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Removal of Methylene Blue Dye from Aqueous Solution Using Modified Ngbo Clay

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Authors' contributions

This work was carried out in collaboration among all authors. All authors participated in all the stages of the experimental work and the manuscript writing. All authors read and approved the final manuscript.

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ABSTRACT

This work focused on the removal of methylene blue dye from aqueous solution using modified clay from Ngbo clay deposit in Ebonyi State, Nigeria. The clay was modified to zeolite by alkaline activation at a high temperature. Characterization of the clay was used to determine the properties of the clay. Batch experimental study was employed to determine the effects of process variables. Different isotherm and kinetic models were used to describe the adsorption process. The result showed that aluminium and iron were the major constituents of the clay. Percentage removal of the dye was significantly dependent on temperature, dosage and time. Maximum percentage adsorption of about 97% has been obtained. Pseudo second order best described the kinetic of the adsorption while Freundlich and Henry isotherm models were best in describing the adsorption isotherm. The study has shown synthesised zeolite as an effective, efficient, benign and cheap alternative sorbent for the adsorption of methylene blue from solutions.

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Keywords: Clay; zeolite; methylene blue dye; adsorption; isotherm models; kinetic models.

1. INTRODUCTION

Varieties of synthetic anionic and cationic dyes are routinely used by chemical industries such as leather, textile, printing, cosmetics, pulp and paper [1]. These dyes are applied to the materials from these industries to give them different colours [2]. Studies have shown that thousands of tons of dyes are manufactured annually and applied in several industrial processes [3]. Wastewater from these industries contains different amount of dyes and constitute severe amount of water pollution if it is discharged into water bodies without adequate treatment. Muhammad et al. [4] reported that more than 10% of the total dye applied is lost in the effluent after the dyeing process. The effluents from these industries are often very rich in colour because they contain the residue of these dyes and chemicals. The discharge of these effluents damages the aesthetic nature of the receiving water bodies and imparts toxicity to aquatic life [5]. It also decreases the rate of photosynthesis and creates an imbalance in the aquatic ecosystem [6]. The presence of these dyes destroys the biological processes in water bodies, leads to death of aquatic animals and makes the water bodies unfit for human consumption.

Methylene blue dyes poses serious threat to ecosystem since it has complex aromatic structure resistant to biodegradation or structural modification by common methods such as on exposure to light, temperature or common oxidizing agents [7]. Methylene blue dye is a cationic dye that is more toxic than most other dyes [8]. The non- degradability of methylene blue dyes leads to bioaccumulation in organisms and could be responsible for various dysfunctional observations such as shock, cyanosis, vomiting, eye burns, limb paralysis, and mental confusion [9]. Hence, it is necessary that remnants of methylene blue be removed from industrial effluent before discharging into water bodies.

Various methods have been applied for the removal of dyes and include chemical coagulation, oxidation, flocculation, ozonation, biological treatment, membrane based separation method, photo-catalytic process, sonochemical process, electrochemical process and adsorption [10–17]. Adsorption has proved to be the most economical and simple method of removing dyes from effluent [18]. Other advantages include no harmful by-product, simple design, easy operation and high operational efficiency [1,19-20].

Most of the commercial adsorption systems employ activated carbon as a conventional adsorbent in the treatment of industrial effluent because of its good adsorption capacity. Activated carbon has a controllable pore structure, low acid/base reactivity and large surface area [21]. However, its use is limited due to high purchase cost. Therefore, there is an increased research into the use of non-conventional cost effective raw materials for use as activated carbon/adsorbent in adsorption. The materials include agricultural waste products, natural abundant materials etc. Some materials reviewed include rice husk, oil palm fibre, bamboo, eucalyptus wood, palm kernel shell etc [6,22–25].

Clay is of interest here because it is readily available and abundant. Clay is usually in the form of powder or granules. It has large surface area that is responsible for its high adsorption capacity which can be increased further by activation [26]. This work therefore entails the modification of Ngbo clay and its application as an economic, effective and environmentally friendly solid adsorbent for decontamination of an aqueous solution containing methylene blue dye solution. The clay was characterized to ascertain the nature and properties of the clay. Batch experiments were used to determine the effects of various parameters such as contact time, pH, adsorbent dosage, initial concentration of dye solution and temperature. Equally, kinetic and isotherm studies were carried out.

2. MATERIALS AND METHODS

2.1 Materials' Preparation and Characterization

Clay has been collected from Ngbo Ebonyi State Nigeria, crushed and soaked in distilled water (2000 ml measuring cylinder) to form slurry in order to remove dirt. The slurry has been allowed to freely settle for two days creating clay suspension from a mixture of clay and water. The coarse component of the clay settled at the bottom of the buckets while some of the dirt component remains as supernatant. The supernatant has been carefully decanted and the

sedimentation process has been repeated five times. The fine clay has been dried in an oven at 50°C for three days and the dried cake grounded into fine powder and sieved with 0.75 µm mesh size under dry conditions. Thereafter, 50g of fine dried grounded clay samples has been mixed with 2M NaOH solution, measured into ceramic crucibles and loaded into muffle furnace (Model SX-5-12) pre-heated at 600°C temperature for 3 hours before it was removed and placed in a desiccator with silica gel to cool.

