



Research Article

Adsorptive Performance of Termite Mound Clay for Removal of Eosin Yellow Dye from Aqueous Solution

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ABSTRACT

This study investigates the adsorption capacity of termite mound clay (TMC) for the removal of eosin yellow (EY) dye from aqueous systems. Scanned Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) were used along with some physicochemical properties to characterize the termite mound clay. The adsorption experiment was studied by batch adsorption method with the influence of parameters which include initial dye concentration, contact time, dosage, pH and temperature. The surface of TMC showed functional groups similar to kaolin with flake-like and rough morphology. Efficiency of 99.12% (12.39 mg/g) was achieved at a pH of 3, indicating a pH-dependent adsorption process. The fitness of the equilibrium data into Langmuir isotherm ($R^2=0.9446$) with maximum monolayer adsorption capacity of 23.47 mg/g revealed a monolayer coverage of EY on the surface of TMC. The uptake of the dye molecules was rapid within 10 min and the kinetic study was best described by pseudo-second-order model with a rate constant (k_2) of 0.271 g/(mg.min). Thermodynamically, the process required little or no heat as a rise in the temperature resulted to decrease in the adsorption capacity and efficiency of TMC on the removal of EY dye. TMC showed potential as a cost-effective adsorbent for the treatment of dye-contaminated water.

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INTRODUCTION

Dye is a coloured organic compound that chemically bonds to the substrate to which it is being applied. It has received wider applications in various areas including textile, leather, paper, paint and food industries (Kandisa and Saibaba, 2016). Effluents from these dye-consumed industries are being released into the aqueous system. These dyes are more resistant to biodegradation in the environment, especially in water bodies because of their complex structures. Consequently, dye is one of the major pollutants of great environmental concern (Bello *et al.*, 2022; Iqbal *et al.*, 2021). Since these dyes are extremely harmful to microbial populations and even carcinogenic to mammals, it is vital to remove them from industrial effluents prior to their discharge into water bodies (Kandisa and Saibaba, 2016).

Eosin yellow (EY) is a water soluble, acidic and anionic azo dye which belongs to fluorescein class (Rahman, 2017). It has been widely utilized in biological staining due to its ability of strong absorption by red blood cells. It is also used to be applied in food before it was banned due to its toxicity (Derayea and Nagy, 2018; Mittal *et al.*, 2013). It is highly toxic to system of living organism. The dye is said to cause allergic reaction to skin and eye, affect several organs like liver, kidney and thereby results in many diseases. Its metabolites are also known to be carcinogenic (Mittal *et al.*, 2013; Tukaram Bai *et al.*, 2019).

A variety of methods categorised as physical, chemical and biological have been employed for the removal of dyes from the contaminated water

bodies. As a result of the non-biodegradable and recalcitrant nature of dyes to chemical and biological methods, adsorption which is a physical approach is successful at removing dye (Alam *et al.*, 2021). While modern methods like membrane separation and photo-degradation are fairly good at treating contaminated water, their high operational costs in terms of electricity and the need for periodic cleaning prevent their widespread adoption. Therefore, adsorption is frequently used for the treatment of dyes containing wastewater and is thought to be a cost-effective and easy design process (Akbal, 2005; De Gisi *et al.*, 2016). The choice of a low-cost adsorbent is important in making adsorption an inexpensive method. Low-cost sorbents can be prepared from locally and abundantly available materials like industrial by-products, sludge, agricultural and household wastes, sea materials, clay and clay minerals (De Gisi *et al.*, 2016; Sadegh *et al.*, 2019).

Clay is a naturally occurring substance composed primarily of fine-grained minerals that is pliable at the appropriate water content and hardens when dried or burned (Nwosu *et al.*, 2013). Clay and clay minerals occur on the earth's crust abundantly. They are characterized by the following physicochemical properties such as crystalline structure, colloidal particle size, high surface area, and swelling capacity. It also has high sorptive/electric charge properties (Aboudi Mana *et al.*, 2017; Costanzo, 2001). Consequently, clay has been widely applied as an adsorbent for water treatment and as a catalyst or support catalyst in several organic syntheses (Nagendrappa, 2011; Shahadat *et al.*, 2021).

Although termites are among the most destructive pests in the world, some smallholder farmers in Africa who lack access to capital use termite mound soil, which improves soil fertility, as a substitute for NPK fertilisers (Beyene and Getu, 2021). Termite mound is made up of clay, which consists of inorganic material minerals and water (Alabadan *et al.*, 2016). Its plasticity is further enhanced by the secretion from the termite during the building process (Assam *et al.*, 2016). This has facilitated its alternative application in sustainable brick production (Legese *et al.*, 2021). The process of building termite mounds also substantially modifies soil physicochemical properties and can change the mineralogy of clay. The elemental analysis of the mounds as revealed by literature are majorly iron, aluminium, silicon and titanium as well as their oxides which is likely to be responsible for their adsorptive property (Ayanda *et al.*, 2019; Fufa *et al.*, 2014). Various termite mounds from different locations have been extensively used as adsorbents for the elimination of heavy metals from aqueous solution (Abdus-Salam and Bello, 2015; Abdus-Salam and Itiola, 2012; Fufa *et al.*, 2014). Its experimental utilization in the recovery of dye-laden water has also been reported (Ayanda *et al.*, 2019).

However, the broad classification of dye into ionic (cationic and anionic) and non-ionic (vat and disperse) dyes calls for more experimental information about its removal from aqueous solution. It is against this backdrop that this study investigated the adsorptive performance of termite mound clay on water containing eosin yellow dye.

