



Kinetic and Equilibrium Modeling of the Adsorption of Amaranth from aqueous solution onto Smectite Clay

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Abstract

The capability of smectite clay (Sa01) was investigated as a low cost adsorbent for the adsorption of a hazardous water soluble dye amaranth from aqueous solutions. Using the batch mode process, the effect of contact time, initial dye concentration (C_0), adsorbent dosage and pH was also investigated at room temperature of 25°C. It was observed that after 40 minutes equilibrium was attained and the percentage removal of dye decreased as initial pH was increased. Increasing the mass of the adsorbent resulted to an increase in the percentage of dye uptake (%R). The adsorption percentages were higher at lower initial concentrations of amaranth. 83.6% of dye was adsorbed at pH 2, adsorbent mass of 1.0 g and initial dye concentration of 30 mg/L. Langmuir and Freundlich isotherm models were used to explain the experimental data. Although equilibrium data were found to follow both isotherm models, the Freundlich model had the better correlation with the experimental data ($R^2 = 0.9894$). Pseudo-first-order and pseudo-second-order kinetic models were used to analyze the adsorption kinetics. The pseudo-second-order kinetic model was found to agree well with the experimental data ($R^2 > 0.99$).

Keywords: Adsorption, amaranth, smectite clay, isotherm, kinetics.

Introduction

Different stages in the dyeing and finishing processes in textile industries produce large amount of wastewater with intense colour. Though only a small proportion of water pollution is due to these dye wastewaters released into the environment, their colours make them visible even in small quantities¹. It is reported that over 100.000 commercially available dyes exist and the global annual production of synthetic dyes is more than 7×10^5 metric tons². Without prior treatment, discharging dye-bearing wastewater into natural streams and rivers can be problematic to the aquatic environment as light penetration and photosynthesis are reduced, and are also hazardous and toxic to aquatic life³.

Azo dyes with the azo group band (-N=N-) are used in many textile industries and are of synthetic origins. Amaranth, an azo dye known widely for its use in colouring textile materials, paper, wood, leather etc, was also used as colouring agent and decoration for food stuff like jams, jellies, ketchups and cake for a longtime. However, it has been prohibited for a few years now in many countries due to its carcinogenicity and other toxic effects. It is now well known that a prolonged intake of amaranth can result into tumor, allergy, respiratory problems and birth defects^{4,5}.

Several methods exist for the treatment of coloured wastewater. These include: adsorption, coagulation and flocculation, biological treatment, advanced oxidation processes, photo catalytic process etc. Adsorption processes is known to be

efficient, economical and superior to other techniques for colour removal from coloured effluents because of initial costs, simplicity of design, ease of operation and insensitivity to toxic substances. The most popular adsorbent throughout the water/wastewater treatment and other industrial applications is activated carbon but due to high cost of carbon makes it an expensive process⁶⁻⁸. This has pushed many researchers to search for low cost adsorbents. The following materials have been used as low cost adsorbent for colour removal and varying results have been obtained. These include Coal, fly ash, wood, silica gel, agricultural wastes (bagasse pith, maize cob, coconut shell, rice husk, etc.), cotton wastes and clay materials (bentonite, montmorillonite, etc.)⁹. However as mineral constitutes of clay show wide spatial compositional variation from one place to another, smectite clay from sabga in the North West region of Cameroon was selected for the present study.

The objectives of this study therefore was aimed at investigating the possibility of removing Amaranth from its aqueous solutions using locally available low cost adsorbent (smectite clay)(Sa01), and to predict the transport mechanisms involved at the solid-liquid interface using some equilibrium and the kinetic models.

Material and Methods

Adsorbent: The clay material obtained from Sabga in the North West region of Cameroon (Sa01) was used by Tonle et al, 2003¹⁰. The physicochemical characteristics of Sa01 are represented in table 1 and table 2 respectively.

Sa01 was air dried, ground into fine powder using a mortar and was then passed through an 80 µm mesh opening size sieve. The sieved powder was kept in an oven at 110°C for 24 hours, removed and cooled in a dessicator before use.

