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Removal of Congo Red and Rhodamine B Dyes from Aqueous Solution by Raw Sarolangun Bentonite: Kinetics, Equilibrium and Thermodynamic Studies

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Abstract

Water pollution due to the existence of colored substance in the waterbody has become a major environmental issue nowadays. In this work, the raw bentonite grounded from Sarolangun has been utilized as a natural abundant adsorbent material for removal of Congo red and Rhodamine B dye from aqueous solution. The raw bentonite was characterized by XRD, FT-IR, and XRF. The results showed that the raw bentonite was mainly composed of montmorillonite mineral. The effect of some operational parameters including contact time, initial dye concentration, and temperature. The kinetics, equilibrium, and thermodynamic adsorption was also systematically investigated. The results indicated that the adsorption of both Congo red and Rhodamine B on the raw bentonite was best fitted with pseudo-second-order kinetics model. The adsorption isotherm study indicated that the adsorption process was well described by Freundlich isotherm model, and the adsorption thermodynamic investigation revealed that the adsorption process of the both dyes was spontaneous and an exothermic process. These finding showed that the raw Sarolangun bentonite has promising properties to be a good low-cost adsorbent for dyes removal.

INTRODUCTION

The presence of hazardous organic dye molecules in the aquatic environment due to the industrial wastewater has become a major issue in the environmental concern [1–3]. Annually, dye effluents have been discharged in huge amount by various industry such as textile, leather, paper, cosmetics, plastics, and food production or processing as a waste or a byproduct reaction [4]. Commonly, synthetic dye used in the industry have carcinogenic, teratogenic and even mutagenic properties to the living creatures. Subsequently, improper disposal of dye effluent directly to the environment without sufficient treatment can cause various environmental and health problems. Moreover, these dye molecules are highly natural stable which can cause disruption of light penetration into the water body [5]. Hence the photosynthetic mechanism of the aquatic plant will be disrupted. Therefore, any attempts to reduce and remove dye molecule concentration in the wastewater is highly appreciated in order to build a sustainable environment.

Recently, various methods have been widely developed and available for wastewater treatment in order to remove dye molecules contaminant. Some of the most investigated methods are including photocatalytic degradation, membrane separation, chemical oxidation, coagulation, flocculation, and adsorption [6]. Among these mentioned methods, adsorption is deemed as the most feasible method for dye removal from wastewater regarding to its ease in operation, simplicity, and high efficiency [7]. Moreover, the widespread of the adsorption utilization

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for wastewater treatment was espoused by the availability of various low-cost and natural abundant adsorbent materials such as activated carbon, clays and clay minerals, and even industrial or agricultural waste and by product [8–11].

Clays and clay minerals have been widely utilized in the broad fields since the ancient times. In the last decades, the limit of the clay utilization has grown further as a promising material for used as an adsorbent for treating various kinds of pollutant in the environment [12]. The forward and broad use of clays and clay minerals was resulted by their variety of structural and surface charges properties, high surface area, high physical and chemical stability, and high surface area [13]. One of the most used clays is bentonite which is mainly composed of montmorillonite mineral. Bentonite is a smectite class clay in which it has 2:1-layer structure with alumina sheet stacked by two silica sheets in sandwich form. Bentonite layers are negatively charged due to the partial isomorphous substitution in which all the layers held together with inter-balancing cation such as Na^{+}, Ca^{2+}, and Mg^{2+} [14].

In this current work, we have examined the composition and structural properties of raw natural bentonite that obtained from Sarolangun District of Jambi Province, Indonesia as a low-cost adsorbent material for environmental purpose. The raw natural bentonite samples have been characterized by using X-Ray fluorescence (XRF), X-Ray powder diffraction (XRD), and FT-IR. The raw natural bentonite then utilized as an adsorbent material for removal of two hazardous dye namely congo red and rhodamine B.