MATERIALS AND METHODS

Materials

Analytical grade chemicals were used and were prepared for experimentation using deionized water. The termite mound clay (TMC) was obtained as a bulk sample within the University of Ilorin premises and transported to the Laboratory for application.

Methods

Preparation of Eosin Yellow Dye as Adsorbate

The preparation of 1000 mg/L of eosin yellow dye was done by dissolving 1 g in 1000 mL of deionized water, from which other required concentrations were prepared. Three concentrations, 5 mg/L, 10 mg/L and 15 mg/L were scanned to obtain wavelength at maximum absorbance (λ_{max}) using Uv-visible Spectrophotometer (Jenway, 7305).

Preparation of Termite Mound Clay as Adsorbent

The termite mound clay used as adsorbent was ground to smaller particle sizes and sieved to 200 μm particle size. This raw powder termite mound clay was then stored for application.

Characterization

The functional groups and surface morphology of termite mound clay were determined using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) respectively. Other physicochemical properties which include moisture content, colour, texture, pH and bulk density were also investigated.

Adsorption Experiments

The batch adsorption experimental method was adopted to investigate the influence of pH, initial dye concentration, dosage, contact time and

temperature on the adsorption process. In a typical procedure, 0.2 g of termite mound clay (TMC) was added to 25 mL of eosin yellow (EY) dye, agitated on a mechanical shaker for 120 min, filtered and analysed for the remnant dye in the filtrate with a UV-Visible spectrophotometer. The operational conditions for the adsorption parameters are as follow: initial concentration of

$$q_e = \frac{C_o - C_e}{m} \times V \quad (1)$$

$$q_t = \frac{C_o - C_t}{m} \times V \quad (2)$$

$$\%_{adsorbed} = \frac{C_o - C_e}{C_o} \times 100 \quad (3)$$

Where C_o , C_e and C_t (mg/L) are the initial concentration, concentrations at equilibrium and concentration at any time (t) respectively; q_e and q_t (mg/L) are the quantities adsorbed at equilibrium and any time respectively; V (L) is the volume of the dye solution and m (g) is the mass of the adsorbent used (Bello *et al.*, 2022).

EY dye (50 to 250 mg/L), adsorbent dosage (0.1 to 0.4 g), contact time (5 to 120 min), and temperature (25 to 45 °C). The amount of dye adsorbed at equilibrium and at any time was determined using equations 1 and 2 respectively. Similarly, the percentage adsorbed (% adsorbed) was determined using equation 3.

Isotherm Study

In order to evaluate the equilibrium adsorption data for further understanding of the mechanism of EY removal onto the termite mound clay, Langmuir and Freundlich isotherm models were used.

Table 1: Isotherm equations

Isotherm model	Equation	Plot	Reference
Langmuir	$\frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m}$ $R_L = \frac{1}{1 + K_L C_o}$	$\frac{1}{q_e}$ vs $\frac{1}{C_e}$	(Chen, 2015)
Freundlich	$\log q_e = \log K_f + \frac{1}{n} \log C_e$	$\log q_e$ vs $\log C_e$	(Bello <i>et al.</i> , 2021)

where q_e (mg/g) is the quantity of EY dye adsorbed; q_m (mg/g) is the monolayer adsorption capacity; C_o and C_e (mg/L) are the initial and equilibrium dye concentrations respectively; K_L (L/mg) is the Langmuir constant; R_L is the dimensionless separation factor that indicates the type of Langmuir isotherm; k_f (mg/g) is the Freundlich constant and n is the empirical

parameter which is related to the sorption intensity.

Kinetic Study

The adsorption data from effect of time were subjected to pseudo-first-order and pseudo-second-order models to understand the kinetic mechanism of the process.

Table 2: Kinetic equations

Kinetic model	Equation	Plot	Reference
Pseudo-first-order	$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t$	$\log(q_e - q_t)$ vs t	(Pathania <i>et al.</i> , 2017)
Pseudo-second-order	$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e}t$	$\frac{t}{q_t}$ vs t	(Liu <i>et al.</i> , 2019)

where q_e and q_t (mg/g) are the amounts of EY dye removed at equilibrium and any time, t (min), respectively; k_1 (min^{-1}) is the pseudo-first-order rate constant and k_2 [$\text{g}/(\text{mg} \cdot \text{min})$] is the rate constant of pseudo-second-order adsorption.

Table 3: Thermodynamic equations

Model	Equation	Plot	Reference
Van't Hoff	$\ln k_c = -\frac{\Delta H^0}{RT} + \frac{\Delta S^0}{R}$	$\ln k_c$ vs $\frac{1}{T}$	(Abdus-Salam and Adekola, 2018)
	$k_c = \frac{C_{ads}}{C_e}$		
	$\Delta G^0 = \Delta H^0 - T\Delta S^0$		

where ΔG^0 (kJ/mol), ΔH^0 (kJ/mol) and ΔS^0 (J/mol.K) are changed in Gibbs free energy, enthalpy and entropy respectively; R (8.314 J/mol.K) is the gas constant; and T (K) is the temperature. K_c is the equilibrium constant; C_e and C_{ads} (mg/L) are the concentration of the dye in solution and the concentration on the adsorbent at equilibrium respectively.

Thermodynamic Study

Thermodynamic equations were applied to the data obtained from the effect of temperature to further understanding the adsorption process.

RESULTS AND DISCUSSION

Characteristics of Eosin yellow (EY) Dye Solution and Calibration Curve

Table 4 shows some characteristic properties of the eosin yellow (EY) dye solution. The wavelength at maximum absorbance (λ_{max}) obtained was 517 nm. It has been reported that EY exhibits strong absorption band in the visible region (Lihong *et al.*, 2009). The molar absorptivity (ϵ) obtained from the calibration curve using Beer-Lambert equation was 0.932 $\text{L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ with R^2 of 0.975.

