



Original Article

## Removal of Acid Red 88 from an Aqueous Solution Using Kaolinite Clay by Adsorption Process.

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Dyes are hazardous contaminants normally found in industrial wastewaters necessitating costly and complex technologies for their removal. In this study, clay was used as an adsorbent in the removal of anionic acid red 88 (AR 88) dye from industrial wastewaters by adsorption. The dye removal efficiency and adsorption capacity of clay was studied by batch experimentation with respect to parameters of dye initial concentration, clay dosage, contact time, and pH. The dye removal efficiency of AR 88 improved with increasing dosage ( $r = 0.9826$ ,  $p < 0.05$ ), contact time ( $r = 0.9821$ ,  $p = 0.000683$ ) and decreased with increasing initial concentration ( $r = -0.9168$ ,  $p = 0.02844$ ) and pH ( $r = -0.9666$ ,  $p = 0.007297$ ) of the solution. At initial AR dye concentration of 100 mg/L, the removal efficiency and adsorption capacity of 89.7% and 4.38 mg/g, respectively were achieved after equilibrium time of 30 minutes under the optimum conditions of pH 2 and clay dosage 20 g/L. The study thrived in applying an efficient and low-cost adsorbent that could be used as a substitute to high-cost conventional commercial adsorbents in treating industrial wastewater with AR dye coloration.

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## INTRODUCTION

There is a continuous growth of textile, food, cosmetics, paper, plastics, printing, medicine, and other industries that demand the colouring of their products. At present, there are over 9000 dyes types included in the colour index for various chemical applications in the colouring industries (Q. Wang et al., 2020). Acid red 88 (AR88) is a synthetic anionic red azo dye commonly employed by textile and other industries associated with colouring their products (Abdi et al., 2019). This huge consumption of these synthetic dyes and water by industries as their main raw materials results into a high pollutant load for produced effluents. The discharge of these industrial dye effluent into the environment is associated with environmental pollution like endangering of aquatic life and ecosystem and deterioration of the quality of water (Yusof et al., 2020).

Similarly, these synthetic dyes threaten human health, for example, they are mutagenic, carcinogenic, and poisonous (Thirumoorthy & Krishna, 2020; Q. Wang et al., 2020). Additionally, undesirable colour changes caused by these dyes of industrial effluent can impede sunlight dispersion into fresh water bodies, dissolved oxygen depletion in the water resulting into hinderance to photosynthetic activities by aquatic plants (Mcyotto et al., 2021). Hence, it is absolutely necessary to remove these dyes from industrial wastewater before being discharged into the environment.

Current treatment methods for removing dyes from industrial wastewater are miscellaneous and include biological, chemical, and physical techniques. Among the widely used methods include; adsorption (Melnyk et al., 2021; Q. Wang et al., 2020; Yusof et al., 2020) photocatalytic oxidation (Badvi & Javanbakht, 2021; Chaker et al., 2021; S. Wang et al., 2020), coagulation (Feng et al., 2021;

Mcyotto et al., 2021), membrane technologies (H. Liu et al., 2020; Semiz, 2020), biodegradation (Paz et al., 2017; Shoukat et al., 2019), ion exchange (Joseph et al., 2020), and electrochemical methods (Afanga et al., 2020; J. Wang et al., 2020). Different hybrid systems of activated sludge, electrochemical oxidation coagulation, and other treatment methods have also similarly proven high efficiency in the treatment of dye industrial wastewater (Gholizadeh et al., 2020; Mukhlis et al., 2020; Saad et al., 2020; Y. Wang et al., 2020). Due to limitations of these treatment methods in terms of high cost and difficulty in operation, adsorption emerges to be the most suitable treatment method for dye effluents due to its effectiveness in removing different types of pollutants, simple design, and low operating costs associated with it.

Several low cost and readily available adsorbents on the market like rice husk (Bhatti et al., 2020), rice straw (Q. Liu et al., 2020), *Zea mays* cob (Singh et al., 2017), fish scales (Kabir et al., 2019), orange peels (AbdurRahman et al., 2013), chitosan (Sarkar et al., 2012), activated carbon (Kuang et al., 2020), sawdust (Suganya et al., 2017), carboxymethylcellulose-clay composite (Sirajudheen et al., 2020), sand (Sharma et al., 2019), citrus Limetta Peel (Singh et al., 2017), juncus effusus (Xia et al., 2020), carbohydrate polymeric adsorbent of rice flour (RF) (Munjur et al., 2020), carbohydrate polymeric adsorbent of wheat flour (WF) (Hasan et al., 2021), graphene Oxide (Almoisheer et al., 2019), and starch-based nanocomposites (Li et al., 2020), have been applied in the removal of dye from industrial wastewater. However, it is crucial to identify a reliable and low-cost alternative adsorbent for sustainable adsorption treatment of industrial dye wastewater.