EXPERIMENTAL

Materials and Instrumentation

Bentonite clay that utilized in this work was obtained from bentonite deposit located in Sarolangun District of Jambi Province, Indonesia. The congo red and rhodamine B dye used as textile wastewater model were obtained directly from the local household textile industry located in the Tuan Kentang Village, Palembang. HCl and NaOH as pH adjusted of the dye solution were purchased from Merck Millipore in analytical grade and used as received without further purification. The ionized water used all activities in this work was prepared by using Pureit® instrument. The chemical composition of the natural bentonite sample by XRF analysis was carried out using PANanalytical XRF, type Minipal 4. The functional group analysis was conducted using Shimadzu FTIR Prestige-21 with KBr pellet method. The characterization was conducted using Rigaku Miniflex 600 equipped with Ni-filtered CuKα radiation at scanning speed 5° min^{-1}.

Adsorbent Preparation

The obtained raw bentonite lump was grounded and washed for several times using deionized water. The raw bentonite samples was then subjected to the pre-treatment process according to the work reported by Ismadji at al [14]. The pretreated bentonite was further washed with deionized water for several times followed by drying at 105 °C for 24 h. The dried bentonite then crushed with porcelain mortar and sieved to pass through 200 mesh ASTM standard sieve. The chemical composition of adsorbent was characterized by X-Ray fluorescence (XRF). The crystallinity of bentonite sample was investigated by X-Ray Diffraction (XRD), and the functional group of bentonite clay was characterized by FT-IR spectroscopy.

Adsorption Experiments

The stock solution (1000 mg/L) of congo red and rhodamine B dye was prepared by dissolving a gram of solid congo red and rhodamine B with 1000 mL of deionized water. The standard and working solution of the dyes was prepared by diluting a certain volume of the stock solution into desired concentration with deionized water. The standard curve of congo red and rhodamine B standard solution was obtained by measuring the absorbance values of each standard solution at 497 nm for congo red and 544 nm for rhodamine B.

The effect of some operational parameters on the adsorption process was evaluated by conducting various adsorption experiment with different operational conditions. In this work, the effect of contact time, initial concentration of dyes, and temperature were studied. The batch adsorption process was fully conducted in a 100 mL of canonical flask containing 50 mL of dye solution. In the contact time effect investigation, 0.05 g of adsorbent was contacted with 50 mL of dye solution with concentration 100 mg/L. The mixture then was shaking for different time ranging from 0 to 90 min. The effect of initial dye concentration was studied by conducting the adsorption experiment with different initial dye concentration and for the thermodynamic study, the adsorption was conducted in different temperature.

The remaining concentration of the dye solution after the adsorption process finished, was measured by using double beam UV-Vis spectrophotometer based on the standard curve method. The adsorption capacity of the dye on the adsorbent was calculated with the following equation:

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

Where q_e is the adsorption capacity (mg/g), m is the amount of adsorbent (g), V is the volume of dye solution (L), C_e is the remaining dye concentration (mg/L), and C_o is the initial dye concentration (mg/L).

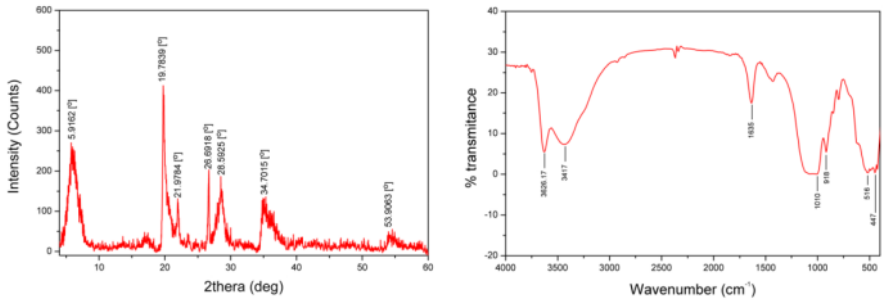
RESULTS AND DISCUSSION

Characterization of the adsorbent

The chemical composition of natural bentonite sample according to the XRF elemental analysis is presented in TABLE 1. According to the value of each metal oxide composition, it can be explained that the composition of natural bentonite is dominated by SiO_2 and Al_2O_3 as the silica and alumina phase of the montmorillonite mineral [15]. The other constituent such as calcium, vanadium, and potassium were in trace concentration and recognized as the impurities mineral. The value of alumina and silica ration was calculated as 1:2.5. This value corresponds to the structure of montmorillonite as smectite mineral in which the alumina layer was stacked by two silica layers [14].