Some physical properties and characterization of the clay was done by using the standard methods reported by [1,6,27].

The chemicals used were of analytical grade. Methylene blue dye has IUPAC name of 3, 7-bis(dimethylamino)-phenazathioniumtetramethylthionine chloride with molecular formula $C_{16}H_{18}N_3SCl_3H_2O$ and molecular weight 373.9 g/mol. The structure of methylene blue has been shown in Fig. 1. Stock solution (100 mg/l) of methylene blue dye has been made by dissolving a measured exact amount into distilled water of pH 7. Solutions for other studies have been taken and diluted as required before use.

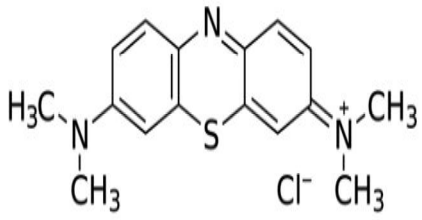

Fig. 1. Structure of methylene blue dye

2.2 Batch Adsorption Experiment

Investigation into the sorption of methylene blue dye onto synthesised zeolite (modified clay) has been carried out using batch adsorption method. Exactly 0.1 g of the zeolite has been measured into a series of 250 cm^3 conical flasks with 100 cm^3 methylene blue dye solution of different concentrations and pH. The conical flasks containing the mixture have been equilibrated mechanically in a mechanical shaker (Remi equipment) at fixed speed of 150 rpm at different temperatures until equilibrium was attained. About 5cm^3 of the solution was withdrawn at pre-determined time intervals and centrifuged for 4 minutes at 500r/min for easy separation. This precipitates the small amount of the zeolite that might have been dispersed in the solution. The concentration of the methylene blue dye has been determined spectrophotometrically at wavelength of 670 nm using Jenway U–V spectrophotometer model 6105.

2.3 Effect of Process Parameters

The effect of pH on the adsorption of methylene blue dye onto the synthesised zeolite has been studied by varying the pH from 2- 10 effected using solutions of HCl and NH_3 while keeping other parameters constant. The effects of adsorbent dosage and temperature have been investigated by varying the adsorbent dosage from 0.5 g to 5.0 g and the temperature from 20 to 40°C. Concentrations ranging from 100 to 900 mg/l were employed in the batch analysis.

The amount of methylene blue dye removed at equilibrium q_e (mg/g) was evaluated using Equation 1, the percentage methylene blue dye (%) adsorbed by the synthesised zeolite has been determined using Equation 2 while the quantity of methylene blue dye q_t (mg/g) adsorbed at any time t has been evaluated using Equation 3 [7,27].

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

$$R\,\% = \frac{(C_o - C_t)}{C_o} \times 100 \quad (2)$$

$$q_t = \frac{(C_o - C_t)}{m}(V) \quad (3)$$

Where C_o(mg/L), C_e (mg/L), C_t (mg/g), represents the initial concentration of methylene blue dye, equilibrium concentration of methylene blue dye and concentration of methylene blue dye at any time t respectively. V and m represent the volume of aqueous media and mass of the dry adsorbent respectively.

2.4 Kinetic and Isotherm Modelling

The adsorption process has been modelled using linear adsorption models of Langmuir, Jovanovic, Freundlich and Temkin.

The Langmuir adsorption is efficiently used for the attraction of adsorbates ions from solution. Langmuir linear formula is represented as given in Equation 4 [18].

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad (4)$$

Where q_e is the amount of dye adsorbed per gram of the zeolite adsorbent, C_e is equilibrium concentration dye solution, K_L is Langmuir isotherm constant related to the affinity of binding sited (l/mg) while q_m is maximum coverage, milligram of dye solution per gram of adsorbent.

The logarithmic equivalent of Freundlich isotherm model is described according to Equation 5.

$$\log(q_e) = \log(K_f) + {}^{1}/_{n} \log(C_e) \quad (5)$$

where 1/n and K_f are Freundlich constants related to adsorption intensity of the adsorbent and adsorption capacity respectively.

Henry's one parameteric isotherm has been used to relate the equilibrium adsorbate concentrations in the liquid and adsorbed phases. The model is given in Equation 6.

$$q_e = K_{He} C_e \quad (6)$$

where q_e is amount of the adsorbate at equilibrium (mg/g), K_{He} is Henry's adsorption constant, and C_e is equilibrium concentration of the adsorbate on the adsorbent (mg/l).

The linear form of Temkin isotherm model is shown in Equation 7.

$$q_e = \beta ln A_t + \beta ln Ce \quad (7)$$

Where β is related to the heat of adsorption and A_t is Temkin constant.

The Jovanoic isotherm model can be expressed in linear form as given in Equation 8.

$$\ln q_e = \ln q_{max} - K_j C_e \quad (8)$$

where q_e is amount of adsorbate in the adsorbent at equilibrium (mg/g), q_{max} is maximum uptake of adsorbate obtained from the plot of ln q_e versus C_e.

The kinetics of sorption of methylene blue dye onto synthesised zeolite has been studied by plotting the Equation of line best fit applied to pseudo-first and pseudo second order models.

Adsorption kinetics has been applied to Lagergren pseudo-first order rate Equation.