Clay is a phyllosilicate material located in most parts of Egypt, it has a wide range of applications

due to its physicochemical properties such as ion exchange capabilities, high surface area, and consequently they are used for adsorption of dyes (Belachew & Bekele, 2020). Natural clays are gaining importance as low-cost adsorbents because of their abundant availability, easy accessibility and high adsorption capabilities for polar molecules and cations. The net negative charges on clay facilitate the adsorption of basic dyes from aqueous solutions (Akar & Uysal, 2010). Natural clays exhibit a negative structure charge, which facilitates it to adsorb positively charged dyes but induce a low adsorption capacity for anionic dyes (Dikmen et al., 2015). For instance, Belachew and Bekele (2020) applied bentonite clay in the enhanced adsorption of Congo red dye from an aqueous solution and achieved up to 94.9% percentage removal. Similarly, Khan (2020) assessed the potential of natural Saudi red clay on the removal of methylene blue dye from aqueous solution by adsorption and

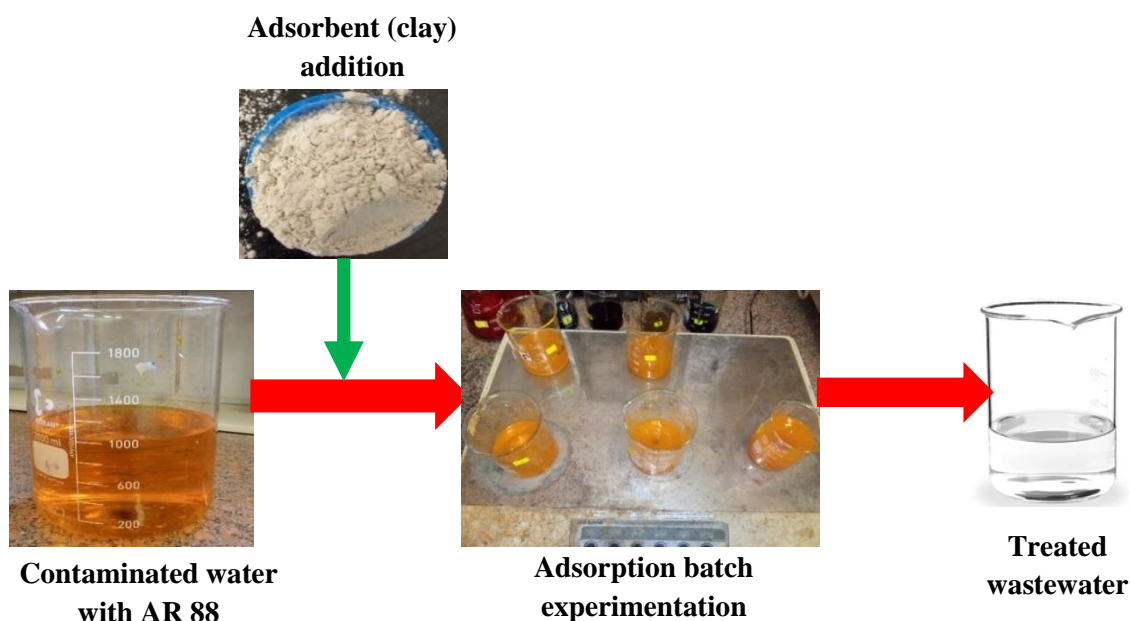
results showed that it could achieve up to 95% removal.

In the present study, clay has been assessed as an environmentally friendly and economically feasible adsorbent in removing acid red 88 (AR 88) dye from an aqueous solution by the adsorption method. The adsorption of acid red 88 was investigated under batch mode adsorption and the effects of factors; adsorbent dose, pH, initial dye concentration, and contact time were investigated. The performance of clay as an adsorbent has been compared with other reported works.

## METHODOLOGICAL APPROACH

This section entails the methodological approach and materials adopted in this research as adsorbents and adsorbate, their procedure of preparation and steps for conducting adsorption batch experiments (see *Figure 1* below) as well as the adopted analytical methods in the study.

**Figure 1: Steps for conducting adsorption batch experiments**



## Materials and Chemicals

Clay was used in this study as obtained in its raw form without any chemical or physical made

modification on it. All reagents used were analytical grade and were not subjected to any additional form of modification. Acid red (AR) 88 dye was obtained

from a dye manufacturing company in Borg Arab Alexandria. Distilled water was used to prepare all aqueous solutions in the study. Clay was acquired from a ceramic factory in Borg Arab, Alexandria, Egypt.

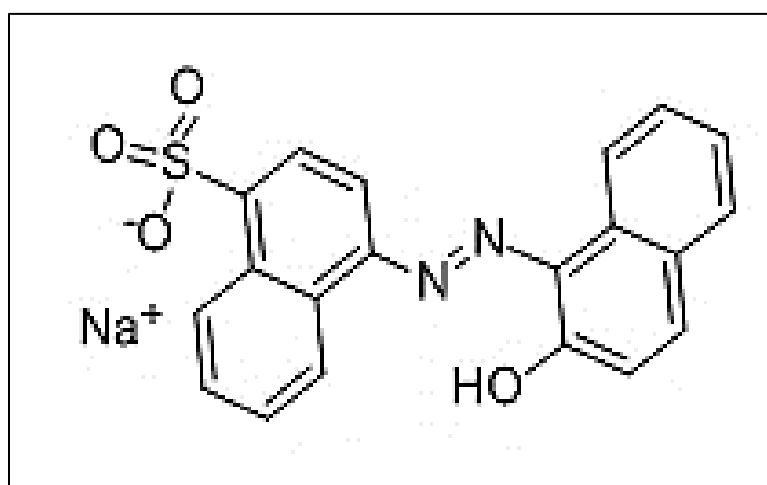
### Adsorbent Sample

The adsorbent used in this study was clay as obtained in its raw form without any modification on it.