Table 4: Characteristics of eosin yellow dye solution

Parameter	Value
Wavelength at maximum absorbance (λ_{max})	517 nm
Molar Absorptivity (ϵ)	0.932
R^2	0.975

Characteristics of Adsorbent

Table 5 presents some physicochemical properties of termite mound clay (TMC) used as an adsorbent. The physical appearance of the TMC is brown with a fine texture. The pH in the water was 6.7, a value close to neutrality. The moisture content of the TMC adsorbent was 12.0 %. The bulk density was 1.29 g/cm^3 which is within the range reported by Beyene and Getu, (2021) and Robert *et al.* (2007). The surface morphology of TMC by scanning electron microscopy (SEM) is shown in Figure 1. The morphology of the TMC is described with different irregular flake-like particles and pore distributions.

Figure 2 shows the surface functional groups obtained from Fourier Transform Infrared Spectroscopy (FTIR). The peaks at 3695.73 cm^{-1}

and 3620.51 cm^{-1} were attributed to Al-OH and OH inner hydroxyl respectively; which are typical characteristic peaks of kaolin (Saikia and Parthasarathy, 2010). The OH stretching of absorbed water was responsible for the slightly broadband at 3433.41 cm^{-1} ; while the H-O-H bending of water appeared at 1633.27 cm^{-1} . The observed peak at 1021.99 cm^{-1} was due to Si-O stretching. The band at 908.15 cm^{-1} was attributed to OH deformation linked to Al^{3+} . The bands at 787.71 cm^{-1} and 687.90 cm^{-1} were assigned to Si-O quartz. Al-O-Si or Fe-O and Si-O-Si bending could be responsible for the absorption band at 538.58 cm^{-1} and 467.64 cm^{-1} respectively. The assignments were done according to the reports of Ayanda *et al.* (2019) and Saikia & Parthasarathy (2010).

Table 5: Physicochemical properties of TMC

Properties	Observation
Colour	Reddish-brown
Texture	Fine
pH in water	6.7
Moisture content (%)	12
Bulk density (g/cm^3)	1.29

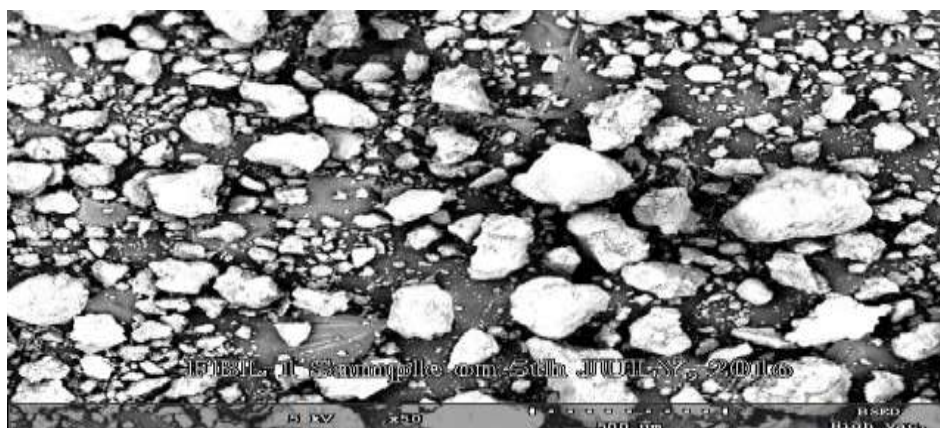


Figure 1: Scanning Electron Micrographs of TMC

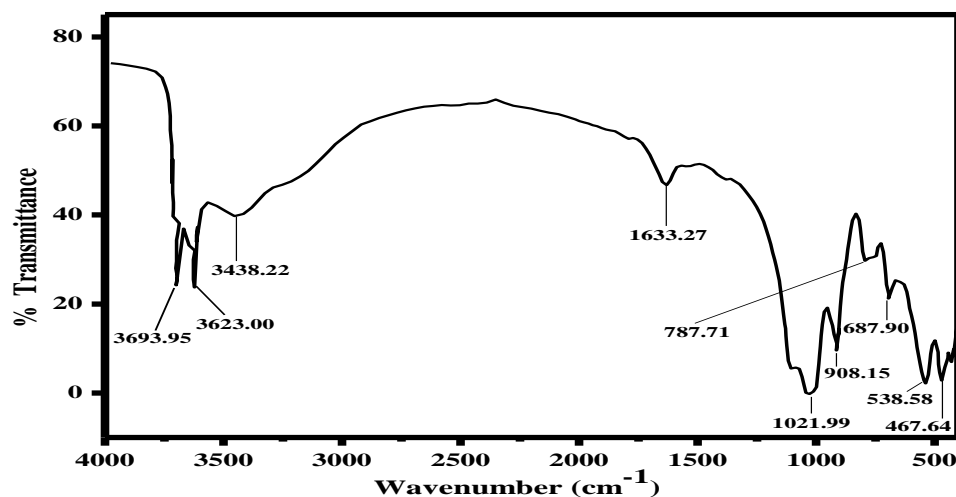


Figure 2: FTIR Spectrum of termite mound clay

Adsorption Experiment

Effect of Initial pH

pH is an important factor controlling the adsorption process (Jawad *et al.*, 2016). The uptake of EY dye onto termite mound clay was investigated in a range of pH 3 to 8 and the result of its effect is depicted in Figure 3. The quantity of dye adsorbed decreases with an increase in the

pH of the dye towards an alkaline medium. The maximum quantity of EY adsorbed by TMC was 12.39 mg/g (99.13 %) at a pH of 3. The extent of adsorption may be due to the charge on the clay relative to that on EY dye which is an anionic dye. The decrease in the quantity adsorbed is a consequence of the increase in negative charge species; causing repulsion with the SO_3 group of the dye molecule and competition with the adsorption sites (Nejib *et al.*, 2015).

