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Sorption characteristics of peat of Brunei Darussalam V: removal of Congo red dye from aqueous solution by peat

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ABSTRACT

Peat collected from Panaga, Brunei Darussalam, shows strong affinity towards the adsorption of Congo red (CR) dye from aqueous solution. The adsorption capacity of Bruneian peat under optimized conditions of shaking time, settling time, and medium pH is determined to be 10.1 mg g^{-1} at equilibrium. Adsorption of CR on Bruneian peat follows monolayer adsorption with small contribution to multilayer formation qualifying the validity of the Redlich–Peterson model, which is further supported by error function determination. In addition, changes in peat structure and surface were studied using scanning electron microscopic and X-ray fluorescence techniques. Kinetics studies were in agreement with the pseudo-second-order model and the adsorption reaction proceeds according to intra-particle diffusion.

Keywords: Adsorption; Congo red; Peat; XRF; Kinetics

1. Introduction

Dyes are usually colored substances, used in many industries, such as textile, plastic, paper, and leather to color their products. Consequently, effluents of these industries contain dyes, some of which are carcinogenic and mutagenic [1]. Further, the color of water affects its ecosystem by inhibiting the penetration of sunlight, thereby reducing the photosynthetic action [2]. Hence, the color in wastewater is considered as a pollutant and needs to be removed before discharging into water streams.

Adsorption is a very effective separation technique for wastewater treatment. Although activated carbon is the most efficient and a popular adsorbent, the cost of production of activated carbon is very high. This has promoted the use of alternative materials as adsorbents to replace activated carbon and to search for various biomasses available in the environment [3–6]. In this context, peat is a viable alternative, and it is a good adsorbent because of its porous structure and affinity to be attracted to ions. Peat is a kind of soil formed in wetland conditions containing partially decomposed plant materials and animal remains. Peat has many polar functional groups, such as aldehyde, carboxylic acid, and alcohol present in compounds in peat, such as lignin and humic acid. These polar groups help peat to remove environmental pollutants, such as dyes and heavy metals effectively from

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solutions. The use of peat for dye and heavy metal removal has been reported [7–9].

Brunei Darussalam is a blessed country with a huge area containing peat lands. Peat from different sites around Brunei Darussalam has been successfully used as a low-cost biosorbent for the removal of Cu(II) [9,10] and toxic dyes, such as methylene blue [11], crystal violet [12], and malachite green [13]. As an extension of our research, this study aims to investigate peat collected from Panaga, Belait district in Brunei Darussalam, for its potential to remove Congo red (CR) dye from aqueous solution. CR is the first synthetic azo dye, also called acidic diazo dye, which exhibits a main adsorption band at 488–500 nm in aqueous medium. Dye molecules (Fig. 1) in acidic medium tend to aggregate and form the tautomerism state. Protonation of CR could occur at amino or azo nitrogens [14].

2. Materials and methods

2.1. Materials

Representative samples of peat were collected locally from Panaga area in Brunei Darussalam. They were dried at 60°C for 3–6 days in an oven until there was no mass loss, sieved to obtain particles sized between 355 and 850 µm, and used for all experiments. CR (M.W. 696.67 g mol^{-1} and $C_{32}H_{24}N_6O_6S_2{\cdot}2Na$) was purchased from Sigma. Solutions were prepared in double distilled water by dissolving appropriate amount of the dye.

2.2. Instrumentation

The concentration of dye solutions was determined using a UV–vis spectrophotometer (Shimadzu/Model UV-1601PC). The surface morphology of peat, before and after treatment with CR dye solution, was studied by scanning electron microscopy (SEM) using Tescan Vega XMU Scanning Electron Microscope at 20 kV. Before SEM analysis, peat particles were coated with gold by SPI-MODULE^{TM} Sputter Coater. X-ray fluorescence (XRF) PANalytical Axios^{max} was used for elemental analysis.

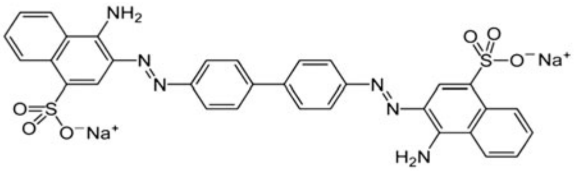

Fig. 1. Structure of CR dye.

