

Determination of the Optimal Process Conditions for the Acid Activation of Ngwo Clay in the Bleaching Of Palm Oil

S .O Egbuna¹., C. N Mbah²., T. O Chime¹.

¹Department of Chemical Engineering, Enugu State University of Science and Technology, ESUT, Enugu. ²Department of Material and Metallurgical, Engineering, Enugu State University of Science and Technology, ESUT, Enugu.

ABSTRACT

In this work, the optimal adsorption parameters for the adsorption of Carotenoid in the bleaching of palm oil was investigated. Ngwo clay, a local adsorbent obtained from Ngwo town in the South-Eastern province of Nigeria, was used in the study. The palm oil used was also obtained from a local market in Enugu in the same region. The purpose of the work was to develop a model to optimize the efficiency of a local adsorbent that will be cheap and environmentally friendly, for the removal of pigments during refining of vegetable oils. The clay was first, acid activated and characterized, and used in the investigation. Central Composite Design (CCD) package was used to optimize the effects of process parameters of Temperature, Time and Clay Dosage on the bleaching efficiency of Palm Oil. A linear model was predicted and optimized based on BBD. This gave bleaching time of 40min., Temperature of 99.83°C, and Clay dosage of 4%, at a predicted bleaching efficiency of 83%. The optimum conditions were validated to obtain an experimental value of 82.5% with 1.7% error condition.

Key words: BBD, ANOVA, Ngwo Clay, Palm Oil, bleaching efficiency, Acid activation

I. INTRODUCTION

Palm oil is one of the various types of vegetable oils belonging to the group called Lipids, because of its fatty acid content. [1] and [2], defined it as triglycerides extracted from plants. Most fats contain some colouring matters, either as a natural constituent, or as a discolouration produced during the processing, [2]. The natural pigments present in vegetable oils are mainly the caroteniods, giving yellow and red colours, and the chlorophylls, which give green colours, [3]. [4], observed that colour deterioration can also take place during the extraction process, especially in the local method of extraction used in most parts of the Eastern region of Nigeria. Removal, or reduction of colour and other pigmented components, otherwise called bleaching, according to [5], is necessarily, not only because a pale- coloured fat has an appeal of purity, [6], but also because the colours of the fat can influence the appearance of prepared food, and even more importantly, the pigment present may affect the flavour and stability of the fats and foods made from them. The decourloration (bleaching), could be achieved by chemical treatment, [7], by heat treatment, , [8], or by adsorption method, [9]; the most effective and widely used being the latter. Two methods are available for the refining of palm oil, namely, the physical and chemical methods. The refining operations, here include, degumming, neutratralization of free fatty acid, (FFA), for chemical process, using caustic soda, bleaching, to improve flavour and stability of the finished product, and deodorization to further reduce colour, and FFA, and improve stability. [10], noted that bleaching is the most important step in palm oil refining, especially in the physical process, since it is at this stage that most of the contaminants and oxidative products of Aldehydes and Ketones, are removed. [11] also observed that contact time, temperature, oil to adsorbent ratio, pressure, and other parameters, are the conditions that affect the performance of the bleaching process. Bleaching is done by using thermally activated clay, [12], or by acid activated clay, [13]. [14], said that the quality of raw material, (palm oil), is very important, since it relates directly to the processing cost, and product's shelf life. [15], opined that colour and FFA content continue to fall with increasing bleaching earth, but the peroxide value is minimized after 30 minutes. They noted that the continuing fall in colour is attributed to the heat bleaching effect.

Bleaching temperature is one of the factors that affect the performance of the bleaching and degumming processes of raw palm oil. [10] have also observed that bleaching temperature has a great effect on the colour, FFA, PV and AV of oil being processed, and the keeping quality of the oil as shown in Figs. I and II, respectively. They noted that bleached oil colour tends to continuously fall as the temperature is increased, but

the deodorized oil colour reached a maximum when the bleaching temperature is about 100°C. Oil – adsorbent ratio, is also of importance in determining the optimum performance condition of clay during bleaching, as it affects the efficiency of degumming and bleaching processes. The higher the clay dosage the more the colour reduction; the optimum clay dosage will however, depend on the quality and nature of the impurities in the raw palm oil [13]. [16] and [13], have developed the adsorption mechanism(model) for the adsorption of carotenoids on activated clay based on adsorption theory of Langmuir isotherm. [17], had earlier worked on the optimal bleaching performance of acid activated Ngwulangwu clay, using Central Composite Design (CCD) to optimize the process variables.

In this work,

- The optimal process conditions for palm oil bleaching was determined using Ngwo clay as an adsorbent
- BBD via Response Surface Method, was used to find the most suitable conditions to optimize the process variables
- The interaction between the variables was analyzed by applying the ANOVA method of analysis.

II. Materials and Methods

The clay and the oil used in the investigations were obtained locally from Ngwo Town, in Enugu, South East province of Nigeria. Tetraoxosulphate (VI) acid is used for the activation and petroleum ether was analytically graded.