### Preparation of Adsorbate (AR Dye Stock Solution)

Acid red 88 (*Figure 2*) was used in this study as an adsorbate. Acid red 88 stock solution of 5000 mg/L was prepared by dissolving 0.5 g of the dye into 1000 ml distilled water. Subsequent dilutions followed this to prepare the appropriate concentrations as required using distilled water. pH was adjusted by adding 1M NaOH solution and 1M HCl acid solution.

**Figure 2: Chemical structure of Acid red 88**



### Adsorption Experiment

Batch adsorption experiments were performed in 250 ml beakers at a stirring rate of 150 rpm and room temperature of 25 °C. A one factor at a time (OFAT) statistical method was used to study the effect of operational parameters of adsorbent dosage, initial dye concentration, pH, and contact time on the removal of AR dye from aqueous solution within a time interval of 60 mins.

In the first experiment, the adsorbent dosage was adjusted from 1 to 28 g/L at the pH of 4.5 and initial dye concentration (C<sub>0</sub>) 100 mg/L. In the second experiment, the aqueous solution pH was varied between 2 to 10 at an adsorbent dosage of 20 g/L (optimum from the first experiment), and initial concentration = 100 mg/L. The third experiment was conducted by varying the contact time from 0-

60 mins at the optimum pH and adsorbent dosage as obtained from preceding experiments. The fourth experiment was carried out by varying the contact initial AR dye concentration from 100-500 mgL<sup>-1</sup> as adopted by Akar and Uysal (2010) at the optimum pH, adsorbent dosage, and contact as obtained from earlier experiments. Each time, 100ml AR dye wastewater sample was subjected to continuous mixing using a magnetic stirrer. The adsorbent was separated from the solution by centrifugation at 6000 min<sup>-1</sup> for 10 minutes.

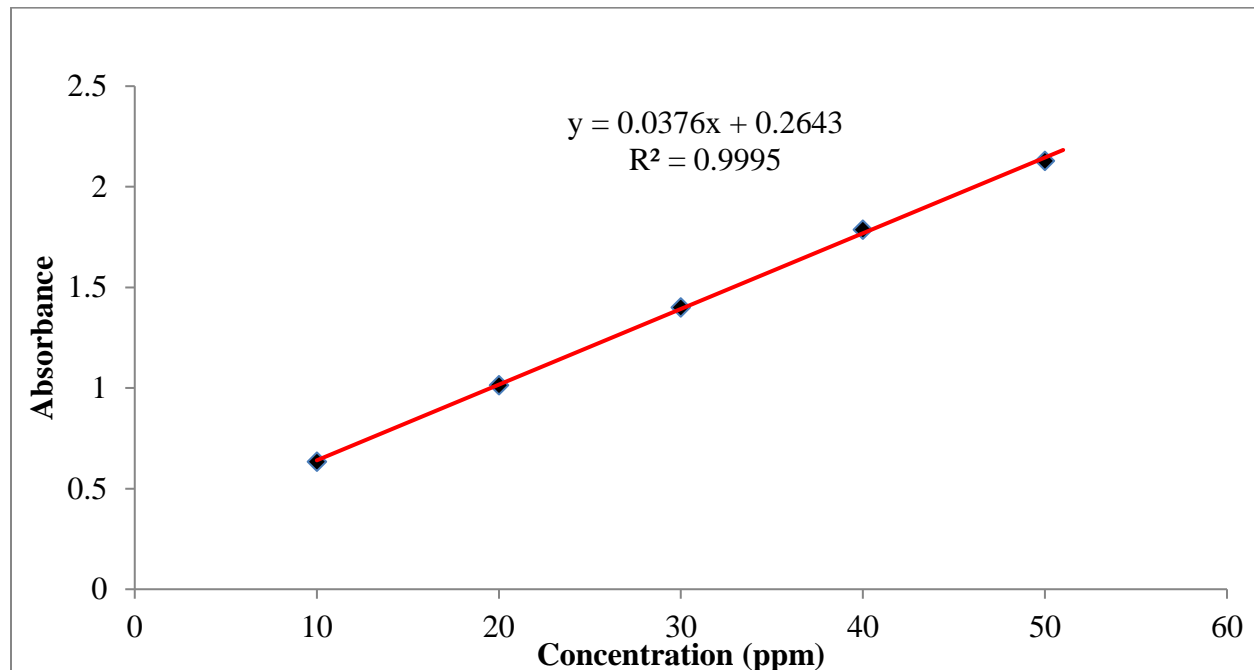
### Analytical Methods

The absorbance of AR solution was measured using a JASCO V-630 UV-visible spectrophotometer (Tokyo, Japan) at a wavelength  $\lambda_{\text{max}} = 450$  nm. The concentration of AR dye was obtained through a calibration curve describing a relationship between

solution concentration (10 – 50 mgL<sup>-1</sup>) and absorbance of AR (Figure 3). The final concentration of dye was estimated using the equation ( $y = 0.0376x +$

0.2643) from the calibration curve. The pH was measured using a digital pH meter JENWAY 370 (Shanghai, China).