TABLE 1. Chemical composition of natural bentonite sample

Compound	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2
Conc. (%)	17±0.5	43.6±0.2	0.71±0.03	0.20±0.005	0.997±0.017	1.87±0.02
Compound	V_2O_5	Cr_2O_3	MnO	Fe_2O_3	NiO	CuO
Conc. (%)	0.11±0.004	0.075±0.002	0.12±0.002	33.39±0.34	0.877±0.039	0.15±0.003



(a) (b)

FIGURE 1. (a) X-Ray diffraction pattern of natural bentonite (b) FT-IR spectra of natural bentonite sample

The XRD analysis toward natural bentonite sample has been carried out in order to investigate the crystalline properties of the contained minerals. FIGURE 1(a) shows the X-ray powder diffraction pattern of natural bentonite at 2theta ranging from 3 to 60°. The main peak of montmorillonite mineral recorded as broad and sharp peak at 2θ around 5.9°, 19.7°, and 34.7° [16]. While the sharp diffraction peak at 2θ around 28.59° and 53.9° were recognized as the quartz mineral as the impurities phase of the bentonite [17,18].

The FTIR spectra of natural bentonite sample is presented in Fig. 1(b). The main band of montmorillonite minerals recorded at wavenumber around 3600, 3400, 1635, 1010, 918, 516, and 447 cm^{-1}. The absorption band recorded at 3626 cm^{-1} reveal the presence of Al(Mg)-O-H stretching vibration of the montmorillonite layer [17]. The band at 3417 cm^{-1} represent the presence of H-O-H stretching vibration of water molecule located in the interlayer of bentonite. The presence of H-O-H bending vibration can be seen as the sharp band recorded at wavenumber 1635 cm^{-1}. The broad band recorded at wavenumber 1010 cm^{-1} represent the presence silicate layer as the Si-O-Si stretching vibration band [19].

Adsorption Kinetics Study

The study on the effect of contact time toward the adsorption performance is an important since this study can describe the adsorption mechanism. The effect of contact time on the adsorption capacity of natural bentonite toward rhodamine B and congo red is presented in Fig. 2 (a). It can be seen in the Fig. 2 (a) that the adsorption equilibrium of congo red and rhodamine B achieved at the first 60 min. After the equilibrium time achieved, there was no considerable increase on the adsorption capacity of natural bentonite toward both congo red and rhodamine B. Furthermore, at the equilibrium state, the value of adsorption capacity on congo red is higher than rhodamine B. Therefore, it can be concluded that congo red has higher adsorption affinity toward natural bentonite rather than rhodamine B.

Since adsorption kinetics study is an important factor to evaluate the rate of dye uptake to the adsorbent, it is a necessary to evaluate the data of contact time effect according to the adsorption kinetics model. Notably, the adsorption kinetics model was conducted to determine the rate controlling step and the mechanism of the adsorption kinetic that occurred during the adsorption process. In this work, two most studied kinetic models namely pseudo-first-order and pseudo-second-order kinetic model were employed to evaluate the kinetic parameter of the experimental data. The pseudo-first-order kinetics model can be displayed as the following linear form [20].

$$\ln(q_e - q_t) = \ln q_e - K_L t \tag{2}$$

Where q_e is the adsorption capacity of natural bentonite (mg/g) at equilibrium state, q_t is the adsorption capacity of natural bentonite at any time "t" (mg/g), k_L is the adsorption rate constant according to the Lagergren kinetic model. The adsorption kinetics parameter related to the pseudo-first-order kinetics model can be calculated based on the slope and intercept value of the plot of $\ln(q_e\text{-}q_t)$ against t from the linear form of equation (2).