2.1 Digestion of Clay Samples.

The mined clay was sun-dried for 24 hours and size reduced to fine particles using mortar and pestle. The ground samples were sieved to remove impurities and then oven dried at 105°C. The samples were then put in contact with hydrochloric acid in a 250 cm³ flask placed in a regulated water bath. The flask was heated while continuously being stirred. 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 Buckner funnel and the clay residue was washed several times with distilled water, followed by filtration until the filtrate was neutral to pH indicator paper.

2.2. Acid Activation of Clay Sample

The acid activation was carried out according to the method described by [18], [19] and, [20]. The clay was ground into powder using pestle and mortar, and sieved through a laboratory test sieve of aperture - 150μ m. The clay sample (100g) was introduced into a 600ml Pyrex Beaker and 250ml of 1M Tetraoxosulphate (VI) acid solution added. The mixture was homogenized in a thermostat bath at a temperature of 95°C for 3hours. The resulting mixture was filtered, washed with distilled water severally to reduce the acidity. The activated clay sample was dried in an electric oven at a temperature of 105°C. The dried activated clay sample was then sieved through a laboratory test sieve of aperture - 150μ m and stored in an air tight container.

2.3 Characterization of Clay Sample

The clay samples were characterized using Atomic Absorption spectrophotometer and X-ray Fluorensce (PW 4030 X-Ray Spectrophotometer). This is shown in Table1.

| PARAMETER | Raw Clay | Bleached Clay |
|------------------------------------|------------|---------------|
| Moisture content % | 7.95 ±0.51 | 6.3 |
| Volatile matter % | 2.46 ±0.04 | .65 |
| Fixed carbon % | 0.84 ±0.08 | .52 |
| Specific Gravity g/cm ³ | 2.57 | 2.38 |
| pH | 5.7 | 5.6 |
| Bulk density g/cm ³ | 1.56 | 0.89 |
| Non Clay Residue (%) | 3.55 | 0.68 |
| TitratableAcidity(mg NaOH/g | 0.78 | 0.46 |
| SiO ₂ | 63.39 | 63 |
| AI ₂ O ₃ | 4.42 | 5.75 |
| Fe ₂ O ₃ | 0.11 | 0.01 |
| CaO | 1.62 | 0.54 |
| MgO | 2.33 | 0.5 |
| Na ₂ O | 4.10 | 0.35 |
| K ₂ O | 3.48 | 1.25 |
| Ignition loss | 11 | 8.0 |

Table 1 The physicochemical properties of raw and activated Ngwo Clay..

2.4 Bleaching Process and Analyses

Bleaching of the palm oil was carried out according to the procedure reported by [21]. 50g of degummed, palm oil was poured into a 50ml Pyrex beaker and heated up to a required temperature of 100°C, for the reaction on a magnetic hot plate. When the magnetic hot plate reached the set temperature, the activated clay sample was added. The process was carried out at 20 -160°C and the contact times were 10, 20, 30 40 an 50min, with clay dosage of 4%, 6% and 8%. At the end of the process, the mixture was filtered through Whitman No 1 filter paper into a test tube until a reasonable amount was obtained. The absorbance of the oil was measured using a UV spectrophotometer as follows: 0.1g of palm oil was diluted in 7.5ml of petroleum ether and the absorbance of the sample determined at 445nm wavelength using the petroleum ether as reference [21]. The percentage of oil bleached was calculated as follows:

| %Bleached= | Absorbance | unbleached | - Absorbance | bleached | - x1 00 | .1 |
|------------|------------|------------|--------------|----------|---------|--------|
| | | Absorbance | bleached | | | |

Table 2 The effect of bleach temperature on Colour, PV, AV and FFA of bleached palm oil; time is constant at 40min.

Tables 2nad 3 show the results of characterization experiments on bleached oil.

