The 3D response surface plots of the proposed model have been shown in figure 4 to figure 6 that represents the analysis of interaction effects of the preferred variables. Figure 4 shows that increase in reaction time and catalyst weight leads to a corresponding increase in yield. The increment of catalyst loading caused significant increase in biodiesel yield at low reaction time. The significant increament of biodiesel yield (32 to 94%) was observed when the reaction time was raised from low level to high level . Low biodiesel content at low reaction time might due to the mass transfer effect of the three –phase system(oil-methanol-catalyst). However, the biodiesel yield was slightly influenced by the rise of catalyst at higher reaction time. It was observed that the yield became steady, when these parameters were increased further than the points indicated (beyond 4 hours and beyond 4wt %). This might indicate that the transesterification reaction has reached equilibrium condition, and further increase may lead to reverse reaction and thus reduce the biodiesel content [11].

Figure 5, shows the interactive effect of catalyst concentration and agitation speed. From the 3D surface plot, increment in catalyst weight and agitation speed leads to an increase in biodiesel yield to certain level. Further increment beyond 4wt% and 400 rpm for catalyst weight and agitation speed, respectively, leads to a decrease in biodiesel yield [21]. Excessive agitation causes splashing and the mixture tend to foam which may result in cavitation corrosion.

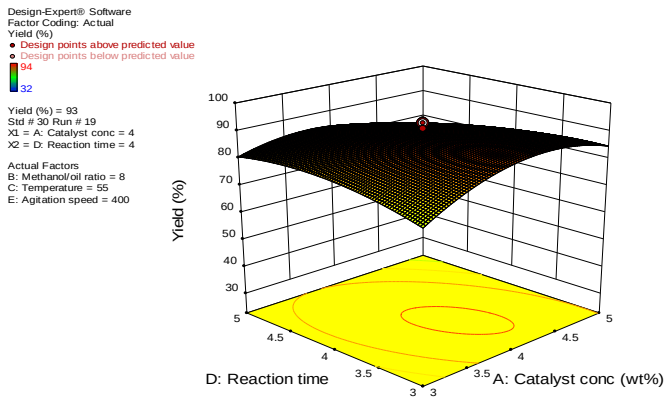

Fig.4.3D response surface plot of biodiesel :effect of catalyst Conc.(A) and reaction time (D) against yield(%)

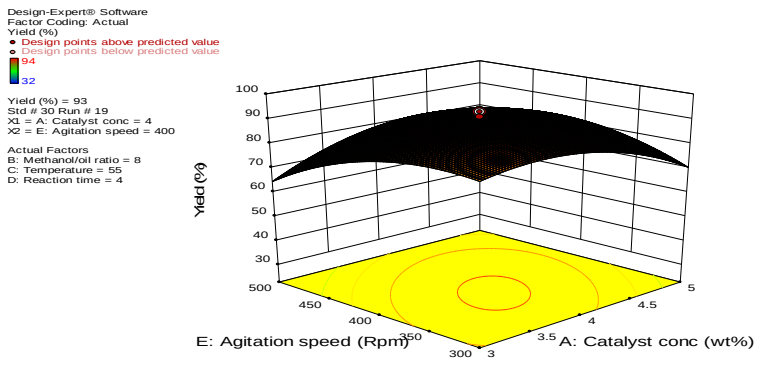

Fig.5.3D response surface plot of biodiesel:effect of catalyst conc.(A)and Agitation speed (E) against yield (%)

Figure 6, shows the interactive effect of methanol/oil ratio and temperature. From the plots, it was indicated that the highest yield (94%) was obtained at temperature of 55^0C and methanol/oil ratio of 8:1[21]. As the temperature increases, the solubility of methanol in the oil increases and so does the speed of reaction. As a matter of fact, at low temperature, methanol is not soluble in the oil; when the stirring is started an emulsion appears [6]. On the other hand, an excessive amount of alcohol above 8:1 makes the recovery of glycerol difficult. This may be attributed to the stoichiometry of transesterification, which requires a 3:1molar ratio of alcohol to triglycerides, since this reaction involves the conversion of one ester and an alcohol towards another, an excess of alcohol is used to drive the reaction near completion .

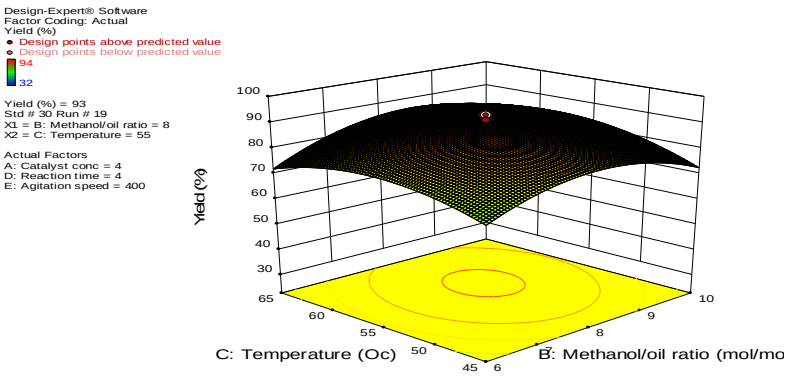

Fig. 6.3D response surface plot of biodiesel :effect of methanol to oil molar ratio (B) and temperature (C) against yield(%)

IV. CONCLUSION

In this work, the parametric optimization of transesterification of neemseed oil was performed. The process parameters (catalyst concentration, methanol to oil molar ratio, reaction temperature, reaction time and agitation speed) were optimized using response surface methodology (RSM) based on central composite design (CCD). Based on the experimental results it was concluded that catalyst concentration, methanol to oil molar ratio, and reaction temperature has significant effect on transesterification of neemseed oil to methyl ester using methanol and calcium oxide derived from waste of animal bone as alcohol and base catalyst.

