

## Optimization of Color Pigments Removal from Palm Oil by Activated Ukpor Clay Using Response Surface Methodology

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**Abstract:** The aim of this study was to investigate the optimum operating process parameters during the adsorption of color pigments from palm oil onto activated Ukpor clay using Response Surface Methodology (RSM). The clay was activated using sulphuric acid solution. The process parameters investigated include; temperature, time, adsorbent dosage, adsorbent/oil ratio and stirring speed. The results showed that the bleaching efficiency was significantly affected by the linear, quadratic and some of the interaction terms. The optimum conditions derived via RSM were: temperature- 152.73 °C; time- 2.60 h; adsorbent dosage- 6.09 g; adsorbent/oil ratio- 0.05g/mL; stirring speed- 39.34rpm; and under these conditions the predicted bleaching efficiency was 82.67% which has good correlation with the experimental value of 82.25%. The high prognosis obtained in this study has shown that 5-level 5 factor fractional central composite design of the RSM is suitable for predicting the optimum adsorption conditions of color pigments from palm oil using activated Ukpor clay.

**Keywords:** Adsorption, analysis of variance, activated clay, bleaching, color pigment, optimum condition, regression

### INTRODUCTION

Bleaching involves solely the removal of variety of impurities like fatty acids, gums, trace metals, phosphatides, etc., followed by decolourization. Bentonites are found in nature abundantly. However, most of them should undergo appropriate physical or chemical treatments such as acid activation, ion exchange and heating in order to promote their surface properties (Rossi *et al.*, 2003; Rozic *et al.*, 2010; Foletto *et al.*, 2011). Acid activation of clays is one of the most important processes that have been performed to achieve structural modifications in the clays for edible oil bleaching. Activated clays show a much higher bleaching capacity when compared to natural clays. During acid activation, initially interlayer captions are replaced with H<sup>+</sup> ions followed by partial destruction of aluminums octahedral sheets with subsequent dissolution of structural captions. The desired changes in the physicochemical properties of clay occurring as a result of acid activation depend considerably on the condition of acid activation such as acid concentration and the duration and temperature of the activation process (Motlagh *et al.*, 2008).

Designing of experiment and standardization of variables affecting the system is very critical in optimization process. Generally this optimization is carried out by using traditional one factor at a time method, which is simple, time and chemicals are

consumed in large quantities. Moreover this method neglects the interaction effects of process variables. Response Surface Methodology (RSM) is a useful model for studying the effect of several factors influencing the response variable by varying them simultaneously and carrying out a limited number of experiments. The two most commonly used designs in RSM are Central Composite Design (CCD) and Box Behnken Design (BBD) (Box *et al.*, 1978). This methodology was widely adopted in industries such as drug and food industry, chemical and biological processes, for the purpose of either producing high quality products or operating the process in a more economical manner and ensuring the process in a more stable and reliable way (Otto, 1999). It has also been successfully applied to different processes for achieving its optimization using experimental designs (Sudamalla *et al.*, 2012; Gunawan and Suhendra, 2008; Alam *et al.*, 2007; Fu *et al.*, 2009; Abali *et al.*, 1997; Yi *et al.*, 2010; Copur *et al.*, 2004; Narayana *et al.*, 2011; Umesh and Sud, 2005; Ata *et al.*, 2001; Yartasi *et al.*, 1999; Li *et al.*, 2010; Dilipkumar *et al.*, 2011; DePaiva *et al.*, 2009).

In this study, 5-level 4-factor fractional Central Composite Rotatable Design (CCRD) was used to evaluate the interactive effects and obtain the optimum conditions for the color pigments removal from palm oil by activated Ukpor clay.

## MATERIALS AND METHODS

(The experiments and analysis were performed at Chemical Engineering lab. Nnamdi Azikiwe University, Awka, within the months of June and July, 2011)

**Clay preparation and activation:** The Ukpok clay was mined from the site in Ukpok town (N: 5° 54' 27.5"; E: 6° 56' 3.7"; A: 137 m) in Nnewi South local government area of Anambra state. The clay was wet at the point of collection and it was possible to remove heavy particles like grasses, stones and decayed organic matters. The clay was further made into paste with water and sieved to remove smaller contaminants. The samples were then air-dried for 3 days, after which they were crushed with mortar and pestle to reduce the particle size. The grinded samples were sieved into different particle sizes using a standard Taylor sieve. The prepared samples were made into large quantities to serve for all the experiments.

The prepared clay samples were then activated using sulphuric acid in the following manner. 100 g of the grinded clay samples were treated with sulphuric acid. The acid was in excess of 10 percent which was calculated to be ten times the amount required for complete dissolution of all cationic components of the crude material.

