

Removal of Safranin-O from Aqueous Solution by Adsorption onto Kaolinite Clay

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Abstract

In this study, Natural Raw Kaolinite (NRK) clay was used as an adsorbent for the investigation of the adsorption kinetics, isotherms and thermodynamic parameters of a cationic dye Safranine-O, also known as Basic Red 2 (BR2) from aqueous solution. The effects of pH, temperature, initial dye concentration and contact time on the adsorption capacity were evaluated and the adsorbent was characterized by XRD, BET and FTIR. The pseudo-first-order, pseudo-second-order kinetic models and the intraparticle diffusion model were used to describe the kinetic data and the rate constants were evaluated. The experimental data fitted very well with the pseudo-second-order kinetic model and also followed intraparticle diffusion model revealing that diffusion is not only the rate-controlling step. The Langmuir Freundlich and Dubinin-Radushkevic adsorption models were applied to describe the equilibrium isotherms and the isotherm constants were also determined. The Langmuir model agrees with experimental data well. The activation energy, change of Gibbs free energy, enthalpy and entropy of adsorption were also evaluated for the adsorption of BR2 onto NRK.

Keywords

Basic Red 2, Kaolinite, Adsorption, Kinetic, Thermodynamic, Surface Area

1. Introduction

Dyes and pigments represent one of the problematic pollutant groups in wastewaters. They are aromatic compounds with various functional groups and can be classified as anionic (direct, acid, and reactive dyes), cationic (basic dyes) and non-ionic (disperse dyes). They are introduced into wastewaters from various sources, ranging

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from the dye manufacturing and textile finishing to rubber, leather, paper, plastics, and cosmetics industries. They can degrade to produce carcinogens and toxic products [1]. Most dyestuffs are designed to be resistant to environmental conditions like: light, effects of pH and microbial attack, etc. In addition, a very small amount of dye in water is highly visible due to their desirable aesthetic aspect [2] [3]. Thus, the removal of dyes from effluents could be considered as an important issue for risk assessment.

Several physical or chemical processes such as flocculation, precipitation, ion exchange, membrane filtration, electrochemical destruction, irradiation and ozonation are used to treat dye laden wastewaters [4] [5]. However, these conventional physicochemical and even biological treatment methods are ineffective, costly and mostly lead to generation of sludge or formation of byproducts that are undesirable [6] [7]. Among the physical methods available, adsorption process has proven to be highly effective in the removal of dyes from wastewater, especially if the adsorbent is inexpensive and readily available [8]. The use of clay minerals over commercially available adsorbents has become popular because they are low-cost, abundant, available, non-toxic and potential ion exchange materials. A few of clay minerals among which are sepiolite [9], kaolinite [10], montmorillonite [11], smectite [12], bentonite [13] and zeolite [14] have been investigated for removal harmful heavy metals and organic pollutant such as dyes.

Kaolinite is a 1:1 aluminosilicate consisting of a tetrahedral and an octahedral sheet. The silicate sheets (Si_2O_5) bonded to aluminum oxide/hydroxide sheets $(Al_2(OH)_4)$ called gibbsite are the successive 1:1 layers which are held together by hydrogen bonding of adjacent silica and alumina sheets. The permanent negative charge is produced because of isomorphic replacement of Si⁴⁺ by Al³⁺ in the silica tetrahedral sheet or of trivalent metal ions (such as Al³⁺) by divalent ions Fe²⁺ and Mg²⁺ in the alumina octahedral sheet, leaving a single negative charge for each substitution. As a result, both the alumina sheet surfaces and the crystal edges have a pH-dependent variable charge caused by broken bonds around the edges and exposed hydroxyl basal [15] [16]. Hence, the kaolinite surface is expected to have two kinds of binding sites that could interact with cations or basic dyes effectively.

Basic dyes are known as the brightest class of soluble dyes used in the textile industries for dyeing acrylic, nylon, silk and wood materials. Their tinctorial value is very high; less than 1 ppm of the dye produces obvious coloration. Basic dye can also cause allerge dermatitis, skin irritation, cancer and mutations [17]-[20]. The present study has been designed to investigate a single stage adsorption of "Safranin-O", a cationic dye, also known as Basic Red 2 (BR2) (Figure 1) onto kaolinite clay mineral from Ubulu Ukwu in Delta State, Nigeria as a model of basic dyes. Of particular interest in this work was to investigate the equilibrium, thermodynamics and kinetics of BR2 removal by adsorption from aqueous solution. The different kinetic models employed to analyze the data for BR2 adsorption are pseudo-first-order, pseudo-second-order and intra-particle diffusion models. Experimental data were analyzed using the Langmuir, Freundlich and Dubinin-Radushkevic adsorption iso-therms.

