

Optimum Activation Conditions of Ughelli Bentonite for Palm Oil Bleaching Using Response Surface Methodology

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Abstract: Natural clays are acquiring prominence as low-cost adsorbent over the past few decades due to their local and abundant availability and capability to undergo modification. The use of these natural clays is hampered by the presence of net negative charge on the surface and small surface area and these factors have led to the need for research and development in the field of modification of clay surfaces to enhance their adsorptive properties. So this work is aimed at optimizing the acid activation conditions of local clay to improve their performance and yield. Bentonite from Ughelli was reacted with different concentrations of sulphuric acid solutions to activate it. The chemical compositions of the natural and acid activated samples were characterized using X-ray Fluorescence (XRF). The main and interaction effects of the process variables- acid concentration, temperature, reaction time, solid/liquid ratio and stirring speed were investigated using the central composite design of the response surface methodology. Characterization results show that acid activation removed the octahedral and tetrahedral cations which led to increase in the surface area of the samples and this increase was dependent on the concentration of the acid used. The natural and acid activated samples were used to bleach palm oil to investigate their adsorption performance. The adsorption experiments showed that the treated samples performed better than the untreated sample. The statistical analysis of the results showed that the linear and quadratic effects of the process variables were highly significant, as well as, the interaction effect of acid concentration and solid/liquid ratio. The optimum conditions for the maximum activation efficiency of 83.10 % was obtained at 6.15 mol/l acid concentration, 132.87°C temperature, 3.73 hours reaction time, 0.052 g/mol solid/liquid ratio, and 388.72 rpm stirring speed. Experiment performed at the predicted optimum conditions yielded bleaching efficiency of 82.96 % which was in good agreement with the predicted value. This study has revealed that Ughellibentonite can be improved by acid activation and serves as a good source of adsorbent for palm oil bleaching.

Key words: activation, bleaching, adsorption, central composite design, regression, ANOVA

INTRODUCTION

Natural clay minerals are well known and familiar to mankind from the earliest days of civilization (Preeti, *et al*, 2007). Because of their low cost, abundance in most continents of the world, high sorption properties, high dissolubility in acidic solutions and potential for ion exchange, clay materials are strong candidates as source of metals and adsorbents. Two structural units are involved in the atomic lattices of most clay minerals one unit consists of closely packed oxygen and hydroxyls in which aluminum, iron and magnesium atoms are embedded in an octahedral combination so that they are equidistant from six oxygen or hydroxyls. The second unit is built of silica tetrahedrons. The silica tetrahedrons are arranged to form a sheet of composition, $\text{Si}_4\text{O}_6(\text{OH})_4$ (Rozić, 2008). Clay deposits are widespread over the regions of Nigeria and are unutilized in the process industries largely because they are not researched upon. These clay deposits can be mined, purified and processed into useful raw materials for the process industries. The structure of these clays can be altered by heating or reaction with strong acids or alkalis to improve their adsorptive and decolourizing properties. The majority of these clays does not possess such properties, but, may be activated by some form of treatment and their bleaching power can be improved. Activation of clays can essentially be accomplished by calcination, reaction with mineral acids/alkalis, or combination of both techniques.

Acid treatment of clays changes the clay structure by creating new pores resulting in an increase of surface acidity through the replacement of cations like Al^{3+} , Fe^{3+} , and Ca^{2+} from the structure with H^+ . During clay activation with mineral acids two basic reactions occur: (1) the acid first dissolves part of Al_2O_3 as well as CaO , MgO ... etc., from the lattice. This causes an opening of the crystal lattice and an increase in internal surface area. (2) The second reaction is the gradual exchange of Al, Ca, Mg ions, located at the surface of the crystal against hydrogen ions from the mineral acid. These metallic ions then react with the negative ions from the mineral acid forming the corresponding salts.

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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 present study, the activation of Ughelli bentonite is studied to determine the optimum process conditions during the modification using response surface methodology. Also, the applicability of the activated samples in bleaching palm oil was investigated

Experimental Procedures:

Acid Activation of the Clay Sample:

The clay material was prepared for activation by air-drying and grinding to a particle size of 0.075 mm. 10 g of the prepared sample was weighed into flask (250ml capacity) and 100 ml of sulphuric acid solution was added. The resulting suspension was heated on a magnetically stirred hot plate at temperature of 90 °C for 2 hours 30 minutes. At the end of the experimental duration, the resulting slurry was poured into a Buchner funnel to separate the acid and clay. The residual clay was washed severally with distilled water until neutral point was obtained with pH indicator. The clay residue was dried in an oven at 80 °C for 4 hours. The dried samples were crushed and sieved again to 0.075 mm particle size. The activation process was repeated with varying acid concentrations of 0.5 – 8 mol/L of H₂SO₄, varying time of 0.5 – 6 hours, varying solid/liquid ratio of 0.01 – 0.1 g/mol, varying stirring speed of 90 – 540 rpm, and varying temperatures of 50 – 180 °C. The clay samples thus prepared were labeled UG0, UG0.5, UG2, UG4, UG6, and UG8, where the numbers indicate the acid concentrations used in the activation step. The chemical composition was determined using X-ray fluorescence (XRF), Philips PW 2400 XRF spectrometer.

Design of Experiment:

The process variables affecting the dissolution of Ughellibentonite in sulphuric acid were investigated using RSM combined with five-level, five-factor fractional factorial design as established by Statistica software (10 trial version), Statsoft, U. S. A. The process variables were reaction temperature of 50 – 180°C, acid concentration of 0.5 – 8mol/l, reaction time of 0.5 – 6.0 hrs, solid/liquid ratio of 0.01 – 0.10g/ml, and stirring speed of 90 – 540rpm. The response variable was chosen as % cations removed. The factor levels were coded as - α , -1, 0, +1 and + α . The range and levels are shown in Table 1.

A total of 30 runs were carried out to optimize the process variables and experiments were performed according to the actual experimental design matrix shown in Table 2. The experiments were performed randomly to avoid systemic error. The results were analyzed using the coefficient of determination, analysis of variance (ANOVA), and response plots. In RSM, the most widely used second-order polynomial equation developed to fit the experimental data and identify the relevant model terms may be written as:

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_{ii}^2 + \sum \beta_{ij} x_i x_j + \varepsilon \quad (1)$$

Where Y is the predicted response variable, in this study the % cation removed, β_0 is the constant coefficient, β_i is the *i*th linear coefficient of the input variable x_i , β_{ii} is the *i*th 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.