Hundred gram of the sample was mixed with the acid solution (500 mL) of various concentrations (0.5-4 M) and the resulting suspension was heated in a water bath at reflux temperature, which proved to be 108°C for the boiling clay slurry. The heating was done for 3 h under continuous stirring. At the completion of the heating time, the slurry was removed from the bath and allowed to cool. After the cooling, the slurry was filtered via a Buchner funnel and the clay residue was washed several times with distilled water, followed by filtration until the filtrate was neutral to pH indicator paper. The prepared wet sample was then dried in an oven at 120°C over night. The lumps of the prepared clays were crushed and sieved again into particle size of 0.212 mm.

**Adsorption experiment:** Hundred gram of the refined unbleached palm oil was measured out into a 250 mL conical flask and heated on a magnetically-stirred hot plate to 900°C while stirring continuously. Two gram of the sized activated clay samples was then added to

the heated oil and stirred continuously via a magnetic stirrer carefully inserted into the beaker. The whole mixture was heated to a temperature of 1300 C for 45 min. At the completion of the time, the hot oil and clay mixture was filtered under gravity using What man filter paper No. 42 (15 cm diameter), before measuring the absorbance. The bleaching efficiency of the activated clay samples was then determined by measuring the color of the bleached oil using UV-VIS Spectrophotometer (Model WFJ 525) at 450 nm. The bleaching efficiency is defined by the following expression in this study:

$$\text{Bleaching efficiency (\%)} = \frac{A_{\text{unbleac hed}} - A_{\text{bleac hed}}}{A_{\text{unbleac hed}}} \times 100 \quad (1)$$

where a unbleached and a bleached are absorbencies of unbleached and bleached palm oil, respectively, at 450 nm.

**Design of experiment:** In order to examine the combined effect of the five different factors (independent variables): bleaching temperature, bleaching time, adsorbent dosage, adsorbent/oil ratio and stirring speed on bleaching efficiency and derive a model, a central composite factorial design of 25-1 = 16 plus 4 centre points and (2 x 5 = 10) star points leading to a total of 30 experiments were performed. The factors levels with the corresponding real values are shown in Table 1 while the design matrix is shown in Table 2. The matrix for the 5 variables was varied at 5 levels (-α, -1, 0, +1 and +α). As usual, the experiments were performed in random order to avoid systematic error.

**Statistical analysis:** The generated experimental data were analyzed using the Statistical 10 trial version software by Stat Soft Inc. USA and then interpreted. The analytical steps used include: Analysis of Variance (ANOVA), regression analysis and response surface plots of the interaction effects of the factors to evaluate optimum conditions for the bleaching process. The linear, quadratic and linear interactive effects of the process variables on the bleaching efficiency were calculated and their respective significant evaluated by ANOVA test. The p-value was used as the yardstick for measuring the significance of the regression coefficients, values of p greater than 0.05 signified that the coefficient is significant. The experimental data

Table 1: Experimental range of the independent variables, with different levels, to study the bleaching properties of Ukpok clay after activation with sulphuric acid

Independent variable	Symbol	Range and levels				
		-α	-1	0	+1	+α
Bleaching temp (°C)	X <sub>1</sub>	15	70	125	180	235
Bleaching time (h)	X <sub>2</sub>	-1.25	0.5	2.25	4.00	5.75
Adsorbent dosage (g)	X <sub>3</sub>	-4.25	0.5	5.25	10.0	14.75
Adsorbent/oil ratio (mm)	X <sub>4</sub>	-0.01	0.02	0.05	0.08	0.11
Stirring speed (rpm)	X <sub>5</sub>	-135	90	315	540	765

Table 2: Experimental design/plan for bleaching studies

Run order	Bleach temp (°C), X <sub>1</sub>	Bleach time (h), X <sub>2</sub>	Adsorbent dosage (g), X <sub>3</sub>	Adsorbent/oil ratio (g/mL), X <sub>4</sub>	Stirring speed (rpm), X <sub>5</sub>
1	-1	70	-1	0.5	-1
2	+1	180	-1	0.5	-1
3	-1	70	+1	4.0	-1
4	+1	180	+1	4.0	-1
5	-1	70	-1	0.5	+1
6	+1	180	-1	0.5	+1
7	-1	70	+1	4.0	+1
8	+1	180	+1	4.0	+1
9	-1	70	-1	0.5	-1
10	+1	180	-1	0.5	+1
11	-1	70	+1	4.0	-1
12	+1	180	+1	4.0	-1
13	-1	70	-1	0.5	+1
14	+1	180	-1	0.5	+1
15	-1	70	+1	4.0	+1
16	+1	180	+1	4.0	+1
17	-2	15	0	2.25	0
18	+2	235	0	2.25	0
19	0	125	-2	-1.25	0
20	0	125	+2	5.75	0
21	0	125	0	2.25	-2
22	0	125	0	2.25	+2
23	0	125	0	2.25	0
24	0	125	0	2.25	0
25	0	125	0	2.25	0
26	0	125	0	2.25	0
27	0	125	0	2.25	0
28	0	125	0	2.25	0
29	0	125	0	2.25	0
30	0	125	0	2.25	0

were fitted to the second-order polynomial regression model and the adequacy of the model tested by the coefficient of determination (R<sup>2</sup>) value as compared to the adjusted R<sup>2</sup> value.