The pseudo-second-order kinetics model can be written as the following linear equation [20].

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \cdot t \tag{3}$$

Where k_2 is the adsorption rate constant related to the pseudo-second-order kinetics model. The kinetics parameter according to the pseudo-second-order model can be obtained as the slope and intercept value of the plot t/q_t versus t.

The kinetic parameters that determined according to the both kinetic models are presented in TABLE 2. According to the magnitude of correlation coefficient (R^2), the both kinetic models have good enough correlation with R^2 higher than 0.9. However, the R^2 value of pseudo-second-order model is higher than pseudo-first order model. Hence, it can be informed that the pseudo-second-order model is fit better to the experimental data rather than the pseudo-first-order model. Moreover, the value of q_e according to the both models showed that the pseudo-first-order model has higher deviation to the q_e of the experimental data for both rhodamine B and congo red.

TABLE 2. Adsorption kinetic parameter for rhodamine B and congo red on natural bentonite

Dye	Pseudo-first-order			Pseudo-second-order		
	k_L (min^{-1})	q_e(mg/g)	R^2	k_2(kg.g^{-1}min^{-1})	q_e(mg/g)	R^2
Rhodamine B	0.0369	133.5971	0.9022	0.0562	169.4082	0.9872
Congo red	0.0708	208.3449	0.9310	0.0705	179.5120	0.9916

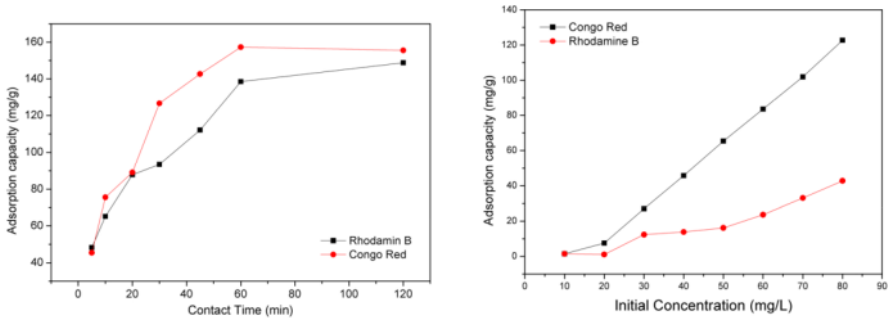


FIGURE 2. (a) Effect of contact time on the adsorption capacity of natural bentonite (b) Effect of initial concentration on the adsorption performance of natural bentonite

Adsorption Isotherm Study

Study on the adsorption equilibrium has been carried out by investigating the effect of initial dye concentration on the capability of natural bentonite adsorption. The results of this experiment are depicted in Fig. 2(b). It showed that by increasing the initial dye concentration, the adsorption capacity of natural bentonite toward the both dyes systematically increased. However, the adsorption capacity of natural bentonite toward congo red is higher than toward the rhodamine B. This finding was in agreement as described in the adsorption kinetic subsection.