Variables	Symbol	Range and Levels				
		- α	-1	0	+1	+ α
Acid concentration (mol/l)	X ₁	-3.25	0.5	4.25	8.0	11.75
Temperature (°C)	X ₂	-15	50	115	180	245
Reaction time (hrs)	X ₃	-2.25	0.5	3.25	6.0	8.75
Solid/liquid ratio (g/mol)	X ₄	-0.035	0.01	0.055	0.1	0.145
Stirring speed (rpm)	X ₅	135	90	315	540	765

Table 2: Experimental design matrix and the experimental and predicted values for cations removal (%).

Run order	Acid Concentration (mol/l), X ₁		Activation Temperature (°C), X ₂		Activation Time (hrs), X ₃		Solid/Liquid Ratio (g), X ₄		Stirring Speed (rpm), X ₅		Cations Removed (%)	
	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Exp	Pred
1	-1	0.5	-1	50	-1	0.5	-1	0.01	+1	540	49	47.34
2	-1	0.5	-1	50	-1	0.5	+1	0.1	-1	90	38	40.42
3	-1	0.5	-1	50	+1	6.0	-1	0.01	-1	90	45	44.96
4	-1	0.5	-1	50	+1	6.0	+1	0.1	+1	540	40	43.46
5	-1	0.5	+1	180	-1	0.5	-1	0.01	-1	90	52	53.17
6	-1	0.5	+1	180	-1	0.5	+1	0.1	+1	540	47	49.41
7	-1	0.5	+1	180	+1	6.0	-1	0.01	+1	540	57	54.97
8	+1	0.5	+1	180	+1	6.0	+1	0.1	-1	90	48	51.64
9	+1	8	-1	50	-1	0.5	-1	0.01	-1	90	55	54.93
10	+1	8	-1	50	-1	0.5	+1	0.1	+1	540	58	54.51
11	+1	8	-1	50	+1	6.0	-1	0.01	+1	540	60	58.75
12	+1	8	-1	50	+1	6.0	+1	0.1	-1	90	49	51.63
13	+1	8	+1	180	-1	0.5	-1	0.01	+1	540	61	62.21
14	+1	8	+1	180	-1	0.5	+1	0.1	-1	90	53	53.81
15	+1	8	+1	180	+1	6.0	-1	0.01	-1	90	56	56.42
16	+1	8	+1	180	+1	6.0	+1	0.1	+1	540	69	66.92
17	-2	-3.25	0	115	0	3.25	0	0.055	0	315	57	58.65
18	+2	11.75	0	115	0	3.25	0	0.055	0	315	70	71.54
19	0	4.25	-2	-15	0	3.25	0	0.055	0	315	59	58.12
20	0	4.25	+2	245	0	3.25	0	0.055	0	315	68	70.44
21	0	4.25	0	115	-2	-2.25	0	0.055	0	315	55	54.73
22	0	4.25	0	115	+2	8.75	0	0.055	0	315	65	67.32
23	0	4.25	0	115	0	3.25	-2	-0.035	0	315	62	59.96
24	0	4.25	0	115	0	3.25	+2	0.145	0	315	52	57.83
25	0	4.25	0	115	0	3.25	0	0.055	-2	-135	50	52.65
26	0	4.25	0	115	0	3.25	0	0.055	+2	765	65	66.83
27	0	4.25	0	115	0	3.25	0	0.055	0	315	79	81.65
28	0	4.25	0	115	0	3.25	0	0.055	0	315	80	81.65
29	0	4.25	0	115	0	3.25	0	0.055	0	315	80	81.65
30	0	4.25	0	115	0	3.25	0	0.055	0	315	81	81.65

Bleaching Experiment:

The bleaching experiments were carried out in a batch process. 50 g of the refined palm oil were charged into a 250 ml beaker and 2 g of the activated clay samples were also added. The mixture of clay and oil were placed in a water bath and heated to a temperature of 80 °C for 30 minutes under continuous stirring. At the end of the reaction, the slurry formed was filtered through a dry filter paper. The bleaching capacity of the acid activated clays was then determined by measuring the colour of the bleached oils using a UV-Vis spectrophotometer (Shimadzu UV mini 1240) at wavelength of 450 nm. The bleaching efficiency of the acid activated clay was calculated in this study using the following equation:

$$\% \text{ Bleaching Efficiency} = \frac{A_{\text{unbleached}} - A_{\text{bleached}}}{A_{\text{unbleached}}} \times 100 \quad (2)$$

Where $A_{\text{unbleached}}$ and A_{bleached} are the absorbencies of the unbleached and bleached oils, respectively.

Statistical Analysis:

The generated experimental data were analyzed using the Statistica 10 trial version software by StatSoft 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 activation process. The linear, quadratic, and linear interactive effects of the process variables on the activation 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 less than 0.05 signified that the coefficient is significant. The experimental data were fitted to the second-order polynomial regression model and the adequacy of the model tested by the coefficient of determination (R^2) value as compared to the adjusted R^2 value.

RESULTS AND DISCUSSIONS**Characterization:**

The changes in the chemical composition of the natural and acid activated Ughelli bentonite at different acid concentrations are shown in Table 3. The content of SiO₂ was observed to increase as the acid

concentration increased up to 6mol/l and decreased with further increase in concentration and this could be due to the formation of mullite which protects the clay layers from further acid attack. The contents of the octahedral cations (Al₂O₃, Fe₂O₃, and MgO) decreased intensely as the acid concentration increased and they increased with further attack after 6mol/L concentration. The behaviour showed by the Al₂O₃, Fe₂O₃, and MgO contents with progressive acid treatment is related to the progressive dissolution of the clay mineral. The octahedral sheet destruction passes the cations into the solution, while the silica generated by the tetrahedral sheet remains in the solid phase due to its insolubility (Dias *et al*, 2003). Pesquera *et al* (1992) suggest that this free silica generated by the initial destruction of the tetrahedral sheet, is polymerized by the effect of such high acid concentrations and is deposited on the undestroyed silicate fractions, thereby protecting it from further acid attack.

Table 3: Chemical analysis and specific surface area of the natural and acid activated Ughelli bentonite samples determined by XRF.