### RESULTS AND DISCUSSION

**Evaluation of regression model for bleaching efficiency:** The correlation between the experimental process variables and the bleaching efficiency was evaluated using the CCD modeling technique. Second order polynomial regression equation fitted between the response (bleaching efficiency, (Y)) and the process variables: reaction temperature (X<sub>1</sub>), reaction time (X<sub>2</sub>), adsorbent dosage (X<sub>3</sub>), adsorbent/oil ratio (X<sub>4</sub>) and stirring speed (X<sub>5</sub>). From Table 3, the ANOVA results showed that the quadratic model is suitable to analyze the experimental data. The model in terms of the actual values of the process parameters is given by:

$$Y = 80.446 + 4.458X_1 + 2.458X_2 + 1.375X_3 - 3.125X_4 + 3.042X_5 - 2.063X_1X_2 - 1.125X_1X_3 + 2.438X_1X_4 + 2.063X_1X_5 + 1.438X_2X_3 + 1.875X_2X_4 + 1.125X_2X_5 - 0.125X_3X_4 + 1.313X_3X_5 + 2.125X_4X_5 - 4.390X_1^2 - 4.390X_2^2 - 5.265X_3^2 - 6.015X_4^2 - 5.890X_5^2 \quad (2)$$

To develop a statistically significant regression model, the significance of the regression coefficients

was evaluated based on the p-values. The coefficient terms with p-values more than 0.05 are insignificant and are removed from the regression model. The analysis in Table 3 shows that linear, quadratic and the interaction terms of temperature and time; temperature and adsorbent/oil ratio; temperature and stirring speed; time and adsorbent dosage; and adsorbent dosage and stirring speed are significant model terms. The model reduces to Eq. (3) after eliminating the insignificant coefficients:

$$Y = 80.44 + 4.46X_1 + 2.46X_2 + 1.38X_3 + 3.13X_4 + 3.04X_5 - 2.06X_1X_2 + 2.44X_1X_4 + 2.06X_1X_5 + 1.44X_2X_3 + 1.31X_3X_5 - 4.39X_1^2 - 4.39X_2^2 - 5.27X_3^2 - 6.02X_4^2 - 5.89X_5^2 \quad (3)$$

The analysis of variance indicated that the quadratic polynomial model was significant and adequate to represent the actual relationship between bleaching efficiency and the significant model variable as depicted by very small p-value (0.0000). The significance and adequacy of the established model was further elaborated by high value of coefficient of determination (R<sup>2</sup>) value of 0.9874 and adj. R<sup>2</sup> value of 0.9623. This means that the model explains 98.74% of the variation in the experimental data. The adequate correlation between the experimental values of the

response variable and predicted values further showed the adequacy of the model as illustrated in Fig. 1.

Figure 2 shows the linear and quadratic effects and the interaction of the independent variables, at the 95% of confidence level, on the percentage bleaching efficiency. It can be seen that all the variables were significant at the linear and quadratic levels. The largest main effect observed in the color pigment removal was the variable adsorbent/oil ratio at quadratic level, followed by stirring speed (quadratic level). These variables have negative effect on the response variable, that is, their increase reduces the bleaching efficiency. The figure also shows that temperature has the largest positive effect at the linear level followed by stirring speed (linear) and time (linear). Moreover, the interactive effects between the independent variables were also significant.

**Response surface plots:** The interactive effects of the process variables on the percent bleaching efficiency

were studied by plotting three dimensional surface curves against any 2 independent variables, while keeping other variables at their central (0) level. The 3D curves of the response (bleaching efficiency) and contour plots from the interactions between the variables are shown in Fig. 3 to 12. The interactive effects of temperature and time on the bleaching efficiency are negative as shown in Table 3. The response surface plots of the interactive effect of temperature and time are shown in Fig. 3. The bleaching efficiency increased with simultaneous increase in reaction temperature and time to about 190°C and 4 h, respectively and thereafter decreased with the increase in temperature and time. The same trend was observed in Fig. 4 to 6. Increase in reaction time resulted in corresponding increase in bleaching efficiency as was evident in Fig. 3 and 7 to 9. Fig. 8 shows that as the adsorbent dosage increased, less time was required for maximum bleaching efficiency to be achieved and the same trend was observed in Fig. 9, at lower adsorbent/oil ratio,