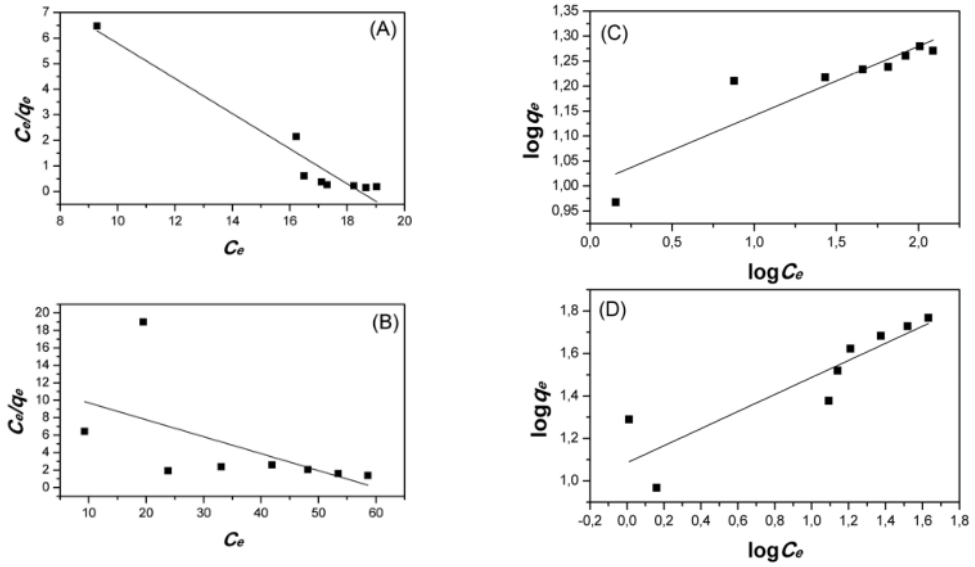


FIGURE 3. Plot of Langmuir adsorption isotherm model on (A) congo red, (B) rhodamine B, and Freundlich model on (C) congo red, (D) rhodamine B.

The adsorption equilibrium properties of congo red and rhodamine B on natural bentonite has been studied according to the effect of initial concentration data by employing the data with the Langmuir and Freundlich adsorption isotherm model. This adsorption isotherm study was aimed to investigate the mechanism of interaction

between the adsorbent and adsorbate that occurred during the adsorption process. Theoretically, the Langmuir adsorption isotherm model is described in the following equation [21].

$$q_e = q_m \frac{C_e}{1 + bC_e} \tag{4}$$

Where q_m is the maximum adsorption capacity (mg/g), b is the Langmuir constant related to the adsorption energy (L/mg). The above Langmuir equation can be rewrite to its linear form as follow.

$$\frac{1}{} \tag{5}$$

The Freundlich adsorption isotherm model empirically is well known model employed for low concentration. The mathematical equation of Freundlich isotherm model is presented in the following equation.

$$q_e = k_F C_e^{1/n} \tag{6}$$

Where k_F is the Freundlich constant related to the adsorption capacity ((mg/g)/(mg/L)$^{1/n}$), n is the Freundlich constant related to the adsorption intensity. The Freundlich isotherm model (eq. 6) can be transformed to its linear logarithmic form as follow.

$$\log q_e = \frac{1}{n} \log C_e + \log k_F \tag{7}$$

TABLE 3. Adsorption isotherm parameter of congo red and rhodamine B adsorption

Dye	Langmuir Model		Freundlich Model		
	k_L	R^2	k_F	n	R^2
Congo red	0.0542	0.9416	1.54 x 10^{-6}	0.1655	0.8359
Rhodamine B	0.0166	0.3196	0.0107	0.5006	0.8024

The adsorption isotherm parameter according to the Freundlich model can be calculated by measuring the value of slope and intercept from the plot of $\log q_e$ versus $\log C_e$ (Fig. 3 (c) and Fig. 3 (d)). Otherwise, the adsorption isotherm parameter according to the Langmuir model can be calculated as the slope of intercept of plot C_e/q_e versus C_e (Fig. 3 (a) and Fig.3 (b)). TABLE 3 shows the adsorption isotherm parameter of congo red and rhodamine B on natural bentonite adsorbent. The obtained data showed that the magnitude of coefficient correlation (R^2) for Langmuir isotherm model is lower than 0.5. This finding describes that the adsorption isotherm nature of congo red and rhodamine B on natural bentonite was not appropriate to be described by Langmuir model. Otherwise, for Freundlich model, the magnitude of R^2 for both dyes was approaching unity (>0.8). This result indicated that the adsorption experimental data is better described with Freundlich model. This finding gives an information that the adsorption of congo red and rhodamine B on natural bentonite occurred in multi layers system rather than the mono layer [22].