Chemical composition (%)	Clay samples					
	UG0	UG0.5	UG2	UG4	UG6	UG8
Al ₂ O ₃	24.5	22.8	17.5	11.7	5.1	6.4
SiO ₂	52.3	56.9	63.7	70.3	76.4	73.5
Fe ₂ O ₃	10.8	7.3	4.63	2.9	0.7	0.91
Na ₂ O	0.96	0.78	0.43	0.28	0.09	0.16
MgO	3.78	2.45	1.42	0.99	0.14	0.19
K ₂ O	0.61	0.38	0.23	0.14	0.05	0.08
TiO ₂	2.56	1.74	1.04	0.87	0.04	0.15
LOI	4.27	3.17	2.42	1.73	0.76	0.84
Surface area (m ² /g)	112	198	254	302	387	359
Si/(Al + Fe + Mg)	1.34	1.75	2.70	4.51	12.86	9.80

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

Source	Coeff.	Sum of Squares	df	Mean Square	F-value	Std. Err.	t(15)	p-value
Model	80.85	4684.86	20	234.243	76.94	1.316	61.46	0.000000
(1)Acid concentration (mol/l)(L*)	4.63	513.375	1	513.375	56.118	1.235	7.49	0.00002
Acid concentration (mol/l)(Q**)	-4.74	634.691	1	634.691	69.379	1.139	-8.33	0.000000
(2)Temperature (K)(L)	2.79	187.042	1	187.042	20.446	1.235	4.52	0.0011
Temperature (K)(Q)	-4.74	634.691	1	634.691	69.379	1.139	-8.33	0.000000
(3)Reaction time (hrs)(L)	1.29	40.042	1	40.042	4.377	1.235	2.09	0.0629
Reaction time (hrs)(Q)	-5.62	890.358	1	890.358	97.327	1.139	-9.87	0.000000
(4)Solid/liquid ratio (g/ml)(L)	-2.21	117.042	1	117.042	12.794	1.235	-3.58	0.0050
Solid/liquid ratio (g/ml)(Q)	-6.37	1143.858	1	1143.858	125.037	1.139	-11.18	0.000001
(5)Stirring speed(rpm)(L)	3.13	234.375	1	234.375	25.620	1.235	5.06	0.00049
Stirring speed (rpm)(Q)	-6.24	1099.405	1	1099.405	120.178	1.139	-10.96	0.000001
1L by 2L	-0.94	14.062	1	14.062	1.537	1.512	-1.23	0.24334
1L by 3L	0.19	0.562	1	0.562	0.062	1.512	0.25	0.2480
1L by 4L	1.69	45.562	1	45.562	4.981	1.512	2.23	0.049698
1L by 5L	1.56	39.062	1	39.062	4.270	1.512	2.07	0.065679
2L by 3L	1.44	39.062	1	39.062	3.614	1.512	1.90	0.086468
2L by 4L	0.94	14.062	1	14.062	1.537	1.512	1.24	0.243341
2L by 5L	0.31	1.563	1	1.563	0.171	1.512	0.41	0.688125
3L by 4L	0.56	5.062	1	5.062	0.553	1.512	0.74	0.474052
3L by 5L	0.69	7.562	1	7.562	0.827	1.512	0.91	0.384627
4L by 5L	0.44	3.062	1	3.062	0.335	1.512	0.53	0.575664
Error		91.481	10	9.148				
Total SS		4361.355	30					

R² = 0.9790 Adj. R² = 0.9371 *Linear effect **Quadratic effect

Evaluation of Regression Model for Activation Efficiency:

The correlation between the experimental process variables and the activation efficiency was evaluated using the CCD modeling technique. Second order polynomial regression equation fitted between the response (activation efficiency, (Y)) and the process variables: acid concentration (X₁), reaction temperature (X₂), reaction time (X₃), solid/liquid ratio (X₄) and stirring speed (X₅). 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:

$$Y = 80.85 + 4.63X_1 + 2.79X_2 + 1.29X_3 - 2.21X_4 + 3.13X_5 - 0.94X_1X_2 + 0.19X_1X_3 + 1.69X_1X_4 + 1.56X_1X_5 + 1.44X_2X_3 + 0.94X_2X_4 + 0.31X_2X_5 + 0.56X_3X_4 + 0.69X_3X_5 + 0.44X_4X_5 - 4.74X_1^2 - 4.74X_2^2 - 5.62X_3^2 - 6.37X_4^2 - 6.24X_5^2 \tag{3}$$

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 4 shows that linear, quadratic, and the interaction terms of temperature and time are significant model terms. The model reduces to Equation (4) after eliminating the insignificant coefficients.

$$Y = 80.85 + 4.63X_1 + 2.79X_2 - 2.21X_4 + 3.13X_5 + 1.69X_1X_4 - 4.74X_1^2 - 4.74X_2^2 - 5.62X_3^2 - 6.37X_4^2 - 6.24X_5^2 \quad (4)$$

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^2) value of 0.9790 and adj. R^2 value of 0.9371. This means that the model explains 97.90% of the variation in the experimental data. The adequate correlation between the experimental values of the independent variable and predicted values further showed the adequacy of the model as illustrated in Figure 1.

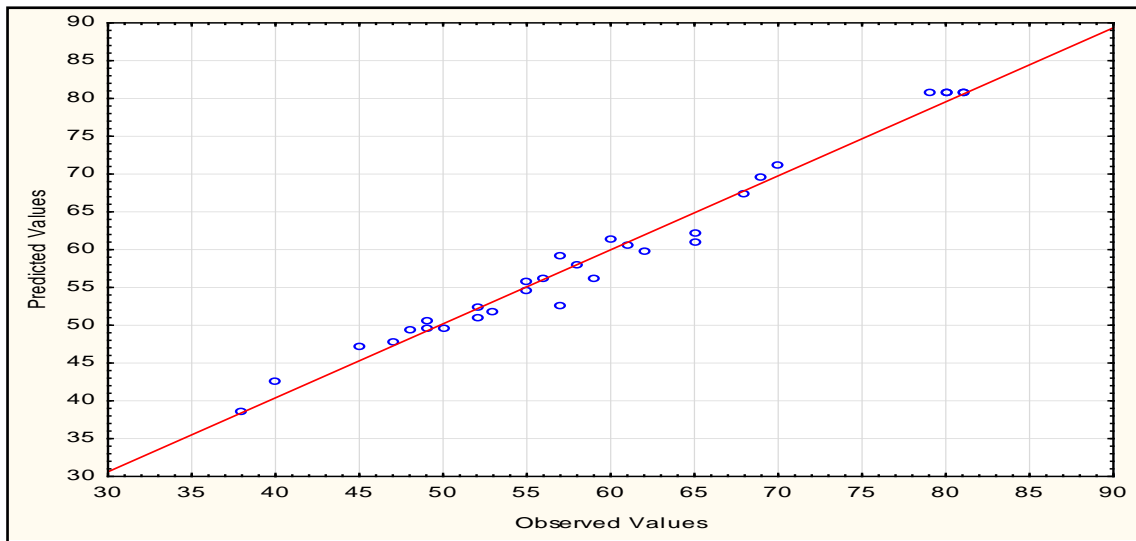


Fig. 1: Plot of the predicted versus the observed values of the activation efficiency.