Table 3: ANOVA for analysis of bleaching efficiency, values of regression coefficients and their significant test and effects

Source	Coeff.	SS	Degree of freedom	Mean Square	F-value	S.E.	t (10)	p-value
Model	80.4460	3356.43	20	167.82	124.65	0.9950	80.8480	0.0000
Reaction temperature (L)	4.4583	477.042	1	477.042	91.1554	0.9339	9.5475	0.0000
Reaction temperature (Q)	-4.3902	543.34	1	543.34	103.824	0.8617	-10.1894	0.0000
2. Reaction time (L)	2.4583	145.042	1	145.042	27.7153	0.9339	5.2645	0.0004
Reaction time(Q)	-4.3902	543.34	1	543.34	103.824	0.8617	-10.1894	0.0000
3. Adsorbent dosage (L)	1.3750	45.375	1	45.375	8.6705	0.9339	2.9446	0.0147
Adsorbent dosage (Q)	-5.2652	781.507	1	781.507	149.334	0.8617	-12.2202	0.0000
4. Adsorbent/Oil ratio (L)	-3.1250	234.375	1	234.375	44.7855	0.9339	-6.6922	0.0001
Adsorbent/Oil ratio(Q)	-6.0152	1020.007	1	1020.007	194.908	0.8617	-13.9609	0.0000
5. Stirring speed(L*)	3.0417	222.042	1	222.042	42.4288	0.9339	6.5137	0.0001
Stirring speed(Q**)	-5.8902	978.054	1	978.054	186.891	0.8617	-13.6708	0.0000
1L by 2L	-2.0625	68.062	1	68.062	13.0057	1.1438	-3.6063	0.0048
1L by 3L	-0.5625	5.063	1	5.063	0.9674	1.1438	-0.9835	0.3485
1L by 4L	2.4375	95.063	1	95.063	18.165	1.1438	4.2620	0.0017
1L by 5L	2.0625	68.062	1	68.062	13.0057	1.1438	3.6063	0.0048
2L by 3L	1.4375	33.063	1	33.063	6.3177	1.1438	2.5135	0.0307
2L by 4L	0.9375	14.063	1	14.063	2.6871	1.1438	1.6392	0.1322
2L by 5L	0.5625	5.063	1	5.063	0.9674	1.1438	0.9835	0.3485
3L by 4L	-0.0625	0.063	1	0.063	0.0119	1.1438	-0.1093	0.9151
3L by 5L	1.3125	27.562	1	27.562	5.2668	1.1438	2.2949	0.0446
4L by 5L	1.0625	18.062	1	18.062	3.4515	1.1438	1.8578	0.0928
Error		52.333	10	5.233				
Total SS		4161.097	30					

R<sup>2</sup> = 0.9874 Adj. R<sup>2</sup> = 0.9623 \*Linear effect \*\*Quadratic effect