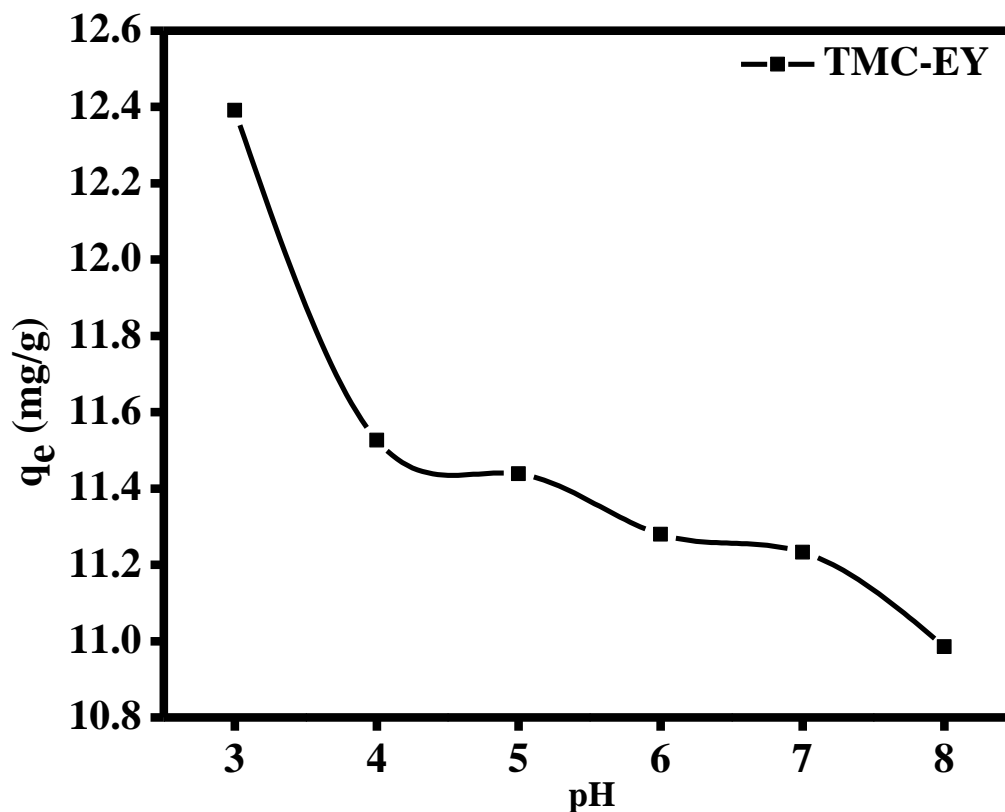


Figure 3: Effect of EY solution pH (initial conc. = 100 mg/L; adsorbent dosage = 0.2 g; contact time 120 min and temp.= 25 °C)

Effect of Initial Concentration and Isotherm Study

The influence of the initial concentration depicted in Figure 4a was investigated by varying the concentration of EY dye from 5 mg/L to 100 mg/L. The quantity of EY adsorbed on the adsorbent increases with an increase in the initial dye concentration. At the minimum adsorbate (EY) concentration (5 mg/L), 0.57 mg/g

representing 91.40% was adsorbed. While at the 100 g/L which was the maximum concentration of EY studied, 12.1 mg/g representing 96.8% was adsorbed. This is due to the increase in the gradient between the adsorbent and the adsorbate (Akkaya Sayılı, 2015). A similar linear trend of increase in adsorption capacity with an increase in the initial dye concentration was reported by Mittal *et al.* (2013).