The linear form of the Equation is given in Equation 9.

$$log(q_e - q_t) = log q_e - \left(\frac{K_1}{2.303}\right) t \quad (9)$$

Where t is time of contact (min), qt is amount of methylene blue adsorbed at time t, qe is amount of methylene blue adsorbed at equilibrium (mg/g), K_1 pseudo first order rate constant.

The pseudo-second-order kinetic model assumes that chemisorption dictates the rate controlling step. The model is illustrated in Equation 10 [27].

$$\frac{t}{q_t} = \frac{1}{K_2} q_e{}^2 + \frac{t}{q_e} \quad (10)$$

Where K_2 is second order rate constant (g/mg min) and q_e adsorption capacity at equilibrium evaluated as intercept and slope of the linear plot of t/qt versus t.

3. RESULTS AND DISCUSSION

3.1 Preparation and Characterization of the Materials

Some physical properties of the clay have been determined and presented in Table 1. The apparent porosity and bulk density were 29.49% and 1.76 g/cm^3 respectively while the moisture content was 3.33% which is quite lower than the 13.9% reported by [1]. The pH was slightly alkaline at 7.50.

Table 1. Physical properties of the clay

Property	Value
Total shrinkage (%)	7.50
Apparent porosity (%)	29.49
Water absorption (%)	17.32
Transverse strength (KGF/cm^2)	37.20
Apparent density (g/cm^3)	2.42
Bulk density (g/cm^3)	1.76
Moisture content (%)	3.33
pH	7.50
LOI	10.52

3.2 Characterization of Raw Clay

The elemental composition of the clay has been analysed using atomic absorption spectrophotometer (AAS). The result in Table 2 indicated that the major constituents of the clay were aluminium, iron, potassium and calcium. Traces of cobalt, chromium, lead and copper were observed among others.

Table 2. AAS analysis of Ngbo clay

Element	Actual concentration (ppm)	Absorbance (Å)
Al	61.0645	0.1893
Ca	15.0090	0.1213
Pb	0.3114	0.0021
Co	0.4246	0.0071
Mn	2.9949	0.5965
K	15.2574	1.2480
Na	1.5641	0.2444
Cr	0.2339	0.0093
Zn	1.4662	0.2701
Cd	0.0296	0.0043
Fe	24.7391	0.6671
Ni	0.0500	0.0048
Cu	0.0362	0.0048
Mg	0.9289	0.2929

The scanning electron microscope (SEM) image results of the Ngbo clay are presented in Fig. 2 at different magnifications. The SEM image result of the clay showed cracking or peeling morphology and presence of tubular or rod material attributed to halloysite, clinochlore, mica and muscovites. The dark and bright patches witnessed in the image have been attributed to presence of imbedded chemicals and completely dried portion of the samples respectively.

3.3 Adsorption Studies

3.3.1 Effect of adsorbent dosage at different temperature

The results of effect of adsorbent dose at different temperature and percentage removal of dyes on the modified clay are shown in Figs. 3 and 4. The removal efficiency increased up to 79% within 60 minutes on increasing the adsorbent dosage from 0.5 to 5 g (for initial dye concentration 100 mg/L, solution volume 100 mL) at 20°C. Then, under the same conditions, the removal efficiency increased up to 89% within 60 minutes at 30°C and up to 94.5% within the same 60 minutes at 40°C. This shows that increase in temperature increases the percentage removal of methylene blue. This is because increase in temperature usually leads to an increase in the equilibrium capacity of an adsorbent [28]. Furthermore, increase in temperature increases the diffusion rate of the dye particles across the boundary layer by decreasing the viscosity of the solution [20].

The amount of methylene blue dye adsorbed (adsorption capacity q_e) equally increased as the adsorbent dosage increased from 0.5g to 5.0g. This is probably on account that more adsorption sites become available when adsorbent dosage is increased. This ensures that more adsorbate ions can adhere to it [29].

