



## Adsorptive removal of anionic dye from aqueous solutions by mixture of Kaolin and Bentonite clay: Characteristics, isotherm, kinetic and thermodynamic studies

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### ABSTRACT

The textile effluents are considered as potential source of water contaminations. Thus the effective methods were adopted for the removal of dyes and colorants from the textile effluents. In the present research, the removal of textile dye Congo red was carried out by bentonite clay blend with kaolin. The kaolin-bentonite clay (KBC) was used as adsorbent. The adsorption properties of KBC towards Congo red were investigated, at various temperatures  $303-318 \pm 2$  K under the optimized conditions. The adsorption equilibrium data were fitted in Langmuir, Freundlich and Dubinin-Radushkevich adsorption isotherm models and the values of the respective constants were evaluated by employing standard graphical method. From the graph, it was founded that Langmuir model is the best fitted isotherm. Feasibility of adsorption process (RL) and sorption energy ( $E_s$ ) was also determined. The pH of adsorbent was estimated by pH drift method. Kinetics of dye removal was investigated that it follows pseudo second order rate constant. The surface morphology of adsorbent was observed by the Scanning Electron Microscope (SEM).

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### INTRODUCTION

During the recent years, the rapid progress in industrial activities leads to excessive discharge of textile effluents which is associated with the incomplete exhaustion of dyes onto fiber during dyeing processes [1]. The textile finishing industries generates a large amount of wastewater which contains dyes and heavy metals which causes environmental pollution. It is estimated that approximately 100,000 units of textile dyes used and their annual production in the world wide are more than  $7 \times 10^5$  metric tons [2]. Reactive dyes pose the great problems in terms of color, which is exacerbated by the dominance of cotton. The human eyes can detect about 0.005 mg/L of reactive dye in waste water, and therefore, presence of dyes exceeding this limit would not be permitted on aesthetic ground [3].

Reactive dyes are widely used in industries including textiles, leather, printing, plastics and foods etc. Most of them present in the textile effluents which are responsible for the contamination of water and disturbing the ecosystem [4]. They have highly visible colors; even their small portion released in the environment may cause the appearance of intense color. The critical attentions of public and local legislation are needed to remove the coloring materials from textile effluents before discharging into the environment [2]. The presences of chromophore in the reactive dyes have a greater capability for the direct reactions with a substrate of the fiber and make them more permanent. The colors of reactive dyes are not easy to remove by effluent treatment processes and in many cases it's not easily degradable [5]. Although some existing technologies may have certain efficiency for the removal of reactive dyes but their operational costs are so great. On the other hand, low cost technologies don't allow a wishful color removal or have certain disadvantages, required more complex treatment plan.

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Synthetic dye stuffs can exist in the effluents of wastewater from different industries such as textiles, paper, leather, plastics, etc [6]. Discharge of wastewater into natural streams and rivers from the industries using dyes causes severe environmental problems. Even small quantities of dyes can color large water bodies, which is not only effects on aesthetic merit but also reduces light penetration and photosynthesis. In addition, most of dyes are either toxic or mutagenic and carcinogenic [7]. For these reasons, the removal of dyes from process or waste effluents becomes environmentally important. Conventional wastewater treatment plants are not suited to remove the dyes due to their non-biodegradable features [8]. Therefore, alternative methods have been developed to remove the dyes from wastewater, namely, coagulation and flocculation, membrane separation, oxidation or ozonation, electro-coagulation, and adsorption. Among these methods, adsorption currently appears to offer the best potential for the entire treatment [9]. Although activated carbon is the most widely used adsorbent for wastewater treatment; it is very expensive and has high operating costs due to the high price of the activated carbon and to the high water flow rate always involved.

Hence, researches have been directed to other non-conventional procedures for the color removal that will be effective with their low cost [10]. Adsorption is one of the most effective physical processes for the removal of textile effluents [11]. Many adsorbing materials have been tested to lower the dye concentration from aqueous solutions, such as activated carbon, rice husk, silica, chitin, fly ash, saw dust, bagasse pith, and orange peel [12]. Application of natural adsorbents has received much attention for the adsorption of various pollutants from aqueous medium.