**Figure 3: Calibration curve for Acid red 88 dye**



The adsorption figures were used to estimate the AR removal efficiencies (*Equation 1*) and the adsorption capacities of clay (*Equation 2*).

$$E = \frac{(C_i - C_e)}{C_i} \times 100\% \quad (\text{Kuang et al., 2020}) \quad (1)$$

$$q_e = \frac{V(C_i - C_e)}{m} \quad (\text{Melnyk et al., 2021}) \quad (2)$$

Where **E** is the removal efficiency; **q<sub>e</sub>** is equilibrium adsorption capacity and it represents the quantity of dye per unit mass of adsorbent (mg·g<sup>-1</sup>); **C<sub>i</sub>** and **C<sub>e</sub>** are the initial and equilibrium concentration of AR dye in the solutions respectively (mg/L); **V** is the solution volume (L); and **m** is the amount of adsorbent (g).

## RESULTS AND DISCUSSION

This section explains in detail the outcomes of the studied clay adsorbent towards its ability to remove AR dye from an aqueous solution. Additionally, the chapter discusses the effect of pH, adsorbent dosage, initial dye concentration and contact time on the performance of clay as an adsorbent.

### Effect of Adsorbent Dosage

The effect of adsorbent dosage was examined in order to establish the optimum dosage. The effect of adsorbent dose was studied by 100 mL of AR concentrations 100 mgL<sup>-1</sup> under different adsorbent doses (1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22, 24, 26, and 28 g) at a pH of 4.5.

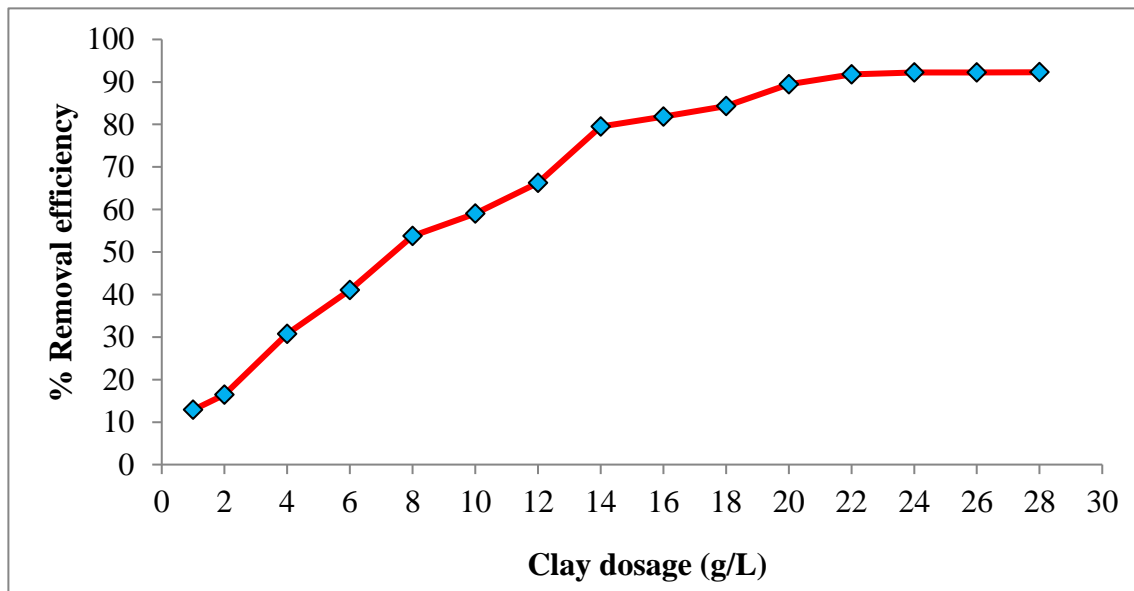
AR dye removal efficiency (Figure 4a) was significantly improved from 12.9 to 89.5% with increasing clay dosage from 1 to 20 g/L,

respectively ( $r = 0.9826$ ,  $p < 0.05$ ). This increase is due to the increased mass of adsorbents resulting into an increased total number of active sites (Kumi et al., 2020). The increase in clay dosage from 20 to 28 g/L attained an insignificant improvement in dye adsorption efficiency ( $r = 0.7925$ ,  $p = 0.109827$ ). Beyond a saturation adsorbent dosage (20g/L), the available number of AR dye molecules in solution was not enough to completely combine with all effective adsorption sites on the clay, resulting in a surface equilibrium state and a reduction in the adsorption capacity per unit mass of adsorbent. This