Table- 1
Physical characteristics of Smectite (Sa01)

Colour	Dirty white
BET surface area	86 m ² /g
Total pore volume	0.15 cm ³ /g
Micro pore volume	0.01 m ³ /g
Average particle size	8 µm
Mineral composition (%)	Montmorillonite (79.2); quartz (10); fedspar (5); hematite (2.35); limonite (0.1)
Cation exchange capacity (CEC)	0.78 meq/g

Preparation of adsorbate: All the experiments were conducted with artificial wastewater solution prepared by dissolving amaranth dye in distilled water. The synthetic dye amaranth (06409) (C.I. Acid Red 27; C.I. 16185; CAS N^o: 915-67-3; Molecular Formula: C₂₀H₁₁N₂Na₃O₁₀S₃; Molecular Weight: (604.46) was got from Fluka, Spain and used as it was obtained. Figure 1 shows the chemical structure of amaranth.

A 1000 mg/L stock solution of amaranth was prepared. By dilution with distilled water, standard solutions were also prepared. The concentrations of these solutions were measured with a UV spectrophotometer at a wavelength of maximum absorption of 520 nm. A calibration curve of absorbance versus concentration of the dye was plotted. The pH of the solutions was adjusted with either 0.1 M HCl or 0.1 M NaOH.

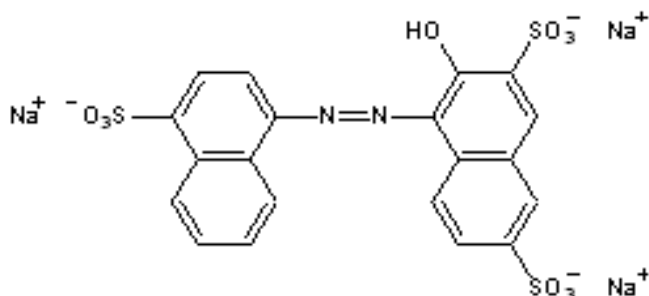


Figure-1
Structural formula of amaranth

Batch mode adsorption studies: Batch mode adsorption studies were performed at ambient temperature of 25°C to

investigate the effect of contact time, adsorbent dose, pH and initial dye concentration. Solutions containing known concentrations of adsorbate and accurately weighed amounts of adsorbent were introduced in 250 mL capacity flasks, sealed and agitated with a magnetic stirrer at given time intervals. Filter paper was used to separate the adsorbates from the adsorbent and the concentrations of dye in the filtrate determined by the UV-visible spectrophotometer.

Calculations of adsorption capacities Q_t and Q_e at time t and equilibrium respectively were calculated based on the following equations:

$$Q_t = \frac{(C_0 - C_t)V}{m} \quad (1)$$

$$Q_e = \frac{(C_0 - C_e)V}{m} \quad (2)$$

The percentages of amaranth dye adsorbed (%R) were also determined by the equation:

$$\%R = \frac{(C_0 - C_t)}{C_0} \quad (3)$$

where C₀ is the initial concentration (mg/ L), C_t and C_e are the residual concentrations (mg/ L) of amaranth at time t (minutes) and equilibrium respectively. V is the volume of aqueous solution (in litres) and m, the mass (g) of Sa01 used in the experiment.

Effect of contact time: The contact time was varied in the range 0 – 120 minutes between the adsorbate and adsorbent to study the time-dependent behaviour of the adsorption of amaranth onto Sa01. The initial concentration of amaranth was kept at 50 mg/L and the smectite dose was 0.5 g/25 ml at pH = 2. At the end of each time, the solution was filtered rapidly and the concentration of amaranth in the filtrate determined.

Effect of pH: The effect of pH on the adsorption of amaranth on Sa01 was investigated. The initial dye concentration was kept at 50 mg/L and adsorbent dose at 0.5 g/25 mL. The desired initial pH of the solutions was adjusted by adding 0.1 M HCl or 0.1 M NaOH to vary the pH range from 1 to 6.5. The suspensions were agitated for 40 minutes at room temperature of 25°C (contact time necessary to attain equilibrium).

