FUDMA Journal of Sciences (FJS)
ISSN online: 2616-1370
ISSN print: 2645 - 2944
Vol. 7 No. 2, April, 2023, pp 52 - 56
DOI: https://doi.org/10.33003/fjs-2023-0702-1709

Al-Fe LAYERED HYDROXIDE WITH BENTONITE CLAY AND APPLICATION FOR THE REMOVAL OF DYE FROM AQUEOUS SOLUTIONS

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ABSTRACT

The research was aimed to investigate the effect of decolourisation of Alizarin yellow dye from aqueous solutions using Al-Fe layered Hydroxide clay.Batch adsorption technique was employed to determine the optimum values of some parameters i.e initial dye concentration (10 – 50 mg/l), contact time (10 – 50 minutes) and adsorbent dose (0.1 – 0.5 g). The results obtained indicate that the dye concentration decreases with increase in contact time and adsorbent dosage.The Adsorption isotherms observed showed that the Langmuir adsorption Isotherm was best fitted having the maximum decolourisation of 76.89 mg/g and R^2 value of 0.986. The Al-Fe layered Hydroxide prepared was characterized by SEM, FT-IR and XRD before and after the adsorption process. The SEM results showed the porosity was roughly filled after the adsorption. The FT_IR spectra showed the presence of OH, C=O and N-CH at 3625 cm^{-1}, 1633 cm^{-1} and 2806 cm^{-1}. The XRD results indicate that the crystalline structure was not altered after the adsorption process. In this research, a low cost and effective adsorbent was used for the removal of Alizarin Yellow dye in an aqueous solution.

Keywords: Al-Fe layered Hydroxide, Adsorption, Alizarin Yellow

INTRODUCTION

Most dyes are pollutants frequently found in waste waters of several industries such as mining, metal processing, petroleum refining, textile, industries and printing (Fu and wang 2011). These pollutants have negative impact on the environment and modify the physical and chemical characteristics of water and soil (Houda *et al.,* 2011) and the properties of aquatic plantsand animals (Das *et al.,* 2011). The presence of dyes in industrial waste water, irrigation and drinking water resources also has high negative impacts on human health due to their toxicity (Amin*et al.,*2013). So the elimination of dyes from waste water is one of the most important environmental problems for research (Fu and wang 2011). A lot of techniques have been developed for removal of dyes from waste water, among which adsorption is the most efficient (Zhao *et al.,* 2014). Different adsorbent was tested for removal of dyes, such as agricultural waste, activated carbon among others (Mahinda *et al.,* 2017).

In this work Al-Fe layered hydroxide with bentonite clay was aimed to synthesize for the removal of alizarin yellow dye. This adsorbent is easily available and economically favorable. Alizarin yellow is a mordant azo dye. It is form by diazo coupling reaction with the molecular formula $C_{13}H_8O_5Na$, and molecular mass 309.21 g mol^{-1} and its λ max is 370 nm, it is a slightly brown powder soluble in cold water (Rabiat *et al.,*2015). It is used to colour wool, lather and nylon and causes gastro intestinal tract disorder and may produce nausea and vomiting when inhaled (Abdelhalim *et al.,* 2015).

MATERIAL AND METHOD

Preparation of adsorbent

The Al-pillared clay was prepared by dissolving 5% of the clay suspension in water; the solution was then mixed with the pillaring solution to provide 20 meq of Al^{3+}/g of the clay. The beaker containing the solutions mixture was placed in an ultrasonic bath at ambient temperature (300K/27^{O}C) for 20 minutes. The product was centrifuged at 4500 rpm for 5 minutes and was washed with distilled water 5 times until the filtrate was free of chloride ions. This was tested using silver nitrate ($AgNO_3$ test). The resulting residue was dried in an oven at 60^{0}C for 4 hours. The dried pillared bentonite clay was grounded in to fine powder using mortar and pestle to about more than 40 mesh size. The intercalated sample was calcined in a furnace at 500^{0}C for 6 hours (Palinko *et al.,* 1997).

Fe Dopping

The Al-pillared clay was dopped with Fe (III) according to(Palinko *et al.,* 1997) by dissolving 4g of the calcined layered clay in 20 cm^3 of 20% $FeCl_3$ solution. It was ultrasonicated at 30^{0}C for 5minute, and then dried in an oven at 60^{0}C for 24 hours, after which it was grounded and calcined again in a furnace at 500^{0}C for 6 hours.