## MATERIALS AND METHODS

### Materials

The kaolin light gray sample used in this investigation was collected from a natural deposit, located in Ikere Ekiti in Ekiti State and the Bentonite clay was also obtained from Ise Ekiti Ekiti State (Nigeria). The sample of kaolin and bentonite powder was characterized using infrared (FT-IR), X-ray diffraction (XRD) and scanning electron microscopic (SEM) techniques. The X-ray diffraction pattern of powder was recorded on a Phillips-1730 (PAN analytical) X-ray diffractometer using Cu K $\alpha$  radiation ( $\lambda=1.54\text{\AA}$ ).

Congo red (CR), a diazo dye was used as a surrogate indicator to simulate industrial wastewater in order to evaluate the adsorption capacity of kaolin-bentonite in this study. The Congo red obtained from dye store, was used as received without any purification. The chemical

formula of CR is C<sub>32</sub>H<sub>22</sub>N<sub>6</sub>Na<sub>2</sub>O<sub>6</sub>S<sub>2</sub> with Color Index 22120 and molecular weight of 696.663 g/mol.

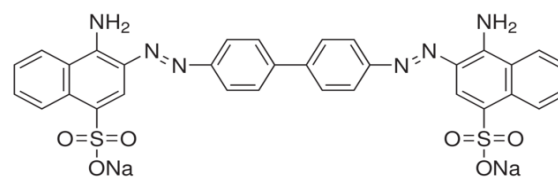


Figure 1. Structure of Congo red

### Adsorption experiments

A stock solution of 1g/L was prepared by dissolving the appropriate amount (1g) of CR in a liter of deionized water. The working solutions were prepared by diluting the stock solution with deionized water to give the appropriate concentration of the working solutions. The pH of the solutions was adjusted by addition of either HCl or NaOH solutions. The UV-Vis Spectrophotometer (UV.1700 Pharmrspac) was used to determine the concentrations of Congo red dye in solution. pH measurements were done using Orien pH meter. The concentration of the residual dye was measured using UV-visible spectrometer at a  $\lambda_{max}$  corresponding to the maximum adsorption for the dye solution ( $\lambda_{max}= 497\text{ nm}$ ) by withdrawing samples at fixed time intervals, centrifuged and the supernatant was analyzed for residual Congo red (CR). Calibration curve was plotted between absorbance and concentration of the dye solution to obtain absorbance-concentration profile. The amount of adsorbed CR (mg/g) was calculated based on a mass balance equation as given by:

$$q_e = (C_o - C_e)V / W \quad (1)$$

where  $q_e$  is the equilibrium adsorption capacity per gram dry weight of the adsorbent, mg/g;  $C_o$  is the initial concentration of CR in the solution (mg/L);  $C_e$  is the final or equilibrium concentration of CR in the solution (mg/L);  $V$  is the volume of the solution (L); and  $W$  is the dry weight of kaolin-Bentonite clay (g).

The sorption capacity at time  $t$ ,  $q_t$  (mg/g) was obtained as:

$$q_t = (C_t - C_o)V / W \quad (2)$$

Where  $C_o$  and  $C_t$  (mg/L) were the liquid phase concentrations of solute at initial and a given time  $t$ ,  $V$  was the solution volume and  $W$  the mass kaolin (g).

## RESULTS AND DISCUSSION

### Characterization of adsorbent

SEM micrograph of the untreated kaolin sample suggests a very cohesive material (Figure. 2) and Bentonite in Figure. 3. While Figure. 4 is the SEM

micrograph of kaolin-bentonite sample. The KBC micrograph confirms that the material is forming micron-size agglomerates. High magnification micrograph of the same structure shows that the micro-size particles are composed of individual platelets, which conglomerate into large size particles.