2. Materials and Methods

2.1. Adsorbate and Adsorbent

The adsorbate, Saffranin-O, a cationic diazo dye with the molecular characteristics shown in **Table 1**, was supplied by BDH chemicals Poole England certified for use as a chromosome stain in Flemming triple stain with a minimum dye content of 80%. It is referred to as BR2 in this work. The chemical structure of BR2 is given in **Figure 1**. The adsorbent, kaolinite clay was obtained from Ubulu-Ukwu, Delta State, Nigeria.



Figure 1. Molecular structure of Safranin-O (BR2).

Table 1. Some molecular properties of Safranin-O (BR2).				
Properties	BR2			
C.I.	50,240			
Colour	Redish			
Dye	80%			
Formula	$C_{20}H_{19} N_4Cl$			
Molecular Weight (FW)	350.85 g·mol ^{−1}			
рКа	5.28			
Valence	+1			
$\lambda_{ m max}$	520 nm			

2.2. Preparation of Adsorbent

Stones and other non-clay particles were removed from the kaolinite clay sample by hand picking. It was then crushed and sieved through a meshed sieve to further remove the larger non-clay fractions. 100 g of the clay sample was dispersed in distilled deionized water and stirred continuously with a stirrer for 6 h. Two layers were formed—a settlable heavier particulate layer and a colloidal suspension which is the kaolinite clay. The suspended layer was carefully and completely decanted after which the colloidal layer is recovered by centrifugation. It was further purified by using Renolds and Moores [21] method. This involves the stirring of the mixture in 30% hydrogen peroxide solution till all effervescence has ceased. The mixture was kept standing overnight to allow any carbonaceous substance to be oxidized and with this treatment any organic substances remaining in the clay is removed. The supernatant was decanted and the kaolinite clay washed thoroughly with distilleddeionized water to remove traces of hydrogen peroxide. The peroxide free kaolinite clay was recovered by centrifugation and oven dried at 353 K to obtain the natural raw and untreated kaolinite clay sample (NRK). The dried sample was carefully crushed and sieved to produce the desired particle size. The X-Ray Diffraction (XRD) powder pattern of NRK was recorded on a Philips PW 3064/60 diffractometer (CuK α radiation; $\lambda = 1.54060$ Å) using a generator voltage of 40 kV and a generator current of 40 mA. A step size of $0.001^{\circ}(2\theta)$ was used with a scan step time of 3.175 s per step. The infrared spectrum of NRK was obtained on a SHIMADZU Fourier Transform Infrared spectrometer under dry air prepared as KBr pellets. BET surface area and porosity measurements of clay sample were determined by quanta chrome quadrawin version 5.05 (Quantachrome, Germany). The samples were outgassed overnight (20 h) at an outgassing temperature of 150.0°C. The specific surface area was calculated on the basis of the BET equation installed in the machine.

2.3. Preliminary Experiments

Preliminary experiments demonstrated that equilibrium was established in less than 1 h. A 2.50 g NRK was dispersed in 500 ml dye solution of 100 mg/L concentration. One milliliters (1 ml) of sample were withdrawn from the batch adsorption system at predetermined time intervals with the use of a micro-pipette (Dragon-Med Pipettor) and the supernatant was centrifuged at a speed of 2500 rpm in 15 min to separate the used adsorbent from the remaining dye solution. The unadsorbed dye concentrations were determined from their absorbance characteristics in the UV-vis spectrophotometer [surgifriend SM7504UV/visible 911]. A linear correlation was established from the plot of BR2 dye concentration and the absorbance at $\lambda_{max} = 520$ nm, to give a correlation coefficient $r^2 = 0.994$. The adsorption capacity of the dye BR2 was calculated using the following relationships:

$$qt = \frac{\left(C_o - C_t\right) \times V}{W} \tag{1}$$

$$\% = \frac{\left(C_o - C_t\right) \times 100}{C_o} \tag{2}$$

where $q_t \pmod{g}$ is the amount of BR2 dye adsorbed at contact time $t \pmod{t}$, $C_0 \pmod{L}$ is the initial dye concentration, $C_t \pmod{W}$ is the dye concentration at time t, V (L) is the volume of the solution and W (g) is the mass of clay in the solution.