Preparation of Adsorbate

1.0 g of Alizarin Yellow was taken in 1000 ml measuring flask and dissolved in distilled, making volume up to the mark. This was 1000 ppm stock solution of dye. Standard solution of the dye was prepared by successive dilution of stock solution.

Batch Adsorption

Experiment on adsorption of alizarin yellow on Al-Fe layered hydroxide bentonite clay was carried out by batch adsorption on accordance to the procedure reported by (Ibrahim and Ahmad 2017). The influence of various parameters such as initial dye concentration (10 - 50 mg/l), Contact time (10 – 50 minutes), temperature (30 –70^{O}C), adsorbent dose (0.1 – 0.5 g) were studied at constant agitation speed of 100 rpm in triplicate. 1000 ppm stock solution was prepared by dissolving 1.0 g of alizarin dye in 1000 ppm volumetric flack and serial dilution was conducted using the relation

$$C_IV_I = C_2V_2 \quad (1)$$

Where;
C_1 = Initial dye concentration, C_2 = Final dye concentration,
V_1 = Initial volume of dye and
V_2 = Final volume of dye.

Infrared absorption spectroscopy (FT-IR)

The spectra of Al-Fe layered hydroxide were recorded on a carry 630 FT- IR Agilent. All spectra (32 scans at 8.0 cm^{-1} resolution) were recorded at a range 4000 – 650 cm^{-1}.

Scanning electron microscopy (SEM)

SEM was conducted in order to investigate the surface morphology of Al-Fe layered hydroxide before and after adsorption. The samples were analyzed using the microscopic operated at an accelerated voltage of 15kv and 500 magnification using phenom SEM 800 – 07374 model.

X- Ray diffraction (XRD)

The crystallinity of the Al-Fe layered hydroxide was analyzed on a Bruncker X- ray diffractometer and the intensity at 2ϴ angle, XRD analyses were conducted using powdered samples. The diffraction patterns were recorded on a Thermo scientific Switzerland with model number 197492086 ARL X'TRA X- ray diffracttometer, using Ni-filtered Cu Ka radiation (k =1.5405 A^{O}) and scintillation counter at 40 V and Ma at ambient temperature. All the samples were scanned at 2h diffraction angle ranging from 0 to 120.The powdered sample with uniform surfaces were exposed to X- rays and the scattering angles of the diffracted X- rays with respect to the angle of the incident beam were measured.

RESULTS AND DISCUSSION

Effect of initial Dye Concentration

The effect of initial dye concentration shows that adsorption capacity decreases from 85.56% - 63.80% as dye concentration increases from 10 to 50 mg/l. the trend is that of the result of the progressive decreases in the electrostatic interaction between the Alizarin Yellow Dye and the adsorbent active sites. Moreover, this can be explained by the fact that less adsorption site were being covered as the dye concentration decreases. Besides, lower initial concentrations to an increase in the affinity of the Alizarin Yellow Dye towards the active sites. The decline in the adsorption capacity is due to the availability of smaller number of surface sites on the adsorbent for a relativity large number of adsorbing species at lower concentration. The experimental results of adsorption of Alizarin Yellow Dye on Al-Fe layered Hydroxide at various initial concentrations as shown in figure 2. Its reveal that, the actual amount of dye absorbed per unit mass of Al-Fe layered Hydroxide decreases with increases in dye concentration. Adsorption is maximum when the initial concentration of Alizarin Yellow dye were 10 mg/l. as the concentration increases, all the adsorption sites are being filled up and there remains unabsorbed dye, hence the decrease in percentage adsorption. This result is in favor of only monolayer coverage and suggests to the application of the Langmuir isotherm model. Since 95.7% adsorption occurs when the initial concentration was 10 mg/l, Al-Fe layered Hydroxide appears to be very effective adsorbent in removing dye (Lakshmi *et al.,* 2015).