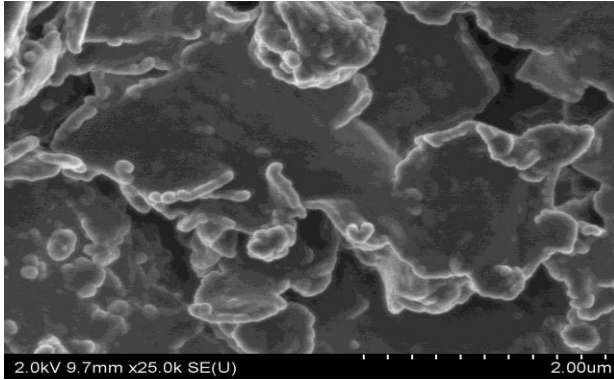


Figure 2. SEM of kaolin

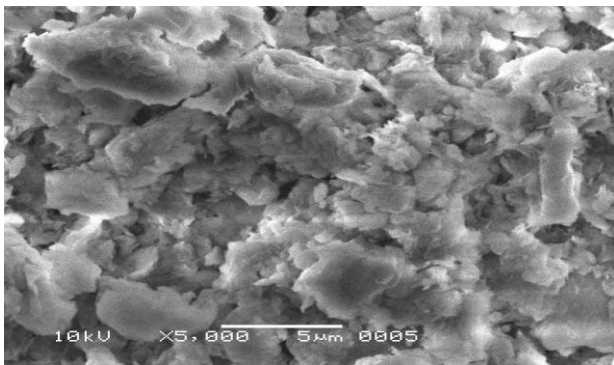


Figure 3. SEM of Bentonite clay

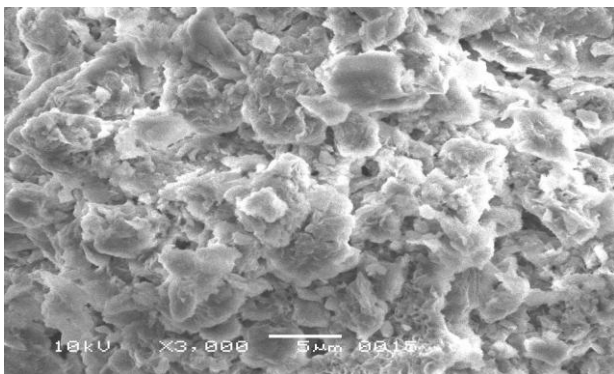


Figure 4. SEM of kaolin-bentonite clay

Figure 5 represents the XRD diffractogram of kaolin-Bentonite clay. The XRD analysis of the natural showed that the material is crystalline and some prominent peaks (degree) synonymous with kaolinite 23.12, 26.99, 35.05, 40.99, 43.79, 46.03, 55.62, 60.09

and 68.52 were identified (Figure 5b). The kaolin-bentonite Clay retained some of the prominent kaolin peaks but more peaks were introduced between the 20 values of 20° and 30° and a slight increase in the basal spacing (>3 nm) of the lattice structure was observed. In the kaolin, the d-spacing of the different 2θ values were mainly below 3 nm. The new found peaks and the broad increase in the d-spacing values were ascribed to the intercalation of the CaO in the KBC. Borgnino *et al.*, [13] postulated that changes in the basal spacing depend on the charge, size, and hydration behavior of the ion or molecule that is located in the interlayer and on interactions between ions and the phyllosilicate layers. The ionic radius of Ca<sup>2+</sup> is 0.99 Å, whereas the single bond covalent radius of Ca<sup>2+</sup> is 1.74 Å. Consequently, the hydrated form of the Ca–O bond length is 1.74 Å for the case of Ca (OH)<sub>2</sub>. The smaller size of Ca<sup>2+</sup> can in principle explain the minimal increase in the basal spacing. Reduction in phyllosilicate basal spacing by addition of Fe<sup>3+</sup> has been reported by Chen *et al.*, [14] who attributed this effect to strong attractive forces between iron and the silicate sheets of montmorillonite. Other authors, on the contrary, have observed a slight increase in basal probably due to the presence of polymeric species of iron within the interlayer[15].