2.3. Method

The experiments were carried out by shaking the mixture of 25.0 mL of CR dye solution of known concentration and 0.050 g of peat at 250 rpm. Then, the concentration of CR in supernatant solution was determined using UV–vis spectrophotometer at the wavelength 497 nm. Percentage removal of dye in each experiment was calculated by the following equation,

$$\% \text{ Removal} = \frac{C_0 - C_e}{C_0} \times 100 \tag{1}$$

where C_0 and C_e are the initial and equilibrium concentrations (mg L^{-1}) of the dye in solution, respectively.

2.4. Determination of zero point charge of peat

The salt addition method was used to determine the point of zero charge in 0.1 M KNO_3 solutions [15]. Solutions of different pHs, whose initial pH was adjusted to pre-determine values between 2 and 12, were prepared using 0.1 M NaOH and 0.1 M HCl solutions. An aliquot of 25.0 mL of each pH-adjusted KNO_3 solution and 0.050 g of peat was mixed and shaken for 24 h and the final pH was measured.

2.5. Effect of pH and kinetics studies

To determine the effect of pH on the adsorption capacity of peat, pH of dye solutions was adjusted and the extent of dye removal at each pH was determined. Adsorption kinetics was studied at ambient pH by shaking 25.0 mL of dye solutions of concentration 50.0 mg L^{-1} after the addition of 0.050 g peat to each solution followed by withdrawing samples at different intervals from each solution before the equilibrium state was reached.

2.6. Equilibrium studies

Samples of peat, each weighing 0.050 g, were mixed with 25.0 mL CR solutions of different initial concentrations ranging from 5 to 400 mg L^{-1}. The mixture was shaken for 1.5 h and allowed to settle for 30 min to achieve equilibrium. The type of adsorption was identified using adsorption isotherm analysis.

3. Results and discussion

3.1. Effect of shaking and settling time

All adsorption isotherm studies should be conducted after the system has reached equilibrium. Hence, the contact time (shaking and settling times) is an important parameter that should be optimized in adsorption experiments, because at shorter contact time periods, the system may not have reached equilibrium while the experiment become tedious at longer contact time, and subsequently, it would be difficult to apply adsorption results in real situation. In this research, both the shaking time and the settling time were optimized. Fig. 2 shows the effect of shaking time on adsorption of CR by peat, which indicates that the CR dye is quickly and effectively removed by peat obtained from Panaga, Brunei Darussalam. Equilibrium was reached at 90 min at which about 55% of 10.0 mg L^{-1} dye was removed under the experimental conditions employed. Compared to rice husk ash [16] and kaolin [17] which required 60 and 40 min contact time, respectively, peat requires a slightly longer time to reach equilibrium when treated with CR.

After optimizing the shaking time, settling time needs to be optimized to ensure that the complete equilibrium has reached. For this purpose, solutions containing peat were allowed to settle after the optimum shaking time period of 90 min. Fig. 2 shows the variation between the extent of removal of CR by peat and the time allowed for settling. Although there is no significant variation in the percentage removal, the optimum settling time can be taken as 30 min for subsequent experiments.

3.2. Effect of initial dye concentration

Although the removal of total amount of the dye is increased with the increase in initial dye concentration, the percentage removal exponentially decreases (Fig. 3). This indicates the availability of a limited number of adsorption sites on the surface of peat. Results of such experiments would be important in the design of large-scale treatment systems.

3.3. The point of zero charge and pH effects

The point of zero charge (pH_{PZC}) of an adsorbent is important to understand the adsorption mechanism. Adsorption of a cation is favored at $pH > pH_{PZC}$, while the adsorption of an anion is favored at $pH < pH_{PZC}$ [18]. An acceptable method to determine the pH_{PZC} of a substrate is to plot a graph between the change in pH (ΔpH) and initial pH and obtain the point where $\Delta pH = 0$ [19]. According to Fig. 4, the pH_{PZC} is determined to be 2.0 for peat investigated in this study. This is slightly lower than that of peat obtained from Johor (Malaysia), Narathiwat (Thailand), and Muthurajawela (Sri Lanka) which show values between 3.3 and 3.8 [20], Priyantha and Sampath—personal communication], indicating the inherent acidity of peat soil of Brunei Darussalam [21].