| Temper | ature °C | Colour in 1 inch cell | | Peroxide | Anisidine | Free fatty | | |
|------------|----------|-----------------------|----------|----------------|-----------|---------------|-----------------|----------|
| | | Bleached o | il | Deodoriz | ed oil | value | value | acid |
| | 20 | 14.2 | | | 3.8 | 6.5 | 3.60 | 0.62 |
| | 40 | 13.8 | | | 3.7 | 6.0 | 3.65 | 0.60 |
| | 60 | 13.5 | | | 3.8 | 5.5 | 3.70 | 0.48 |
| 80 | | 13.3 | | 3.6 | | 5.0 | 3.80 | 0.50 |
| 95 | | 12.6 | | 3.5 | | 4.3 | 3.85 | 0.40 |
| 100 | | 11.5 | | 3.4 | | 3.0 | 4.05 | 0.12 |
| Time(days) | | Colour (red), 1" | , | Cell | | Peroxide | alue of deodori | zed oil |
| 110 | • | 9.6 | | 3.5 | | 2.8 | 6.00 | 0.13 |
| 120 | | 9.2 | | 4.8 | | 1.2 | 6.50 | 0.40 |
| 140 | | 8.5 | | 5.1 | | 1.0 | 10.50 | 0.50 |
| 160 | | 8.0 | | 5.5 | | 0.9 | 14.50 | 0.60 |
| | Bleach | ned at 110°C | Ble | ached at 150°C | Blea | ched at 110°C | Bleached | at 150°C |
| 1 | | 3.10 | | 5.8 | | 0.00 | 0.00 | |
| 4 | | 3.20 | 3.20 5.9 | | | 0.46 | 1.80 | |
| 7 | | 3.30 6.2 | | 6.2 | | 0.82 | 2.00 | |
| 14 | | 3.35 6.8 | | | 1.17 | 2.32 | | |
| 21 | | 3.45 | | 7.4 | | 1.50 | 2.80 | |
| 28 | 28 | | | 8.6 | | 1.75 | 3.48 | |

Table 3 The effects of colour and PV on the keeping quality of bleached and deodorized palm oil.

2.5 Experimental Design of Box Benhken Design (BBD)

Box Benhken Design of experiment, also known as (central composite rotatable design) was used to optimize the variables in order to obtain optimum bleaching conditions of palm oil using activated Ngwo clay. Time, temperature and dosage were chosen as independent variables, and the percentage oil bleached was the dependent variable. The experimental range and level of independent variables for bleaching of palm oil were analyzed using BBD. In this study, a set of 20 experiments were carried out. These 20 experiments contain 8 core points, 6 stars like points and 6 null points. The distance of the star like points from core point is given by D = 2n/4, where n is the number of factors (for three factors, D = 2x3/4 The results are shown in table 4, where X_1 a coded variable that represents the temperature, X_2 represents the time and X_3 represents the clay dosage. The design of the experiment was analyzed with the aid of Minitab software. By solving the regression equation and analyzing the response surface contour plots, the optimum values of selected variables were obtained. The most important parameters affecting the efficiency of the bleaching process are *temperature* (X₁), *time* (X₂) and dosage (X₃). In order to study the combined effects of these factors, experiments were performed by varying physical parameters using the experimental design. Experimental and Theoretical values for percentage palm oil bleached were obtained. It can be seen that the optimum percentage oil bleached of 83% was obtained at temperature of about 100°C in 70mins bleaching time and clay dosage of 6grms. The result of analysis of variance (ANOVA) for the response surface quadratic model is determined. The tests for adequacy of the regression models, significance of individual model coefficients and the lack of fit test were performed using the same statistical package. The regression model obtained is given in equation 2.

$\begin{array}{l} Y(\%) = -35.1 + 1.27 X_1 + 2.17 X_2 + 25.24 X_3 - 0.00621 X_1 X_2 - 0.207 X_2 X_3 - 0.124 X_1 X_3 - 0.0041 X_{12} - 0.0197 X_{22} - 0.094 X_{32} \\ \dots \end{array}$

The P values were used as a tool to check the significance of each of the coefficients, which in turn are necessary to understand the pattern of the mutual interactions between the test variables [22]. The larger the magnitude of F-test value, the smaller the magnitude of P-values and the higher the significance of corresponding coefficient by [23]. When the value of P is less than 0.05, It shows that the model terms are significant. The fitness of the model equation was also expressed by the coefficient of determination, R_2 . In this case, X_1 , X_2 , X_3 , X_{12} and X_{22} are the significant model terms. The final mathematical model by eliminating the insignificant terms and interactions is expressed as.

 $Y(\%) = -35.08 + 1.27X_1 + 2.17X_2 + 25.2X_3 - 0.00411X_{12} + 0.0197X_{22} + 0.019X_{22} + 0.0$

2.6 Statistical Analysis

A quadratic polynomial equation by Box Behnken Design was developed to predict the response as a function of independent variables and their interaction. In general, the response for the quadratic polynomials is described by equation 4:

$$Y = \beta_0 + \sum (\beta_i X_i) + \sum (\beta_i X_i)^2 + \sum (\beta_{ij} X_{ij}).....4$$