Effect of adsorbent dosage: The effect of the dose of Sa01 on the adsorption of amaranth was studied by agitating 25 mL of 50 mg/ L dye solutions with varying adsorbent mass from 0.4 g to 2.0 g. All experiments were carried out at constant pH of 2.

Table 2
Chemical Composition of the Sa01

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	LOI
63.36	14.58	4.24	< 0.03	0.23	0.7	0.39	2.34	0.2	< 0.05	13.79

Effect of initial dye concentration and contact time: Batch adsorption was conducted with series of initial dye concentrations ranging from 30 to 70 mg/L at pH = 2 and adsorbent mass of 1g in 25mL of dye solution.

Equilibrium and kinetic modelling: The Langmuir and Freundlich isotherm models were used to describe the interaction between the adsorbate and adsorbent and the better fit isotherm was determined by R^2 values computed by linear regression. Pseudo-first-order and pseudo-second-order kinetic models were applied to the system. The R^2 values and comparison of experimental and theoretical adsorption capacity Q_e were used to verify the kinetic model that explains better the uptake of dye by Sa01.

Results and Discussion

Effect of contact time: The result showed that the percentage of dye adsorbed increased when the agitation time was increased and equilibrium was reached after 40 minutes. The adsorption of amaranth on Sa01 was found to be fast at the beginning and became slower as the contact time was increased until equilibrium was reached. This trend is because the concentration driving force ($Q_e - Q_t$) continuously decreases with time t ¹¹. 40 minutes was adopted as agitation time in subsequent measurements unless otherwise stated.

Effect of pH: The effect of pH on adsorption of amaranth is given in figure 2. The maximum removal of amaranth occurred at pH 1.0 (62.424%) and as the initial pH was increased, the percentage removal decreased. This is because at low pH, the clay surface is positively charged and this will lead to a significantly strong electrostatic attraction between the positively charged smectite surface and the anionic dye molecules thus resulting in an increase in dye adsorption. Also by increasing the pH of the solution, the number of negatively charged sites on the surface increases. Thus the smectite with negatively charged sites will repel the anionic dye. Therefore by increasing the pH, dye uptake decreases^{12,13}. pH value of 2 was selected for all subsequent studies.

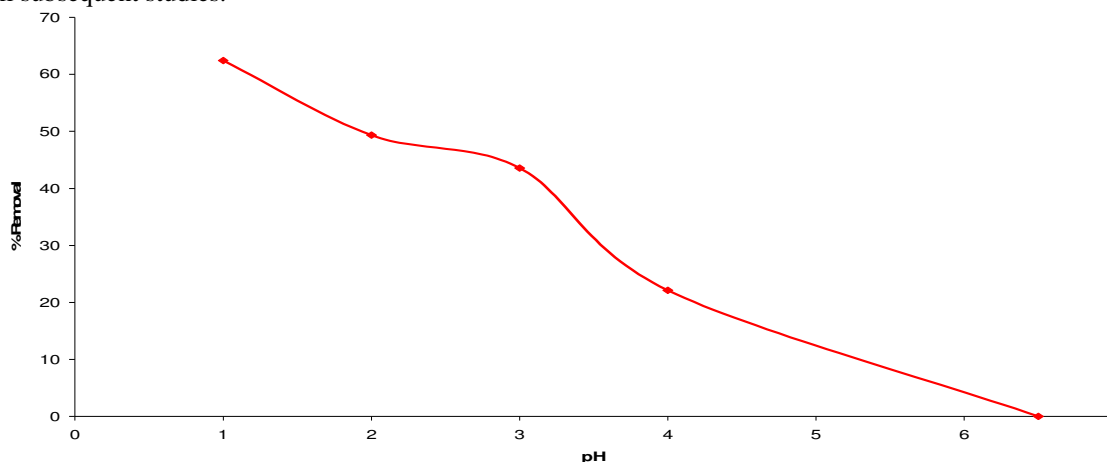


Figure -2

Effect of pH on the adsorption of amaranth on Sa01, (Adsorbent dose = 0.5g/25mL solution)