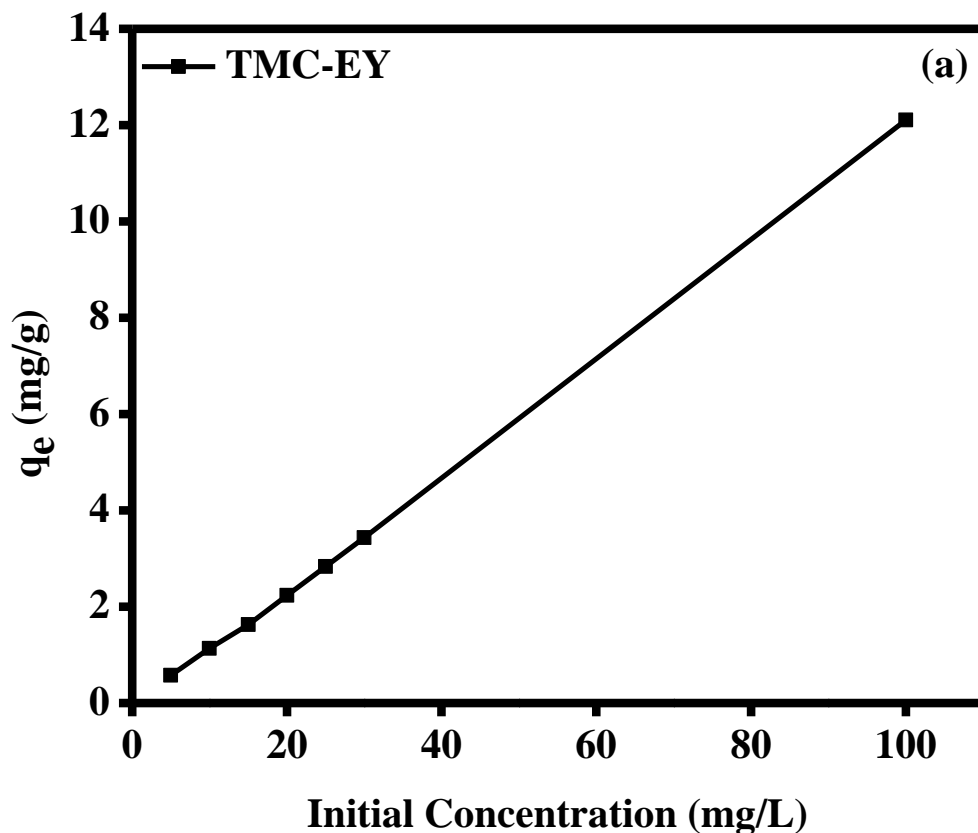


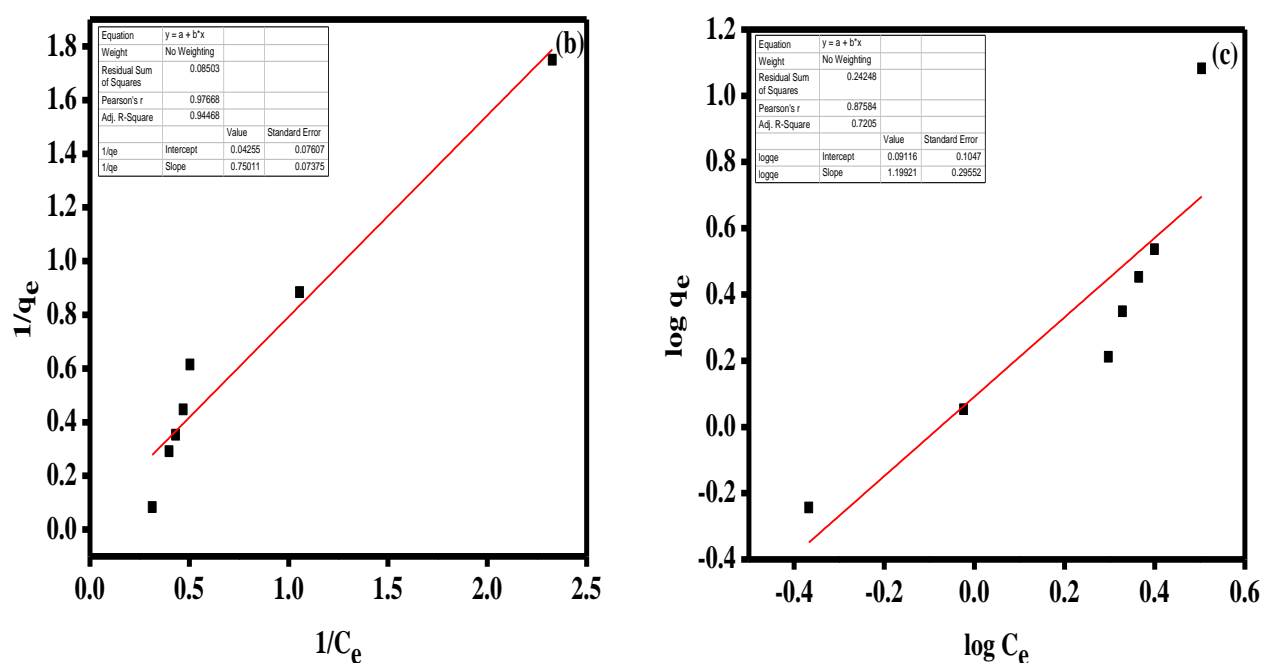
Figure 4: (a) Effect of initial concentration of EY (adsorbent dosage = 0.2 g; contact time 120 min; un-adjusted pH and temp.= 25 °C).

Langmuir and Freundlich adsorption isotherms (Figs. 4b and c) were used to model the equilibrium data and the adsorption parameters obtained are presented in Table 6. The Langmuir isotherm shows the best fitting with R^2 values of 0.9446 compare with that of Freundlich model having 0.7205. The fitness of the adsorption data into the Langmuir isotherm model shows a

monolayer coverage of the EY onto the surface of the TMC (Hameed *et al.*, 2007). From the dimensionless separation factor, R_L which ranges from 0.149 to 0.779, the adsorption is favourable (Kamga, 2019). However, some multi-layer adsorption of EY dye onto TMC adsorbent cannot be ruled out considering the moderate R^2 value obtained from Freundlich model.

Table 6: Isotherm parameters

Isotherm model	Parameter	Value
Langmuir	R^2	0.9446
	q_m (mg/g)	23.47
	K_L (L/mg)	0.05
	R_L	0.149 – 0.779
Freundlich	R^2	0.7501
	$\frac{1}{n}$	1.1992
	n	0.8338
	k_F (mg/g)	-1.0402

**Figure 4:** (b) Langmuir and (c) Freundlich isotherm plots for adsorption of EY dye onto TMC

Effect of Contact Time and Kinetic Study

Adsorption of eosin yellow (EY) was investigated at various contact time intervals for its kinetic study. The observed effect of contact time is depicted in Figure 5a. The adsorption was rapid within the first 10 min at which 11.28 mg/g (90.28 %) of EY dye was adsorbed onto the surface of TMC. The quantities adsorbed

increased rapidly after which further increase in the contact time had no significant effects on the quantity of dye adsorbed. The availability of the adsorption sites together with a high concentration gradient between the adsorbents and the adsorbate molecules account for a faster rate at the early stage (Senthil Kumar *et al.*, 2010). The process slowed down steadily towards

equilibrium at 90 min, suggesting reduction of the adsorption sites as the time increases till the expiration of the reaction time (120 min).