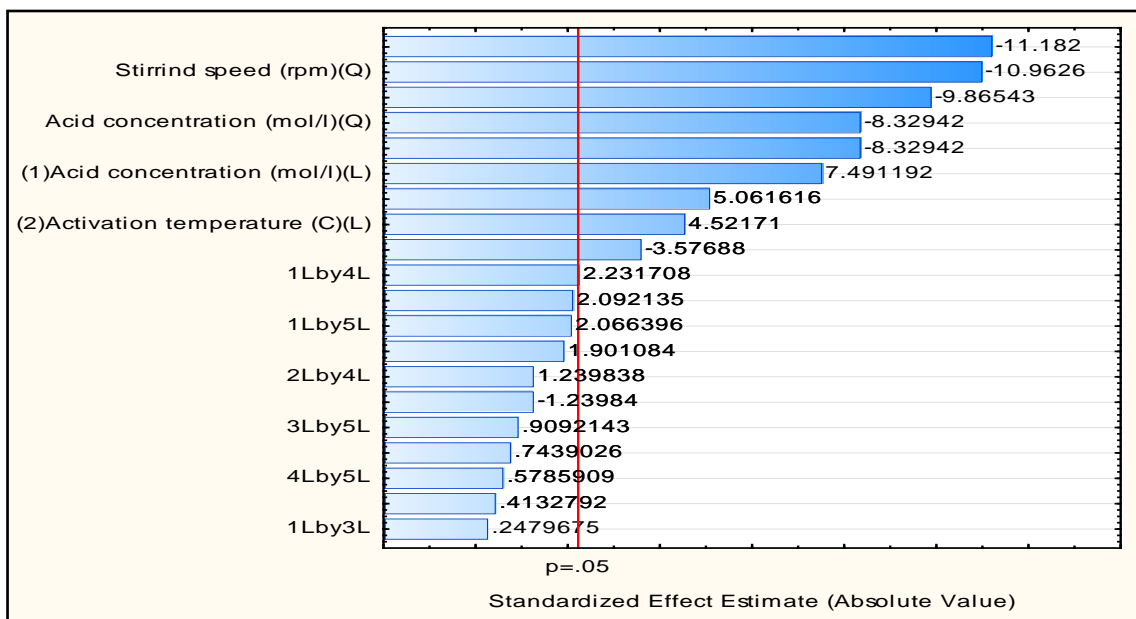


Fig. 2: Pareto's chart to estimate the linear (L), quadratic (Q), and linear interaction (L by L) effects on the activation efficiency.

Figure 2 shows the linear, quadratic and the interaction effects of the independent variables, at the 95% of confidence level, on the percentage activation efficiency. It can be seen that all the variables were significant at the linear and quadratic levels, except the linear effect of time. The largest main effect observed in the cations removal was the variable solid/liquid 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 activation efficiency. The figure also shows that acid concentration has the largest positive effect at the linear level followed by the stirring speed and then temperature (linear). Moreover, the interactive effects between the other independent variables were not significant.

Response Surface Plots:

The interactive effects of the process variables on the percent activation efficiency were studied by plotting three dimensional surface curves against any two independent variables, while keeping other variables at their central (0) level. The 3D curves of the response (activation efficiency) and contour plots from the interactions between the variables are shown in figures 3 – 12. The interactive effect of acid concentration and temperature on the activation efficiency is negative (Table 3), that is, increasing both variables reduces the activation efficiency, and this could be attributed to the fact that excess acid and high temperature destroy the crystalline structure of the bentonite, thereby reducing its adsorptive capacity (Bonilla *et al*, 1981). The response surface plot of the interactive effect of acid concentration and temperature is shown in Figure 3, the activation efficiency increased with simultaneous increase in acid concentration and temperature to about 3.89mol/l and 120 °C and thereafter decreased with further increase in their values. The same trend was observed for the interactive effects of acid concentration and time; acid concentration and solid/liquid ratio; acid concentration and stirring speed and the response surface plots are shown in Figures 4, 5 and 6, respectively. The interactive effects of temperature and time on the activation efficiency are positive as shown in Table 3. The response surface plots of the interactive effect of temperature and time are shown in Figure 7. The activation efficiency increased with simultaneous increase in reaction temperature and time. The activation efficiency started to increase from 40 °C and 0.5 hr and continued as long as both variables are increased. Increase in reaction time resulted in corresponding increase in activation efficiency as was evident in Figures 4, 7, and 8. Figure 9 shows that as the solid/liquid ratio increased, more acid concentration was required for maximum activation efficiency to be achieved and the same trend was observed in Figure 8, at high stirring speed, temperature less than 80 °C was required to give the maximum activation efficiency. The effect of stirring speed was observed to be similar to that of temperature effect. High stirring speed gave rise to increased activation efficiency as was evident from Figures 10, 11 and 12. The optimum conditions are: acid concentration 6.15mol/l; reaction temperature 132.87°C; reaction time 3.73 hours; solid/liquid ratio 0.05 g/ml; stirring speed 388.72 rpm; and the optimum activation efficiency at this optimum condition was predicted to be 83.10%. Experiments were carried out at these optimum conditions to validate the predicted optimum values. The experimental value of 82.96% agreed closely with that obtained from the regression model.