2.4. Adsorption Study

2.4.1. Effect of Initial pH

Effect of initial solution pH was investigated using various pH values in the range of 3 - 11. The pHs of solutions were adjusted to the desired pH by adding drops of 0.1 M NaOH or HCl. To 30 ml of aqueous solution containing 100 mg/L BR2 dye, 0.5 g of NRK was added.

2.4.2. Effect of Adsorbent Dose

The effect of adsorbent dose variation on BR2 dye adsorption was studied by adding 0.05, 0.10, 0.15, 0.20, 0.30, 0.45, 0.70, 0.90 and 1.35 g of NRK to 30 ml aqueous solution having an initial BR2 concentration of 300 mg/L.

2.4.3. Effect of Initial Dye Concentration

The effect of the initial BR2 concentration variation was investigated by taking a fixed amount of the clay samples, 150 mg, and the concentration of dye adjusted from 20 to 300 mg/L at a volume of 30 ml.

2.4.4. Effect of Temperature

The effect of temperature was examined using 2.50 g adsorbents added to separate volumes of 500 ml solutions containing BR2 concentrations of 50, 100 and 200 mg/L at different solution temperature varying between 20°C to 55°C.

2.5. Adsorption Kinetics

The influence of contact time on the amount of BR2 adsorbed was investigated at a varied time interval at a constant adsorbent weight of 0.15 g and varied concentrations of 20, 50, 100, 150 and 170 mg/L of BR2 at a given temperature. Kinetic models, *i.e.* the pseudo-first order, the pseudo-second order and intraparticle diffusion models were considered for interpreting the experimental data obtained:

2.5.1. Pseudo-First-Order Model

The pseudo-first order kinetic model of Lagergren was used in order to estimate the adsorption capacity of the adsorbent [22]:

$$\frac{\mathrm{d}q_t}{\mathrm{d}t} = k_1 \left(q_e - q_t \right) \tag{3}$$

where q_e and q_t (mg·g⁻¹) are the amounts of dye adsorbed onto the adsorbent at equilibrium and at various times t (min); k_1 (min⁻¹), the rate constant of the first-order model for the adsorption process; integrating and applying the boundary conditions (t = 0 and $q_t = 0$ to t = t and $q_t = q_t$),

Equation (3) gives the form:

$$\ln\left(q_e - q_t\right) = \ln\left(q_e\right) - k_1 t \tag{4}$$

The rate constant k_1 and q_e were obtained from the slope and intercept of the linear plots of $\ln(q_e - q_t)$ against t, respectively.

2.5.2. Pseudo-Second-Order Model

The pseudo-second-order [23] reaction model is expressed as

$$\frac{\mathrm{d}q_t}{\mathrm{d}t} = k_2 \left(q_e - q_t\right)^2 \tag{5}$$

where k_2 (g·mg⁻¹·min⁻¹) is the rate constant of the pseudo-second-order model for the adsorption process q_e (mg/g) and q_t (mg/g) have the same definition as above for Equation (4). Integrating and applying boundary conditions (t = 0 and $q_t = 0$ to t = t and $q_t = q_t$), Equation (5) becomes;

$$q_t = \frac{t}{\left[\left(\frac{1}{k_2 q_e^2} + t/q_e\right]}$$
(6)

While the linear form is

$$\frac{t}{q_t} = \frac{1}{\left(k_2 q_e^2\right)} + \frac{t}{\left(q_e\right)}$$
(7)

The rate constant k_2 (g·mg⁻¹·min⁻¹) and q_e (mg/g) were obtained from the intercept and slope of the linear plots of t/q_t against t, respectively.

2.5.3. The Intraparticle Diffusion

The intraparticle diffusion equation was used accordingly which is written as

$$q_t = k_p t^{0.5} + I \tag{8}$$

where q_t is same as defined as above, k_p , the intraparticle diffusion rate constant (mg·g⁻¹·min^{-0.5}) and I, give an idea about the thickness of the boundary layer, *i.e.*, the larger the intercept the greater is the boundary layer effect [24]. To follow the intra-particle diffusion model, a plot of q_t against $t^{0.5}$ should give a linear line where a slope is k_n and intercept I.