The kinetic information was obtained from the kinetic treatment of data with pseudo-first-order and pseudo-second-order models as shown in Figs 5b and c. Using the R^2 value (Table 7), the pseudo-second-order model gave a better description of the adsorption process than the

pseudo-first-order. Also, the calculated quantity adsorbed ($q_{e, \text{cal}}$) from this pseudo-second-order agrees with the one obtained experimentally ($q_{e, \text{exp}}$). The applicability of pseudo-second-order revealed that chemical interaction was involved in the adsorption process of EY onto the adsorbent (Bello *et al.*, 2021; Pathania *et al.*, 2017).

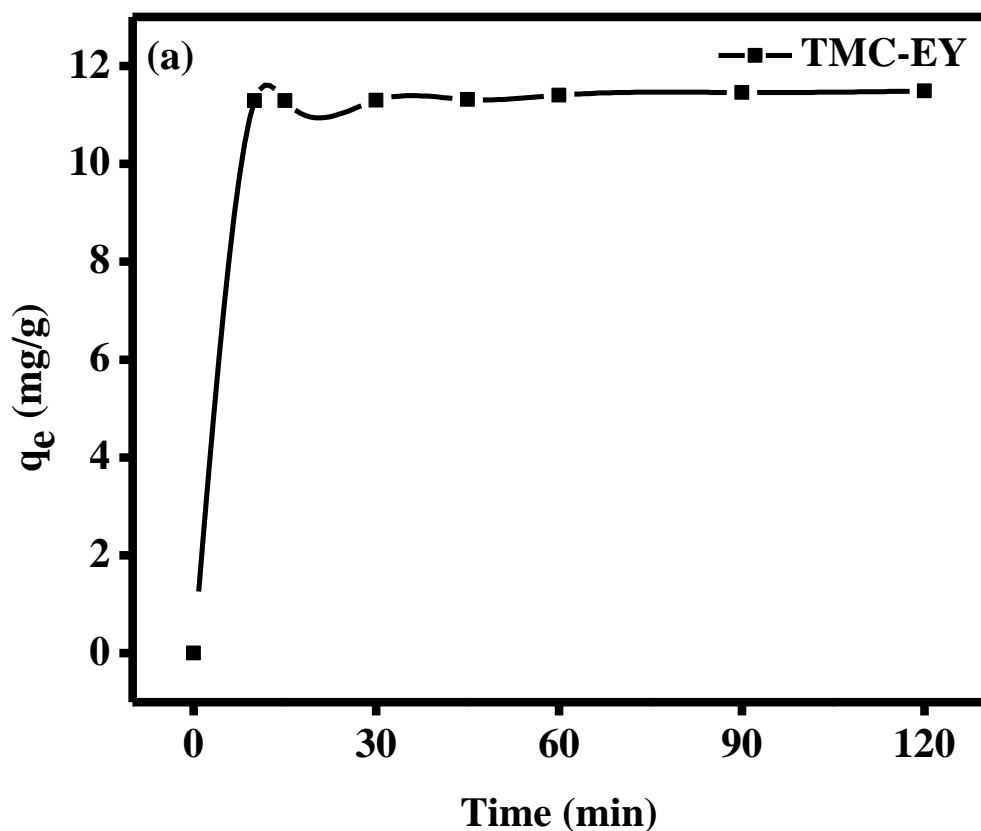


Figure 5a: Effect of contact time (initial conc. = 100 mg/L, adsorbent dosage = 0.2 g; un-adjusted pH and temp.= 25 °C)

Table 7: Kinetics parameters

Kinetics model	Parameter	Value
Pseudo-first-order	$q_{e, \text{exp}}$ (mg/g)	12.10
	R^2	0.8495
	$q_{e, \text{cal}}$ (mg/g)	-
	k_1 (min^{-1})	-0.0024
Pseudo-second-order	R^2	0.9999
	$q_{e, \text{cal}}$ (mg/g)	11.49
	k_2 (g/mg.min)	0.2713

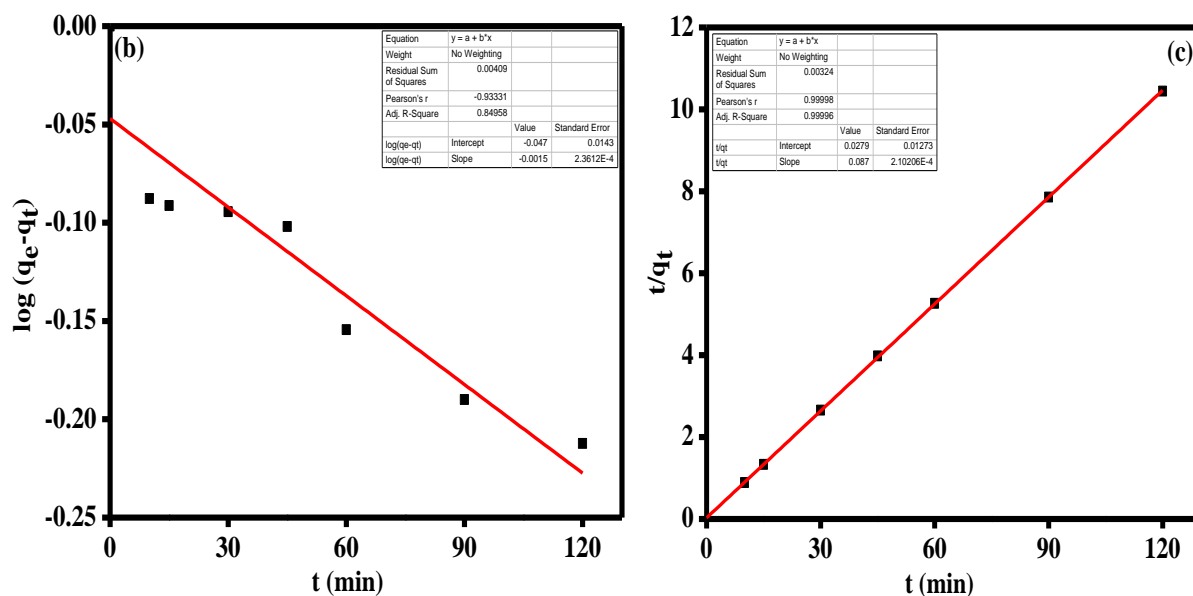
**Figure 5:** (b) Pseudo-first-order and (c) Pseudo-second-order kinetic plots for adsorption of EY dye onto TMC**Effect of Adsorbent Dosage**