Effect of adsorbent dosage: The influence of the mass of Sa01 (0.4 – 2 g/25 ml) on the removal of amaranth at constant initial dye concentration (50 mg/L), contact time (40 minute) and pH = 2 is shown in figure 3. The result showed that as the adsorbent mass was increased from 0.4 g to 2.0 g, removal of dye also increased from 44.222% to 83.032%. Gandhi et al (2012)¹⁴ observed the same trend. However, the adsorption capacity decreased from 1.402 to 0.527 mg/g. The increasing trend in adsorption percentage as adsorbent dosage increases might be attributed to the increase of surface area and presence of additional available sites¹⁵. The adsorbent dosage was inversely proportional to the adsorption capacity of Sa01. This is because increase in adsorbent dosage might have led to the overlapping of surface area due to the restricted area made available and therefore led to reduction of effective surface area which plays an important role in dyes uptake^{16,17}.

Effect of initial concentration and contact time: Figure 4 illustrates the adsorption of amaranth for different initial concentrations range 30 – 70 mg/L at pH 2 as a function of contact time. An increase in the amount of dye adsorbed (mg/g) was observed when the initial concentration of solution was raised. As initial dye concentration was increased from 30 to 70 mg/L, the adsorption capacity increased from 0.636 to 1.553 mg/g. The concentration provides an important driving force to overcome all mass transfer resistance of the dye between the aqueous and solid phases. Hence the adsorption process is improved at higher initial concentration of dye¹. The time required for the system to attain equilibrium increased with the increase in initial concentration. That is from 40 minutes to 100 minutes for initial concentration from 30 to 70 mg/L respectively. The uptake of amaranth was rapid in the beginning of the adsorption process. The adsorption rate decreased gradually and became constant when the system reached equilibrium. The same trend was observed by B.H. Hameed et al, 2009¹.

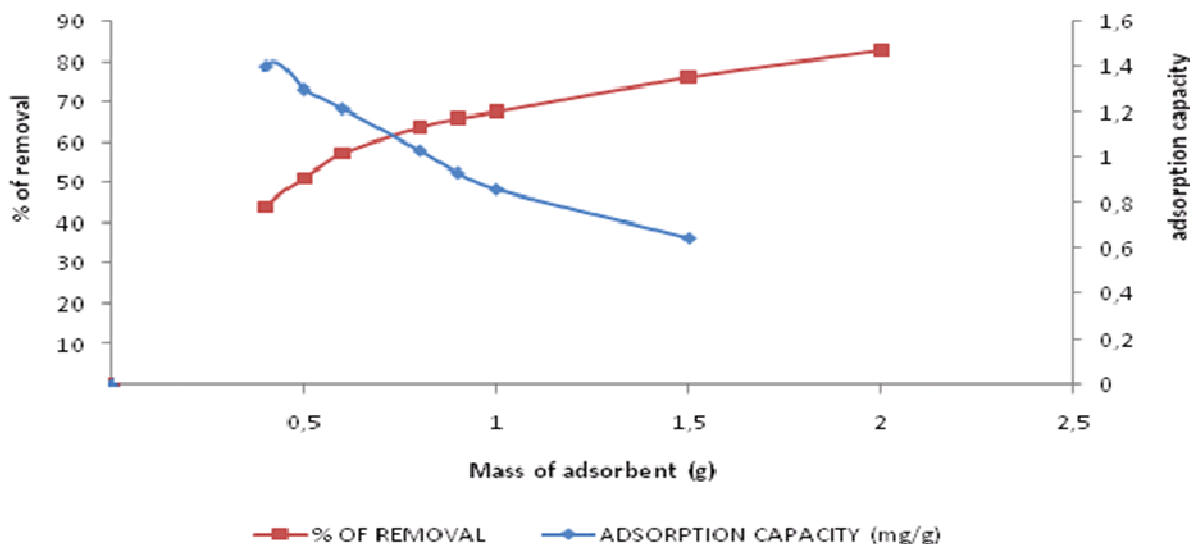


Figure 3

Effect of adsorbent dosage on amaranth adsorption on Sa01, (Initial concentration = 50 mg/L, pH = 2)