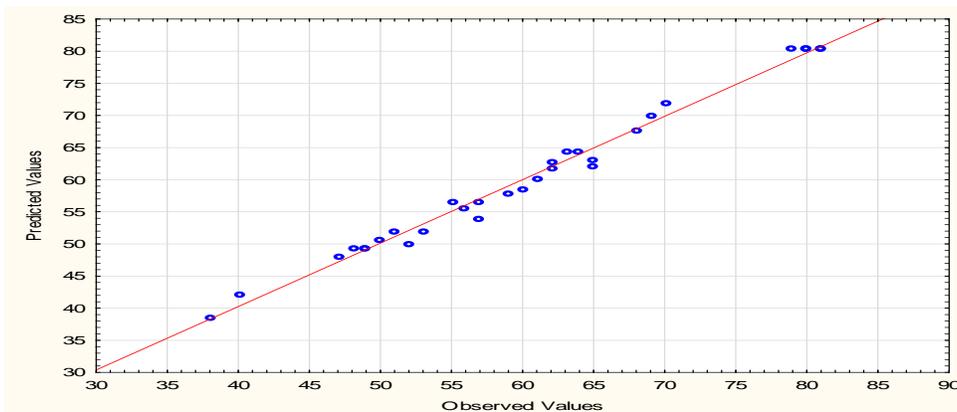


Fig. 1: Plot of the predicted values versus the observed experimental values

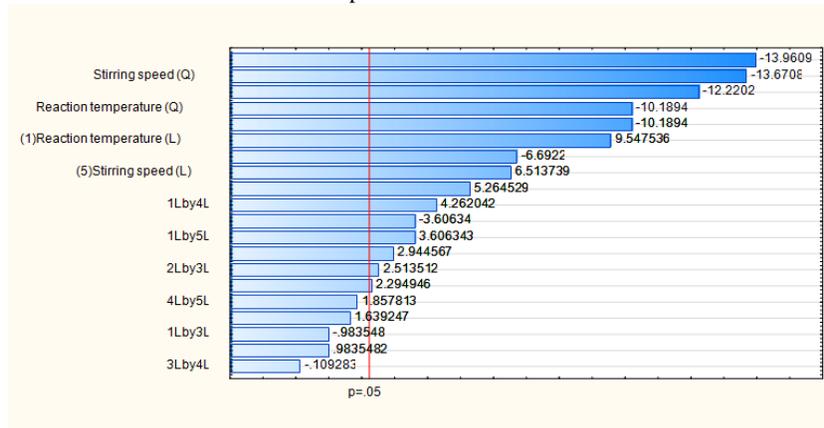


Fig. 2: Pareto's chart to estimate the linear (L), quadratic (Q) and linear interaction (L by L) effects on the bleaching efficiency

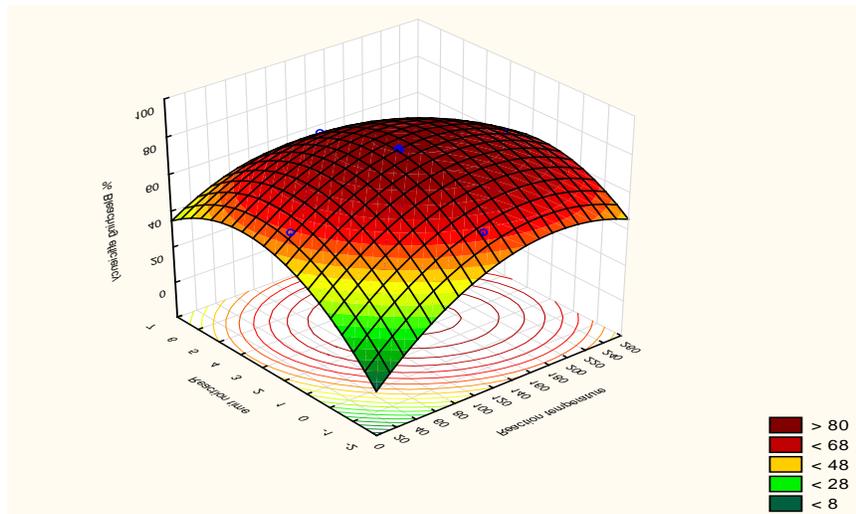


Fig. 3: Effect of temperature and time on bleaching efficiency at constant adsorbent dosage, adsorbent dosage/oil ratio and stirring speed

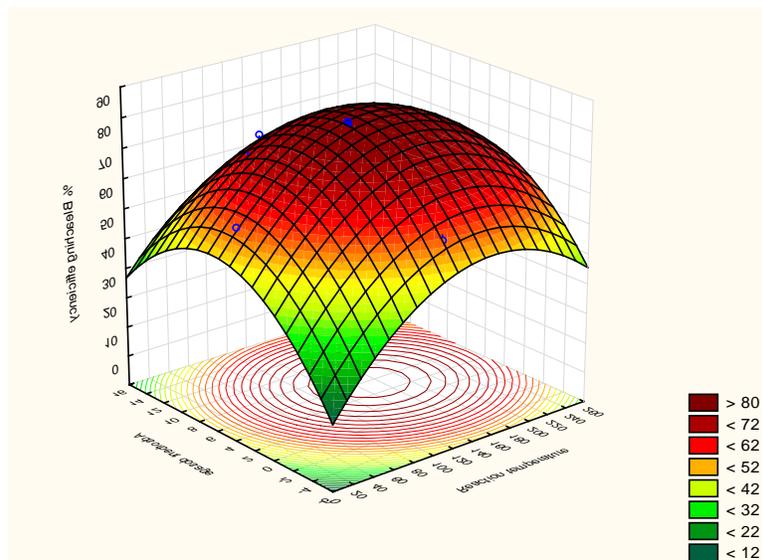


Fig. 4: Effect of temperature and adsorbent dosage on bleaching efficiency at constant time, adsorbent/oil ratio and stirring speed