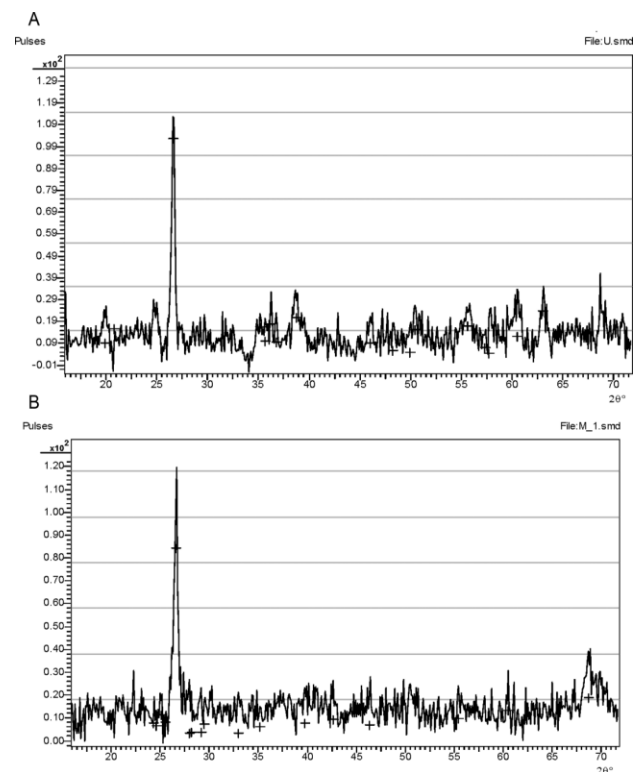
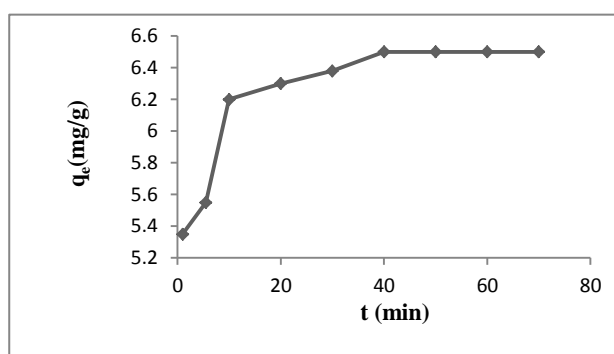


Figure 5. (a) XRD analysis of the Natural Bentonite. (b) XRD analysis of the Kaolin-Bentonite clay

### Effect of contact time

The effect of contact time on the amount of anionic dye, CR, adsorbed by kaolin-Bentonite at room temperature (27°C) was examined at 100 mg/L concentration of dye with 0.1g kaolin-Bentonite dose. As can be seen from Figure. 6, when the equilibrium time increased, the amount of adsorption was not drastically increased. The adsorption of CR occurred very quickly from the beginning of the experiments during the first 50 min, then a slight increase until 80 min where the maximum adsorption of CR onto clay was observed; it can be said that beyond this there is almost no further increase in the adsorption and it is thus fixed as the equilibrium time.



**Figure 6.** Effect of contact time ( $C_0 = 100$  mg/L, m kaolin-bentonite = 0.1g, pH=6.9)

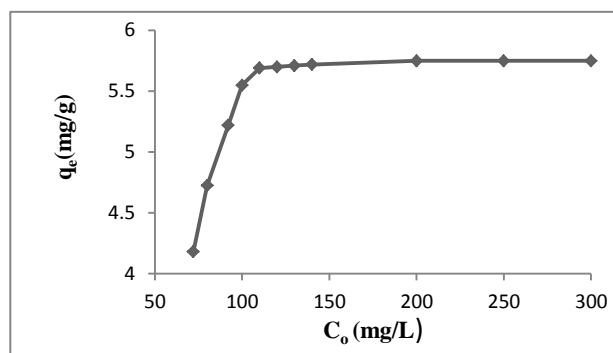
### Effect of initial dye concentration

The effect of initial dye concentration under equilibrium conditions was investigated. By considering the amount of dye adsorbed per unit of kaolin-Bentonite (mg/g), adsorption capacity of kaolin-Bentonite increased as the initial dye concentration increased. The maximum adsorption capacity attained for kaolin-Bentonite 5.66 mg/g (see Figure. 7). The adsorption was preceding the saturation of kaolin-Bentonite at 100 mg/L of dye. It can be proposed that an increase in the initial dye concentration leads to an increase in mass gradient between the solution and adsorbent, and thus acts as a driving force for the transfer of dye molecules from bulk solution to the particle surface. An increase in the proportional dye adsorption is attributed to the equilibrium shift during the clay adsorption process.