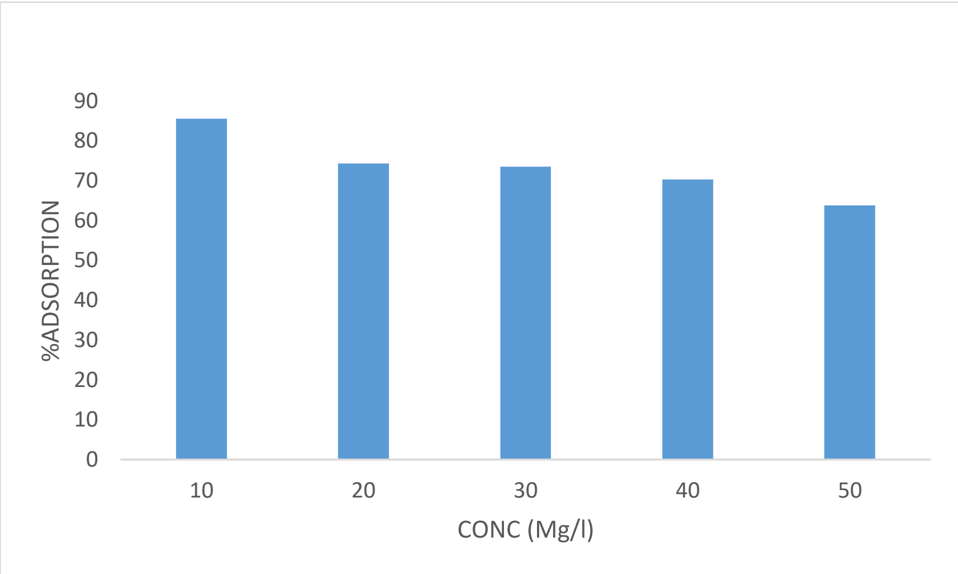

Figure 1: Effect of initial Dye Concentration

Effect of Contact time

The contact time has a vital role in adsorption studies, it helps in determining the maximum time required for the removal of Alizarin Yellow dye by Al-Fe layered hydroxide. It has been observed that, the adsorption increases asthe contact time increased, the maximum adsorption was 90.80% at 50 minutes and 66.95% at 10 minutes. This result was in line with the work of (Rabia *et al.,* 2015).

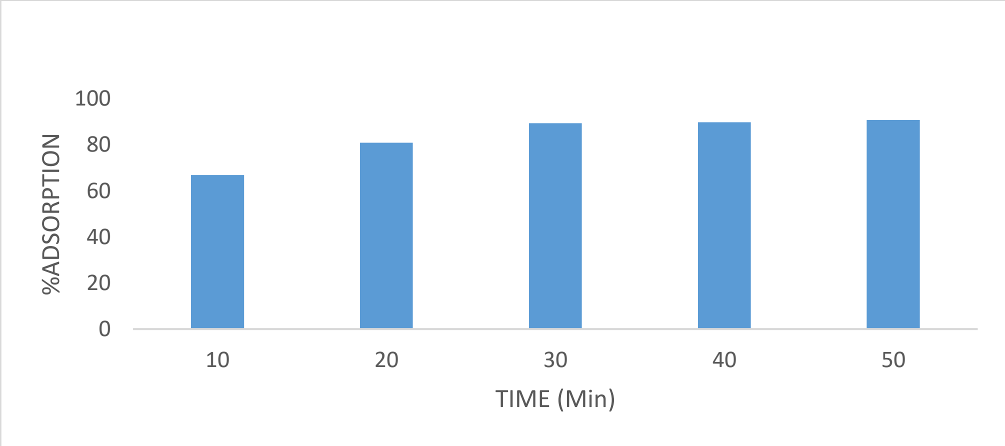

Figure 2: Effect of Contact time

Effect of adsorbent Dose
Adsorbent dose is an important factor in the determination of Dye uptake by adsorbent. It shows that, the percentage removal of alizarin yellow dye increases with increased in adsorbent dose. It has been observed that,amount of dye uptake increased from 56.0% -74.0% with 0.1 g- 0.5 g dose of Mg-Fe layered Hydroxide adsorbent. This because of the availability of more space site for the adsorption to take place which is in line with the work of (Lakshmi *et al.,* 2015) and (Rabia *et al.*, 2015).