2.6. Adsorption Isotherms

2.6.1. Langmuir Isotherm

The Langmuir isotherm has been widely applied to pollutants sorption processes. A basic assumption of the Langmuir theory is that the adsorption takes place at specific homogenous sites in the adsorbent. Moreover, when a site is occupied by a solute, no further adsorption can take place at this site [25]. The Langmuir adsorption isotherm can be written as follows

$$q_e = \frac{K_L q_{\max} C_e}{1 + K_L C_e} \tag{9}$$

where q_e (mg·g⁻¹) is the amount of BR2 adsorbed onto clay samples at equilibrium, q_{max} (mg·g⁻¹) is the theoretical monolayer capacity, K_L (L/mol) is the Langmuir equilibrium constant related to the affinity of binding sites and energy of adsorption and C_e (mg·L⁻¹) is the equilibrium solution concentration. The linear form of the Langmuir isotherm equation is written as:

$$\frac{C_e}{q_e} = \frac{C_e}{q_{\max}} + \frac{1}{K_L q_{\max}}$$
(10)

The values of q_{max} and K_L are evaluated from the intercept and the slope of the linear plot of experimental data of C_e/q_e versus C_e respectively.

2.6.2. Equilibrium Parameter

One of the essential characteristics of the Langmuir equation is that it could be expressed by dimensionless constant called equilibrium parameter (R_L) . R_L is calculated using the following equation:

$$R_L = 1/(1 + K_L C_O) \tag{11}$$

where K_L (dm³·mol⁻¹) is the Langmuir constant and C_0 (mg·L⁻¹) is the highest initial dye concentration. The parameter indicates the type of isotherm to be; irreversible ($R_L = 0$), favorable ($0 < R_L < 1$), linear $(R_L = 1)$ or unfavorable $(R_L > 1)$ [26].

2.6.3. Freundlich Isotherm

The Freundlich isotherm which is used to describe heterogeneous systems is expressed by the following empirical equation:

$$q_e = K_F C^{1/n} \tag{12}$$

where K_F (L·g⁻¹) and *n* (dimensionless) are the Freundlich adsorption isotherm constants. By integrating Equation (12) a linear form of the Freundlich equation is obtained;

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{13}$$

2.6.4. Dubinin-Radushkevic (D-R) Isotherm

The adsorption data were analyzed using the linear form of the *D-R* isotherm, [27] below:

$$q_e = q_{\max} \exp\left(-BD\left[RT\ln\left(1+1/C_e\right)\right]^2\right)$$
(14)

where $q_e \pmod{g}$ is the amount of BR2 adsorbed, $q_{\max} \pmod{g}$ is the *D-R* monolayer adsorption capacity, *T* (K) is the temperature and $C_e \pmod{g}$ is the equilibrium concentration of BR2 left in solution. Reducing Equation (14) and taking the logarithm of both side of the equation

$$\ln q_e = \ln q_{\rm max} - BD\varepsilon^2 \tag{15}$$

BD (mol²·kJ⁻²) is a constant related to sorption energy E (KJ·mol⁻¹), via the following relationship

$$E = \frac{1}{(2BD)^{0.5}}$$
(16)

The mean adsorption energy (E) is the Gibbs free energy change when 1 mol of the ion is transferred to the surface of the solid from infinity in the solution and ε is the Polanyi potential, which is related to the equilibrium concentration as:

$$\varepsilon = RT \ln\left(1 + 1/C_{e}\right) \tag{17}$$

The *BD* and q_{max} , Equation (15) are determined from slope and intercept of the plots of $\ln q_e$ versus ε^2 respectively.

3. Result and Discussion

3.1. Characterization of Adsorbent

The mineralogical composition of NRK determined from X-ray diffractogram shown in Figure 2. The clay was



Figure 2. X-ray diffraction pattern of NRK.

found to contain predominantly kaolinite and quartz minerals with some small amounts of illite, gibbsite and goethite (**Table 2**). The data suggests that the NRK consists mixture of kaolinite-illite clay mineral and this was inferred from the peaks with higher percentage relative intensities (**Table 2**). The percentages of elemental composition on the clay mineral suggests that it contains some little amount of nitrogenous compound (**Table 3**) and BET nitrogen adsorption method for NRK specific surface area and pore determination is shown in **Table 4**. Specific (BET) surface area is the total surface area of a solid that adsorbs a gas per unit weight of the solid. While the external surface area of a solid is the area of such solid which is attributed to the pores in other words it is the exposed surface of a solid residing almost exclusively inside the micropores for adsorption as such for a microporous solid/materials it is expected that the value for external surface area (ESA) be higher than the value for the specific surface area (SSA) (**Table 4**).