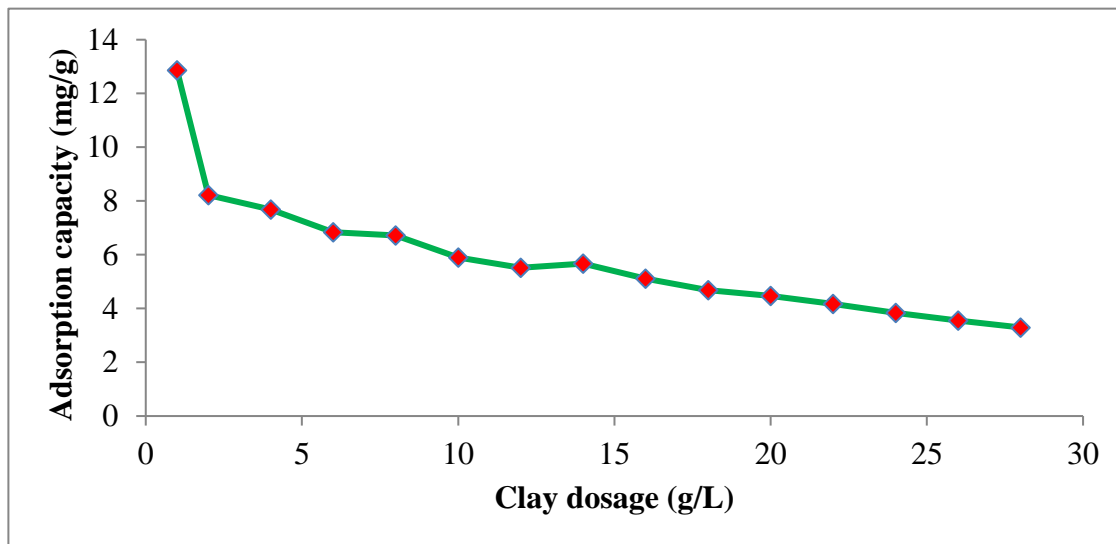
behaviour could be because any extra addition of adsorbent dosage beyond the optimum value causes an overlapping of adsorption sites, leading to the deficient use of functional groups (Fawzy et al., 2018). However, the adsorption capacity (Figure 4b) had negative correlation ( $r = -0.872733$ ,  $p < 0.05$ ) with the adsorbent dose, which could be because some of the adsorption pores remained unoccupied during the adsorption process (Kuang et al., 2020). Accordingly, clay dosage of 20 g/L was chosen for further studies.

**Figure 4: Effect of adsorbent dosage**

(a)



(b)



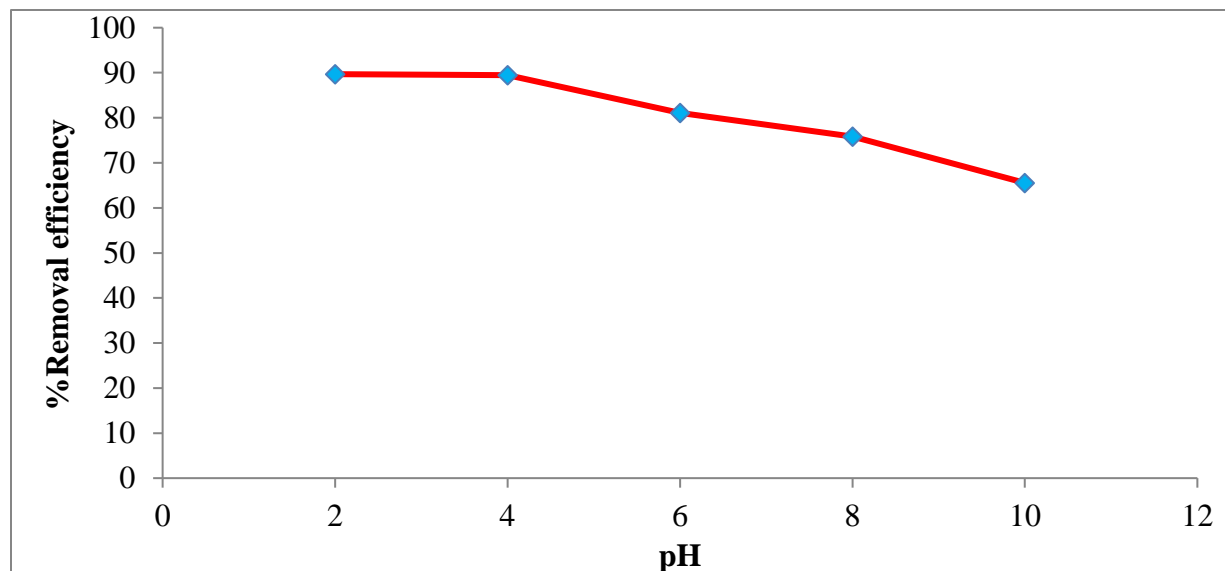
*pH = 4, initial dye concentration = 100mgL<sup>-1</sup>, contact time = 60 min, mixing speed =150 rpm) (a) The % removal efficiency; (b) the adsorption capacity*

**Effect of Solution pH**

The pH of a Solution is one of the important parameters governing the adsorption performance

of a given adsorbent (Akar & Uysal, 2010). The variation in Acid red dye percentage removal efficiency of clay is shown in *Figure 5*.

**Figure 5: Effect of solution pH on Acid red dye removal efficiency**



*(Adsorbent dosage 20g/L, initial dye concentration = 100mgL<sup>-1</sup>, contact time = 60 min, mixing speed =150 rpm)*



The acid dye removal efficiency was observed to decrease notably with increasing pH. The removal efficiency decreased from 89.7% to behaviour 65.6 % with increasing pH values from 2.0 to 10.0 respectively ( $r = -0.9666$ ,  $p = 0.007297$ ). However, insignificant removal efficiency was observed with an increase in the pH from 2 to 4 ( $p = 0.32025$ ). High adsorption capacity observed in the acidic pH values (prevailing of  $H^+$  ions) could be due to the electrostatic attraction forces between dye anions and positively charged parts on the clay surface.