The optimum conditions derived via RSM for base catalyzed transesterification of methyl ester from neem oil were: 4% catalyst weight, 8:1 methanol oil molar ratio, reaction temperature 55^0C ,reaction time of 4hr and agitation speed of 400rpm. The predicted yield at these conditions is 90.34%. An additional experiment was performed to confirm the optimum conditions obtained and this shows that the predicted values (95.6%) were in agreement with the experimental values (94%). The fuel properties of the methyl ester are within the American Standard for Testing Material (ASTM D 6751) specification for biodiesel.

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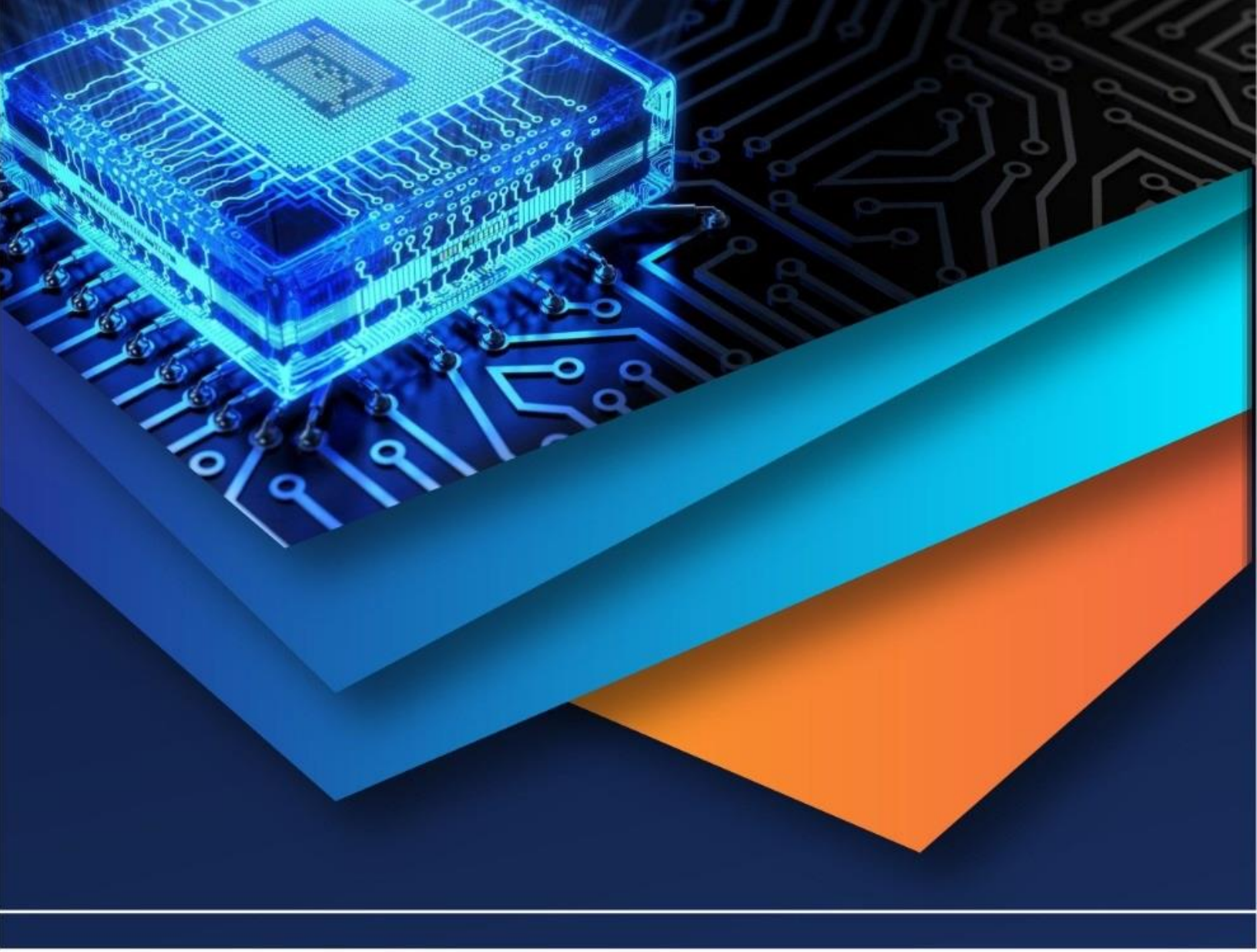

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