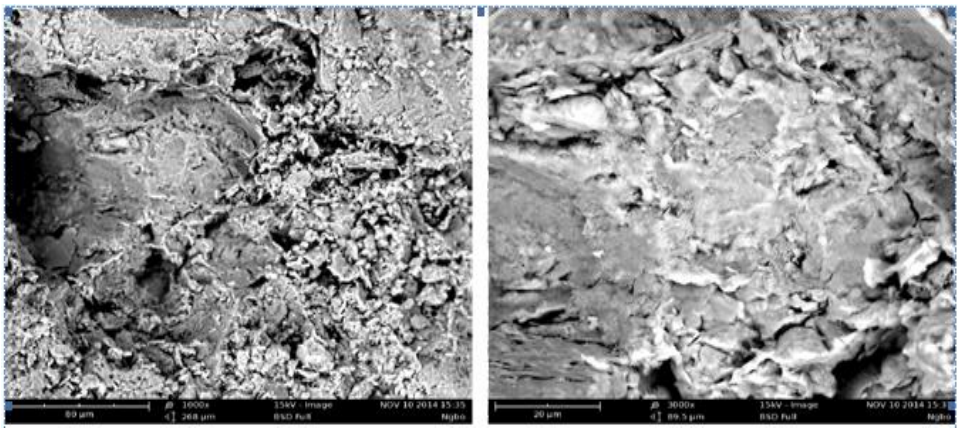

Fig. 2. SEM analysis of the clay

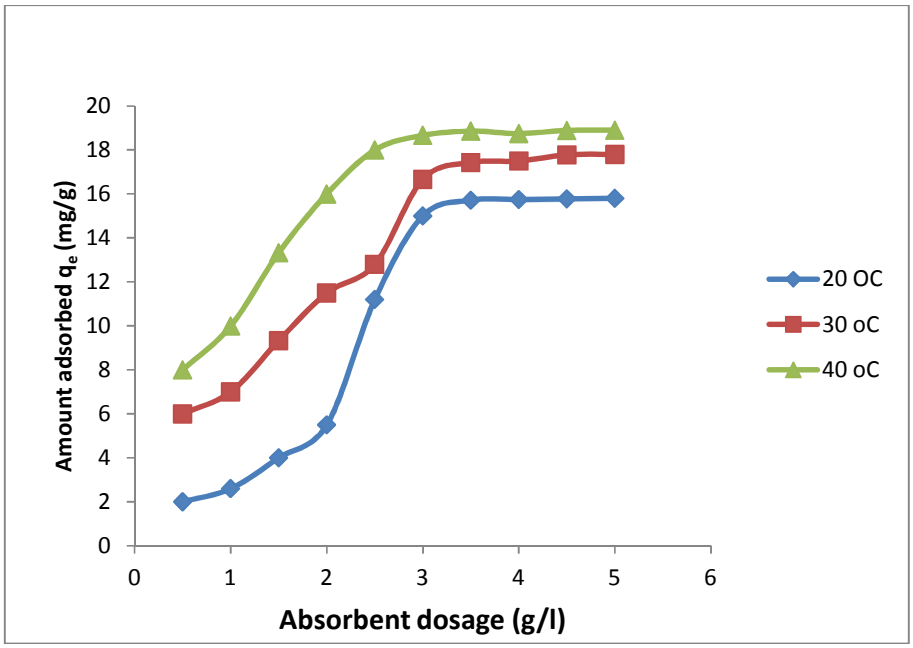

Fig. 3. Effect of adsorbent dose at different temperatures at initial concentration of 100mg/l and volume of 100 ml

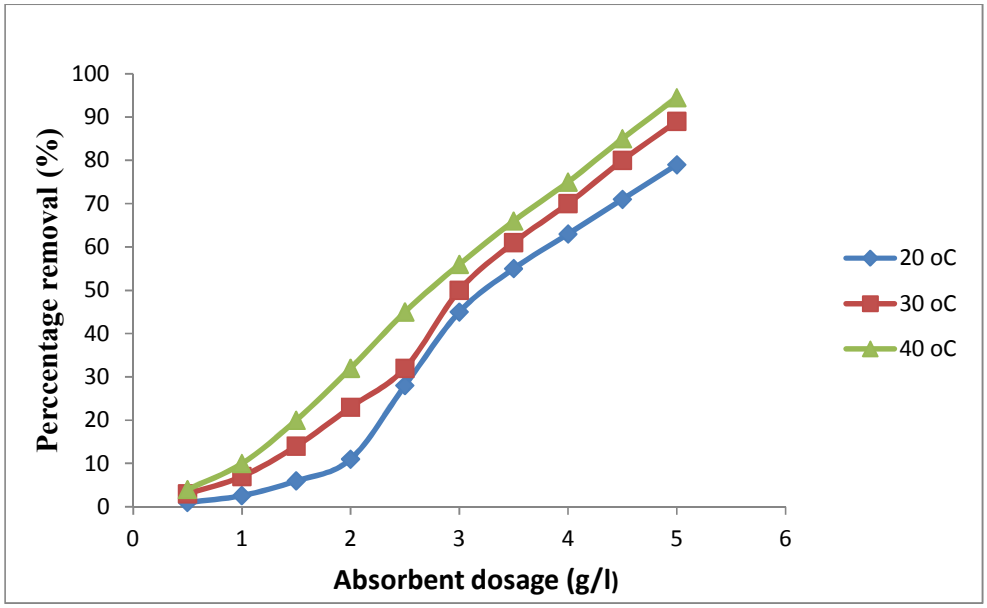

Fig. 4. Percentage removal of methylene blue at different temperatures at initial concentration of 100 mg/l and volume of 100 ml

3.3.2 Effect of contact time

The effect of contact time on adsorption of methylene blue dye on the modified clay was shown in Fig. 5. The plot indicated that the adsorption capacity of the methylene increased with increasing time and reached maximum removal at the adsorption equilibrium. [18] reported that an adsorption process involves the adsorbate first migrating to the boundary layer, then diffusing into the adsorbent surface and finally into the porous adsorbent structure. This normally takes some time. The highest rate of adsorption capacity was attained at 80, 60 and

40 minutes for 20°C, 30°C and 40°C temperatures respectively. There was less significant increase in adsorption capacity after the time due to the fact that there were less active sites in each clay dosage; therefore, an increase in contact time led to increased removal efficiency until the sites were saturated. The removal of dye is controlled by the rate of movement of the adsorbate from the outer sites to the interior sites of the adsorbent.