The abundant presence of  $OH^-$  ions in basic solution due to an increase in pH can be competitive for available active sites with the dye anions for effective adsorption (Zhu et al., 2010). Chen & Zhao (2009) observed that negatively charged

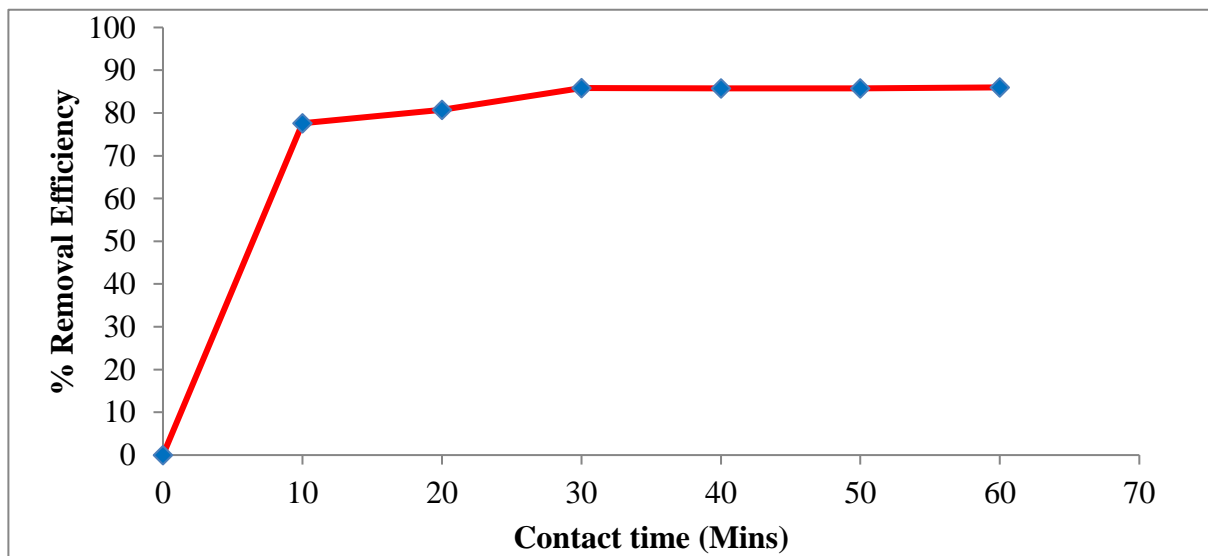
surface site on the clay does not favour the adsorption of anionic dye due to electrostatic repulsion. Accordingly, an optimum pH of 2 was adopted for subsequent experimental work.

### Effect of Contact Time

*Figure 6(a)* shows the variation removal efficiency with contact time while *Figure 6(b)* shows the variation of adsorption capacity of AR on clay with contact time. AR dye adsorption efficiency significantly increased to 77.6% in the first 10 minutes of agitation ( $r = 0.9821$ ,  $p = 0.000683$ ). However, a relatively constant removal performance was observed with an incremental increase in the contact time above 30 minutes ( $r = 0.8779$ ,  $p = 0.317806$ ).

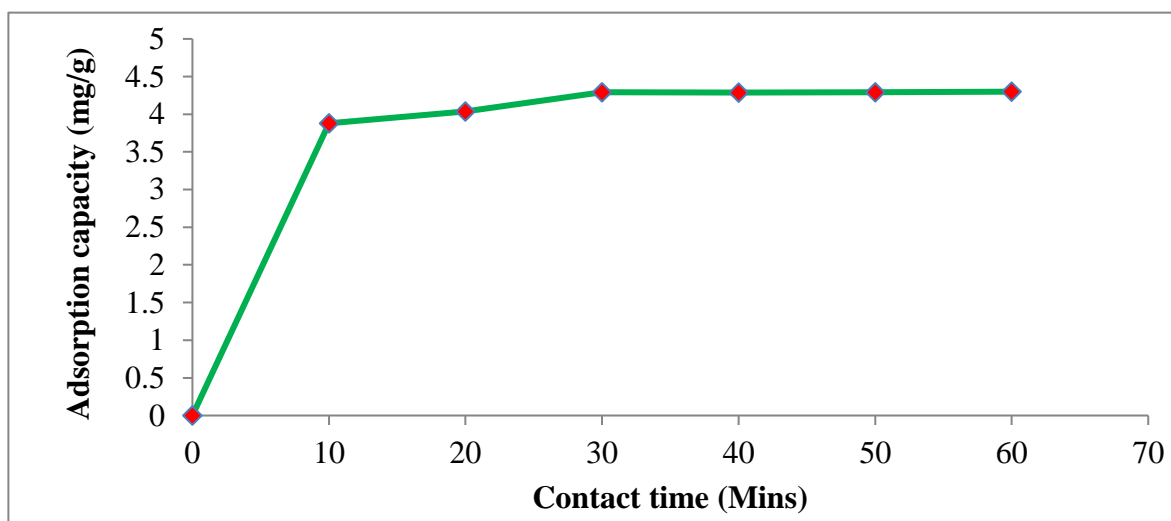
**Figure 6: Effect of contact time**

(a)