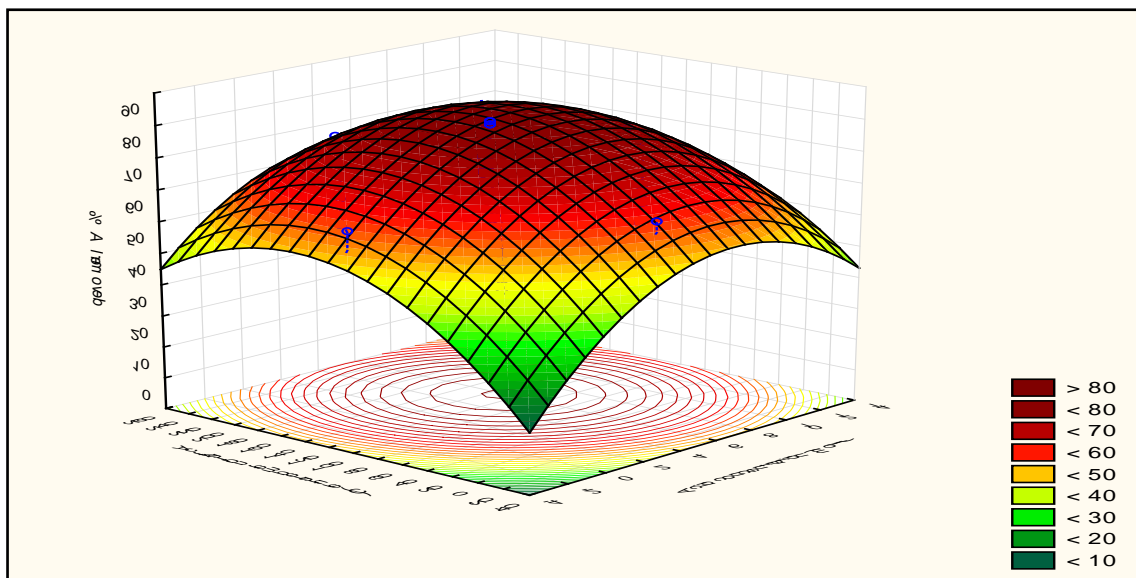


Fig. 3: Effect of acid concentration and temperature on activation efficiency at constant reaction time, solid/liquid ratio, and stirring speed.

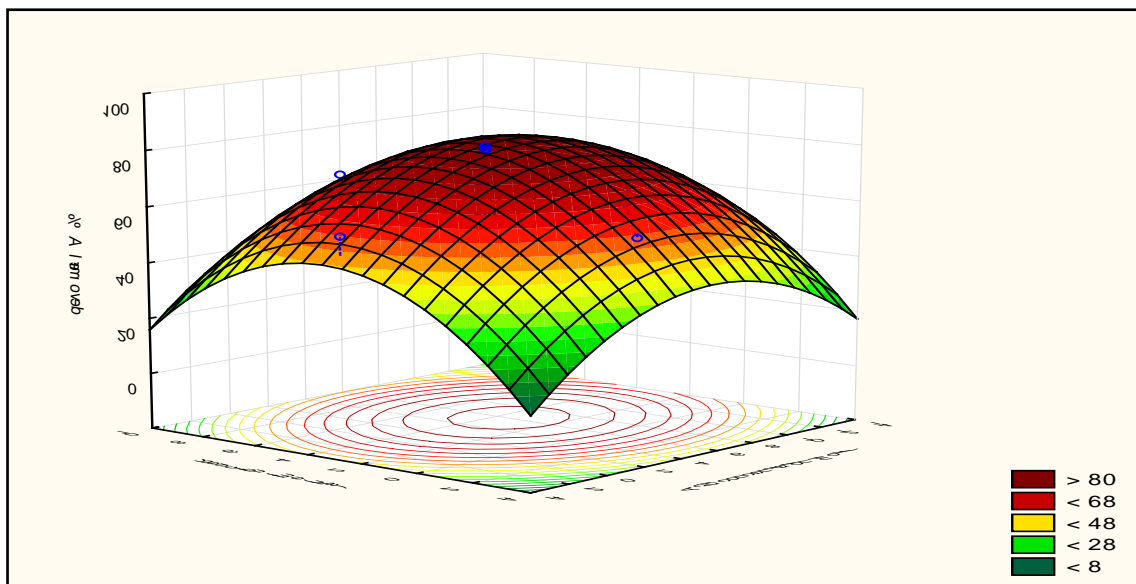


Fig. 4: Effect of acid concentration and reaction time on activation efficiency at constant reaction temperature, solid/liquid ratio and stirring speed.

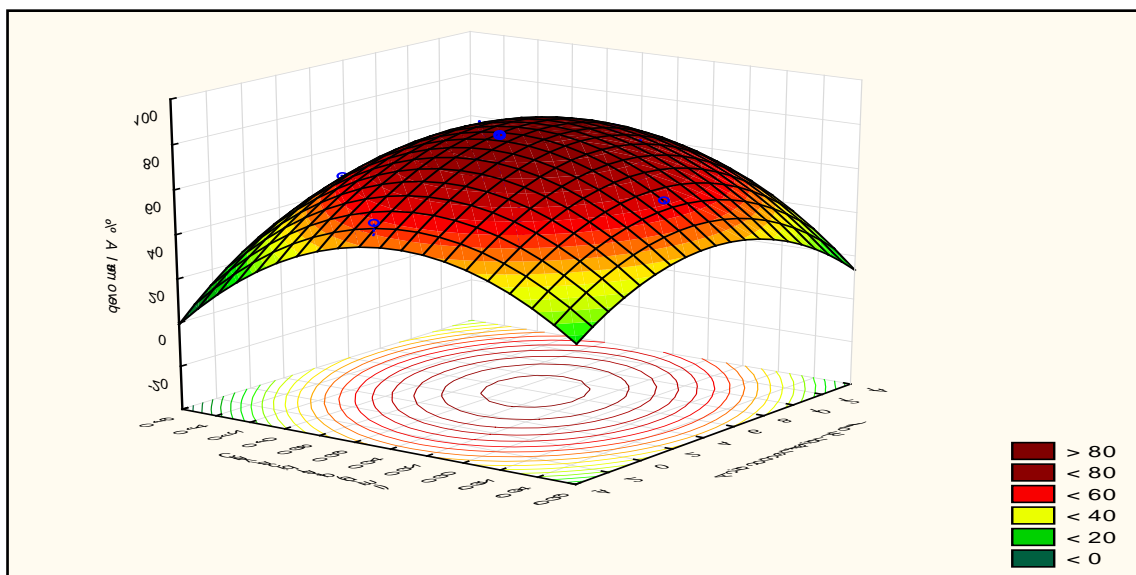


Fig. 5: Effect of acid concentration and solid/liquid ratio on activation efficiency at constant reaction temperature, reaction time and stirring speed.