3.3.3 Effect of pH at different temperatures

The effect of changes in solution pH on the adsorption of methylene blue on the modified clay has been investigated at different temperatures as shown in Fig. 6. The result indicated that a major significant increase in percentage removal has been observed as the solution pH increased from 2 to 4. Thereafter, a relatively small increase in removal percentage

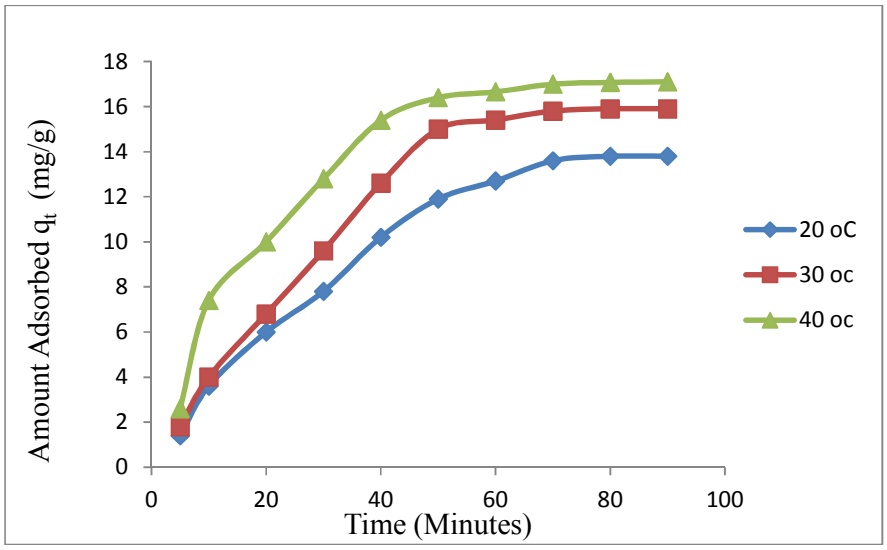

Fig. 5. Effect of contact time on adsorption at initial concentration of 100 mg/l and volume of 100 ml

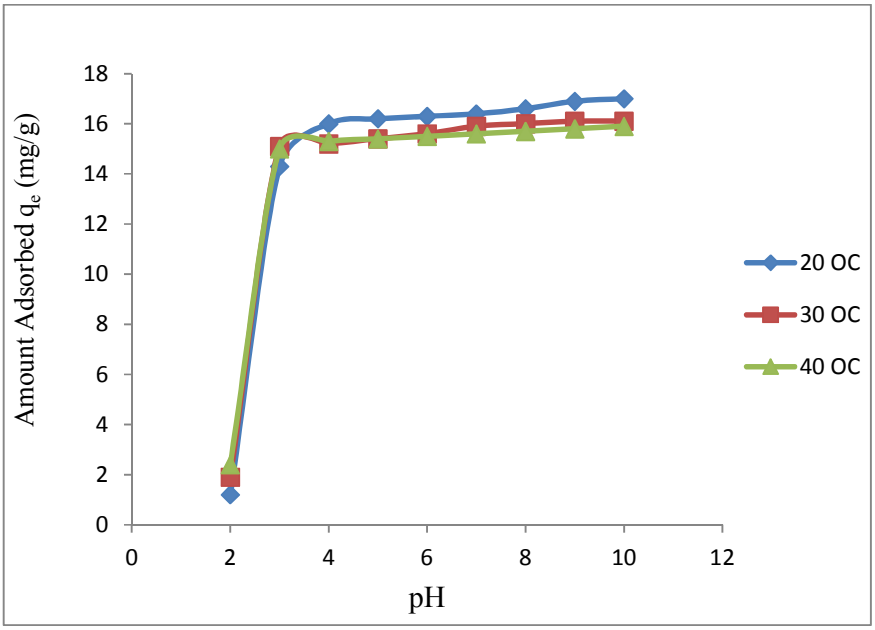

Fig. 6. Effect of pH on the adsorption of methylene blue at initial concentration of 100 mg/l and volume of 100 ml

was observed as the solution pH increased further from 4 to 10. Hence, the adsorption of methylene blue dye was almost independent of pH at solution pH higher than 4. This may be to strong hydrophobic interactions between the particles.

3.3.4 Effect of initial concentration

Solution concentrations ranging from 100 mg/l to 900mg/l have been employed in determining the effect of concentration on the percentage removal of methylene blue dye as shown in Fig. 7. The plot showed that the amount of methylene blue adsorbed per gram of adsorbent increased with increase in methylene blue concentration. This is because the ratio of the adsorbate molecules to the adsorbent increased as the initial dye concentration increased, resulting to an increase in the quantity adsorbed. The percentage removal of the dye decreased as the initial concentration increased. The same trend has been reported by [27]. This was probably because as the initial concentration of the dye was increased, more dye molecules were adsorbed but the ratio of the dye molecules in the solution to the adsorbed molecules decreased.

3.4 Isotherm Studies

Adsorption isotherm explains the relationship between the amount of an adsorbate adsorbed per unit mass and the concentration at equilibrium at constant temperature [30]. The equilibrium studies have been carried out by fitting the experimental data to the isotherm models already described.

