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Research Article

OPTIMIZATION OF COLOUR PIGMENTS REMOVAL FROM PALM OIL USING ACTIVATED OGBUNIKE KAOLINITE

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ABSTRACT

Response surface methodology (RSM) and genetic algorithm (GA) were employed to determine the optimum conditions for the removal of pigments from palm oil using Ogbunike clay activated with hydrochloric acid. The physicochemical characterization of the clay showed that it exists mainly as kaolinite. The process variables and ranges used in the experimental design were 75 – 150 ^oC bleaching temperature, 1.50 – 3.00 hours bleaching time, 1.25 -5.50 g clay dosage and 0.05 – 0.40 mm particle size. The analysis of variance (ANOVA) showed that a second order polynomial regression equation was adequate for fitting the experimental data. The model statistical tests carried out showed a good correlation between the experimental and predicted values (R^2 = 0.9964). About 73.35 % pigments were removed using RSM while 71.34% pigments were removed using genetic algorithm at the optimum conditions. Hence, Ogbunike kaolinite proved to be a good adsorbent for pigments removal from palm oil.
Keywords: Optimization, ANOVA, bleaching, genetic algorithm, kaolinite.

1. INTRODUCTION

Palm oil is an important source of dietary for people in the western part of Africa and this oil has some storage and use difficulties. Palm oil thickens on storage at ambient temperature and has a very low smoke point, which makes it unsuitable for frying. These difficulties can be reduced if the oil is bleached to remove the impurities. Impurities present in palm oil can be greatly reduced by adsorption process or bleaching by using clay mineral adsorbents [1].

Kaolinite is a 1:1 dioctahedral clay mineral that has two layered structure comprising of silicon-oxygen tetrahedral sheet linked to aluminium octahedral sheet through –Al-O-H-O-Si hydrogen bonding. One surface of the layer comprises basal oxygen atoms belonging to the tetrahedral sheet, while the other surface is made up of OH groups from the octahedral sheet. It

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has triclinic symmetry with stacking sequence of identical layers with a translation of $-\alpha/3$. The thickness of each single layer of tetrahedral and octahedral sheets in kaolinite is about 7Å. The Si-O bond lengths for the apical tetrahedral oxygen atoms are shorter whereas that of Al-O bonds are longer than correspon ding average bond lengths [2].

The application of statistical experimental design techniques in adsorption process development can result in improved product yields, closer confirmation of the output response to nominal and target requirement, reduced process variability, reduced development time and overall cost [3]. Response surface methodology (RSM) comprises sophisticated statistical and mathematical techniques, which are used for process improvement and optimization [4]. The main objective of response surface methodology is to determine the optimum operational conditions for a process [5].

Genetic algorithms consist of evolutionary algorithms that use techniques inspired by evolutionary biology such as inheritance, mutation, selection, and crossover (also called recombination). The evolution usually starts from a population of randomly generated individuals and happens in generations. In each generation, the fitness of every individual in the population is evaluated, multiple individuals are selected from the current population (based on their fitness), and modified to form a new population [6]. Genetic algorithms have gained popularity over traditional optimization techniques because they can solve discontinuous or non-differentiable fitness functions efficiently [7-9].

Although some work have been done on the removal of pigments from palm oil using activated clay, there is no study related with the application of genetic algorithm in modeling the process. In the present study, a 5-level-4-factor central composite design (CCD) of RSM and genetic algorithm were developed and their performances were evaluated to optimize the removal of pigments from palm oil using activated Ogbunike kaolinite. The independent practical variables, such as bleaching time, bleaching temperature, clay dosage and particle size, were investigated to determine their effects on the bleaching efficiency.

2. MATERIALS AND METHODS

2.1. Materials

The kaolinite sample used in this research was sourced from Ogbunike (N: 6°10'0"; E: 6°54'0"; A:161 m) in Anambra State of Nigeria. The crude palm oil (CPO) was bought from local oil mill at Ezema village, Ojoto in Idemili South local government area of Anambra State, Nigeria.

2.1.1. Characterization of Clay Sample

X-ray fluorescence analysis of the clay sample was carried out using ARL 9400XP+ Wavelength-dispersive XRF Spectrometer while FTIR analysis was performed using Shimadzu FTIR-8400S spectrophotometer.