Where Y is the response (yield); β_o is the intercept coefficient, β_i is the linear terms, β_{ii} is the squared terms and β_{ij} is the interaction terms, and Xi and Xj are the un-coded independent variables [24]. Analysis of variance

| Exp. Run | Std. | FACTORA | FACTOR | FACTOR C | RESPONSE Y |
|----------|-------|------------------------|------------------------------------|------------------------|-------------------|
| | value | (X ₁) mins | B (X ₂) ⁰ C | (X ₃) grms | |
| | 19 | 50.00 | 50.00 | 4.00 | 35.30 |
| | 213 | 50.00 | 80.00 | 6.00 | 22.00 |
| | 316 | 50.00 | 80.00 | 6.00 | 52.00 |
| | 45 | 30.00 | 80.00 | 4.00 | 59.00 |
| | 517 | 50.00 | 80.00 | 6.00 | 59.30 |
| | 67 | 30.00 | 80.00 | 8.00 | 12.20 |
| | 710 | 50.00 | 100.00 | 4.00 | 70.00 |
| | 88 | 70.00 | 80.00 | 8.00 | 61.10 |
| | 914 | 50.00 | 80.00 | 6.00 | 8.00 |
| | 104 | 70.00 | 100.00 | 6.00 | 83.00 |
| 1 | 6 | 70.00 | 80.00 | 4.00 | 57.00 |
| 2 | 3 | 30.00 | 100.00 | 6.00 | 12.20 |

(ANOVA) was applied to estimate the effects of main variables and their potential interaction effects on the oil

yield.

| 1311 | 50.00 | 60.00 8.00 | 8.00 |
|------|-------|-------------|-------|
| 1415 | 50.00 | 80.00 6.00 | 56.10 |
| 152 | 70.00 | 60.00 6.00 | 8.00 |
| 1612 | 50.00 | 100.00 8.00 | 12.20 |
| 171 | 30.00 | 60.00 6.00 | 55.60 |

Table 4. Experimental Results as obtained from the lab

2.7 Model Fitting and Statistical Analysis

Results obtained from the experiments (observed and predicted) are summarized as follows:

[□] The results were used to develop a second order polynomial equation (in coded units) that could relate oil yield to the parameters studied. The following quadratic equation model explained it.

^{\Box} From the experimental design and the experimental results obtained. The second order response functions representing Y is the response for palm oil yield, X₁ the coded value of variable temperature (T), X₂ the coded value of variable ratio methanol to oil (M), X₃ the coded value of variable weight of catalyst (W), and X₄ the coded value of variable reaction time (t). The closer the value of R₂ to unity, the better the empirical models fit the actual data. On the other hand, the smaller the value of R₂, the lesser will be the relevance of the dependent variables in the model in explaining the behavior of variations,[25]. Thus, the predicted values match the observed values reasonably well, with R₂ of 0.94.

III. RESULTS AND DISCUSSION

3.1 Characterization of Ngwo Clay

Table 1 shows the results of the characterization of both raw and activated Ngwo clay . the silicate content of both clays is good showing that it can be a good source of silica in the production of glass , as well as in floor tiles work. The increase from 61.39 tp 63 after activation may be as a result of non solubility of the compound. The other metal oxides were reduced in value, probably because of the clay's mineral dissolution in the acid.

3.2 Regression model

The three variables that affect the efficiency of bleaching of palm oil using activated Ngwo clay examined in the optimization study were; Temperature, ($^{\circ}$ C), Time, (Minutes), and Clay Dosage (%). Central Composite Rotatable Design of Box Benkhen was used in the work. The BBD was used to develop correlation between these operating variables and the efficiency of bleaching. The experimental and predicted values are presented with the complete design matrix in table 4. the quadratic terms were estimated by using the runs at the Center points. Selection of the model was based on the highest order, where the additional terms were significant, and the model was not adjudged insignificant lack of fir, and where the Adjusted R-squared and Predicted R – squared are in agreement, (within 0.2 of each other). The linear model was assumed based on these conditions.

3.3 Design Matrix Using BBD For Palm Oil Bleaching

Table 5 shows the results of the analysis of variance.

| Source | Sum of square | df | Mean Square Value | F-Value | P-Value |
|--------------------|---------------|---------|-------------------|---------|---------|
| Model | 7662.04 | 6 | 1277.01 | 4.35 | 0.0203 |
| A-Time | 614.25 | 1 | 614.25 | 2.09 | 0.01785 |
| B-Temperature | 621.28 | 1 | 621.28 | 2.12 | 0.1762 |
| C-Absorbent Dosage | 2041.60 | 1 | 2041.60 | 6.96 | 0248 |
| AB3504.64 | 1 | 3504.64 | 11.95 | 0.0062 | - |
| AC-647.70 | 1 | 647.70 | 2.21 | 0.1681 | - |
| BC-232.56 | 1 | 232.56 | 0.79 | 0.3941 | - |
| Residual | 2933.07 | 10 | 293.31 | - | - |
| Lack of Fit | 810.72 | 6 | 135.12 | 0.25 | - |
| Pure Error | 212295.12 | 4 | 530.59 | - | 0.9330 |
| Cor Total | 10595.12 | 16 | - | - | - |
| | | | | | |

Table 5. The Analysis of Variance Table (ANOVA TABLE)

AFAL OTT

--- -

The relative effects of each of the variables to the total variability was interpreted using ANOVA, because total variance of the process is equal to the sum of the component variaces if the factors are acting separately, [26]. ANOVA has its advantage in its use to rank the factors effects on the total variation in order of magnitude. This has effect of showing the points of effort in the variability reduction, so as to maximally improve the process within a minimum time and effort, [27].