3.2. FTIR Spectra Analysis of the Adsorbent

Fourier transformed infrared spectra in the range 4000 - 500 cm⁻¹ is shown in **Figure 3**. The band at 3620 cm⁻¹ corresponds to the stretching vibration of the internal hydroxyl group of kaolinite while those at 3653 and 3697 cm⁻¹ corresponds to the interlayer hydroxyl stretching mode [28]. While those at 3653 cm⁻¹, 3697 cm⁻¹ corresponds to interlayer hydroxyl stretching mode. The band at 2359 cm⁻¹ is assigned to the C-H stretching vibration

	· · ·	
Composition	d-spacing [Å]	Relative intensity (%)
Kaolinite	7.166	14.32%
Kaolinite	4.486	7.91%
Quartz	4.263	27.63%
Kaolinite	3.582	11.75%
Quartz + illite	3.351	100.00%
Dolomite	2.701	<5%
Quartz	2.568	6%
Goethite	2.504	<5%
Goethite	2.461	<5%
Goethite + gibbsite	2.343	<5%
Gibbsite	2.242	<5%
Gibbsite	2.133	<5%
Dickite	1.983	<5%

Table 2. XRD compositiona	l analysis of NRK cla	y sample used	d in this study.
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Fable 3. Elemental composition of purified NRK clay sample used in this study.						
Clay	Н	S	Ν	С	C/N	
NRK	1.147%	0.258%	1.33%	0.12%	0.0928	

Table 4. BET surface area and pore data for NRK.

Parameter	Value
Specific surface area (SSA)	$27.8 \text{ m}^2 \cdot \text{g}^{-1}$
Total pore volume	$0.054 \text{ cm}^3/\text{g}$
Average pore size	2.67 nm
Average pore radius	7.713 Å
External surface area (ESA)	$34.61 \text{ m}^2 \cdot \text{g}^{-1}$



and the Si-O vibrational band can be observed at 1008 cm⁻¹ and 1033 cm⁻¹. The band at 1631.83 cm⁻¹ is assigned to the O-H stretching vibration of H₂O of NRK [29].

3.3. Effect of Initial pH

Figure 4 shows the effect of pH on BR2 adsorption from aqueous solution onto NRK. The results showed that the percentage dye adsorption by the raw kaolinite increased significantly as pH increased. This pH effective-adsorption could have resulted from the fact that the pH of the dye solution can significantly affect the surface charge of NRK and the degree of ionization and speciation of BR2 molecule [30]. In addition, the negative charges on NRK which are mainly derived from the broken bonds around the edges and exposed hydroxyl basal strongly depend upon pH [31].

The increase in BR2 adsorption over the pH range may be related to two possible mechanisms: electrostatic interaction and chemical reaction between clay mineral and the dye molecules [32]. There is a significant increase in electrostatic attractions between positive charges of the cationic dye and the negative charges on the surface of NRK, thereby increasing dye adsorption. As the pH of the system increases, the number and strength of negatively charged sites on NRK increases and the number of positively charged sites on the surface decline which translates into a progressive increase in adsorption capacity of NRK. Similar observations have been made by other researchers [33]-[35].

3.4. Effect of Adsorbent Dose Variation

The percentage BR2 removal (%) and amount adsorbed, q_e , per weight (g) NRK is shown in **Figure 5**. A close observation showed that an increase in adsorbent dose from 50 mg to about 2.70 g resulted in a gradual and progressive decrease in adsorption capacity of NRK clay with respect to BR2 with a corresponding increase in the percentage dye adsorbed. This implied that the number of adsorption sites increased as adsorbent mass increases and this can be attributed to two possibilities; the increase in the adsorbent surface area and availability of more adsorption sites. However, the further increase in the amount of the adsorbent did not affect the uptake capacity significantly (**Figure 5**). Such a phenomenon was similar to those observed by Porkodi and Vasanth (2007) [36]; and Tahir and Rauf (2006) [37]. Consequently, the equilibrium adsorption capacity, q_e , decreased from 27.3 to 1.65 mg/g, whereas, the percentage dye removal efficiency indicated that there was 99.1% increase in the dye removal as the adsorbent dose is increased from 50 mg to 2.70 g.

3.5. Kinetic Studies

The amount of BR2 removed at various initial dye concentrations by NRK as a function of time at constant adsorbent weight is graphically illustrated in **Figure 6**. From this figure, it was observed that the amount of BR2



Figure 4. Effect of pH variation on the adsorption of BR2 onto NRK.