The Langmuir isotherm has been modelled by plotting C_e/q_e against log C_e in Fig. 8. The Langmuir constants, q_e and K_L have been determined from the slope and intercept respectively. q_e is related to the maximum adsorption capacity while K_L explains the magnitude of the rate of adsorption [31]. Maximum adsorption capacity q_m of 0.230 has been obtained while in the adsorption of bromoscresol green dye, q_m of 0.0489 has been reported [20].

Linear plot of log(q_e) against log (C_e) in Fig. 9 has been employed in describing the Freundlich isotherm model. The Freundlich constants K_f and n have been obtained from the intercept and the slope of the plot respectively. K_f is the Freundlich constant that gives an indication of the adsorbent capacity and show the strength of the relationship between the adsorbent and the adsorbate while n is associated with the intensity of the adsorption process [18]. The correlation coefficient was 0.999 indicating strong correlation between then experimental data and the isotherm model.

Henry adsorption model is a one parametric linear adsorption model that operates at low concentration of adsorbate and without consideration of surface coverage. Plot of q_e against C_e has been used to investigate the Henry's model in Fig. 10. High correlation coefficient was obtained. This is because Henry's model is a simple one parametric model.

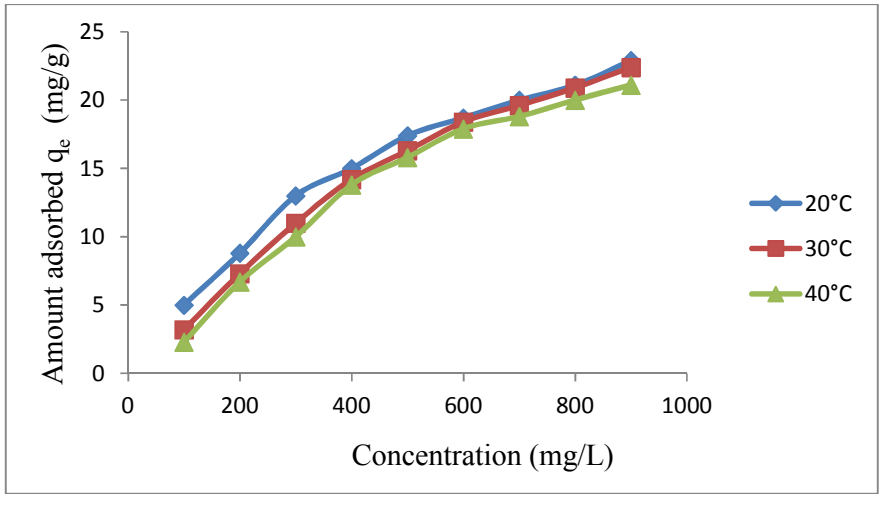

Fig. 7. Effect of concentration on the adsorption of methylene blue

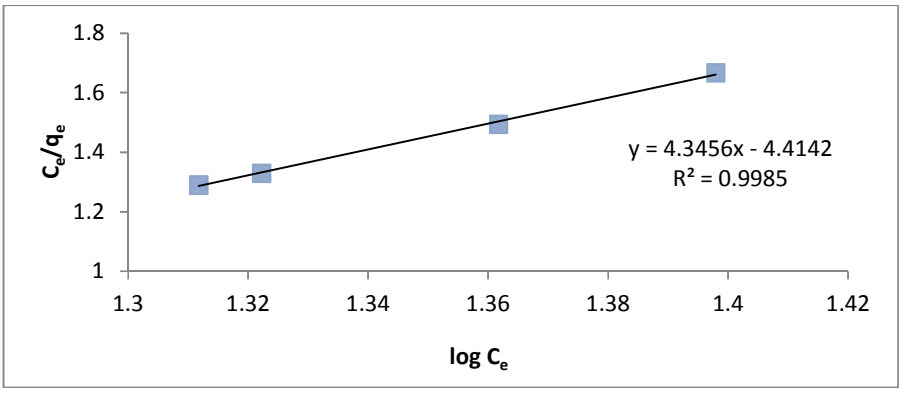

Fig. 8. Langmuir isotherm plot for 30°C

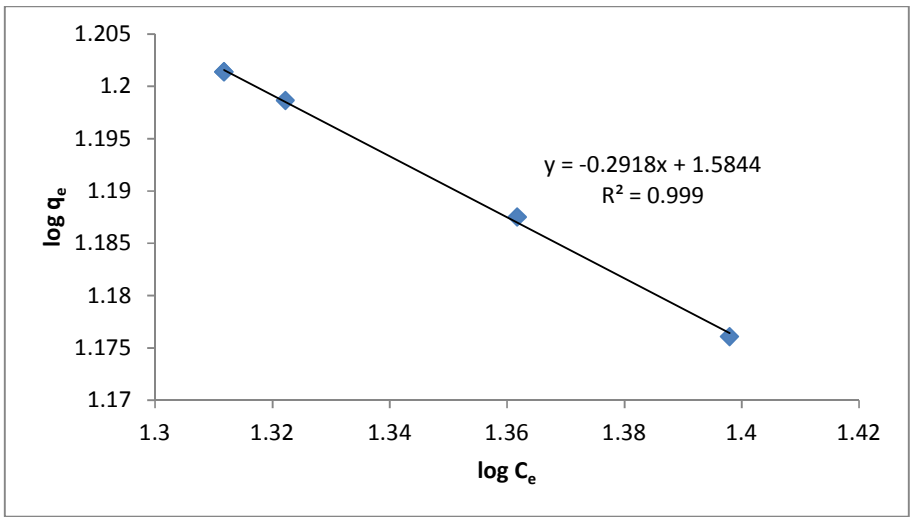

Fig. 9. Freundlich isotherm plot for 30°C

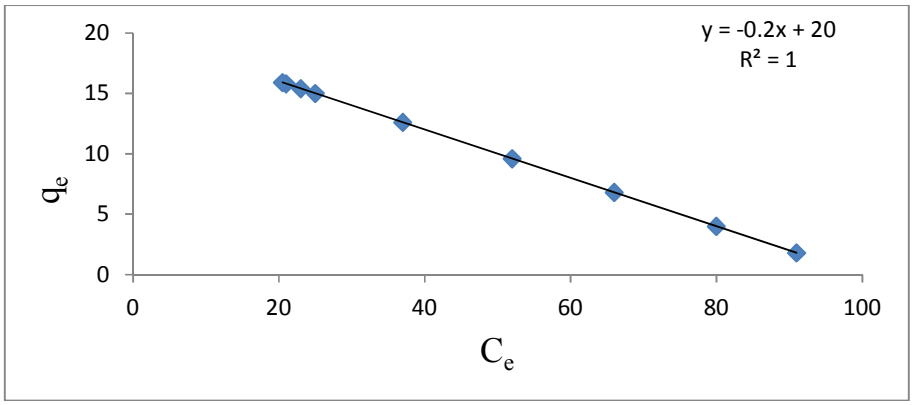

Fig. 10. Henry's isotherm plot for 30°C