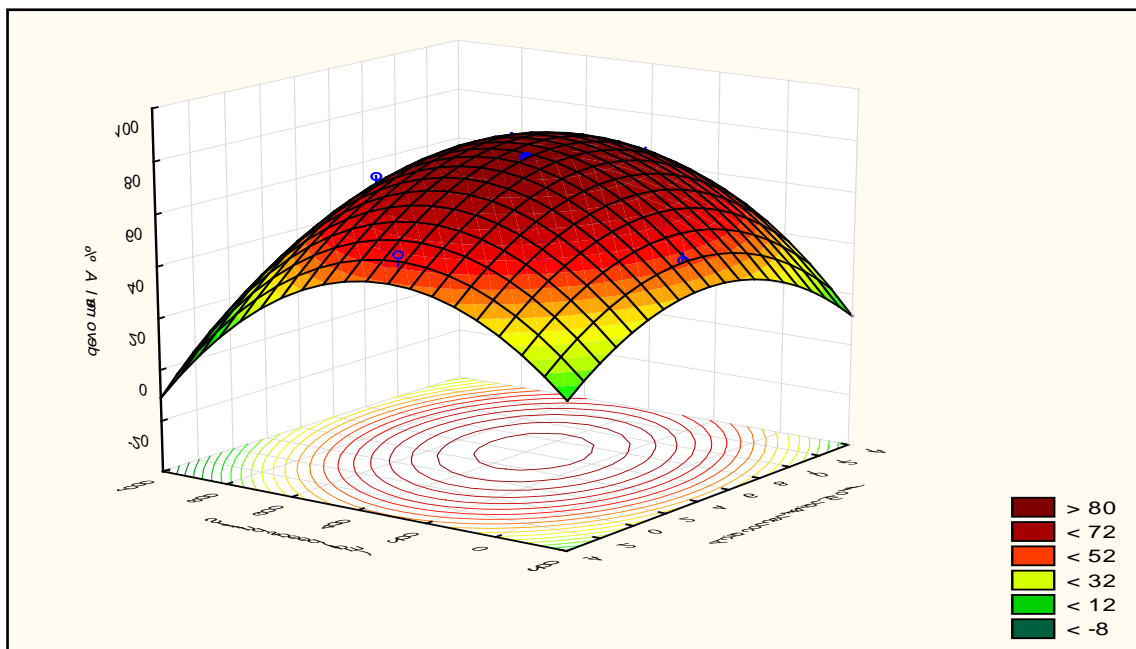


Fig. 6: Effect of acid concentration and stirring speed on activation efficiency at constant reaction temperature, reaction time and solid/liquid ratio.

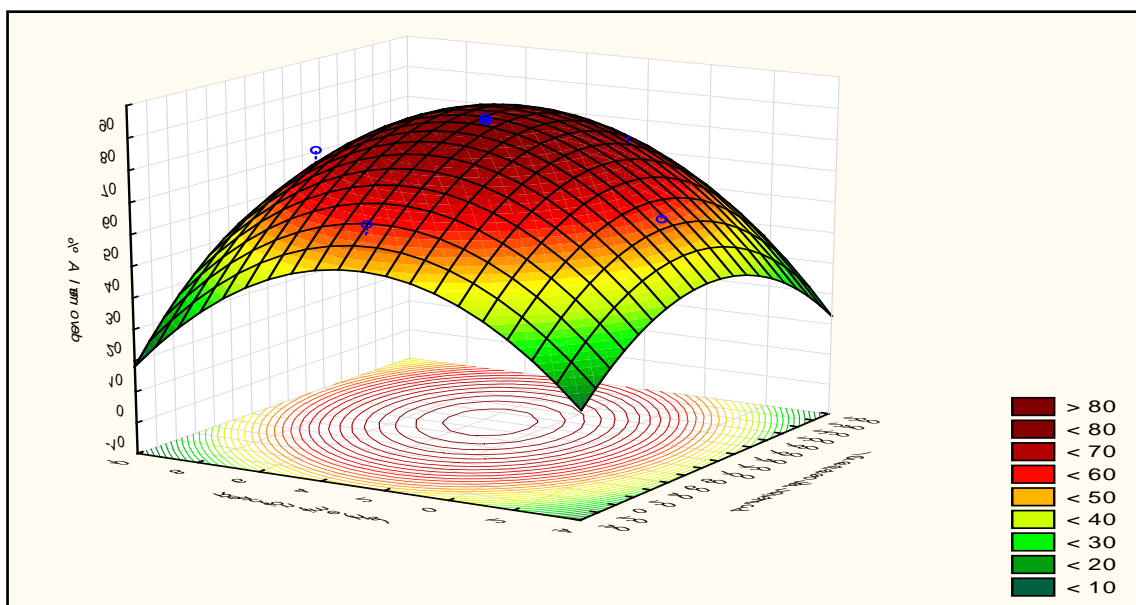


Fig. 7: Effect of temperature and reaction time on activation efficiency at constant acid concentration, solid/liquid ratio and stirring speed.

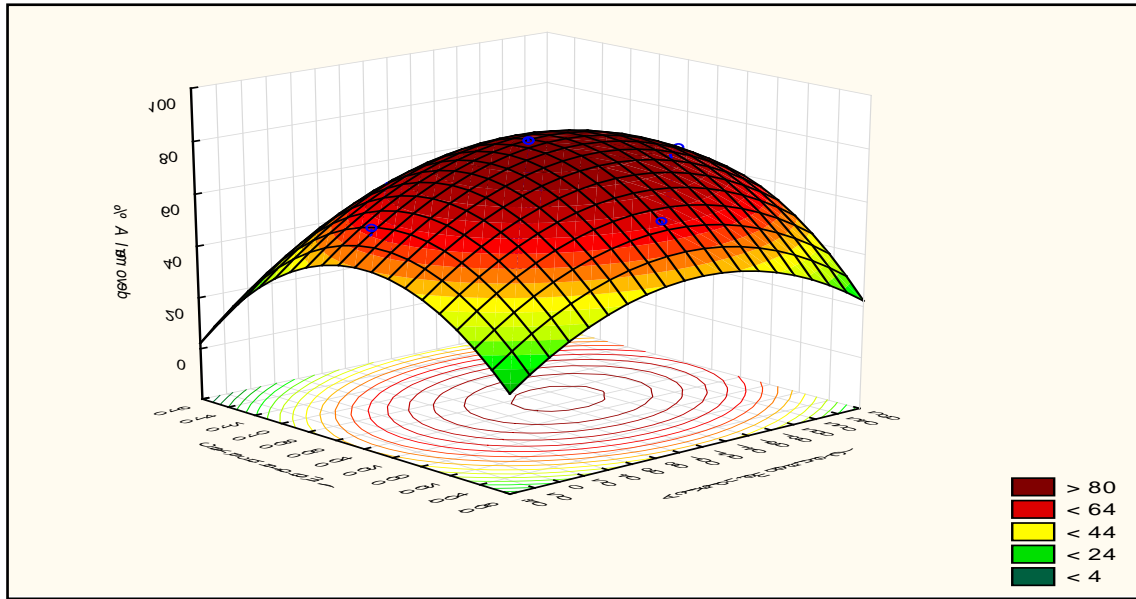


Fig. 8: Effect of temperature and solid/liquid ratio on activation efficiency at constant acid concentration, reaction time and stirring speed.