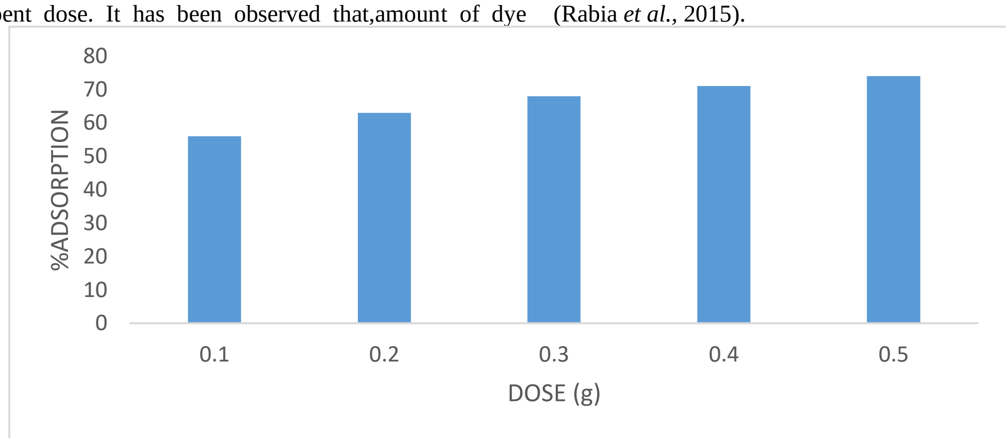

Figure 3: Effect of adsorbent Dose

Fourier Transform infrared (FT-IR)
The FT-IR Spectra of Al-Fe layered Hydroxide in figure 1 showed Hydroxyl absorption band at 3625 cm^{-1}. Another absorption band with an unsaturated function at 1633 cm^{-1}correspond to C=O, while N-CH peaks was observed at 2806cm^{-1}.

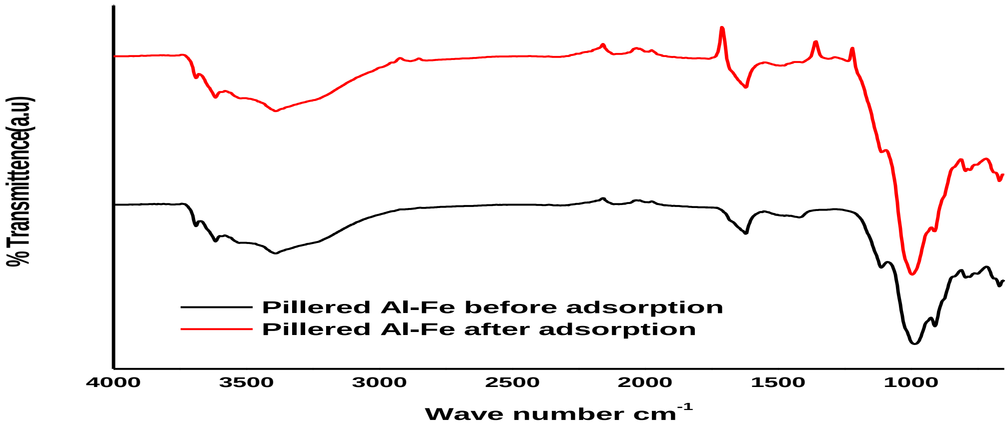

Figure 4: FT-IR Spectra of Al-Fe layered Hydroxide

Scanning Electron Microscopy (SEM)
Figure 5 a and b shows the SEM Micrograph of Mg-Fe layered Hydroxide before and after adsorption in which more tightly packed pattern pores were shows after adsorption and more rough surface before adsorption.

Figure 5**:** SEM Micrograph of Al-Fe layered Hydroxide before (a) and (b) after adsorption

X-ray Diffraction Spectroscopy (XRD)

Figure 6, Showed XRD Studies of the adsorbent Al-Fe layered Hydroxide before and after adsorption of dye, were carried out using Bruncker X- ray diffractometer 40 V/ 30 mA.Before adsorption, many peaks were observed that are attributed for two responses before adsorption Quartz (20.7^0, 26.5^0, 50^0) and Magnesiochloritiod at (19.7, 42.4^0, 59.8^0 and 67.9^0) and also after adsorption there are manypeaks with only two response of Quartz (20.7^0, 26.5^0, 50^0) and Chloritiod (19.7, 34.6^0, 68^0).The XRD result confirmed the pillaring was done successively by Mg-Fe layered hydroxide. Therefore, the diffraction reflection corresponding to Al-Fe layered hydroxide, verifying that the crystalline structure was not altered after pillaring process. This observation was also in line with the work of (Baloyi *et al.*, 2018).