(b)



(Adsorbent dosage 20g/L, initial dye concentration = 100mgL<sup>-1</sup>, mixing speed =150 rpm, pH = 2). (a) The % removal efficiency; (b) the adsorption capacity

In the initial stages, the high number of unoccupied sites results into a rise in the concentration gradient between the dyes molecules in aqueous solution and that on the clay adsorbent surface. There was no substantial adsorption of AR dye by clay with increasing the agitation time from 30 to 60 min. Thus, the adsorption capacity could be described as a fast-initial rate followed by a slower adsorption rate. The slower adsorption rate could be due to the accumulation of AR molecules on the binding sites at equilibrium, causing saturation of the clay surface (Fawzy et al., 2018). This may also be attributed to the availability of the positively charged surface of the adsorbent which caused rapid electrostatic adsorption of anionic dye AR from the solution at a pH of 2 (Khan, 2020). Similarly, Akar and Uysal (2010) found that untreated clay achieved  $98.10 \pm 0.34\%$  removal efficiency of AR 88 dye within 15 min at pH 2.0 in the batch mode.

#### Effect of Initial AR Dye Concentration ( $C_0$ )

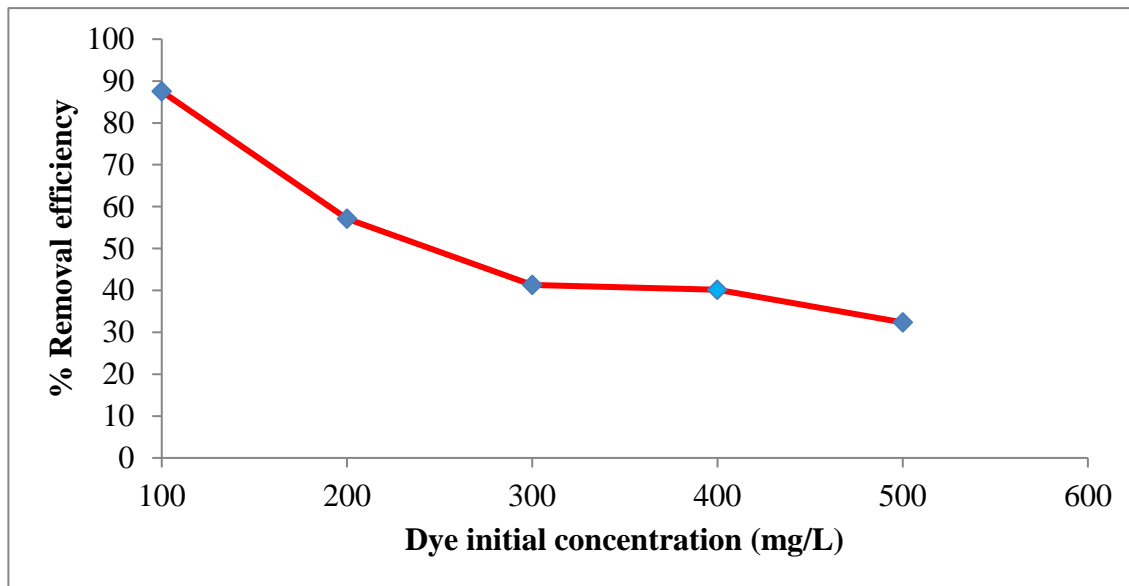
The effect of different initial dye concentrations (100, 200, 300, 400, 500 mgL<sup>-1</sup>) on the % removal efficiency and adsorption capacity was studied using the same mass of clay (20g), dye solution pH

of 2, and mixing speed 150 rpm). As shown in *Figure 7(a)*, the AR dye removal efficiency substantially decreased from 87.5 to 32.4% with increasing initial concentration ( $C_0$ ) from 100 to 500 mg/L, respectively ( $r = 0.9168$ ,  $p = 0.02844$ ). This result indicated that AR dye molecules were hardly removed at high initial concentrations. At low  $C_0$ , the ratio of the available binding sites to the adsorbate molecules was high, enabling the interaction of AR molecules with the clay surface thus achieving high removal efficiency. On the contrary, the decrease in removal efficiency at high initial concentration could be attributed to the lack of sufficient adsorption sites to accommodate further dye molecules present in the aqueous solution (Kabir et al., 2019).