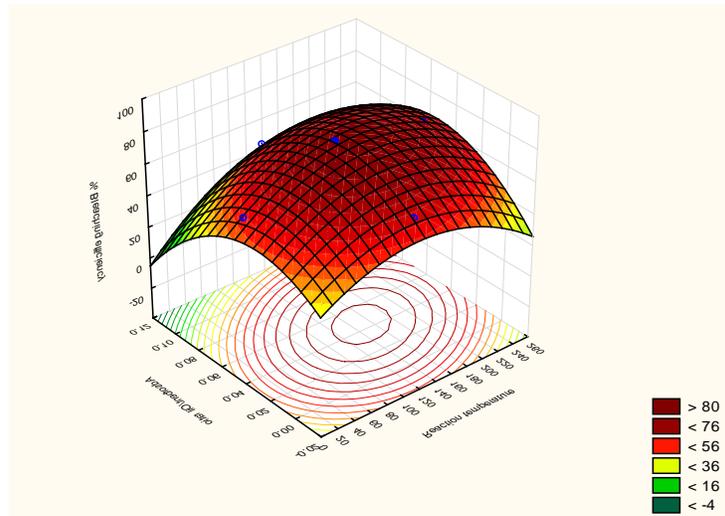


Fig. 5: Effect of temperature and adsorbent/oil ration on bleaching efficiency at constant time, adsorbent dosage and stirring speed

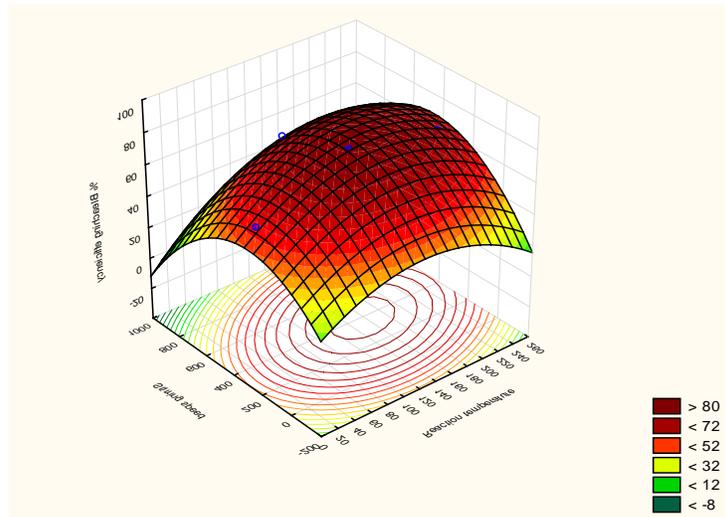


Fig. 6: Effect of temperature and stirring speed on bleaching efficiency at constant time, adsorbent dosage and adsorbent/oil ratio

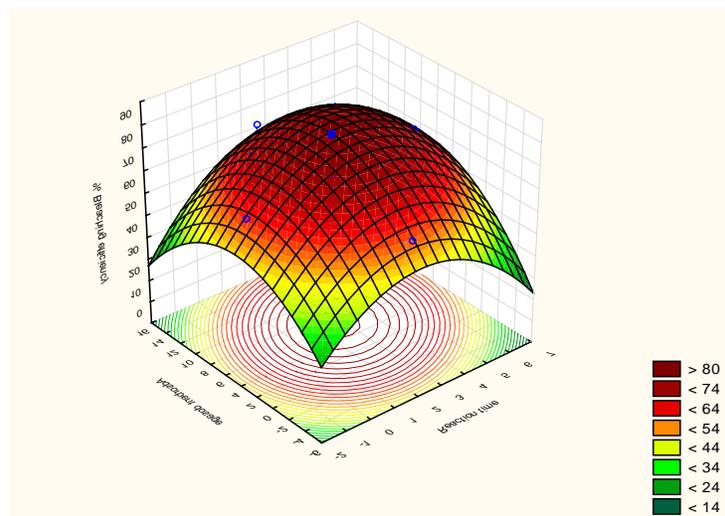


Fig. 7: Effect of time and adsorbent dosage on bleaching efficiency at constant temperature, adsorbent/oil ratio and stirring speed

a time of less than 2 h gave the maximum bleaching efficiency. The effect of adsorbent dosage was observed to be similar to that of temperature effect. High adsorbent dosage gave rise to increased bleaching efficiency as was evident from Fig. 4, 8, 10 and 11. It was observed from Fig. 5, 9, 11 and 12, that bleaching efficiency increased at lower adsorbent/oil ratio. This is because there were enough adsorption sites for the

adsorb ate molecules and this resulted to lower time to achieve maximum bleaching efficiency. The effect of stirring speed on the bleaching efficiency was observed to be positive and this is because high speed increased the kinetic energy of the adsorb ate molecules, thereby causing frequent collision of the adsorb ate and adsorbent molecules. The optimum conditions are: reaction temperature 152.73°C; reaction time