Figure 5. The variation of adsorbent dose at a constant BR2 dye concentration.



Figure 6. Kinetic curves of BR2 retention by NRK clay at different initial BR2 concentrations.

uptake, increased with time at all the initial dye concentrations. All the curves have similar characteristics, showing a fast adsorption, between time t = 0 and 40 min after which, there is a slower adsorption profile until equilibrium was attained and the removal of BR2 by NRK clay became constant. For a contact time of 180 min, percentage dye removal was 66.6% for 170 mg/L dye solution, 70.9% for 150 mg/L dye solution, 85.4% for 100 mg/L dye solution, 88.4% for 50 mg/L dye solution and 88.8% for 20 mg/L dye solution. The sorption reaction at all initial dye concentrations was found to be rapid.

For the kinetics study, two kinetic models were tested to explain the data presented in **Figure 6**, Viz; pseudo-first order, and pseudo-second order models. The lower values of R^2 and the difference between the experimental and calculated adsorption equilibrium values showed that the pseudo-first order model failed to describe the adsorption kinetics. Meanwhile, the agreement of experimental data with the calculated value is corroborated by the correlation coefficient (R^2) , meaning that pseudo-second order model (**Figure 7**) best describe the kinetics of the adsorption process (**Table 5**). The values of the rate constants (k_2) were found to decrease from 0.189 to 0.023 mg/(g·min) as the initial concentration increased from 20 to 170 mg/L, an indication that the adsorption process is highly concentration dependent, which is consistent with earlier studies [38]-[40].

The kinetic data obtained was also analyzed by the intra-particle diffusion model. The curve (not shown) is multilinear which means that the intraparticle diffusion was not the only rate-limiting step and that there may be other steps involved in control of the rate of adsorption, all of which may be operating simultaneously [41]-[43]. This kind of multi-linearity in the shape of the intraparticle diffusion plot was also observed by Tarun *et al.* (2009) in the use of Saw dust and neem bark as low-cost natural biosorbent for adsorptive removal of Zn(II) and Cd(II) ions from aqueous solutions [44]. These phases in the intraparticle diffusion plot suggest that the adsorption process is complex such that the initial portion of the curved indicates a boundary layer effect; the second portion is then due to intraparticle diffusion while the third linear portion is likely due to chemical reaction [42].



Figure 7. Pseudo-second order kinetic plot for adsorption of BR2 on NRK under different dye concentrations at temperature of 31°C.

Pseudo-first order				Pse	Pseudo-second order		Intraparticle diffusion			
*CO (mg/L)	qe _{exp} (mg/g)	qe _{cal} (mg/g)	$\begin{array}{c} \mathbf{K}_1 \\ (\min^{-1}) \end{array}$	R_1^2	$\frac{qe_{2 cal}}{(mg/g)}$	K ₂ (g/mg/min)	\mathbf{R}_2^2	$\begin{array}{c} \text{Kid} \\ (\text{mg} \cdot \text{g}^{-1} \cdot \text{min}^{1/2}) \end{array}$	$(\text{mg} \cdot \text{g}^{-1} \cdot \text{min}^{1/2})$	R_i^2
20	3.44	0.824	0.0322	0.93	3.460	0.189	0.999	0.073	2.65	0.999
50	9.89	2.000	0.0438	0.974	10.000	0.147	0.999	0.422	7.26	0.941
100	17.07	4.246	0.0530	0.913	17.241	0.072	0.999	1.663	11.93	0.98
150	20.28	10.864	0.0415	0.871	20.408	0.025	0.999	4.79	10.59	0.96
170	22.64	11.803	0.0461	0.866	22.727	0.023	0.998	4.79	10.14	0.96

Table 5. Kinetics parameters for the removal of BR2 dye by
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*Initial BR2 dye concentration.

The slope of the second portion of the plot is used in defining the intraparticle diffusion parameter, k_{id} (mg/g min^{1/2}), as shown in Table 5 and this parameter increases as the concentration of the BR2 was increased.

On the other hand, the intercept (I), of the plot reflects the boundary layer effect. The larger the intercept, the greater the contribution of the surface sorption to the rate limiting step [45] this was also observed as the dye concentration increases (Table 5).