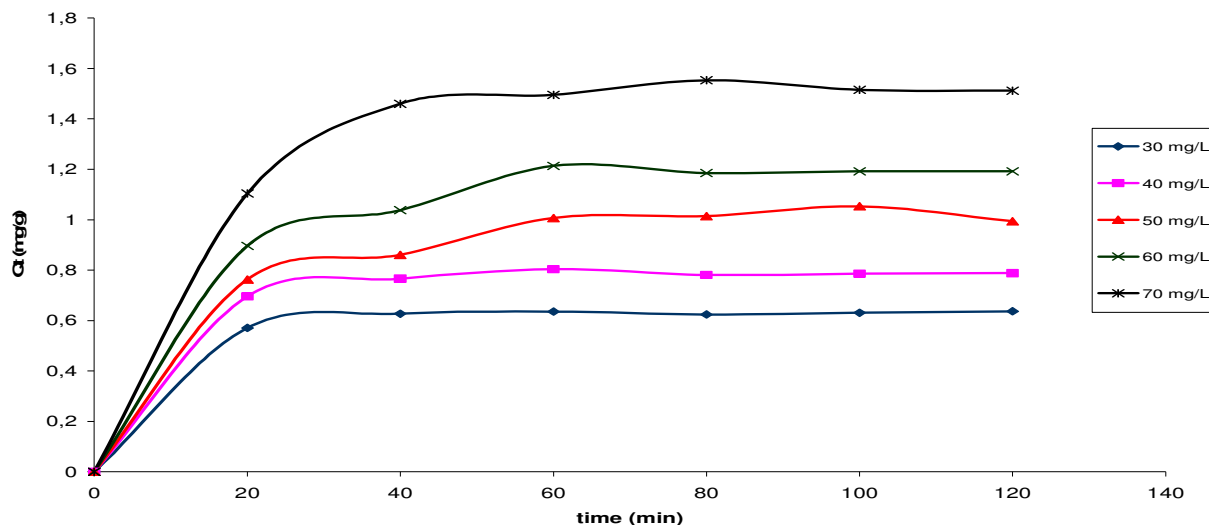


Figure-4

Effect of agitation time and initial dye concentration of amaranth on sa01, (Adsorbent dose = 1 g/25 mL, pH = 2)

Isotherm Modeling: Isotherms describe the equilibrium relation that exist between the concentrations of the adsorbate on the solid phase and in the liquid phase, thus it is critical in optimizing the use of adsorbents¹⁸. The equilibrium data for the adsorption of amaranth over the concentration range from 30 – 80 mg/L at ambient temperature of 25°C, pH 2, contact time of 40 minutes and 1 g/25 mL Sa01 were analyzed using two adsorption isotherm models: The Langmuir and Freundlich isotherm models are expressed by equations (8) and (9) respectively.

Langmuir isotherm model: The Langmuir isotherm is based on the assumption that adsorption occurs on a homogeneous

surface^{16,19}. The linear form of the Langmuir adsorption isotherm equation is:

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_m} + \frac{1}{KQ_m} \quad (8)$$

where C_e is the equilibrium concentration of adsorbate in the solution (mg L^{-1}); Q_e the amount of adsorbate adsorbed on the adsorbent at equilibrium (mg g^{-1}); Q_m the monolayer adsorption capacity (mg g^{-1}) and K is the Langmuir adsorption equilibrium constant (L mg^{-1}), which is related to the energy of adsorption.

The Langmuir constants, Q_m and K were determined from the intercept and the slope of the straight line obtained by plotting C_e/Q_e versus C_e (figure 5).