Adsorption Thermodynamic Study

The adsorption study was systematically conducted at the different temperature ranging from 30 °C to 70 °C in which the result for this study is presented in Fig. 4. As can be seen in the Figure, by increasing the adsorption temperature, the adsorption capacity of congo red and rhodamine B on natural bentonite decreased gradually. This finding informed that the adsorption process was an exothermic.

Further evaluation on the adsorption mechanism can provide deeper information about the kind of interaction between the adsorbent surface and the adsorbate. However, it cannot be obtained by only considering the dependence of the adsorption with temperature. In this case, the spontaneity of the adsorption process, the heat change that occurred during the adsorption process is needed to give more detailed information about the adsorption mechanism. These properties can be investigated through the thermodynamic parameter by measuring value of the

change of free adsorption energy (ΔG), the change of enthalpy (ΔH), and the change of entropy (ΔS). The thermodynamic parameter of the adsorption can be calculated according to the Vant Hoff equation as follow [23]:

$$\ln\left(\frac{q_e}{C_e}\right) = \frac{\Delta S}{R} - \frac{\Delta H}{R} \cdot \frac{1}{T} \quad (8)$$

$$\Delta G = \Delta H - T\Delta S \quad (9)$$

Where R is the gas constant (8.314 J/mol K), T is the absolute temperature (K), q_e is the adsorption capacity at equilibrium state (mg/g), and C_e is the dye concentration at the equilibrium state (mg/L). The thermodynamic parameter of ΔH and ΔS can be determined according to the value of slope and intercept of the plot $1/T$ versus $\ln(q_e/C)$. While the ΔG value can be calculated according to the equation 9.

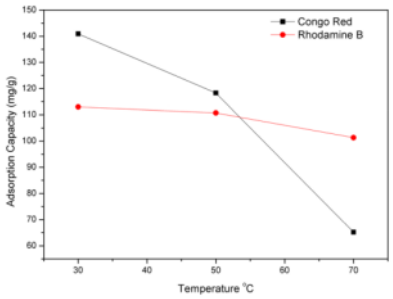


FIGURE 4. Effect of temperature on the adsorption performance

TABLE 4. Thermodynamic parameter of dye adsorption on natural bentonite

Temperature (°C)	Thermodynamic parameters		
	ΔG (J/mol)	ΔH (J/mol)	ΔS (J/mol K)
Congo Red			
30	-4209.2551	-34140.2807	-98.7822
50	-2233.6099		
70	-257.9646		
Rhodamine B			
30	-2466.7641	-5000.5128	-8.3622
50	-2299.5199		
70	-2132.2758		

The thermodynamic adsorption parameters of congo red and rhodamine B on the natural bentonite are presented in TABLE 4. The negative value of ΔG on the adsorption process of both congo red and rhodamine B onto natural bentonite indicated that the adsorption process occurred spontaneously [24]. However, by increasing the adsorption temperature, the negative value of ΔG decreased. This finding showed that the adsorption process is negatively affected by the increasing of temperature. The negative value of ΔH described that the adsorption process of congo red and rhodamine B on natural bentonite are exothermic [6]. While the negative value of ΔS indicated the decrease of the randomness at the solid/solution interface that occurred in the internal surface of the natural bentonite adsorbent [25].

CONCLUSION

The raw natural bentonite from Sarolangun district of Jambi Province, Indonesia has been employed as possible low-cost based adsorbent material. The raw bentonite samples have been characterized and employed as adsorbent for congo red and rhodamine B dye from aqueous solution. The batch adsorption experiment has been conducted in various operational condition in order to evaluate the adsorption parameter of kinetics, equilibrium isotherm, and thermodynamic. The results showed that the adsorption of congo red and rhodamine B on raw natural bentonite

followed the pseudo-second-order kinetic model and the adsorption equilibrium study indicated that the adsorption process was occurred in multi-layer system. The thermodynamic study gives an information that the adsorption process was in spontaneous for the both dye and the process was an exothermic. These results indicated that raw natural bentonite has a good adsorption affinity toward congo red and rhodamine B.

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