Fig. 5 shows the absorption spectra of CR recorded at different pHs. According to the figure, the λ_{max} significantly changes upon acidification, especially below 4.0, indicating that absorbance measurements alone would not provide an accurate measure of the concentration of CR in solution, and hence the removal of CR, unless the medium pH is assured to be above 5.0. As the ambient pH of aqueous solutions of CR is 6.4, measurements at 497 nm do not lead to inaccurate results. When the optimized treatment conditions are applied to the CR solution-peat system, the pH of the peat suspensions is lowered down to about 5 due to the inherent acidity of peat. As the λ_{max} of CR solutions at this pH is not much different from the reported value of 497 nm, quantification of the solutions after adsorption equilibrium at the same λ_{max} would still produce accurate results. For the same reason, the effect of pH on the extent of CR removal was not investigated in detail, and ambient pH was used throughout this research.

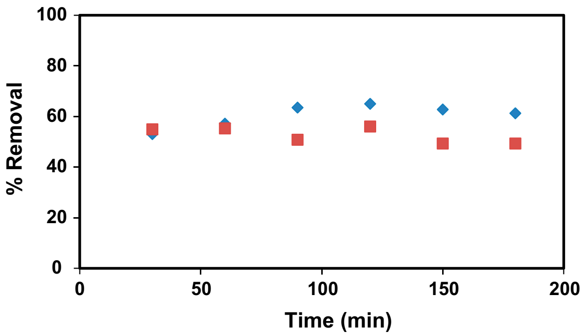

Fig. 2. Extent of removal of CR at different shaking (◆) and settling (■) time intervals by peat (0.050 g peat, 25.0 mL of 10 mg L^{-1} dye solution).

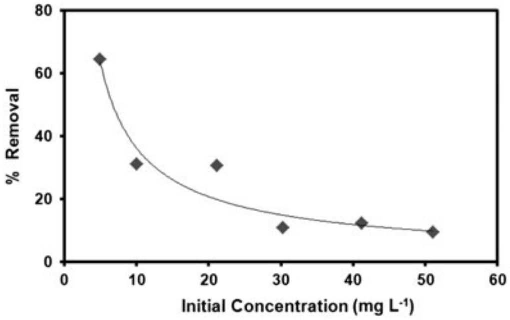

Fig. 3. Percentage removal of CR at different initial concentrations (0.050 g peat, 25.0 mL of 10 mg L^{-1} dye solution).

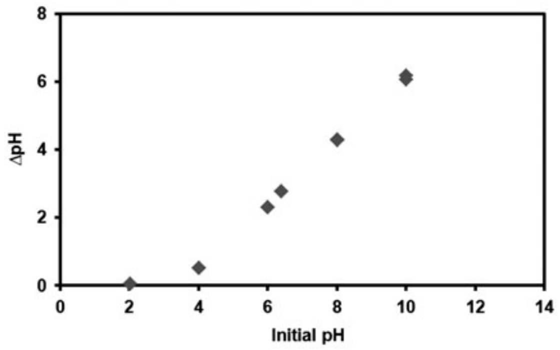

Fig. 4. Initial vs. change in pH plot for the determination of zero point charge of peat.

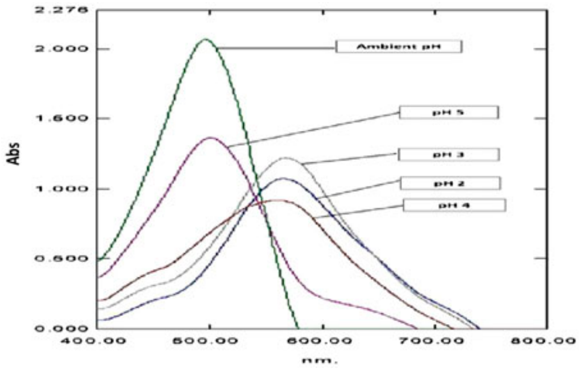

Fig. 5. Absorption spectra of aqueous solution of CR (100 mg L^{-1}) at different pHs.

3.4. XRF analysis

XRF spectroscopy can be applied to determine the elemental composition of peat before and after adsorption. It is found that composition with respect to all the elements is decreased after adsorption (Table 1). The most significant decrease is observed for S and Cl, which could be present as anionic constituents showing a possibility for ion-exchange with anionic CR dye molecules. Further, some anionic constituents together with cationic constituents would leave the surface of peat allowing space for the adsorption of larger dye molecules. Slight decrease of cationic constituents could be attributed to this fact.

