

## Discongruity in the Reclusion of a Neutral Dye, Neutral Red, Using Acid and Base Activated Clays from Odoalamo Viillage.

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### ABSTRACT

Activation of clay from Odoalamo village using acid and base has been done. Adsorptive experiments were done using neutral red dye to assess the effect of activation. The result shows a decrease in adsorptive properties in the base activated clays while the acid activated ones increases. The 1M H<sub>2</sub>SO<sub>4</sub> activated clay gave the highest result for activation of clay.

**Keywords:** Discongruity, adsorption, illite, activation.

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### 1. INTRODUCTION

Ijebu north east local government area of Ogun state, Nigeria is an area with abundant mineral resources part of which are illite clays; also called black clay, in Imope village and kaoline/illite clay; also called brown clay, in Igaran village. Kaolinite is an important industrial mineral and Illite is used for the preparation of mixtures for traditional ceramic. [6] Clay activation is a process to increase the clay character to obtain the desired properties in accordance with its use. Chemical activation can be done by addition of acid or base solution. Generally the acid used is H<sub>2</sub>SO<sub>4</sub> [4, 5] while the base used is NaOH [2,4]. Treatment with mineral acid increases clay surface area and pore volume [3]. The result of such treatment is dependent on temperature of the system, polarity of acid and time [9].

Contamination of the environment by dyes and heavy metals has been on the increase due to industrial revolution of the last few decades. Dye detected in waste streams from textile, paint and Petro chemical industries are the major environmental problem in both developed and developing countries [1]. Consequently, there has been adverse effect on our environment, because of the toxicity of the dye compounds involved [7]. Biosorption, a relatively new technology, has been considered to be an alternative for the uptake of toxic metal ions from waste water. It is more economical than conventional methods and has thus been given much attention in the recent years. The mechanism of the biosorption process is explained in terms of the reaction between anionic groups present in the biomasses and the cationic metal ions. In this study, the disparity in the removal of a neutral dye, neutral red, from aqueous solution using acid activated and base activated natural illite clay from Odoalamo in Ijebu north east was investigated.

## 2. EXPERIMENTAL

### Drying, Crushing and Sieving:

About 30g each of illite natural clays- from Odoalamo, were taken from the soil and dried at 110°C till constant mass. The clay was sieved, crushed (in mortars and with pestles) and sieved again before they were packed in air tight bags and stored.

### Characterization of Clays:

X- ray diffraction (Rigaku D/Max-III C, PW 1800; Tokyo, Japan) analysis was used for determination of the crystalline structure of the clay while Fourier transform infrared spectroscopy (FT-IR), using Pelkin Elmer 3000 MX spectrometer. (Nicolet iS10 FT-IR Spectrometer) was used to identify important stretching and bending bonds in the clay's morphological structures.

### Activation Of Clay – Acid And Base Activation

#### Activation by Bases:

A total of 30g each of the clay was put in 100ml cup glass and 100ml, 2M NaOH is added with constant stirring for eight (8) hours while being heated at 80°C. The same weight of clay were treated the same way but without heating. The Clays were then filtered and washed with distilled water until the wash water read a pH of 7. It is then dried at 100°C to constant weighs, crushed, sieved and packed in air tight bags.

#### Activation by Acids:

Into five (5) 200ml glasses for each clay sample, 50g of clay were added to 150ml each of 1M, 1.5M, 2M, 3M and 4M H<sub>2</sub>SO<sub>4</sub> and stirred continuously for twenty four (24) hours. The clays were then filtered and washed with distilled water until the wash water read a pH of 7. They were then dried at 100°C, crushed, sieved and stored in labelled airtight bags.

### 2.1 Adsorption Experiments To Study The Effect Of Activation

1. Samples of the heated and unheated base activated clays were fed into 100ml borosilicate flasks in bits of 0.25g, 0.50g, 1.00g and 2.00g respectively. 50ml of Neutral red dye was added to each and stirred for 100 minutes. The clays were then separated from the solutions by centrifuge at 3000rpm for 3 minutes and the residual concentration of dyes from the samples was taken with the UV-Vis at 535nm wavelength. The experiment was repeated three times each and average value taken.
2. 0.5g, 1.0g and 2.0g of each clay, previously activated by 1M, 1.5M, 2M, 3M and 4M H<sub>2</sub>SO<sub>4</sub> were added to 50ml Neutral red 50ppm solution in 100ml borosilicate flasks and stirred for 100 minutes each. The clays were then removed from the resulting solution by centrifuge at 3000rpm for 3 minutes. The concentration of dye in the residual solution obtained was got using UV-Vis at a wavelength of 535nm. The experiment was repeated three times each and average value taken.