Freundlich isotherm model: Freundlich model suggested that adsorption process involves heterogeneous adsorption with different classes of adsorption sites^{2,9,20}. The linearized Freundlich isotherm equation that corresponds to the adsorption on heterogeneous surface is given by:

$$\ln Q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (9)$$

where Q_e is the quantity of solute adsorbed at equilibrium (mg of adsorbate per g of adsorbent). C_e is the concentration of adsorbate at equilibrium (mg/L). K_F and n are Freundlich constants related to adsorption capacity and adsorption intensity of the adsorbent respectively. The Freundlich isotherm

constants are calculated from the plot of $\ln Q_e$ versus $\ln C_e$ (figure 6).

Isotherm constants obtained from equations (8) and (9) are represented in table 3. From the correlation coefficients (R^2) obtained, the adsorption process was found to fit well to the Freundlich model than the Langmuir. Thus adsorption of amaranth on Sa01 occurred heterogeneously in majority while homogeneous surface adsorption occurred but in minority¹¹. The maximum adsorption capacity was 1.845 mg/g. Freundlich constants, K_F and $1/n$ were 0.2811 L/g and 0.460 respectively. The Freundlich exponent $1/n$ indicates favourable condition of adsorption of amaranth onto smectite since $0 < 1/n < 1$ ^{2,9,20}.

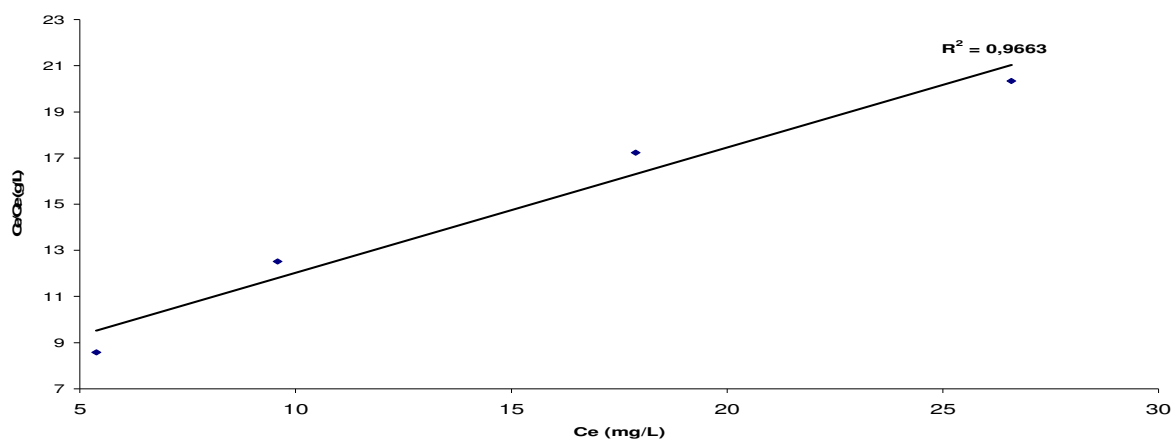


Figure-5
 Linear plot for the Langmuir Isotherm for the adsorption of amaranth on Sa01 (pH = 2)