2.1.2. Acid Activation of Clay Sample

The clay material was prepared for activation by drying it under the sun at an ambient temperature of 35^oC to prepare it for grinding. The clay sample was then pulverized and sieved to a particle of 300μm. 50g of the clay sample was mixed with 250ml of the prepared acid. The resulting suspension was heated on a magnetically stirred hot plate at a temperature of 98^oC for 2.0 hours. The clay residue was washed free of the acid several times with distilled water until a neutral point was obtained with pH meter. The clay was then dried at a temperature of 110^oC for 3 hours, then ground again and sieved with 75μm sieve and stored in desiccators.

2.2. Bleaching Pro cess

100 g of the refined unbleached palm oil was measured out into a 250 ml conical flask and heated on a magnetically-stirred hot plate to an already determined temperature from the experimental design. The required mass of the activated clay sample was then added to the heated oil and stirred continuously via a magnetic stirrer carefully inserted into the beaker up to the bleaching time in the experimental design. At the completion of the time, the hot oil and clay mixture was filtered under gravity using Whatman filter paper No.42 (15 cm diameter), before measuring the absorbance. The bleaching/adsorption 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 450nm. The bleaching efficiency is defined by the expression in Equation (1) [10].

$$Bleaching\ Efficiency\ (\%) = \frac{A_{unbleached} - A_{bleached}}{A_{unbleached}} \times 100 \qquad (1)$$

where $A_{\mathrm{unbleached}}$ and A_{bleached} are absorbencies of unbleached and bleached palm oil respectively, at 450 nm.

2.3. Experimental Design for RSM Modeling

The process variables (Bleaching time, Bleaching temperature, Clay dosage and Particle size) which affect the bleaching efficiency through the bleaching process were examined using RSM. To avoid unnecessary repetition of experiments, a central composite design (CCD) was applied to produce 30 experimental conditions used to investigate the effects of the four chosen factors on the bleaching efficiency. The ranges of the variables investigated are displayed in Table 1, which include 75-150^{o}C bleaching temperature, 1.5-3.0 hours bleaching time, 1.25-5.50 g clay dosage and 0.05-0.40 mm particle size. The design matrix is shown in Table 2. The four input variables were appraised at low (-1), medium (0) and high (+1) levels and, axial points were added for orthogonality of the design. Six center points were used to estimate the lack of fit and pure error of the proposed model. Multiple regressions were applied to fit the coefficient of the quadratic polynomial regression model of the response. The quality of the fitted quadratic polynomial response model was assessed by analysis of variance (ANOVA) and test of significance. In RSM, the most widely used second-order polynomial equation developed to fit the experimental data and identify the relevant model terms is shown in Equation 2.

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{\substack{i=1 \\ j>1}}^{n-1} \sum_{j=2}^{n} \beta_{ij} x_i x_j + \sum_{i=1}^{n} \beta_{ii} x_i^2 + \varepsilon \qquad (2)$$

where Y is the predicted response variable which is the bleaching efficiency in this study, β_0 is the constant coefficient, β_i is the ith linear coefficient of the input variable x_i, β_{ii} is the ith quadratic coefficient of the input variable x_i, β_{ij} is the different interaction coefficients between the input variables x_i and x_j and ε is the error of the model.

Table 1. Experimental range of the independent variables, with different levels, to study the adsorption/bleaching properties of Ogbunike kaolinte after activation with hydrochloric acid

Independent variables	Symbol	Range and Levels				
		-a	-1	0	+1	+a
Bleaching Time (hrs)	A	-0.50	1.50	2.50	3.00	3.50
Bleaching Temp. (^oC)	B	40	75	110	150	200
Clay Dosage (g)	C	-1.50	1.25	2.95	5.50	8.50
Particle Size (mm)	D	-0.05	0.05	0.25	0.40	0.55

Table 2. Experimental design/plan for adsorption/bleaching studies of Ogbunike kaolinite in hydrochloric acid.