Figure 6 shows the effect of adsorbent dosage on the adsorption of EY dye onto TMC. Studying the influence of adsorbent dosage in the adsorption process is pertinent to effectively maximise the adsorbent. It can be observed that the quantity of EY dye adsorbed decreases with

an increase in adsorbent dosage. The reduction is likely attributed to the overlapping of the adsorption active sites resulting from the agglomeration of the particles as the dosage increases (Alghamdi *et al.*, 2019; Etim *et al.*, 2016). The lowest dosage, 0.2 g of TMC yielded the optimum adsorption capacity.

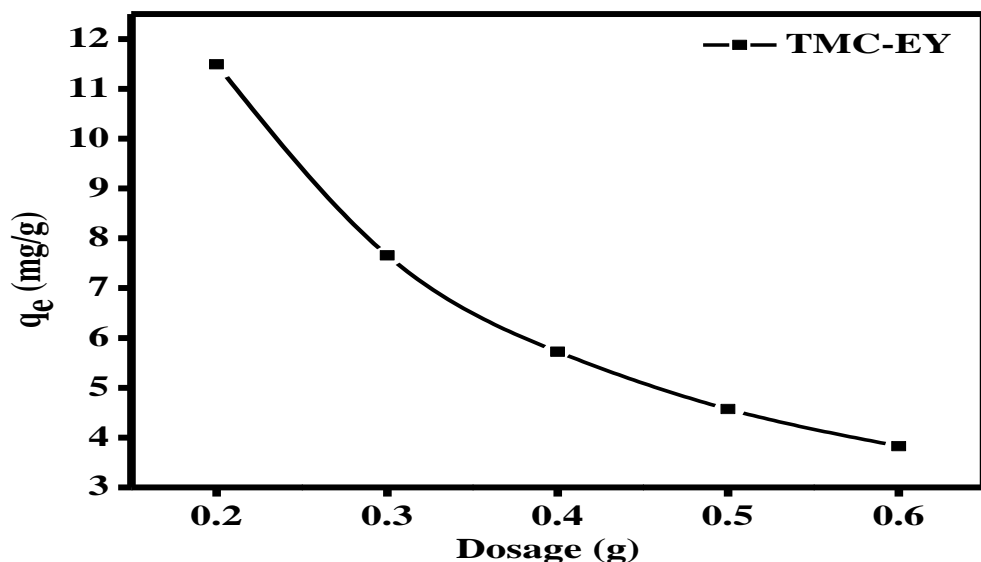


Figure 6: Effect of adsorbent dosage (initial conc. = 100 mg/L, contact time = 120 min; un-adjusted pH and temp.= 25 °C)

Effect of Temperature and Thermodynamic Study

Temperature plays an essential role in the adsorption process, either through its effect on the adsorbent or the adsorbate solution. In Figure 7a, the adsorption capacity decreases with an increase in temperature. The decrease was as a

result of the loss of force of interaction between the adsorbate (EY) and the adsorbent (TMC) as the temperature was raised from 25 °C to 50 °C. A similar trend was reported by Bendaho *et al.* (2017) for the removal of acid dye onto activated Algerian clay.