### 3. RESULT AND DISCUSSION

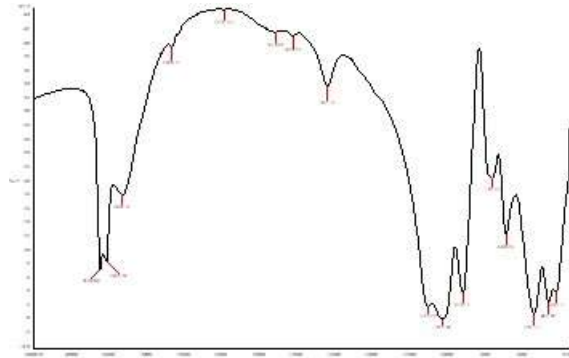


Fig 1 - FT-IR

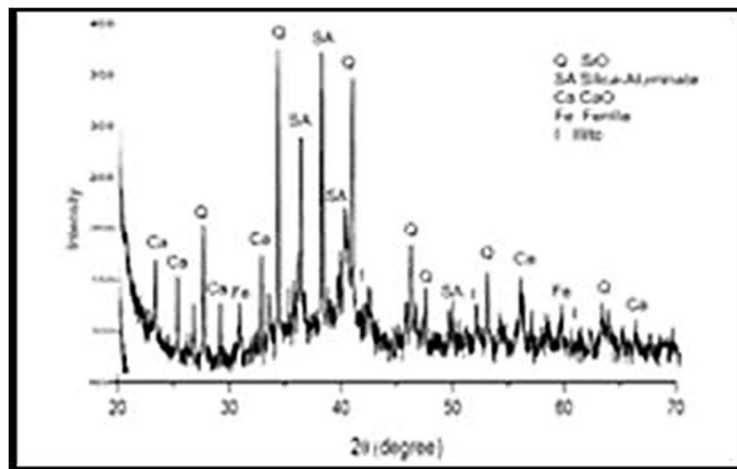


Fig 2 - XRD pattern for BLN

The result of the absorbent performance of the natural and activated (acid and base) clays is presented below:

- $C_o$ (PPM) -- Initial concentration of dye.
- $C_e$ (PPM) -- final concentration of dye.
- $C_o - C_e$ (PPM) -- concentration of dye adsorbed.
- $Q$ (%) -- adsorption percentage.

#### 1. CLN -NATURAL BLACK CLAY.

Mass(g)	$C_o$ (PPM)	$C_e$ (PPM)	$C_o - C_e$ (PPM)	$Q$ (%)	Qmg/g
0.25	50.00	06.09	43.91	87.82	8.78
0.50	50.00	16.50	33.50	67.00	3.35
1.00	50.00	27.77	22.23	44.46	1.11
2.00	50.00	36.97	13.23	26.46	0.33

**2. CLBC – COLD BASE ACTIVATED- 2M NaOH BLACK**

Mass(g)	C <sub>o</sub> (PPM)	C <sub>e</sub> (PPM)	C <sub>o</sub> -C <sub>e</sub> (PPM)	Q(%)	Qmg/g
0.25	50.00	16.67	33.33	66.66	6.67
0.50	50.00	16.99	33.01	66.02	3.30
1.00	50.00	20.50	29.50	59.00	1.48
2.00	50.00	22.42	27.58	55.16	0.69

**3. CLBH – HOT BASE ACTIVATED- 2M NaOH BLACK**

Mass(g)	C <sub>o</sub> (PPM)	C <sub>e</sub> (PPM)	C <sub>o</sub> -C <sub>e</sub> (PPM)	Q(%)	Qmg/g
0.25	50.00	16.29	33.71	67.42	6.74
0.50	50.00	13.87	36.13	72.26	3.61
1.00	50.00	16.80	33.20	66.40	1.66
2.00	50.00	26.60	23.40	46.80	0.59

**4. CLA 1M H<sub>2</sub>SO<sub>4</sub>**

Mass(g)	C <sub>o</sub> (PPM)	C <sub>e</sub> (PPM)	C <sub>o</sub> -C <sub>e</sub> (PPM)	Q(%)	Qmg/g
0.50	50.00	4.52	45.48	90.96	4.55
1.00	50.00	8.42	41.58	83.16	2.08
2.00	50.00	0.46	49.54	99.08	1.24

**5. CLA 1.5M H<sub>2</sub>SO<sub>4</sub>**

Mass(g)	C <sub>o</sub> (PPM)	C <sub>e</sub> (PPM)	C <sub>o</sub> -C <sub>e</sub> (PPM)	Q(%)	Qmg/g
0.50	50.00	1.33	48.67	97.34	4.87
1.00	50.00	3.25	46.75	93.50	2.34
2.00	50.00	2.31	47.69	95.38	1.20