Run	Bleaching Time (hrs) : A		Bleach Temp. (^oC) : B		Clay Dosage (g) : C		Particle Size (mm) : D	
	Coded	Real	Coded	Real	Coded	Real	Coded	Real
1	-1	1.50	-1	75	-1	1.25	-1	0.05
2	-1	1.50	+1	1.50	-1	1.25	-1	0.05
3	+1	3.00	-1	75	-1	1.25	-1	0.05
4	+1	3.00	+1	150	-1	1.25	-1	0.05
5	-1	1.50	-1	75	+1	5.50	+1	0.40
6	-1	1.50	+1	150	+1	5.50	+1	0.40
7	+1	3.00	-1	75	+1	5.50	+1	0.40
8	+1	3.00	+1	150	+1	5.50	+1	0.40
9	-1	1.50	-1	75	-1	1.25	+1	0.40
10	-1	1.50	+1	150	-1	1.25	+1	0.40
11	+1	3.00	-1	75	-1	1.25	+1	0.40
12	+1	3.00	+1	150	-1	1.25	+1	0.40
13	-1	1.50	-1	75	+1	5.50	+1	0.40
14	-1	1.50	+1	150	+1	5.50	+1	0.40
15	+1	3.00	-1	75	+1	5.50	+1	0.40
16	+1	3.00	+1	150	+1	5.50	+1	0.40
17	0	2.50	-2	40	0	2.95	0	0.25
18	0	2,50	+2	200	0	2.95	0	0.25
19	-2	-0.50	0	110	0	2.95	0	0.25
20	+2	3.50	0	110	0	2.95	0	0.25
21	0	2.50	0	110	-2	-1.50	0	0.25
22	0	2.50	0	110	+2	8.50	0	0.25
23	0	2.50	0	110	0	2.95	-2	-1.50
24	0	2.50	0	110	0	2.95	+2	5.50
25	0	2.50	0	110	0	2.95	0	0.25
26	0	2.50	0	110	0	2.95	0	0.25
27	0	2.50	0	110	0	2.95	0	0.25
28	0	2.50	0	110	0	2.95	0	0.25
29	0	2.50	0	110	0	2.95	0	0.25
30	0	2.50	0	110	0	2.95	0	0.25

2.4. Optimization Using Genetic Algorithm (GA)

Genetic algorithm (GA) is a method for moving from one population of chromosomes (e.g., strings of one and zeros) to a new population by using a kind of natural selection jointly with the genetics-inspired operators of crossover, mutation and, and inversion. GA is uniquely different from other search methods in that it uses the probabilistic transition rules in place of deterministic rules and search inside a population [11]. The GA frequently changes the group for the individual solutions of the problem, and these changes are known as evolution. In each step of this evolution, two members of the group are selected randomly as the parent and child, and they are considered for the next generation. In this way the group evolves toward an optimal solution. In the present study, R2007b matlab software (The mathworks, Inc., Ver. 7.5.0.342, MA, USA) was used. The objective function for optimizing pigments removal obtained from RSM was used for the optimization study. By the trial-and-error method, an appropriate option for the initial range, fitness scaling, selection, elite count, crossover fraction, Mutation function, crossover function, migration, algorithm, stopping criteria, and output function were selected, and the optimized solution was obtained.

3. RESULTS AND DISCUSSION

3.1. XRF Analysis

The result of XRF analysis of the clay shows that alumina (Al_2O_3), hematite (Fe_2O_3) and quartz (SiO_2) are present in major quantities while other components are present in trace amounts. The XRF result is shown in Figure 1.