The Model F-value of 4.35 implies that the model is significant. There is only a 2.03% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 that indicate model terms are significant. In this case C, and AB are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. The "Lack of Fit F-value" of 0.25 implies the Lack of Fit is not significant relative to the pure error. There is a 93.30% chance that a "Lack of Fit F-value" this large could occur due to noise.

The "Pred R-Squared" of 0.3807 is in reasonable agreement with the "Adj R-Squared" of 0.5571. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 7.702 indicates an adequate signal. This model can be used to navigate the design space. This is shown in table 6

The post ANOVA information are shoen in Tanle 7

- ...

a.

- -

 Table 7 The post ANOVA information

~ ~

| Coeff. Facto | or Estimate | Stand | ard Difference | 95% C.I Err | or95% C.I L | 4 0 W | High | L |
|--------------------|-----------------|-------|----------------|---------------------|-------------|--------------|----------|----------------------|
| Intercept | 39.4 | 1 | | 4.15 | 30.22 | | 48.73 | |
| A-Time | 8.76 | 1 | | 6.06 | -4.73 | | 22.25 | |
| BTemperature | 8.81 | 1 | | 6.06 | -4.68 | | 22.30 | |
| C-ClayDosage | -15.98 | 1 | | 6.06 | -29.47 | | -2.48 | |
| AB29Bleaching.60 E | fficiency1 % | 68.56 | Actual | 10.52 | 48.68 | | 1 | |
| 330.17684 | | | Optimal p | oint | | | | |
| AC12.73 | 1 | 8.56 | | -6.35 | 31.80 | | 1 | |
| -7.39062 | | | Time | | | | | |
| BC7.63 | 1 | 8.56 | | -26.70 | 11.45 | - | 1 | |
| -2.11562 | | | Temperatu | | | Final | - | tion in |
| -8.64375 | | | Clay Dosa | e | | Terms | of | Coded |
| 0.074000 | | | Time - Ter | nperature | | Factors: | | |
| 0.31813 | | | Time -Ads | orbent Dosage | | | | |
| -0.19063 | | | Temperati | ıre -Adsorbent Dosa | ige | give the fi | | s 8 and 9 llts in |
| | | | | | | terms | of code | ed and |
| Bleachin | ng Efficiency % | 0 | Cod | ed | | actua | 1 | factors |
| 39.47 | | | optiı | num | | respe | ctively. | |
| 8.76 | | | *A | | | | 1 | |
| 8.81 | | | *B | | | Tabl | e 8 | Results |
| -15.98 | | | *C | | | in ter | ms of c | oded |

| | |] |
|-------|------|---------|
| 29.60 | *A*B | factors |
| 12.73 | *A*C | |
| -7.63 | *B*C | |

Table 9, Results in Terms of Actual Factors:

Table 10 shows the results of predicted, actual and residual values.

Table 10 Predicted Values, Actual Values And Residuals

| tandard order | Actual value | Predicted value | Residual |
|---------------|--------------|-----------------|----------|
| 1 | 55.56 | 51.50 | 4.10 |
| 2 | 8.00 | 9.82 | -1.82 |
| 3 | 12.20 | 9.92 | 2.28 |
| 4 | 83.00 | 86.65 | -3.65 |
| 5 | 59.00 | 59.41 | -0.41 |
| 6 | 57.00 | 51.48 | 5.52 |
| 7 | 12.20 | 2.01 | 10.19 |
| 8 | 61.10 | 44.98 | 16.12 |
| 9 | 35.30 | 39.01 | -3.71 |
| 10 | 70.00 | 71.88 | -1.88 |
| 11 | 8.00 | 22.31 | -14.31 |
| 12 | 12.20 | 24.68 | -12.28 |
| 13 | 22.00 | 39.37 | -17.47 |
| 14 | 8.00 | 39.37 | -31.47 |
| 15 | 56.10 | 39.37 | 16.63 |
| 16 | 52.00 | 39.37 | 12.53 |
| 17 | 59.30 | 39.37 | 19.83 |

3.4 Model Graphs

Normal Plot of Residuals

The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line. Expect some moderate scatter even with normal data.