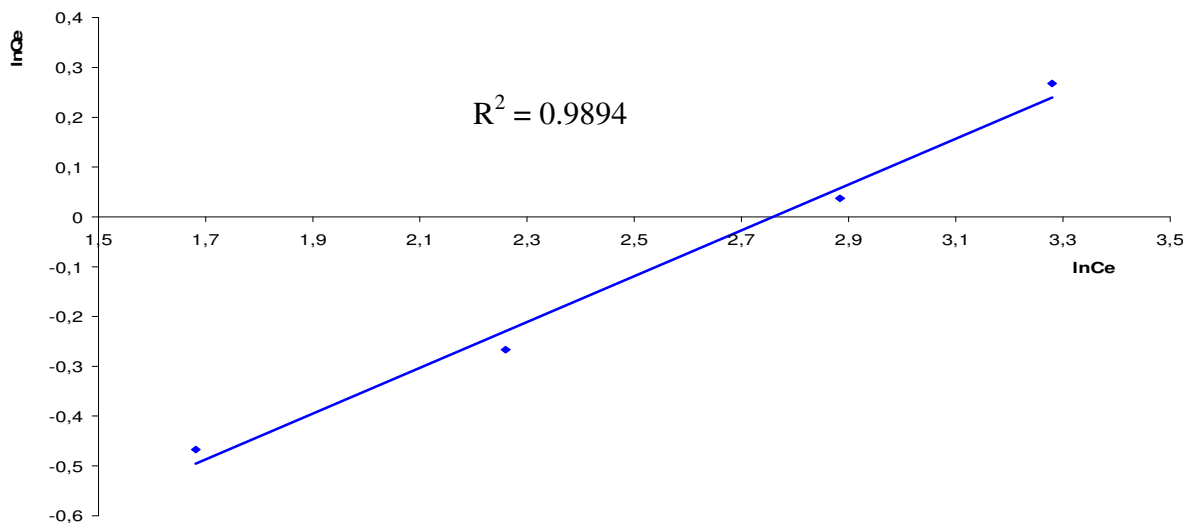


Figure-6
 Linear plot for the Freundlich Isotherm model for the adsorption of amaranth on Sa01 (pH = 2)

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless equilibrium parameter (separation factor) defined by Mackay as:

$$R_L = \frac{1}{1 + KC_0} \quad (10)$$

where K is the Langmuir constant and C₀ is the initial dye concentration (mg/L). R_L values indicate the type of isotherm: to be irreversible (R_L = 0), favorable (0 < R_L < 1), linear (R_L = 1), or unfavourable (R_L > 1)²¹. The R_L values calculated from each initial dye concentration are tabulated in table 4. Each initial concentration shows favourable occurrence of monolayer adsorption with R_L values between 0 and 1 (0 < R_L < 1)^{3, 18, 22}.

Table-3

Langmuir and Freundlich isotherm constants using Sa01 as adsorbent (Sa01 < 80µm).

Isotherm	Parameter
Langmuir	
Q _m	1.845
K _L	0.0822
R ²	0.966
Freundlich	
K _F	0.281
1/n	0.46
R ²	0.989

Table-4

R_L values for different initial concentrations

Initial dye concentration C ₀ (mg/L)	20	40	60	80
R _L	0.289	0.234	0.169	0.135

Kinetic Modeling: Evaluation of the adsorption kinetics of amaranth dye onto Sa01, the pseudo-first order and pseudo-second order models were used.

The correlation coefficients (R²) values and comparison of experimental and theoretical adsorption capacity Q_e were used

to predict the fitness of the kinetic models to the adsorption systems.

Pseudo-first-order kinetic model: For the pseudo-first-order process, the linear form of Lagergen equation was expressed as:
 $\ln(Q_e - Q_t) = \ln Q_e - k_1 t$ (11)

where Q_e and Q_t are the adsorption capacity (mg/g) at equilibrium and at time t respectively. K₁ is the rate constant of the pseudo-first-order adsorption (mg/g.min)²³. The linear plot of ln(Q_e-Q_t) versus t was used to determine K₁, Q_e and R² (table 5).

Pseudo-second-order kinetic model: The pseudo-second order rate equation was expressed in equation (12)

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (12)$$

where K₂ is the rate constant of pseudo-second-order model (g/mg.min) and Q_e is derived from the linear plot of t/Q_t versus t (figure 7)²⁴. The second-order rate constant was used to calculate the initial adsorption rate (h) given by the equation h = K₂Q_e².