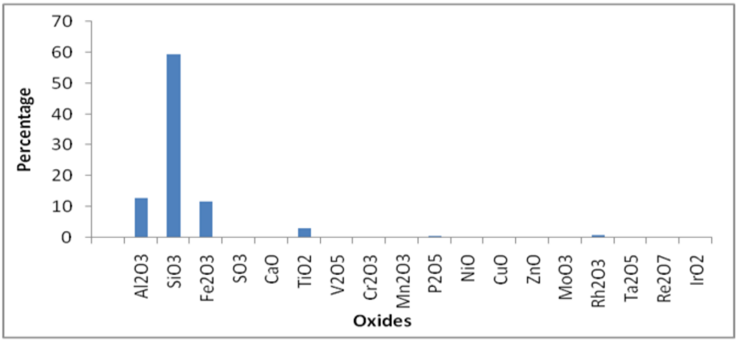


Figure 1. XRF result of Ogbunike kaolinite

3.2. FTIR Analysis

The FTIR spectra of Ogbunike kaolinite is shown in Figure 2. The result shows the functional groups present in the clay. The band at 524 cm^{-1} is attributed to C-C=O bend, C-Br and C-I stretches. The band at 792.77 cm^{-1} is attributed to C-Cl stretch and CH out-of-phase deformation while the band at 1020.38 cm^{-1} is attributed to Si-O-Si and P-O-C anti-symmetrical stretches. The band at 1107.18 cm^{-1} is attributed to C-N, C-O and C=S stretches as well as C-O-C anti-symmetrical stretch while the band at 1635cm^{-1} is attributed to C=O and Al-O-H stretches as well as NH, NH_2 and NH_3 deformation. The band at 2376.31 cm^{-1} is attributed to P-H stretch and NH stretching modes while the band at 3399.65 cm^{-1} is attributed to OH stretch for solids and liquids as well as NH stretch for dilute solution.

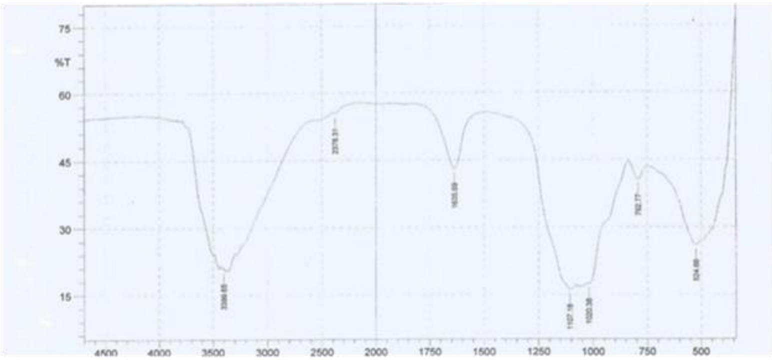


Figure 2. FTIR spectra of Ogbunike kaolinite

3.3. Statistical Analysis

The bleaching efficiency (BE) of acid activated Ogbunike kaolinite was optimized statistically using the response surface methodology (RSM).The data generated from the central composite design was analyzed using the Design Expert 10.0 Trial Version. Mathematical relationship between the dependent variables and the independent variable was evaluated using the software. The second order polynomial regression equation that fitted the data is shown in Equation 3.

$$BE\ (\%) = 39.99 + 6.90*A + 6.40*B + 0.040*C + 6.77*D + 2.26*AB - 2.96*AC + 0.44*AD -1.37*BC+1.32*BD+0.31*CD-1.57*A^2+6.44*B^2-0.23*C^2+3.18*D^2 \quad (3)$$

The adequacy of the predicted model was tested using the sequential model sum of squares and the model summary statistics as shown in Table 3. The model summary statistics shows that the quadratic model is adequate with higher values of R^2 (0.9964), predicted R^2 (0.9884) and adjusted R^2 (0.9931).

Table 3. Model Summary Statistics

Source	Std.Deviation.	R^2	Adjusted R^2	Predicted R^2	PRESS	Remarks
Linear	5.98	0.7503	0.7103	0.6424	1280.78	
2FI	5.66	0.8298	0.7402	0.6470	1264.31	
Quadratic	0.93	0.9964	0.9931	0.9884	41.63	Suggested
Cubic	1.23	0.9971	0.9878	0.8139	666.40	Aliased

Statistical analysis of variance (ANOVA) was performed to determine whether the process variables are statistically significant. The ANOVA results are shown in Table 4. The F-value for each variable, that is, a ratio of the squared deviations to the mean of the squared error, indicates which variable has a significant effect on the bleaching efficiency. The Model F-value of 297.47 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Values of "Prob> F" less than 0.050 indicate the model terms are significant. The "Lack of Fit F-value" of 0.74 implies the Lack of Fit is not significant relative to the pure error. There is a 67.81% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good as it shows the model is well fitted. From the ANOVA results, the bleaching time (A), temperature (B), particle size (D), interactions between the bleaching time and temperature (AB), bleaching time and clay dosage (AC), temperature and dosage (BC), temperature and particle size (BD) and the quadratic terms of bleaching time (A^2), bleaching temperature (B^2) and particle size (D^2) are all significant. The F-values for these variables are greater than the F-values of other variables from Table 5 at 95% confidence interval. This means that, the variance of all these variables is significant compared with the variance of error and all of them have meaningful effect on the bleaching efficiency. The final second order polynomial predictive equation, after the elimination of insignificant terms is given in Equation 4.