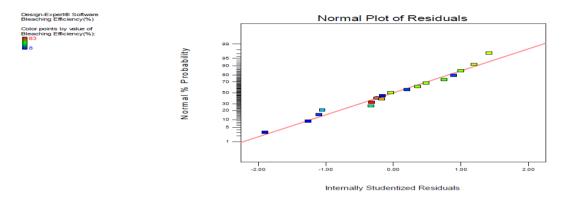


Fig. 1 Normal Plot of Residuals

Residuals vs Predicted Plot

This is a plot of the residuals versus the ascending predicted response values. It tests the assumption of constant variance. The plot should be a random scatter (constant range of residuals across the graph.) Expanding variance ("megaphone pattern <") in this plot indicates the need for a transformation

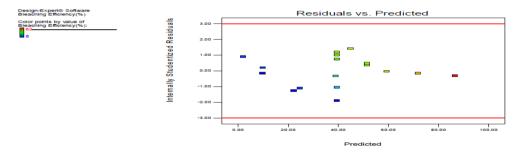


Fig 2 Residuals vs Predicted Plot

Residuals vs Run

This is a plot of the residuals versus the experimental run order. It allows you to check for lurking variables that may have influenced the response during the experiment. The plot should show a random scatter. Trends indicate a time-related variable lurking in the background. Blocking and randomization provide insurance against trends ruining the analysis.

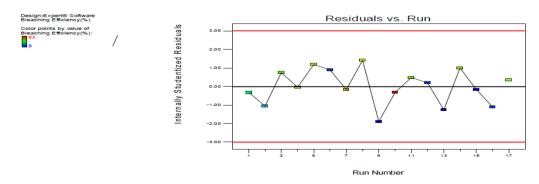


Fig. 3 Residuals vs Run

A graph of the actual response values versus the predicted response values. It helps detect a value, or group of values, that are not easily predicted by the model. The data points should be split evenly by the 45 degree line. If they are not, a transformation comes in (check the Box-Cox plot) to improve the fit.

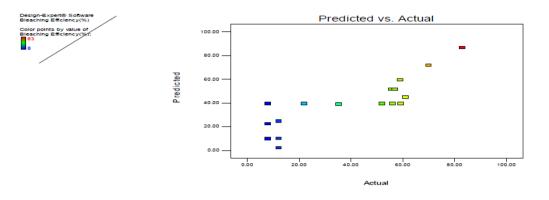
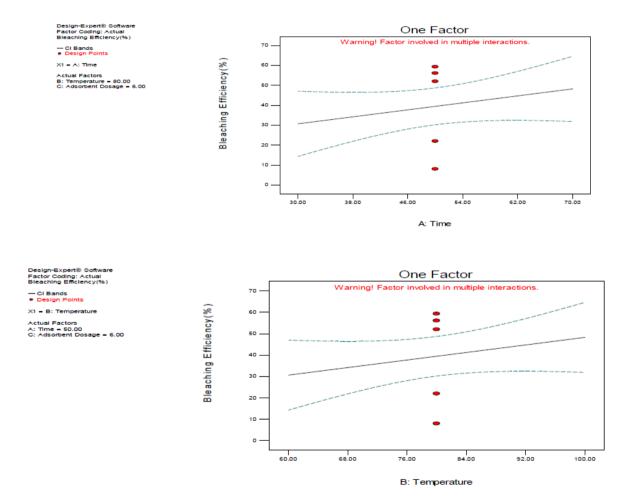


Fig 4 Predicted vs Actual

3.4 Effects of bleaching factors on the bleaching efficiency, (One factor plot)



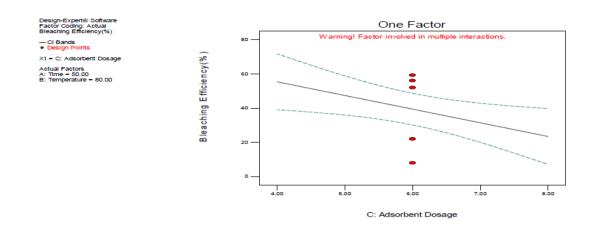


Fig 5 Interactive effect of (a)Time, (b)Temperature, and (c) Clay dosage. (One factor plot) 3.5 Model Graphs: Effects of bleaching factors on the bleaching efficiency, (Two factors plot)

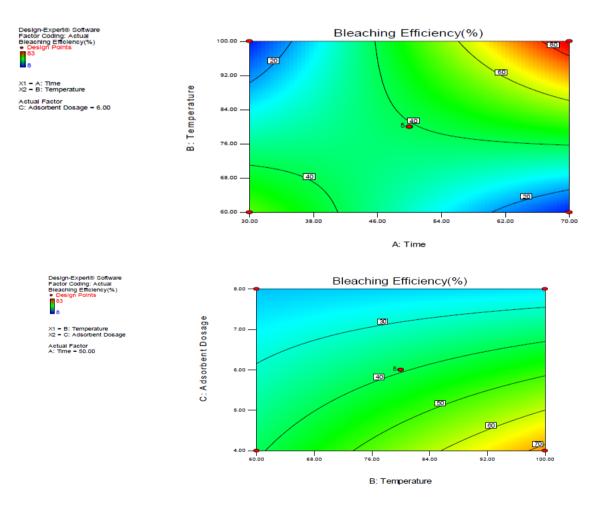


Fig. 6 Interactive effect of (a)Time, (b)Temperature, and (c) Clay dosage. (Two factor plot)