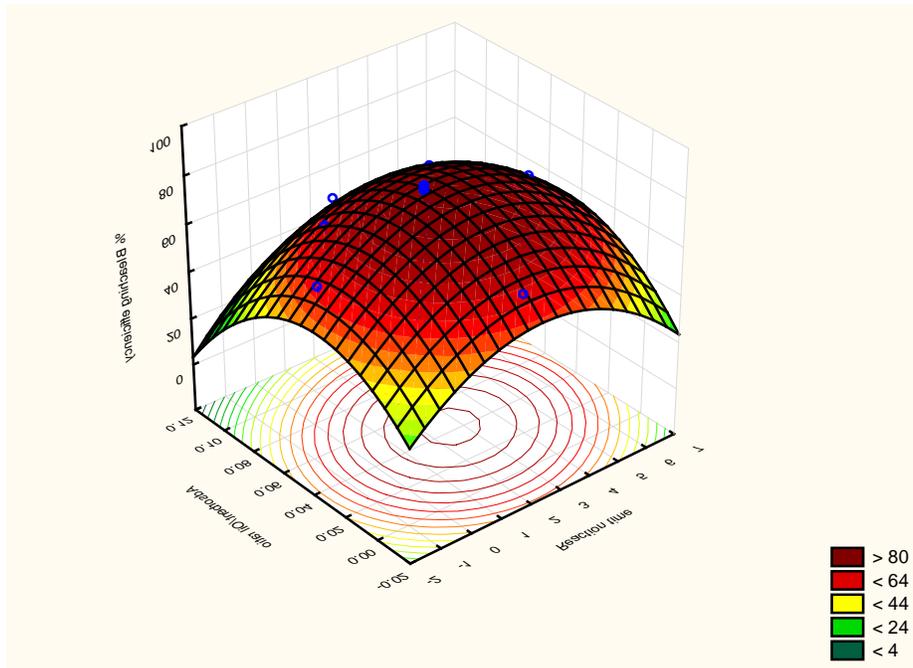


Fig. 8: Effect of time and adsorbent/oil ratio on bleaching efficiency at constant temperature, adsorbent dosage and stirring speed

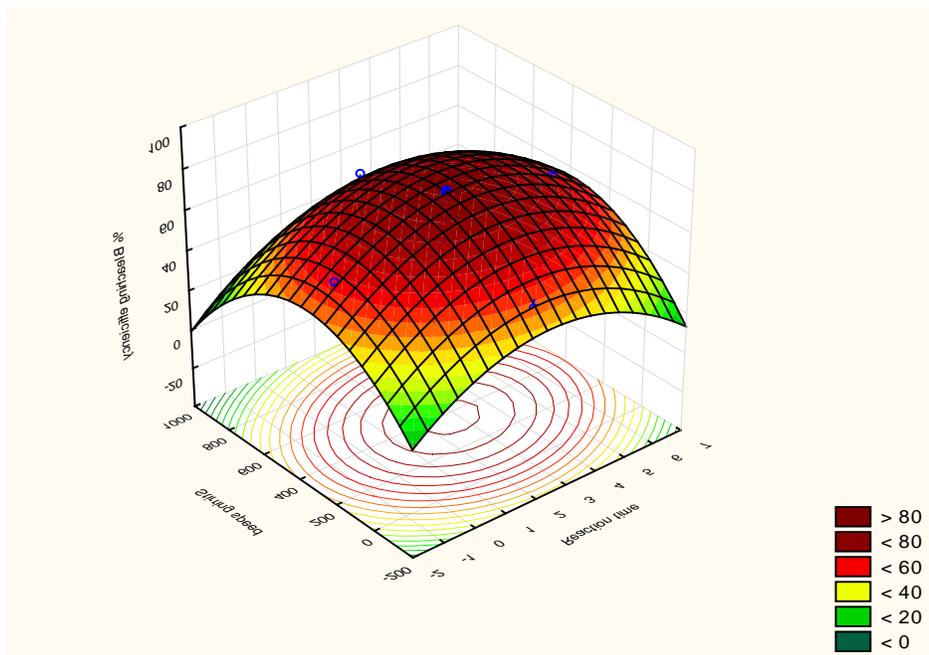


Fig. 9: Effect of time and stirring speed on bleaching efficiency at constant temperature, adsorbent dosage and adsorbent/oil ratio