Table 4. ANOVA for the quadratic model

Source	Sum of Squares	Df	Mean Square	F-value	p-value Prob> F	
Model	3568.89	14	254.92	297.47	< 0.0001	Significant
A-Bleaching Time	950.93	1	950.93	1109.67	< 0.0001	
B-Bleaching Temp.	819.62	1	819.62	956.43	< 0.0001	
C-Dosage	0.031	1	0.031	0.037	0.8510	
D-Particle Size	916.75	1	916.75	1069.78	< 0.0001	
AB	81.95	1	81.95	95.63	< 0.0001	
AC	140.33	1	140.33	163.76	< 0.0001	
AD	3.15	1	3.15	3.67	0.0746	
BC	30.06	1	30.06	35.07	< 0.0001	
BD	27.74	1	27.74	32.38	< 0.0001	
CD	1.56	1	1.56	1.82	0.1970	
A^2	23.01	1	23.01	26.85	0.0001	
B^2	387.66	1	387.66	452.37	< 0.0001	
C^2	0.50	1	0.50	0.59	0.4553	
D^2	94.55	1	94.55	110.33	< 0.0001	
Residual	12.85	15	0.86			
Lack of Fit	7.69	10	0.77	0.74	0.6781	not significant
Pure Error	5.17	5	1.03			
Cor Total	3581.74	29				

$$BE\ (\%) = 39.99 + 6.90*A + 6.40*B + 6.77*D + 2.26*AB - 2.96*AC - 1.37*BC + 1.32*BD -1.57*A^2+6.44*B^2+3.18*D^2 \quad (4)$$

In terms of actual factor values, the bleaching efficiency is obtained as shown in Equation 5.

$$\begin{aligned} BE\ (\%) = & -71.692 + 0.134*\text{Bleaching Time} + 1.089*\text{Bleaching Temp.} + 42.291*\text{Particle Size} \\ & + 8.622E\text{-}004*\text{Bleaching Time}*\text{Bleaching Temp.} - 0.026*\text{Bleaching Time}*\text{Dosage} \\ & - 0.026*\text{Bleaching Temp.}*\text{Dosage} + 0.167*\text{Bleaching Temp.}*\text{Particle Size} \\ & - 2.792E\text{-}004*\text{Bleaching Time}^2 + 5.261E\text{-}003*\text{Bleaching Temp.}^2 + 62.86914* \\ & \text{Particle Size} \end{aligned} \quad (5)$$

The coefficient of variation (CV) value of 2.05 illustrate that the model can be considered reasonably reproducible [12]. The signal to noise ratio which is given as the value of the adequate precision is 74.533 as shown in Table V. It indicates that an adequate relationship of signal to noise ratio exists and that the result can be used to navigate the design space.

Table 5. Summary of regression values

Std. Dev.	Mean	C.V. %	PRESS	Adeq. Precision
0.93	45.21	2.05	41.63	74.533

The experimental data were also analyzed to check the correlation between the experimental and predicted bleaching efficiencies. It can be seen from Figure 3, that the data points on the plot were reasonably distributed near to the straight line, indicating a good relationship between the experimental and predicted values of the response. This shows that the underlying assumptions of the above analysis were appropriate. The result also suggests that the selected quadratic model was adequate in predicting the response variables for the experimental data [10].