3.6. Thermodynamic Parameters

The thermodynamic parameters of the adsorption were determined using the following equations:

$$\ln\left(k_{d}\right) = \frac{\Delta S_{ads}}{R} - \frac{\Delta H_{ads}}{RT}$$
(18)

$$\Delta G_{\rm ads} = \Delta H_{\rm ads} - T \Delta S_{\rm ads} \tag{19}$$

where k_d is the distribution coefficient at different temperatures (293, 303, 313, and 328 K) and is equal to the ratio of the equilibrium amount adsorbed (Ce_A in mg/L) to the equilibrium concentration (Ce_S in mg/l) at different temperatures (*i.e.* $k_d = Ce_A/Ce_S$) and R is the gas constant. The values of ΔH_{ads} and ΔS_{ads} for changes in enthalpy and entropy of reactions were obtained from the slope and intercept respectively of the linear plot of $\ln k_d$ versus 1/T (Figure 8). Equation (19) was applied to calculate the standard change in Gibbs free energies, ΔG_{ads} at different temperatures.

The thermodynamic parameters are shown in **Table 6**. It is obvious that the adsorption of BR2 onto NRK is spontaneous and exothermic as the values for ΔG and ΔH are negative respectively and the entropy change (ΔS_{ads}) showed the disorderliness of the reacting dye particles during the interaction with surface of NRK.

3.7. Equilibrium Studies

The adsorption data were analyzed to see whether the isotherm obeyed the Langmuir [25], Freundlich [26] and Dubinin-Radushkevich (D-R) [27] isotherm models equations. The non linear equations of each models were



Figure 8. Van't Hoff plot for the BR2 dye adsorption onto NRK clay at different temperature.						
Table 6. Thermodynamic parameters for BR2 adsorbed onto NRK clay sample.						
CI						
Clay		$\Delta G(KJ)$	mol)		$\Delta H (KJ \cdot mol^{-1})$	$\Delta S (KJ^{-1} mol^{-1})$
	293 K	303 K	313 K	328 K		
NRK	-10.23	-7.45	-8.45	-7.24	-35.34	-85.72

earlier respectively expressed in Equations (10), (13) and (15) above. The parameter for these isotherm models for the adsorption of BR2 onto NRK is listed in **Table 7**. The fit of the data for BR2 adsorption onto NRK suggests that the Langmuir isotherm model (**Figure 9**) gave better fittings than those of Freundlich but followed closely by *D-R* models, as is obvious from a comparison of the correlation coefficient (R^2) (**Table 7**). The effect of isotherm was also discussed with a view to predict whether an adsorption system is favorable or unfavorable. This is resolved by the use of the essential feature of the Langmuir isotherm expressed by means of " R_L ", Equation (11), a dimensionless constant referred to as separation factor or equilibrium parameter. The value of R_L was calculated using Equation (11) and incorporated in **Table 7**. As shown, the R_L value for BR2 onto NRK at 31°C is 0.3011 (**Table 7**), a value that lies between 0 and 1, interprets that the adsorption



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Langmuir				
q _{max} (mg/g)	16.234			
K _L (L/mg)	0.431			
R _L	0.3011			
R_L^2	0.9987			
Dubinin	-Radushkevic			
q _{max} (mg/g)	16.071			
E ($KJ \cdot mol^{-1}$)	707.106			
$\beta (\mathrm{mol}^2 \cdot \mathrm{KJ}^{-2})$	$1.0 imes 10^{-6}$			
$\mathbf{R}^2_{\mathrm{D-R}}$	0.9932			
Fre	eundlich			
K _F (L/mg)	0.8966			
1/n	0.6206			
R_F^2	0.9125			

Cable 7. Adsorption	isotherm	constants	for BR2	adsorption	onto NRK
a contraction of the second second	10001101111	e o mo cumeo		adoorption	01110 1 1111

process is favorable [46].

In the energy parameter of *D-R* isotherm models, the sorption energy (β_D) , and mean free energy (E_D) value are estimated at $1.0 \times 10^{-0.6} \text{ mol}^2/\text{kJ}^2$ and 707.106 kJ/mol respectively (**Table 7**). Meanwhile the parameter predicting the type of adsorption was evaluated as E_D which is an indication of physisorption or chemisorptions or ion exchange etc. According to Atkins (1999) [47], physisorption is called a non specific adsorption which occur as a result of long range weak Van der Waals forces between adsorbates and adsorbents. The energy released when a particle is physisorbed is of the same magnitude as the enthalpy of condensation. The enthalpy of physisorption was however measured in the region of less than 20 kJ/Mol [47]. He also argued that chemisorption is a specific adsorption and limited to monolayer coverage of the substrate [47]. Here, a covalent bond is formed between the adsorbate and adsorbent. In this study, the estimated value for mean free energy is higher than 20 kJ/Mol and moreover surface coverage in this experiment fitted best into the Langmuir Isotherm (sorption on homogeneous surface). Hence, adsorption is restricted to monolayer coverage as purposed for chemisorption. A comparison of previous adsorption capacities of BR2 adsorbed onto various adsorbents is presented in **Table 8**. It can be seen that NRK in this study has a lower adsorption capacity compared to other adsorbents.