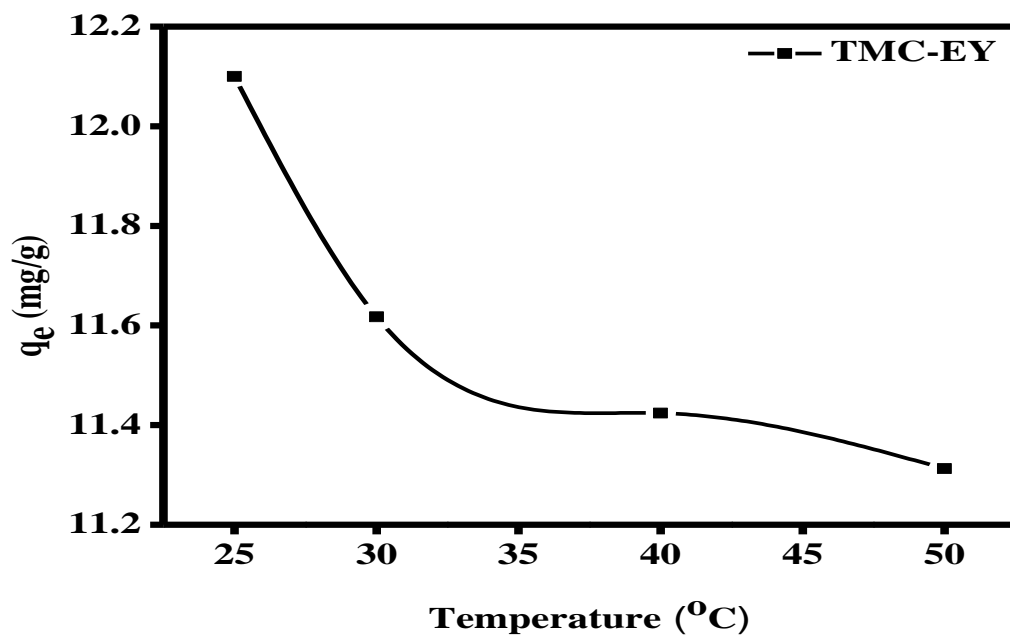


Figure 7a: Effect of temperature (initial conc. = 100 mg/L, adsorbent dosage = 0.2 g; un-adjusted pH and contact time =120 min)

The plot of $\ln K_c$ versus $\frac{1}{T}$ of the Van't Hoff equation is depicted in Fig. 7b and the parameters obtained are presented in Table 8. The negative enthalpy change (ΔH) of -31.98 kJ/mol corroborate the exothermic nature of the process observed in the adsorption experiment. The change in free energy (ΔG) which was negative

implies a spontaneous process with a high affinity between the adsorbate and adsorbent (Khattari and Singh, 2009). Although, the degree of disorderliness (ΔS) is negative and it is an indication of low randomness between the adsorbent-adsorbate (TMC-EY) system (Gao *et al.*, 2010).

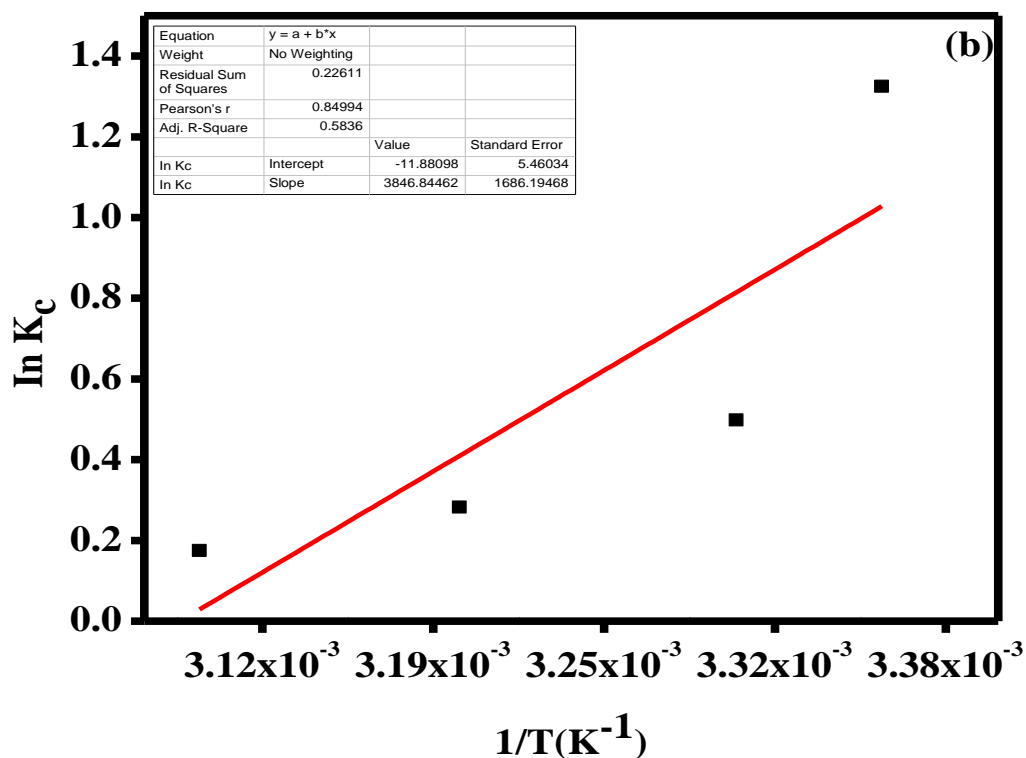


Figure 7b: Plot of $\ln K_c$ versus $\frac{1}{T}$ for adsorption of EY dye onto TMC

Table 8: Thermodynamic parameters

System	Temperature (K)	Parameter		
		ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol.K)
TMC-EY	298	-2.546		
	303	-2.052	-31.98	-98.78
	313	-1.064		
	323	-0.076		

CONCLUSION

This study investigated the adsorptive performance of termite mound clay (TMC) on the removal of eosin yellow (EY) dye from aqueous system. It is posited that the observed surface functional groups of TMC which are similar to that of kaolinite clay and morphology are responsible for its performance. The adsorptive performance is also influenced by the parameters investigated. The adsorption capacity of TMC increased with an increase in the initial concentration of eosin yellow dye in the aqueous solution and rapidly with time within the first 10 min before reaching equilibrium at 90 min. However, it decreased with an increase in pH of the solution, its dosage and the temperature of the

system. A chemisorption interaction was likely to be responsible for TMC-EY system as confirmed by the fitness of the adsorption data into Langmuir isotherm and pseudo-second-order kinetic models. The best adsorptive performance of the TMC for uptake of EY from aqueous solution can be achieved at room temperature as revealed by the thermodynamic study. Consequently, termite mound clay (TMC) can be employed as a low-cost adsorbent for the adsorption of eosin yellow dye from aqueous solution.

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