ding: 83 Bleaching Efficiency(%) X1 = A: Time X2 = B: Temp 100 Actual Factor 80 - 6.00 60 40 20 -20 100.00 70.00 92.00 62 00 54.00 B: Temperature 68.00 38.00 A: Time 60.00 30.00 ng: Bleaching Efficiency(%) 120 X1 = A: Time X2 = C: Adsort 10 Actual Factor B: Temperature = 80.00 80 60 40 20 -20 8.0 70.00 52.00 7.00 54.00 46.00 38.00 C: Adsorbent Dosage A: Time 4.00 30.00 Bleaching Efficien cy (%) 120 X1 = B: Tem X2 = C: Ads 100 Actual Factor A: Time = 50.00 60 20 02.00 84 00 C: Adsorbent Dosage 5.00 в: Temperature 4 00 60.00

3.6 Contour plot of interaction effects of the factors



3.7 Effect of Adsorbent Dosage on Bleaching Efficiency:

The clay dosage was varied from 4.0grms to 8.0grams. It was observed that increasing the clay dosage increased the bleaching efficiency. The results clearly indicate that the bleaching efficiency increases to an optimum value at adsorbent dosage of 4.02g, above which further increase in adsorbent dosage has no significant effect on it, but the value remains constant. This could be explained by the fact that adsorption equilibrium has been reached between the adsorbent/oil mixtures, thereby, preventing further pigment removal by the excess adsorbent dosage. Hence, it can be rightly concluded that absorbent dosage of clay is directly proportional to the bleaching efficiency.

3.8 Effect of Temperature on Bleaching Efficiency:

Fig. 5 shows that the bleaching efficiency is favoured by an increase in process temperature. From the figure it is obvious that bleaching does not proceed to any appreciable degree at low temperatures. But as the temperature is increased from 60° C to 100° C, the bleaching efficiency increased showing that temperature promotes access to further adsorption sites in the adsorbent. The bleaching efficiency increased with contact time at high temperature until it reaches a maximum optimum value of 99.84° C, then it starts to decrease with increasing contact time. These indicate that a further increase in the reaction temperature will not have any or much significant effect on the bleaching efficiency. Hence, temperature varies directly as bleaching efficiency.

3.9 Effect of Time on Bleaching Efficiency:

According to the chart shown above, it is obseved that bleaching does not proceed to any appreciable degree at low contact time or reaction time. But as the time is increased from 30mins to 70mins, the bleaching efficiency increased showing that reaction time favours positively and promotes access to further adsorption sites and decolourization in the adsorbent clay. The optimum time as observed in the study is 39.27min. The bleaching efficiency increased with contact time at high temperature until it reaches a maximum, and starts to decrease with increasing time duration. Consequently, time factor in beaching of palm oil improves bleaching efficiency.

3.10 Optimization

Numerical optimization was applied to search for the design space, and the created model was used to find the factors settings that met the objective of optimal bleaching efficiency. The optimal parameters were selected based on the highest desirability, with 40 minutes, 100°C, and 4% for Time, Temperature and Clay dosage respectively. This gave an efficiency of 83.39% which has a good correlation with the actual value of 82..39%. Validation was done by repeating the bleaching experiments at the predicted optimum conditions as shown in table 7. The results obtained from the optimization of the bleached palm oil parameters showed that the bleaching efficiency is linearly affected by process temperature and the contact time, and quadratic dependent on adsorbent clay dosage, as these can be interpreted from graph and chart shown above. As process temperature increases, duration of time and clay dosage invariably increase the bleaching or degree of decolourization.

IV. CONCLUSION

The optimal process parameters on the efficiency of activated Ngwo Clay on the bleaching performance of palm Oil has been investigated. Response Surface Methodology was used to study the effect of key parameters of Temperature, Time and Clay Dosage on the bleaching of palm oil. Box Benkhen Design, (BBD), a Central Composite Rotatable Design, with 17 assay, was successfully applied in the experimental design in order to optimize the variables. The conditions foe the optimal bleaching efficiency were found to be; temperature, time and clay dosage of 99.84°C, 40min. and 4g respectively. This resulted to 80.39 % of palm oil bleached Graphical response surface and contour plots were used to locate the optimum points this study clearly shows BBD as a good technique for studying the effect of major parameters in the bleaching of palm oil. it has also shown that Ngwo clay is a veritable source of adsorbent for bleaching vegetable oils. The good correlation between the predicted and experimental values shows that the method adopted is good enough for the process and that all the process variables of temperature, contact time, and clay dosage were significant.