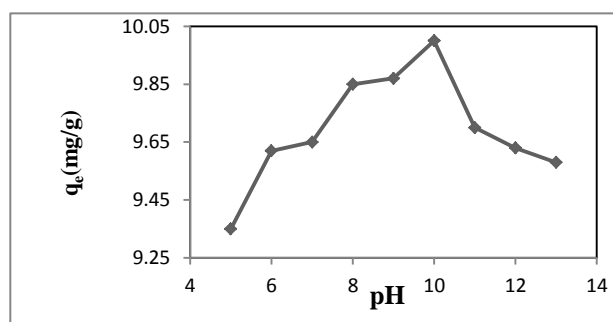
### Effect of pH

The pH of solution has been identified as the most important parameter affecting dye adsorption onto the clay. But the study of the influence of this parameter is not easy mainly under strongly acidic pH, because of the formation of protonated species (bluish color), which possibly leads to a change in the structure of the dye. The negatively charged adsorbent surface sites can

be protonated too. The red color is stable in the pH range of 5–13. In this pH range, the adsorption of CR on kaolin was investigated. Figure.7 shows, the increase in CR removal by kaolin with increasing pH and reached maximum  $q_e$  (max) = 9.95 (mg/g) at pH value of 10 and then decreased at higher pH, the high negatively charged adsorbent surface sites did not favour the adsorption of deprotonated CR due to electrostatic repulsion. Also, an abundance of  $\text{OH}^-$  ions in basic solution creates a competitive environment with anionic ions of CR for the adsorption sites causing a decrease of adsorption [16].



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



**Figure 8.** Effect the pH on the  $q_e$

### Adsorption isotherms

To investigate an interaction of adsorbate molecules and adsorbent surface, two well-known models, the Freundlich [17] and Langmuir [18] isotherms, were selected to explicate dye–clay interactions.

### Langmuir isotherm model

The basic assumption of Langmuir model is that the formation of monolayer takes place on the surface of the adsorbent, indicating that only one dye molecule could be adsorbed on one adsorption site and the intermolecular forces decrease with the distance. It is also assumed that the adsorbent surface is homogeneous in character and possesses identical and energetically equivalent adsorption sites. The model is presented as follows:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (3)$$

The values of  $q_m$  and  $K_L$  of linear expression of Langmuir adsorption isotherm were calculated from the slopes and intercept of the linear plot of  $C_e/q_e$  versus  $C_e$  in Figure. 8 and Table 1 according to the following linear equation:

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

The isotherm was found to be linear over the entire concentration range studies with a good linear correlation coefficient (see Table 1), showing that Langmuir equation represents the best fit of experimental data than the other isotherm equation.

The fact that Langmuir isotherm fits the experimental data very well confirms the monolayer coverage of dye onto kaolin-bentonite particles ( $q_m = 71.43$  mg/g) and also the homogeneous distribution of active sites on the adsorbent, since the Langmuir equation assumes that the surface is homogeneous. The essential features of the Langmuir isotherm can be expressed in terms of a dimensionless constant called separation factor ( $R_L$ , also called equilibrium parameter) which is defined by the following equation.

$$R_L = \frac{1}{1 + K_L C_0} \quad (5)$$

Where  $C_0$  (mg/L) is the initial dye concentration and  $K_L$  (L/mg) is the Langmuir constant related to the energy of adsorption. The value of  $R_L$  indicates the shape of the isotherms to be either unfavourable ( $R_L > 1$ ), linear ( $R_L = 1$ ), favourable ( $0 < R_L < 1$ ) or irreversible ( $R_L = 0$ ). The influence of isotherm shape on whether adsorption is favourable or unfavourable has been considered. For a Langmuir type adsorption

process, the isotherm shape can be classified by a dimensionless constant separation factor ( $R_L$ ), given by Equation. The degree of favorability is generally related to the irreversibility of the system, giving a qualitative assessment of the kaolin-Bentonite-CR interactions. The degrees tended toward zero (the completely ideal irreversible case) rather than unity (which represents a completely reversible case).

### Freundlich isotherm model

While Langmuir isotherm assumes that enthalpy of adsorption is independent of the amount adsorbed, the empirical Freundlich equation, based on sorption on heterogeneous surface, can be derived assuming a logarithmic decrease in the enthalpy of adsorption with the increase in the fraction of occupied sites. The Freundlich equation is purely empirical based on sorption on heterogeneous surface and is given by:

$$q_e = k_f C_e^{1/n} \quad (6)$$

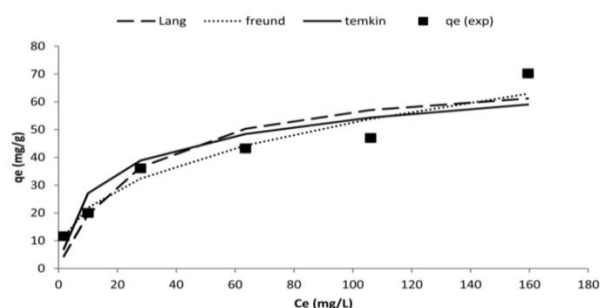
The above equation can be rearranged to obtain a linear form by taking logarithms:

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \quad (7)$$

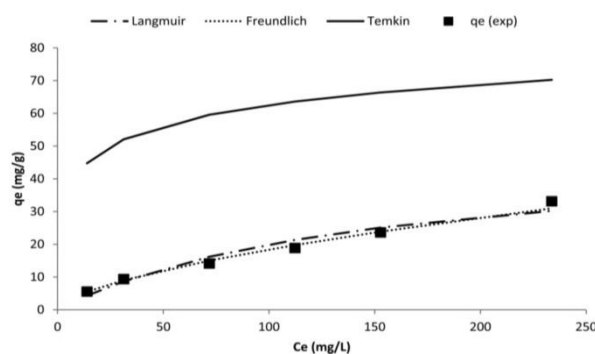
It was revealed that the plot of  $\ln q_e$  and  $\ln C_e$  yields a straight line (Figure. 9). The results are indicated in Table 1. The favourable adsorption of this model can be characterized such that if a value for  $n$  is above unity, adsorption is favourable and a physical process. In the present study the value of  $n$  ( $n = 2.6274$ ) is greater than 1, indicating that the adsorption process is favourable. But the value of correlation coefficient ( $R^2 = 0.9753$ ) is lower than the Langmuir isotherm value.

TABLE 1. Results of the Isotherm Analysis

clay samples	Langmuir			Freundlich		Temkin-Pyzehev			
	$q_m$	$K_a$	$r^2$	$k_f$	$1/n$	$r^2$	B1	kT	$r^2$
KBC	71.43	0.03743	0.9086	9.12575	0.3806	0.9753	11.533	1.0475	0.8718
Kaolin	49.02	0.00687	0.8685	1.08904	0.6139	0.9943	9.0266	10.2394	0.8964



(a)



(b)

Figure 9. (a) Comparison of the experimental and predicted  $q_e$  (mg/g) values for the different isotherm models for the sorption of Congo red onto Kaolin-Bentonite. (b) Comparison of the experimental and predicted  $q_e$  (mg/g) values for the different isotherm models for the sorption of Congo red onto Kaolin

### Kinetic of sorption

Kinetic models have been used to investigate the mechanism of sorption and potential rate controlling steps, which is helpful for selecting optimum operating conditions for the full-scale batch process. Pseudo-first-order and pseudo-second-order kinetic models were used.

### Pseudo-first-order model

The pseudo-first-order rate expression based on solid capacity is generally expressed as follows [19]:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (8)$$

Where,  $q_e$  is the amount of CR adsorbed at equilibrium (mg/g),  $q_t$  is the amount adsorbed at time  $t$  (mg/g),  $k_1$  is the rate constant of first order adsorption (min<sup>-1</sup>). After integration and applying boundary conditions,  $t = 0$  to  $t$  and  $q_t = 0$  to  $q_e$ ; the integrated form of equation becomes:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (9)$$

Value of adsorption rate constant  $k_1$  for the CR adsorption onto kaolin was determined from the straight line plot of  $\log(q_e - q_t)$  against  $t$  (see Figure. 10). The data were fitted with a poor correlation coefficient, indicating that the rate of removal of CR onto kaolin-bentonite does not follow the pseudo-first-order equation.

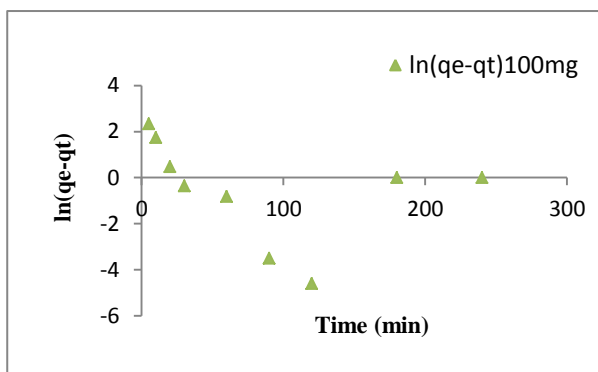


Figure 10. Pseudo-first order plot of CR sorption onto Kaolin-Bentonite clay

### Pseudo-second-order model

Ho and Mc [20] presented the pseudo-second order kinetic as:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (10)$$