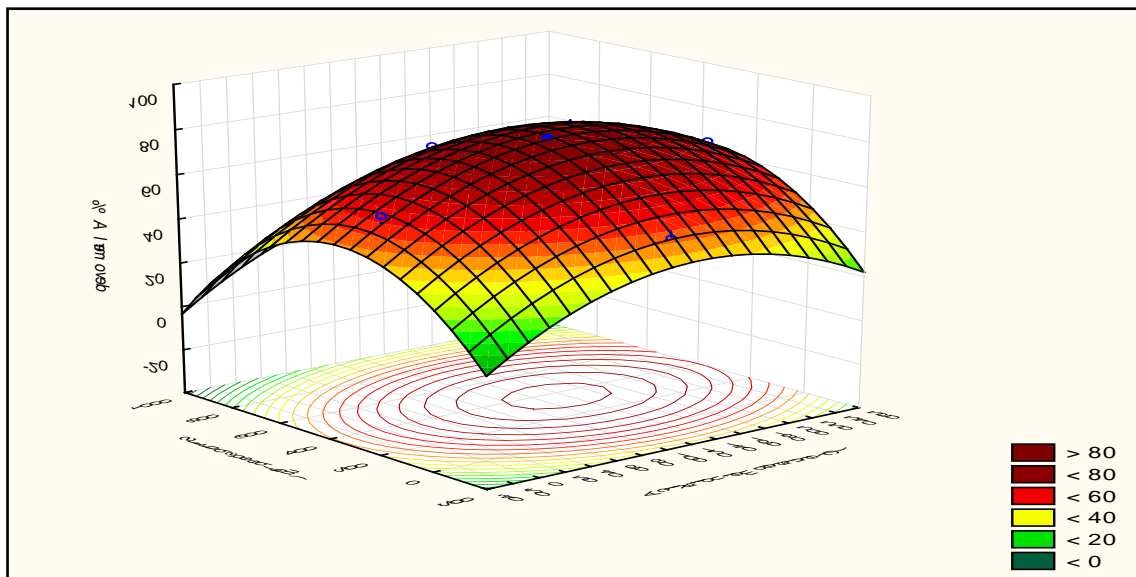


Fig. 9: Effect of temperature and stirring speed on activation efficiency at constant acid concentration, reaction time and solid/liquid ratio.

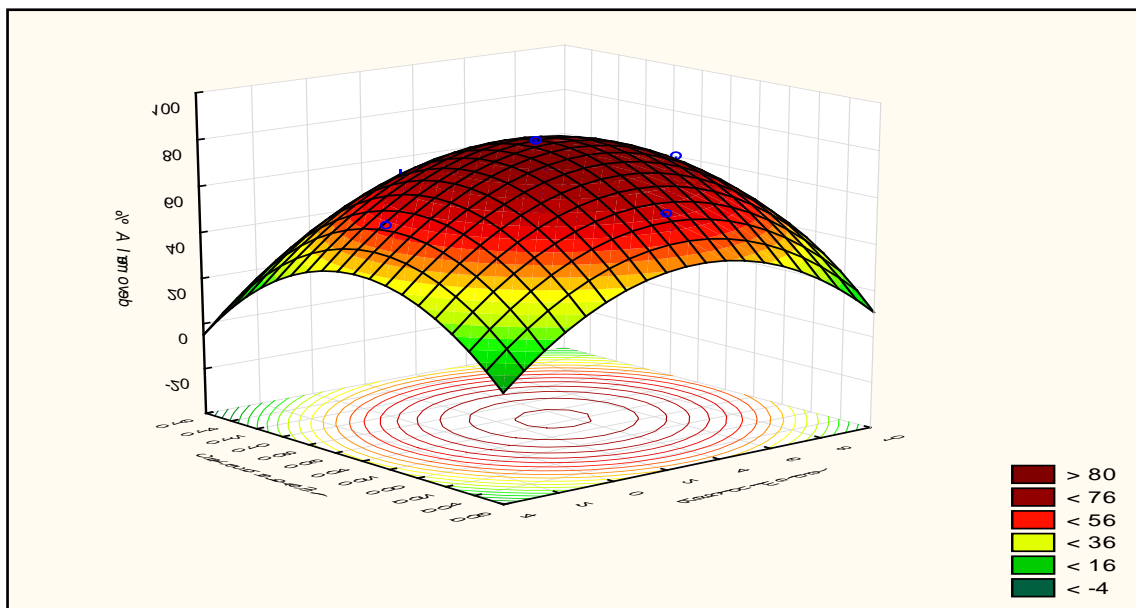


Fig. 10: Effect of reaction time and solid/liquid ration on activation efficiency at constant acid concentration, reaction temperature and stirring speed.

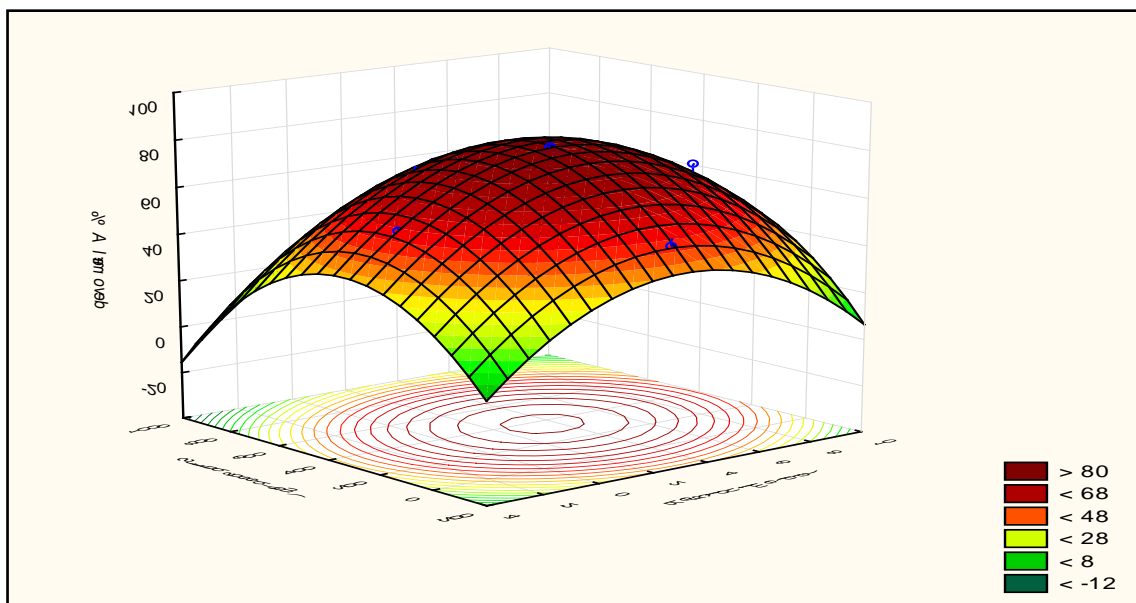


Fig. 11: Effect of reaction time and stirring speed on activation efficiency at constant acid concentration, reaction temperature and solid/liquid ratio.