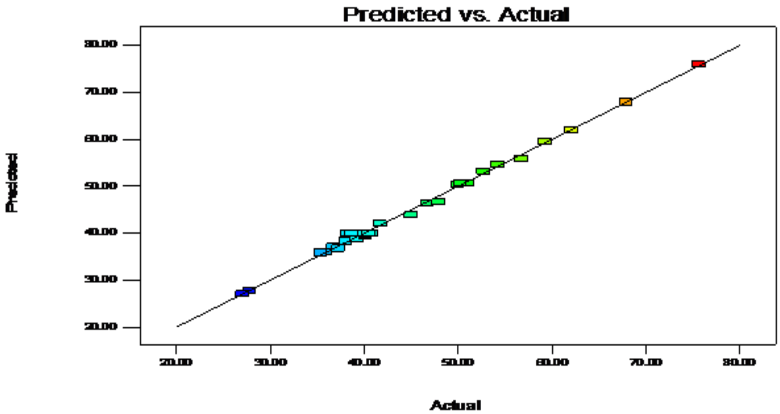


Figure 3. Plot of the predicted values versus the actual experimental values

3.4. Response Surface Plots

The three-dimensional response surface plots obtained as a function of two factors while maintaining all other factors constant at the mid-values are helpful in understanding both the main effects and interactive effects of the factors. The response surface plots are shown in Figures 4 (a-d). The interactive effect of bleaching time and bleaching temperature is shown in Figure 4a. As the bleaching temperature increased from 120 to 150 ^{o}C, the percentage pigments removed increased from 45 to 60 %, while as the bleaching time increased from 120 to 180 minutes; the percentage pigments removed increased from 54 to 60%. Figure 4b shows the interactive effect of bleaching time and clay dosage. The result shows that the percentage pigments removed increased from 42 to 48% as the bleaching time increased from 120 to 180 minutes, whereas the percentage pigment removed increased from 42 to 47% as the clay dosage decreased from 5 to 2g. The response surface plot of bleaching time and particle size is shown in Figure 4c. From the results shown in Figure 4c, the percentage pigments removed increased from 45 to 56 percent as the particle size increased from 0.38 to 0.60 mm; while as the bleaching time increased from 30 to 180 minutes, the percentage pigments removed increased from 40 to 56%. The interactive effect of bleaching temperature and clay dosage is shown in Figure 4d. The result shows that the percentage pigments removed increased from 50 to 53 % as the clay dosage decreased from 5 to 2g; while the percentage pigments removed increased from 43 to 53% as the bleaching temperature increased from 120 to 150 ^{o}C.

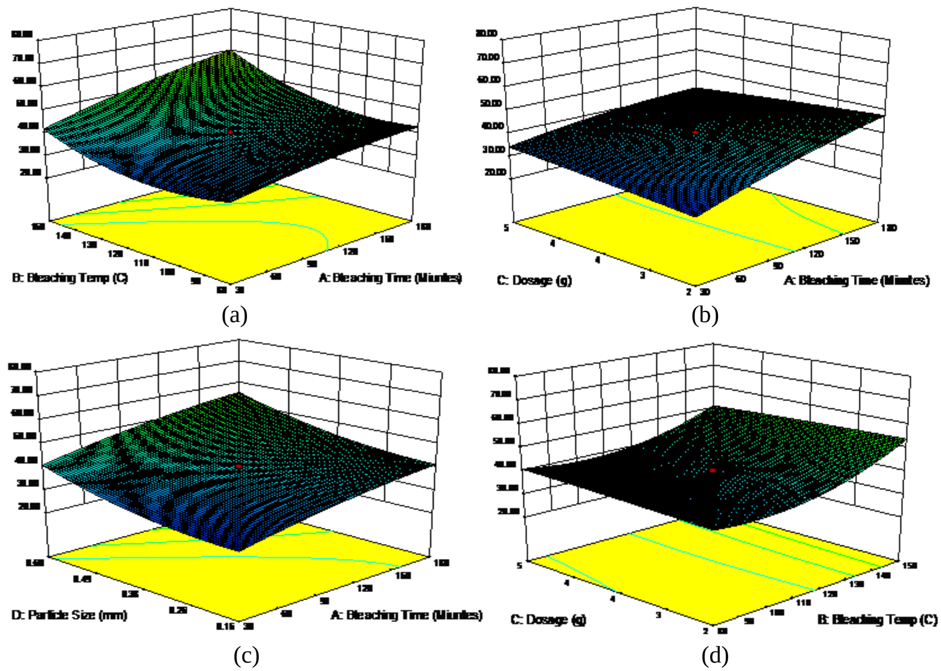
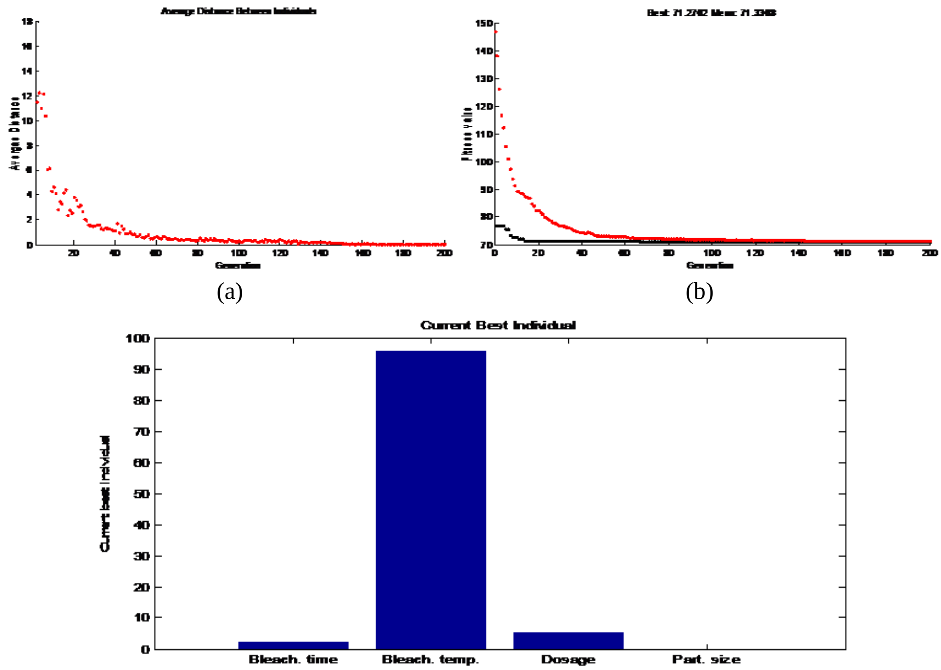