REFERENCES

- [1] S. Parwez, (2006), The Rearson Guide to the B.Sc (Nursing) Entrance Examination. Pearson Education India pp. 109.
- [2] E. Bernardini, (2011), Vegetable oils and fats Processing, Vol.2, interstampa Rome Inc.
- [3] D.D. Brooks, (1999), Bleaching Factors that affect the oil loss. Proceedings of the 1999 PROIM. International palm oil congress, (chemistry and technology. 45-51 S.O Egbuna., A.J Ujam and M.E., Ejikeme, (2013), Comparative Analysis of Diffusion Rate in Palm Oil Extraction Using Different Extraction Solvents, ARPN Journal of Science and Technology; Vol.3 No 11, 1076 - 0 1089.
- [5] G Borner and M Schneider. (1999), practical Experiences with countercurrent bleaching process. Proceedings of 1999 PORIM international Palm oil congress, (chemistry and technology(52-59
- [6] Vov Sonntag, (1979), compsotion and characterization of Individual fits and Oils pp. 289-477
- T.L Mahatta (1975), Technology and refining of oils infacts, small business publications (SBP) building and/45 ROOP Nager, New Delhi
- [8] A.D., Rich (1964), Basic Factors in Bleaching of fats and oils, J.Am. chemical society, 42, 315
- [9] M.K. Gupta (2008), practical guide to vegetable oil processing, (AOCS Press, Urbana, IL).
- [10] S.O Egbuna. and N.A.G Aneke., (2005), Evaluation of Quality stability in a physically refined palm oil, proceedings of the 25th AGM of the NSCHE, Kaduna.
- [11] P.G. Nutting,, (1975), Adsorbent clay, USA ecological surey Bulletin 928 pp. 127
- [12] S.O Egbuna., E Ugadu. and A.J. Ujam (2014), Effect of thermal activation on the physio-chemical properties of white clay a natural local adsorbent. International journal of engineering invention, pp.
- [13] F.K. Hymore (1996), Effects of some additives on the performance of a-acid activated clay in the bleaching of palm oil. Applied clay science (10); 379-385.

- [14] M Wong., (1983), quality control in oil refineries. Proceedings of workshop of quality in the palm oil industry 240-245
- P.D. Howes, S.H Lim, D.B Shaw,. T.C., Soon, P.K Stemp. (1991), Bleaching Earths, Trends and Developments in Bleaching 1991 [15] PORIM. International Palm Oil Conference(Chemistry and technology) 55-75.
- S.O. Egbuna Development of Kinetic model for adsorption of carobnoids, on activated clay in the Bleaching of palm oil. [16] International Journal of Engineering and technology Pp 371-380, 2014.
- [17] E.M Ejikeme, S.O., Egbuna and P.C.N Ejikeme optimal Performance of Acid Activated Ngwulangwu clay, International journal of Engineering and innovative technology, pp 13-19, 2013.
- O. O., James, M.A., Mesubi, F.A., Adekola, E.O., Odebunmi, J.I.D., Adekeye, and R.B. Bale ""Bleaching performance of a Nigerian (Yola) Bentonite. Latin American Applied Research". 34, 45 49, 2008. [18]
- P.C.N,Ejikeme E. Ejikeme, and D.O Onwu. "Optimization of process conditions for the concentration of isopropyl alcohol-water [19] solution using response surface methodology". International Journal of Science & Eng. Research, vol. 4, no. 2.". J. Colloid-Inter. Sci., 292, 381–391, 2013
- [20] M.A., M., Vicente - Rodriquez, M Suarez, J., de Dios Lopes-Gonzalez, and M. A Banares - Munoz, "Characterization, surface area and porosity analyses of the solids obtained by acid leaching of a Saponite. Langmuir", 12, 566 -572 1996
- B. J. Nde-Aga, R Kamga, and J. P. Nguetnkam, "Adsorption of palm oil carotene and free fatty acids onto acid activated [21] Cameroonian clays". Journal of Applied Sciences. 7, 2462 - 2467, 2007.
- [22] A., Shrivastava, P., Saudagar., I., Bajaj, and R. Singhal, "Media optimization for the production of U-linolenic acid by cunninghamella echinulata varielegans MTCC 552 using response surface methodology". International Journal of FoodEngineering, 4(2), 1-2. Silva, J. 2008.
- Z.Alam, S.A. Muyibi, , and N. Kamaldin, "Production of activated carbon from oil Palm empty fruit bunches for removal of [23]
- zinc. Proceedings of Twelfth International Water Technology Conference", IWTC12 2008Alexandria, Egypt, 373-382, 2008. S.V Ghadge, And H. Raheman. "Process optimization for biodiesel production from mahua, (Madhuca indica) oil using [24] response surface methodology". Bioresource Technology 97: 379-384, 2006.
- W Cao, C .Zhang, P. Hong, and H. Ji,.. "Response surface methodology for autolysis parameters optimization of shrimp head [25] and amino acids released during autolysis". Food Chemistry 109: 176-183, 2008.
- G.I.. Onwuka, Food Analysis and Instrumentation Theory and practice. Naphthali Prints Lagos, 2005. [26]
- [27] A. G. Wilbura,., Food Quality Assurance. The AVI Publishing Company Inc., West Port Connecticut, 1983.