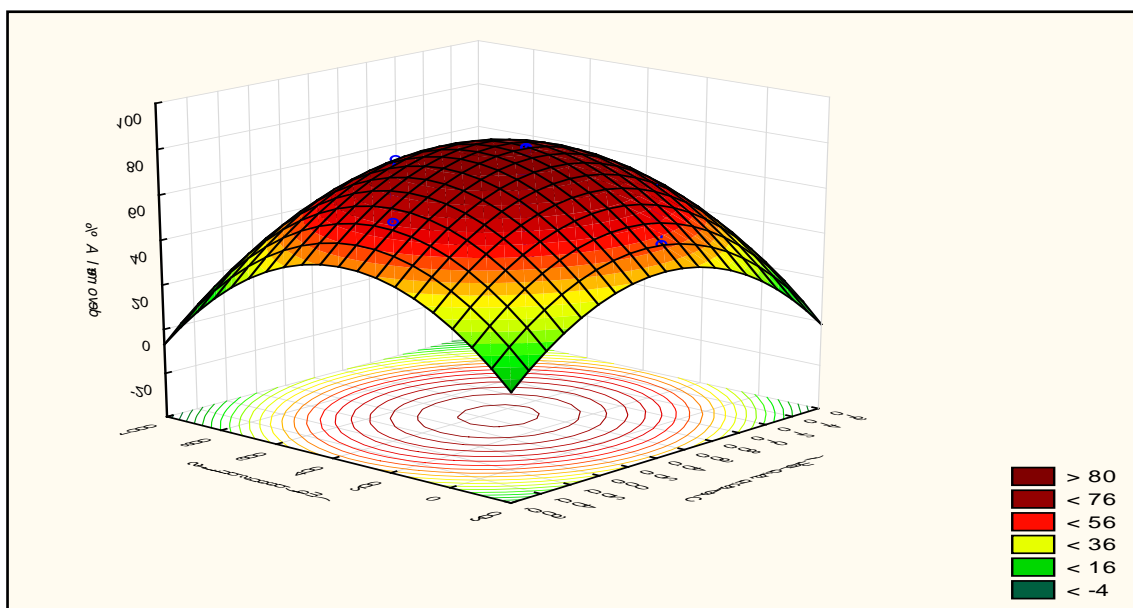


Fig. 12: Effect of solid/liquid ration and stirring speed on activation efficiency at constant acid concentration, reaction temperature and reaction time.

Bleaching Studies:

The results of the bleaching studies performed using the natural and acid activated samples are shown in Figure 13. The figure shows that the bleaching efficiency increased with increase in the acid concentration used in the activation step. The increase reached a maximum with the sample activated with 6mol/l H₂SO₄ and decreased thereafter with further increase in the acid concentration. This decrease in bleaching efficiency is attributed to the small surface area and Si / [Al₂O₃ + Fe₂O₃ + MgO] ratio exhibited by the sample activated with 8 mol/L of H₂SO₄ that led to the destruction of the clay crystalline structure.

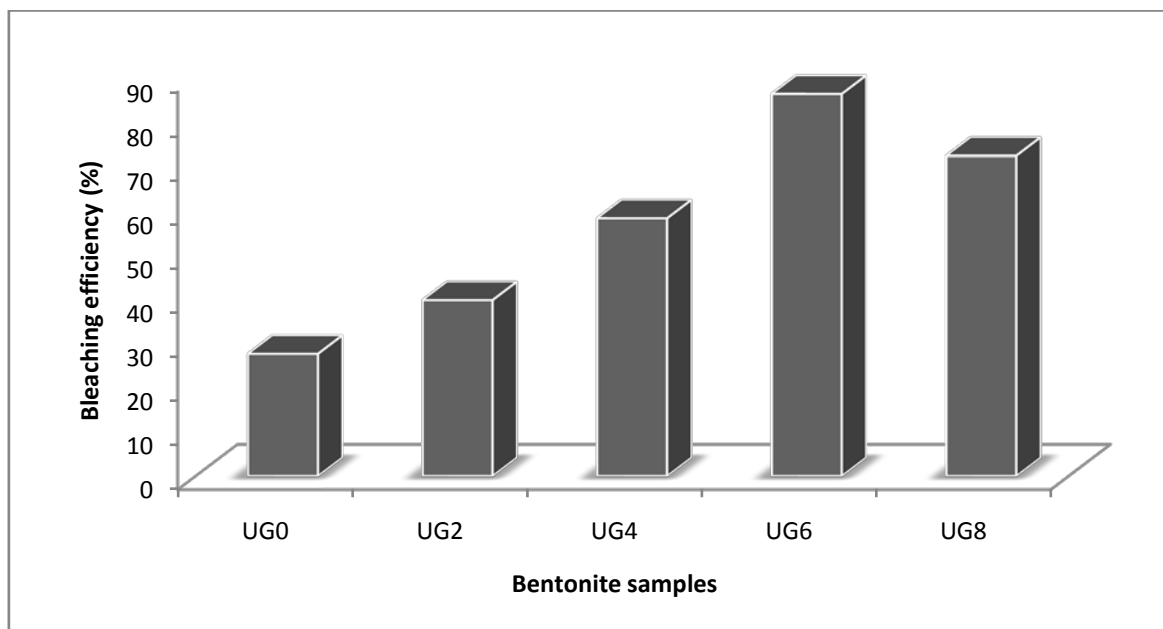


Fig. 13: Plot of the bleaching efficiencies of the natural and acid-activated bentonite samples.

Conclusions:

The optimum activation conditions of bentonite from Ughelli have been studied. Response surface methodology was successfully employed to analyze the activation data. The process parameters investigated were all significant both at the linear and quadratic level and only the interaction effect of temperature and reaction time was statistically significant. The high level of correlation between the predicted and observed

response proved the regression model to be adequate in explaining the variations in the experimental data. The optimum conditions for the maximum adsorption efficiency of 83.10% were determined to be: 6.15mol/l acid concentration; 132.87°C temperature; 3.73 hours reaction time; 0.052 solid/liquid ratio; and 388.72 rpm stirring speed. This study has revealed that effective adsorbent can be produced from Ughellibentonite under optimized process conditions.

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