Where,  $k_2$  is the rate constant of second order-adsorption (g/mg.min). For the same boundary conditions the integrated form of equation becomes:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (11)$$

The initial sorption rate,  $h$  (mg/ g. min), at  $t = 0$  is defined as

$$h = k_2 q_e^2 \quad (12)$$

Where,  $k_2$  and  $h$  values were determined from the slope and intercept of the plots of  $t/q$  against  $t$  (Figure. 11). The correlation coefficient of examined data was found very high ( $R^2 > 0.9999$ ). This shows that the sorption of CR onto Algerian kaolin followed the pseudo-second-order kinetic model. A good agreement with this adsorption model was confirmed by the similar value of calculated  $q_e$  and the experimental one; and calculated Langmuir monolayer adsorption,  $q_m$ . The best fit to the pseudo-second order kinetic indicated that the adsorption mechanism depended on the adsorbate and adsorbent [21].

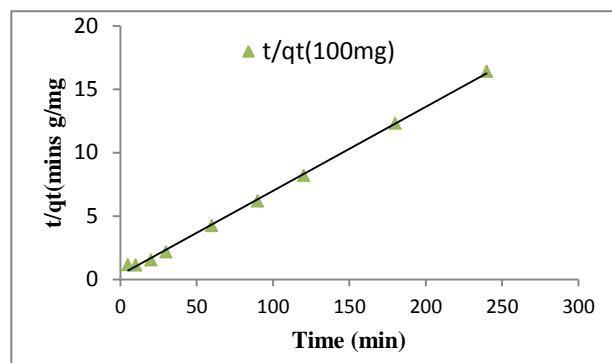


Figure 11. Pseudo-second order plot of CR sorption onto Kaolin-Bentonite clay

### CONCLUSION

The kaolin-bentonite is an effective adsorbent for removal of the toxic anionic dye, Congo red, from aqueous solution. The adsorption was highly dependent on various operating parameters, like: contact time, pH, initial concentration and temperature. The results gained from this study were well described by the Freundlich theoretical but Langmuir equation represented the best fit of experimental data. The kinetic data indicated that the adsorption process was controlled by pseudo-second-order equation. Thermodynamics studies indicated that adsorption of Congo red on kaolin-Bentonite was stable over an extensive range of temperature; it was also exothermic and took place spontaneously in nature. These results show how and why kaolin-Bentonite can be effectively used as a liner in water treatment plants for the removal of dye. Reuse of spent kaolin-Bentonite and recovery of adsorbed dye may be of further scope of research.

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## چکیده

فاضلاب های نساجی بعنوان منبع پتانسیلی از آب های آلوده مطرح شده است. بنابراین روش های موثری برای حذف رنگ های مصنوعی و طبیعی از فاضلاب های نساجی اقتباس شده است. در این تحقیق سعی بر بررسی حذف رنگ قرمز یکجور چای سیاه (کونگو) در نساجی بوسیله ی مخلوطی از بنتونیت خاک رس و خاک چینی دارد. بنتونیت خاک رس (KBC) بعنوان جاذب استفاده شده است. مشخصات جذب سطحی خاک چینی و بنتونیت خاک رس نسبت به قرمزی کنگو، در دماهای مختلف  $\pm 2$  ۳۰۳-۳۱۸ درجه ی کلون تحت شرایط بهینه مورد بررسی قرار گرفت. اطلاعات جذب سطحی، در مدل های هم دمای جذب سطحی لانگمیر، فرنرلیچ و رادوشکویچ دوبینین و مقدار ثابت های نسبی که به وسیله ی روش های گرافیکی استاندارد ارزیابی شده است، منطبق شدند. بوسیله ی گراف نشان داده شده است که مدل لانگمیر خطوط هم دما را بهتر تطبیق داده است. تخمین جذب سطحی فرآیند (RL) و انرژی (ES) امکانپذیر است. PH جاذب بوسیله ی روش دریافت تخمین زده شده است. سینتیک حذف رنگ از ثابت سرعت مرتبه ی دوم فرضی پیروی می کند. مورفولوژی سطح جاذب بوسیله ی اسکن میکروسکوپ الکترونی مشاهده شد.