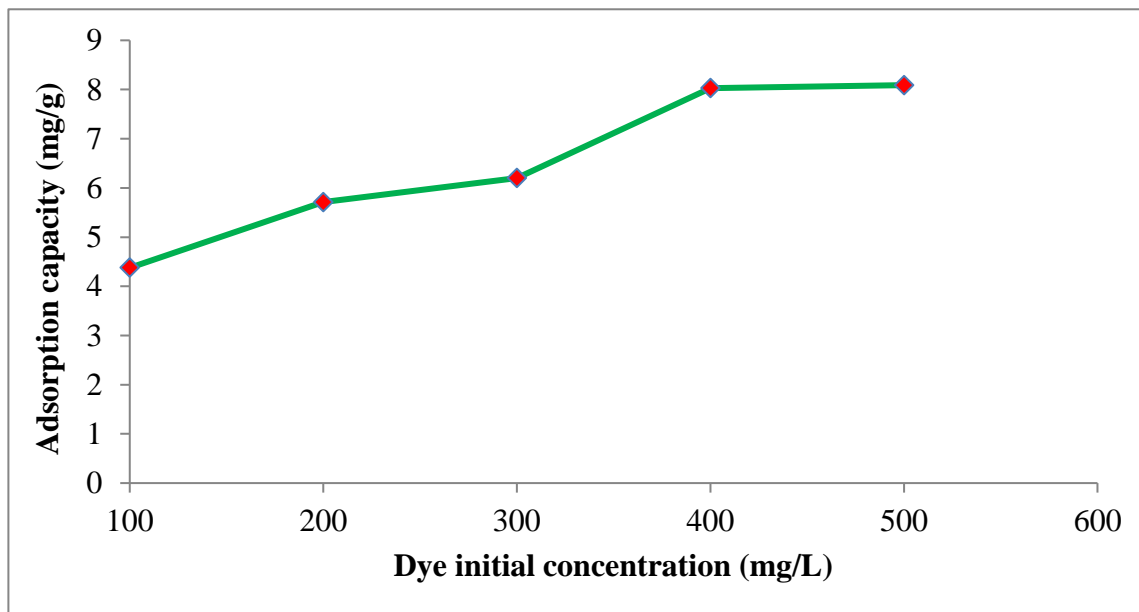
Similarly, the adsorption capacity of clay significantly increased from 4.38 to 8.09 mg/g when the initial concentration was raised from 100 to 500 mg/L, respectively ( $r = 0.9168$ ,  $p = 0.02844$ ) as shown in *Figure 7(b)*. An increase in the diffusion of dye in the aqueous solution results into successive collisions between the dye molecules and clay particles (Kumi et al., 2020).

**Figure 7: Effect of dye initial concentration**

(a)



(b)



(Adsorbent dosage 20g/L contact time = 30 min, mixing speed =150 rpm, pH = 2). (a) The % removal efficiency; (b) the adsorption capacity

**Performance Evaluation of Clay in the Acid Red Dye Removal**

To evaluate the performance of clay as an adsorbent for the treatment of industrial wastewater with AR 88 dye colorization, the results obtained from this study were compared to the performance of other

adsorbents (Calcined alunite, raw alunite, lemna minor biomass, magnetic graphene oxide/cationic hydrogel nanocomposite, porous biochar-supported Fe-Mn composite and persulfate (PS), saw dust,

polyamine-modified magnetic graphene oxide, graphene nanosheet, and chitosan-polyethyleneimine synthesized with calcium chloride).

**Table 1: Comparison of various adsorbents for AR 88 dye removal**

Adsorbent	Efficiency (%)	Dosage (g/L)	pH	Reference
Porous biochar-supported Fe-Mn composite and persulfate (PS)	98.84	Fe-Mn/BC =1 PS = 2	2	(Chen et al., 2020)
Polyamine-modified magnetic graphene oxide	73	1	3	(Abdi et al., 2019)
Calcined Alunite	98	0.2	2	(Tunali Akar et al., 2013)
Raw alunite	93	0.3	2	(Tunali Akar et al., 2013)
Lemna minor biomass	98	6	3	(Balarak et al., 2015)
Magnetic graphene oxide/hydrogel nanocomposite	95	0.3	3	(Dong and Wang, 2016)
Saw dust	84.46	30	3	(Modirshahla et al., 2010)
Graphene nanosheet	84.5	30	3	(Modirshahla et al., 2010)
Chitosan-polyethyleneimine synthesized with calcium chloride	87.9	0.4	3	(Yusof et al., 2020)
Clay	89.7	20	2	This study

The results for each of the respective materials are summarized in table 1, showing the removal efficiency of the AR dye at optimum condition of dosage and pH. The clay adsorbent shows a higher AR dye removal efficiency than saw dust, polyamine-modified magnetic graphene oxide, graphene nanosheet, and chitosan-polyethyleneimine synthesized with calcium chloride and comparable to raw alunite and magnetic graphene oxide/cationic hydrogel nanocomposite. It can thus be construed that the performance of clay is satisfactory and comparable with other alternative adsorbents.

## CONCLUSION

Clay adsorbent can effectively achieve a good performance removal of AR dye from wastewater

minus any surface modification. This study indicated that the adsorption process of AR as an adsorbate and clay as an adsorbent is largely influenced by initial pH, adsorbent dosage, initial dye concentration, and contact time. The removal efficiency decreased with increase in the initial dye concentration. Also, the adsorption was found to be higher for lower pH and the optimum pH for the aqueous solution was 2, achieving AR dye removal efficiency of 87.5% under initial concentration 100mg/L, clay dosage 20 g/L, mixing speed of 150 rpm, and equilibrium time of 30 minutes. Therefore, it may be concluded that this cheap and environment friendly clay can be used as a replacement to high-cost conventional commercial adsorbents including activated carbon, silica gel, zeolites, and polymeric organic resins.

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