**6. CLA 2M H<sub>2</sub>SO<sub>4</sub>**

Mass(g)	C <sub>o</sub> (PPM)	C <sub>e</sub> (PPM)	C <sub>o</sub> -C <sub>e</sub> (PPM)	Q(%)	Qmg/g
0.50	50.00	3.54	46.46	92.92	4.65
1.00	50.00	3.32	46.68	93.36	2.33
2.00	50.00	4.38	45.62	91.24	1.14

**7. CLA3M H<sub>2</sub>SO<sub>4</sub>**

Mass(g)	C <sub>o</sub> (PPM)	C <sub>e</sub> (PPM)	C <sub>o</sub> -C <sub>e</sub> (PPM)	Q(%)	Qmg/g
0.50	50.00	6.92	43.08	86.16	4.31
1.00	50.00	9.66	40.34	80.68	2.02
2.00	50.00	2.42	47.58	95.16	1.19

**8. CLA 4M H<sub>2</sub>SO<sub>4</sub>**

Mass(g)	C <sub>o</sub> (PPM)	C <sub>e</sub> (PPM)	C <sub>o</sub> -C <sub>e</sub> (PPM)	Q(%)	Qmg/g
0.50	50.00	12.93	37.07	74.14	3.71
1.00	50.00	05.02	44.98	89.96	2.25
2.00	50.00	04.75	45.22	90.44	1.13

From tables 1, 2, and 3, it could be observed for the clay that the base activated clays adsorbs less than the natural clays. Furthermore, it appears that the hot activated clays have a little edge over the cold activated ones. This is in agreement with the observation of Bijang et al 2019 [4]. Maziarz and Matusik who stated that alkaline treatment can cause changes in the structure, texture and morphology of clay minerals which conforms to the partial dissolution of the layer structure and subsequently result in the release of  $\text{Si}_4^+$  and  $\text{Al}_3^+$  ion into the solution [8]. 0.25g clay dosage gives the maximum adsorption in the natural clays while for the activated clays it is 1.0g for the clay.

The results for the adsorption by 1M, 1.5M, 2M, 3M, and 4M  $\text{H}_2\text{SO}_4$  acid activated clays are presented in tables 4 and 8. The 1.5M  $\text{H}_2\text{SO}_4$  activated clay adsorbed best for the clay. Activation of clay using acid will produce clay with larger active sites, good porosity and greater surface acidity (10). The neutral dye bonding to the active site of the clay is shown in fig 2. It is suspected that the low adsorption by the alkaline activated clays is due to the low acidic sites on them as they are neutralised by strong base.

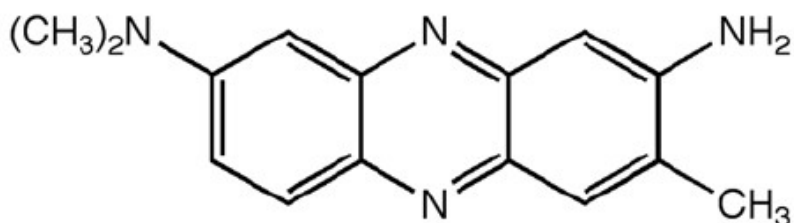


Fig 3.

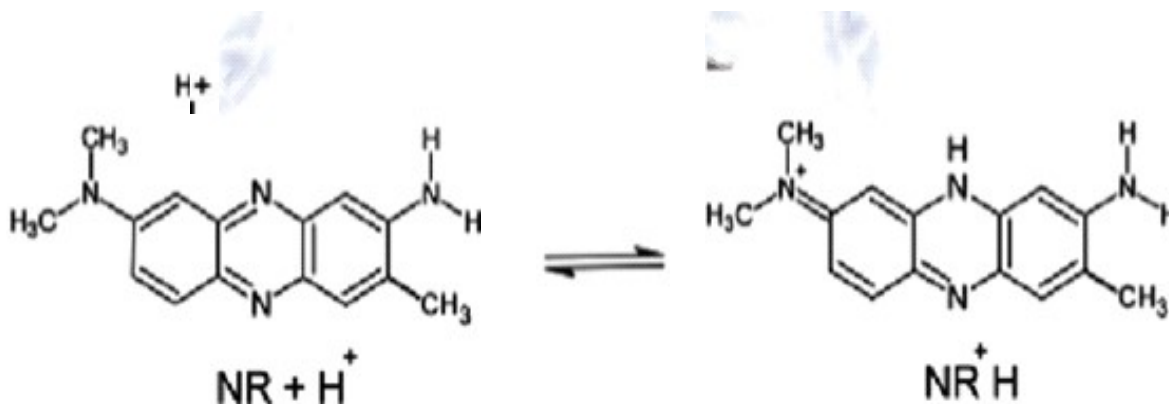


Fig 4.

#### 4. CONCLUSION

The acid activated natural clay from Odoalamo village absorb neutral red dye more than the neutral clays. The base activated forms however have lower adsorptive properties. The 1M  $\text{H}_2\text{SO}_4$  activated clay yield best result for the clay.

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