The rate constants and correlation coefficients (R²) of the two kinetic models are listed in table 5. From the results, pseudo-second-order kinetic model gave R² > 0.99 for all concentrations. The values of the rate constants decreased with increase in initial amaranth dye concentration. The Q_e values calculated from the linear plot of the pseudo-second-order kinetic model were also found to be in agreement with experimental Q_e values (table 5). Since pseudo-second-order kinetic model fitted better with this system than the pseudo-first-order kinetic model, coupled with the high agreement between its calculated and experimental Q_e values it can be suggested that the adsorption was controlled by chemisorptions^{1,17}. This process involves valence forces through exchange of electrons between adsorbate and adsorbent. Also the decrease in rate constant K₂ as the initial concentration of amaranth dye increases reveals the fact that it is faster for the adsorption system with amaranth to reach equilibrium at lower initial concentration. This phenomenon is observed in figure 4¹¹.

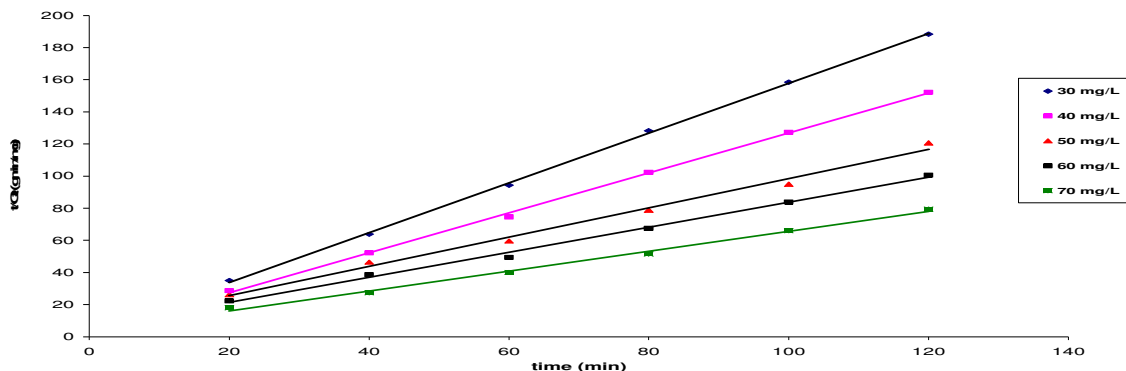


Figure 7
Linear plot for the pseudo-second order kinetics for the removal of amaranth by Sa01 (pH = 2)

Table-5
Comparison of the pseudo-first-order, pseudo-second-order adsorption rate constants and calculated and experimental Q_e values obtained at different initial Concentrations

Models	Parameter	C_0 (mg/L)				
		30	40	50	60	70
$Q_{e, \text{exp}}$ (mg/g)		0.627	0.804	1.007	1.214	1.515
Pseudo-first order	R^2	0.6449	0.8759	0.5556	0.9263	0.7218
	K_1 (h ⁻¹)	0.0242	0.0175	0.0176	0.0295	0.0208
	$Q_{e, \text{cal}}$ (mg/g)	0.0585	0.1086	0.2768	0.5043	0.3366
Pseudo-second order	R^2	0.9996	0.9992	0.9925	0.9964	0.9960
	h (mg/g min)	2.8985	2.5308	7.3961	5.8016	3.7361
	K_2 (g/mg min)	0.8284	0.6112	0.1121	0.1048	0.1027
	$Q_{e, \text{cal}}$ (mg/g)	0.6453	0.8041	1.0984	1.2822	1.6147

Conclusion

The removal of amaranth from aqueous solutions using Sa01 as low cost adsorbent was studied using batch adsorption mode under different conditions. Two types of isotherm models were investigated; the Langmuir and Freundlich Isotherms. The Freundlich adsorption isotherm was found to correlate better the experimental data with $R^2 = 0.9894$. Values of the separation factor from Langmuir isotherm were in the range $0 < R^2 < 1$ and $n = 2.174$ from the Freundlich isotherm, indicated favourable adsorption process.

The experimental data obtained from the study was fitted with two different kinetic models. Based on the R^2 values and the comparison of the experimental and theoretical values of adsorption capacities Q_e , it was concluded that the adsorption process followed pseudo-second order kinetic model with chemisorptions as the rate-limiting step.

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