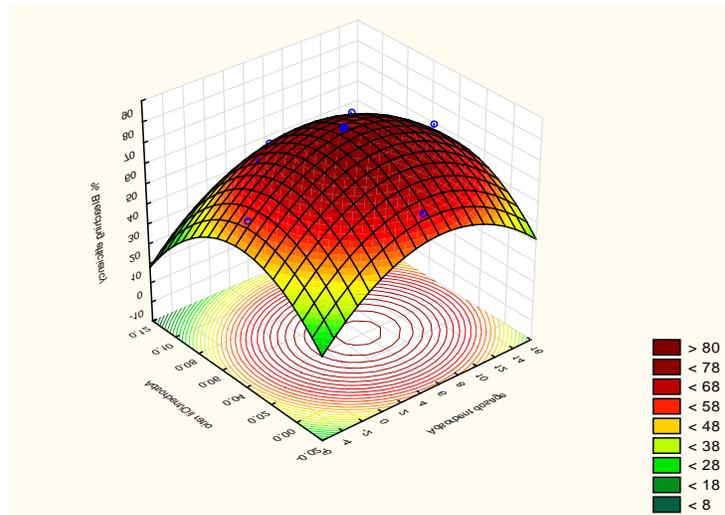


Fig. 10: Effect of adsorbent dosage and adsorbent/oil ratio on bleaching efficiency at constant temperature, time and stirring speed

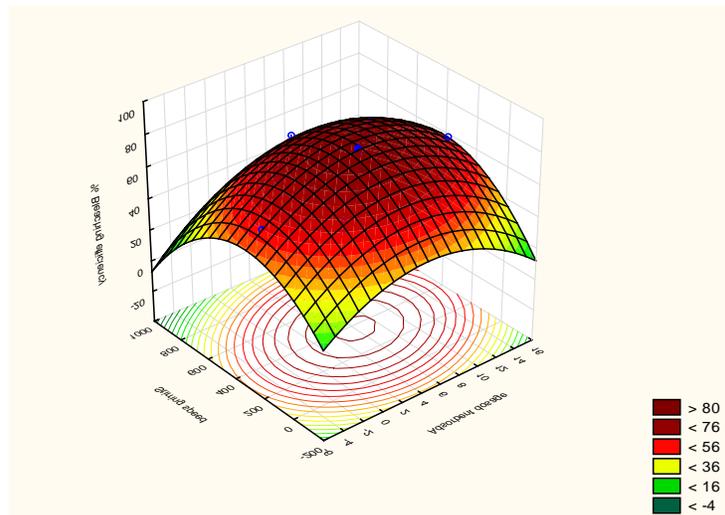


Fig. 11: Effect of adsorbent dosage and stirring speed on bleaching efficiency at constant temperature, time and adsorbent/oil ratio

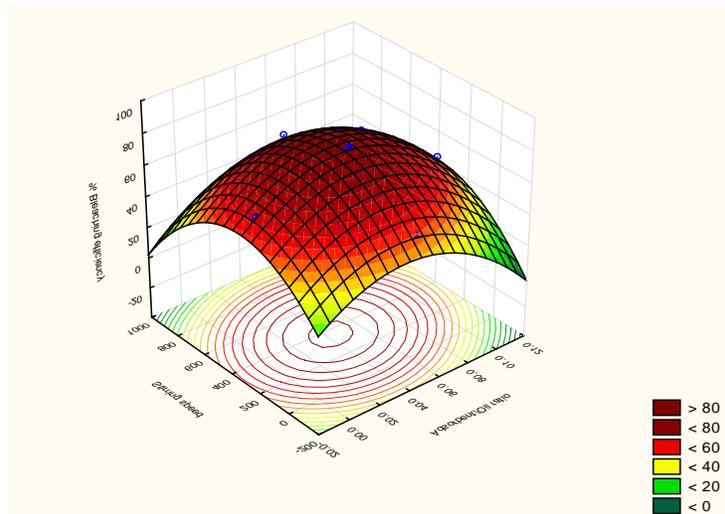


Fig. 12: Effect of adsorbent/oil ratio and stirring speed on bleaching efficiency at constant temperature, time and adsorbent dosage

2.60 h; adsorbent dosage 6.09 g; adsorbent/oil ratio 0.05 g/ml; and stirring speed 397.34 rpm; and the optimum bleaching efficiency at this optimum condition was predicted to be 82.67%. Experiments were carried out at these optimum conditions to validate the predicted optimum values. The experimental value of 82.25% agreed closely with that obtained from the regression model.

## CONCLUSION

The optimization of the bleaching efficiency of palm oil using activated Ukpok clay has been successfully studied. Central composite design of the response surface methodology was used to investigate the relative significant process variables that affect the bleaching efficiency. Among the process variables, reaction temperature and reaction time were found to be the most linear significant variables. The optimum conditions for maximum bleaching efficiency were as follows: reaction temperature 152.73°C; reaction time 2.60 h; adsorbent dosage 6.09 g; adsorbent/oil ratio 0.05 g/mL; and stirring speed 397.34 rpm and the optimum bleaching efficiency was 82.67%. This study has shown that Ukpok clay is a good source of adsorbent for palm oil bleaching and the good correlation between the predicted and experimental values shows that the method of optimization adopted is efficient for the process.

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