4. Conclusions

This study investigated the interaction of a cationic dye with the partially negatively charged surface of natural kaolinite namely BR2 (Safranin-O) onto NRK. At the end, it was found that, more than 90 percent of dye removal was achieved by using 150 mg of the adsorbent for an initial dye concentration of 100 mg·L⁻¹.

Due to strong electrostatic interaction between BR2 and NRK, the pseudo-second-order very well predicted the behaviour of adsorption at different dye concentrations and the intraparticle diffusion models (up to 10 min) played a significant role, but it was not the main rate-determining step during the adsorption.

The experimental data best fit with the Langmuir isotherm showing that the surface of the kaolinite particle in the removal of BR2 has homogeneous distribution of active sites within the clay, equal energy of adsorption and equivalent in nature.

The enthalpy change (ΔH^0) for the adsorption process was $-35.34 \text{ kJ} \cdot \text{mol}^{-1}$, an exothermic reaction, which did not indicate a strong chemical interaction between the adsorbed dye molecules and NRK. The ΔG^0 values were negative therefore the adsorption was spontaneous and the negative value of ΔS^0 suggested a decreased randomness at the solid/solution interface.

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Adsorption					
Adsorbents	Capacities (Q _{max})	References			
K. fragilis	138.20 mg/g	Ivo Safarik, 1995			
Activated Carbon	132.5 - 265 mg/g	Ivo Safarik et al., 1997			
Activated Carbon	131.58 mg/g	Gokturk and Kalue, 2008			
Mango Seed Integuments (Untreated)	34.48 mg/g	Mohamad et al., 2012			
Mango Seed Integuments (Treated)	43.47 mg/g	Mohamad et al., 2012			
Rice Husk	105.9 mg/g	Papita and Rahul, 2012			
Palygorskite Clay	200 mg/g	Dakhil et al., 2013			
Kaolinite (NRK) Clay	16.23 mg/g	This Paper			

Table 8. Reported maximum adsorption capacities in the literature for Safranin-O adsorbed onto different adsorbents.

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Abbreviations/Nomenclatures

BR2: Basic Red 2.

BD: A constant related to sorption energy E ($mol^2 \cdot kJ^{-2}$).

 Ce_{A} : Amount adsorbed at equilibrium (mg·L⁻¹).

 Ce_s : Dye equilibrium concentration in solution (mg·L⁻¹).

 C_0 : Initial dye concentration (mg·L⁻¹).

 C_e : Dye concentration at equilibrium (mg·L⁻¹).

 C_t : Dye concentration at a given time (mg·L⁻¹).

D-R: Dubinin-Radushkevic.

E : Mean free adsorption energy of dye transferred to the surface from infinity $(kJ \cdot mol^{-1})$.

h: Initial rate of adsorption (mol/g min).

 k_1 : Equilibrium rate constant of pseudo-first-order sorption (min⁻¹).

 k_2 : Equilibrium rate constant of the pseudo-second-order sorption (g·mg⁻¹·min⁻¹).

 k_d : Distribution coefficient ratio at different temperatures.

 $k_{\rm F}$: Freundlich adsorption isotherm constant (L·g⁻¹).

 K_L : Langmuir adsorption isotherm constant (L·mol⁻¹).

NRK: Natural Raw Kaolinite.

 q_e : Quantity of dye adsorbed at equilibrium (mg·g⁻¹).

 q_{max} : Maximum adsorbed quantity by the adsorbent (mg·g⁻¹).

 q_t : Quantity adsorbed at a given time (mg/g).

R: Gas constant.

T: Adsorption absolute temperature (K).

V: Volume of dye solution (L).

W: Weight of adsorbent in dye solution (g).

 ΔG_{ads} : Gibbs free energy change of adsorption (KJ·mol⁻¹).

 ΔH_{ads} : Enthalpy change of adsorption (KJ·mol⁻¹).

 ΔS_{ads} : Entropy change of adsorption (J·mol⁻¹·K⁻¹).

 ε : Polanyi potential.



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