Plot of q_e against ln C_e has been used in describing the Temkin isotherm model as shown in Fig. 11. Temkin isotherm is applied under the condition of intermediate mode of sorbate concentration. It considers the fall in the heat of sorption of molecules in the layer to decrease

linearly with area of coverage owing to sorbent and sorbate interaction instead of logarithmically [32]. The correlation coefficient of 0.971 suggests good correlation though the Freundlich gave a better result than it.

The Jovanoic isotherm was modelled by using the linear plot of ln q_e against ln C_e in Fig. 12. The correlation coefficient obtained indicated that the experimental data did not follow the Jovanoic model. The Jovanovic model is predicated on the assumptions contained in the Langmuir model, but in addition the possibility of some mechanical contacts between the adsorbate and adsorbent [33].

All the calculated isotherm values are presented in Table 3 where it was concluded that Freundlich and Henry isotherms best described the equilibrium process.

3.5 Kinetic Studies

For an adsorption process, the kinetic study reveals the rate of adsorbate uptake. The kinetics models have been used to investigate the mechanism of adsorption and the potential rate controlling steps such as mass transport and chemical reaction processes [34]. Among other kinetic models investigated, only the pseudo first and second order gave a reasonable correlation coefficient.

Table 3. Isotherm tables for adsorption of methylene blue at 30°C

Isotherm model	Isotherm values
Langmuir	q_m (mg/g) = 0.230 K_2 (l/mg) = 0.984 R^2 = 0.998
Freundlich	n = 3.436 K_f(l/g) = 38.371 R^2 = 0.999
Henry	K_{He} (l/g) = 0.200 R^2 = 1.00
Temkin	β (J/mg)= 8.835 A_t (l/g) = 132.3 R^2 = 0.971
Jovanovic	q_{max} (mg/g) = 25.636 K_j = 0.021 R^2 = 0.970