Figure 4. Response surface plots for the bleaching of palm oil using activated Ogbunike kaolinite

3.5. Numerical Optimization Using Response Surface Methodology and Genetic Algorithm

The main purpose of this study was to determine optimum conditions for the bleaching of palm oil using activated Ogbunike kaolinite. The optimization tools of central composite design (CCD) of design expert software and genetic algorithm of matlab were employed for the optimization exercise. The model developed from CCD showed that the optimum process conditions were bleaching time of 2.62 hours, bleaching temperature of 106.83^{o}C, clay dosage of 3.32 g and particle size of 0.17 mm. At the above conditions, an optimum bleaching efficiency of 73.35 % was predicted. The bleaching efficiency was validated in triplicate with the average experimental value of 72.16 %. The GA optimization parameters were determined by a trial-and-error method. The best conditions were defined in which the initial range was [1,100], the selection function was stochastic uniform, the values of the elite count and crossover fraction were, respectively, equal to 2 and 0.8, and the mutation and crossover functions were selected as adaptive feasible and two point, respectively. The stopping criteria were selected, with 200 generations and stall generations; output function and level of display were also selected.

Figure 5a shows the average distance between the individuals for each generation. It could be reported that in this case, the group has good variety. Figure 5b shows the mean value of the fitness function of generation 200, which is the value 71.34%. The optimal values for the independent variables viz. the bleaching time, bleaching temperature, clay dosage and particle size were equal to 2.61hrs, 96.20^{o}C, 5.49g and 0.05mm, respectively (Figure 5c). By carrying out three independent experimental replicates under these optimal conditions, the pigments removal predicted was validated with an average value of 71.05 %, which proved GA to be an effective optimization tool.



(a) (b) (c)

Figure 5. The obtained charts for the obtained points by GAs: (a) average distance between the individuals in each generation (b) the best value for the fitness function and (c) the optimal values obtained for the fitness function's independent variables

4. CONCLUSION

The optimum levels of process variables for the removal pigments from palm oil were investigated in this study using the central composite design of response surface methodology and genetic algorithm of matlab. The quadratic model developed showed that the bleaching efficiency was influenced by bleaching time, bleaching temperature, clay dosage and particle size. The optimum bleaching conditions were obtained as 2.62 hours bleaching time, 106.83 ^{o}C bleaching temperature, 3.32 g clay dosage and 0.17 mm particle size and under these conditions, about 73.35 % pigments were removed using RSM. The optimum conditions obtained using genetic algorithm include 2.61 hours bleaching time, 96.20 ^{o}C bleaching temperature, 5.49 g clay dosage and 0.05 mm particle size and at which 71.34% pigments were removed. The results obtained show that Ogbunike kaolinite is a good adsorbent for colour pigments removal from palm oil.

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