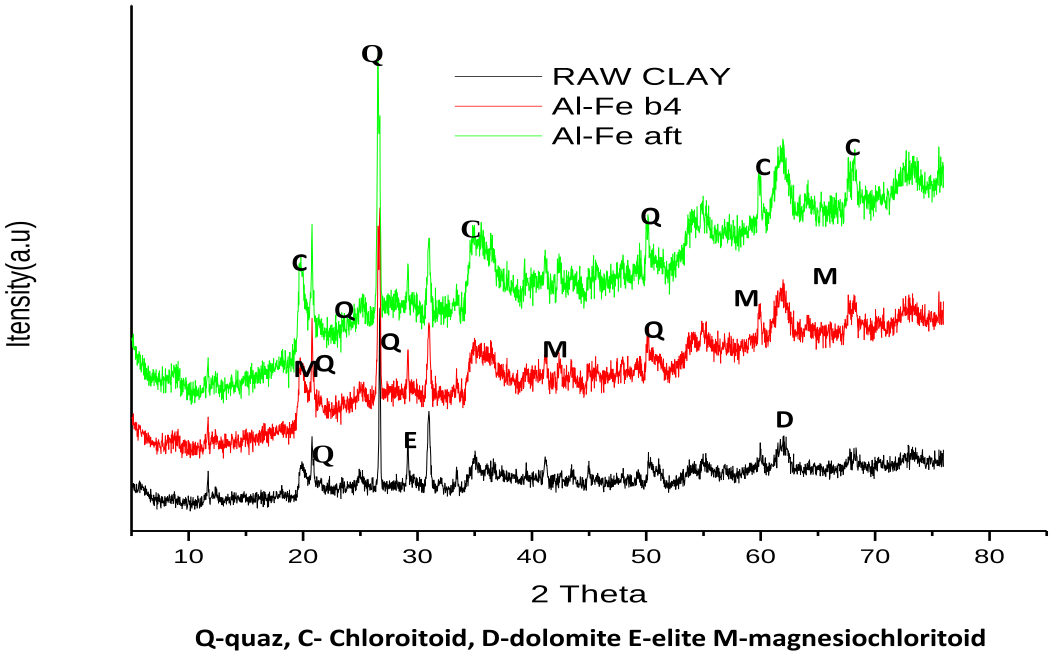

Figure 6: Al-Fe layered hydroxide before and after adsorption

Adsorption Isotherms

The Freundlunch Isotherm assumes heterogeneous surface with a non-uniform distribution of adsorption and describes the adsorption process as the ratio of the amount of solute adsorbed onto adsorbent to residual concentration is not the same at different concentration. It's in liner form is expressed by the following equation.

$$\log qe = \log KF = \frac{1}{nlog} Ce \qquad (2)$$

Where K_f and n are the freundlunch constant which predict the amount of dye adsorbed per gram of adsorbent at equilibrium concentration and strength of the adsorption process, respectively. It was well known that values between 0.1 and 1.0 indicate beneficent of adsorption and also, high Kf values shows the excellent adsorption.

The Langmuir model assumes that all the sites are identical, contain one molecule and are all energetic and sterical. The linear formof the Langmuir isotherm described by equation

$$Ce/_{qe} = \frac{1}{K1QM} + Ce/_{q} \qquad (3)$$

Where qe is the amount of the dye adsorbed(mg/g) toward hydrogen, qm is the maximum adsorption capacity relating for Langmuir adsorption(mg/g), and Ce is the residual concentration of dyes at equilibrium (mg/L) and KL is Langmuir adsorption constant (Abdelhalim *et al.*,2015).

Table 1: Adsorption Isotherm

Adsorption parameter	Parameters	Al-Fe LH
Langmuir	K (Lmg^{-1})	320
	R_l	1.561
	Qm (mgg^{-1})	625
	R^2	0.986
Freundlich	n_f	6.784261
	K_f	7689534
	R^2	0.0646

It can be seen from the Table 1.1 that the values for adsorption of Alizarin yellow dye ontoAl-Fe layered hydroxide satisfied Langmuir Isotherm with R^2 value of 0.986. The amount of dye adsorbed per gram of adsorbent at equilibrium concentrationqmvalue 625 shows the excellent adsorption and R_f value 1.561 indicate beneficent of adsorption.

CONCLUSION

This study shows that Al-Fe layered Hydroxide with bentonite clay was successfully prepared by calcinations at 500 ^{O}C and could be used as an effective adsorbent for removal of alizarin yellow dye in aqueous solution. The adsorption is found to be initial concentration, time and dose dependent. In addition, the correlation of Langmuir adsorption isotherm fits the experiment data most accurately. It was determined that the maximum adsorption capacity is 95.50% making it is an effective adsorbing material.Hence making it useful for the economic treatment of wastewater containing alizarin yellow dye. The results obtained from SEM, FI-IR and XRD indicate that adsorption of alizarin yellow dye on Al-Fe layered Hydroxide was confirmed.

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