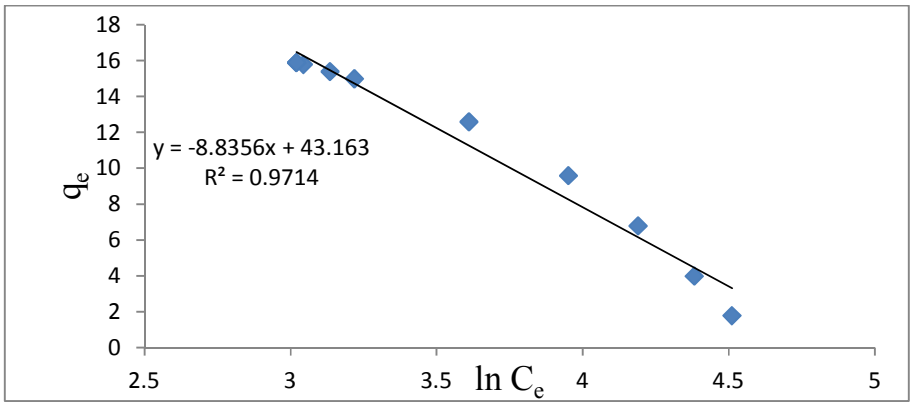

Fig. 11. Temkin isotherm plot for 30°C

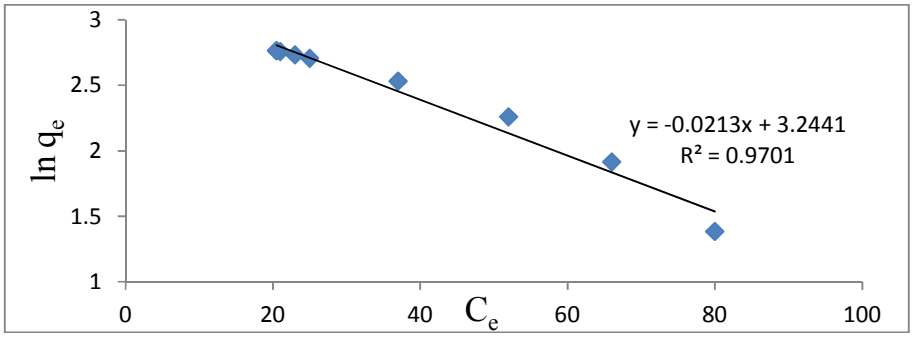

Fig. 12. Jovanovic isotherm plot for 30°C

The pseudo first order kinetics has been modelled by plotting log ($q_e - q_t$) against t of adsorption in Fig. 13. The pseudo first order rate constant K_1 and the equilibrium amount sorbed q_e have been determined from the slope and intercept of the model Equation respectively. The correlation coefficient of 0.787 suggests that the first order kinetic model cannot be used to model the kinetics of the adsorption process.

Linear plot of t/q_e against t has been used to describe the pseudo second order model in Fig. 14. The kinetic constants have been evaluated from the slope and intercept of the Equation. The Pseudo-second- order kinetic model gave a regression coefficient of 0.923, which indicated that a good relationship existed between the model and the experimental data. The high regression coefficient of the Pseudo-second-order model is also an indication that chemisorption was the rate controlling step in the adsorption process [35]. Pseudo second order kinetics has been also found to be the best kinetic model in describing the adsorption of phenol red and orange G [36]. The calculated kinetic constants together with their correlation coefficients are tabulated in Table 4.

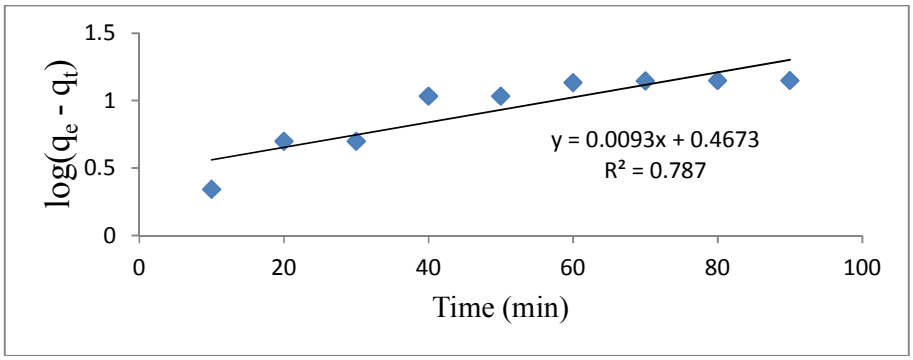

Fig. 13. Pseudo-first –order kinetic plot at 30°C

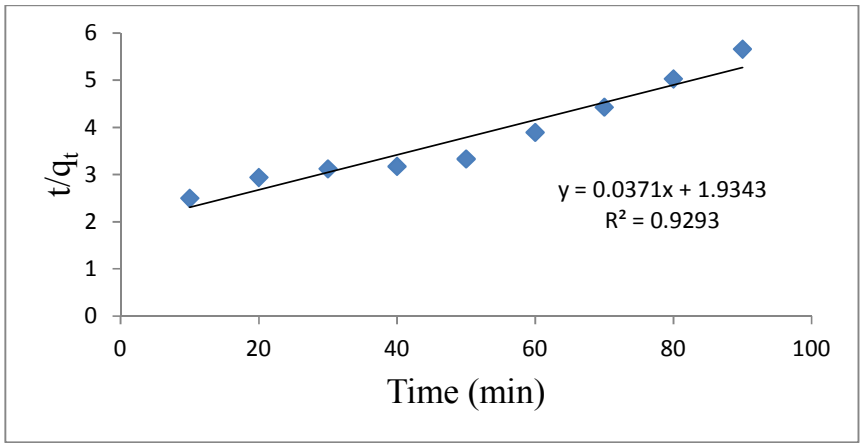

Fig. 14. Pseudo-second –order kinetic plot at 30°C

Table 4. Kinetic table for adsorption of methylene blue at 30°C

Kinetic model	Kinetic value
Pseudo first order	q_{e1} (mg/g) = 1.021 K_1 (min^{-1}) = 1.076 R^2 = 0.787
Pseudo second order	q_{e2} (mg/g) = 27.027 K_2 (g/mg.min) = 377.694 R^2 = 0.929

4. CONCLUSION

The removal of methylene blue dye from aqueous solution using modified clay has been successfully carried out. The characterization of the clay revealed the major constituents of the clay are aluminium and iron with crystalline nature suitable for adsorption of particulates. Contact time, adsorbent dosage and temperature have been found to have significant effect on the percentage removal of the dye whereas changes in solution pH above 4 did not affect the percentage removal insignificantly. Study of adsorption isotherms showed that Freundlich and Henry isotherm models best fitted the experimental data while pseudo second order kinetic model was best in describing the adsorption process.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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