3.5. SEM investigation

The SEM image of a representative sample of peat shows that its surface is irregular and porous (Fig. 6). After treatment of peat with CR followed by thorough washing with distilled water, it has been observed that pore structure has been changed and large pores have become filled indicating that the dye molecules have adhered on the surface of peat.

Table 1
Percentage elemental composition of peat before and after treatment with CR (50 mg L^{-1} dye solution)

Element	Untreated peat	Treated with 50 mg L^{-1} CR
Na	0.93	0.28
Mg	1.06	0.54
Al	6.68	6.01
S	6.34	4.24
Cl	1.03	0.064
K	1.59	1.39
Ca	0.55	0.28

3.6. Equilibrium modeling

Equilibrium studies are important to investigate the mode of adsorption and to determine the adsorption capacity of adsorbents. In the present study, Langmuir, Freundlich, and Redlich–Peterson isotherm models were used to explain the adsorption process. Langmuir model assumes monolayer adsorption on the surface of the adsorbent containing a finite number of identical sites. The linear equation for the Langmuir isotherm [22] is given by,

$$\frac{C_e}{q_e} = \frac{1}{Kq_{\max}} + \frac{C_e}{q_{\max}} \tag{2}$$

where q_e (mg g^{-1}) is the amount of dye adsorbed at equilibrium, $q_{\max}$ (mg g^{-1}) is the maximum amount of dye adsorbed by the adsorbent, C_e (mg L^{-1}) is the equilibrium concentration of the dye, and K (L mg^{-1}) is the Langmuir constant.

The Freundlich model assumes the multilayer formation on the surface of the adsorbent. The linear equation for the Freundlich model [23] is given by,

$$\log q_e = \frac{1}{a} \log C_e + \log K_F \tag{3}$$

where K_F is the equilibrium constant which is indicative of the adsorption capacity and a is the adsorption constant whose reciprocal is indicative of adsorption intensity [24].

The Redlich–Peterson isotherm [25], having three parameters and featuring both Langmuir and Freundlich isotherms, can be given as,

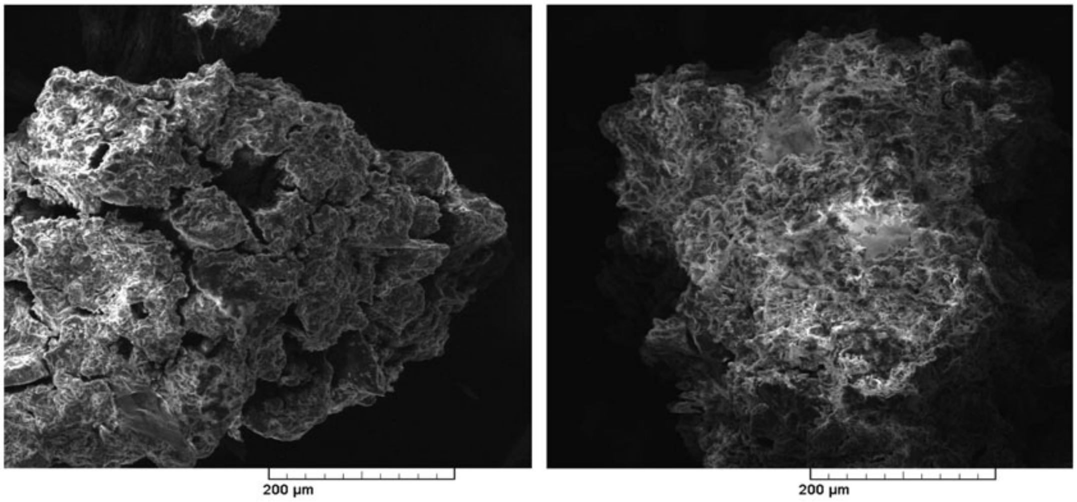

Fig. 6. SEM images of untreated peat (left) and peat treated with 10 mg L^{-1} CR solution (right) at 500× magnification.

$$q_e = \frac{K_R C_e}{1 + a_R C_e^n} \tag{4}$$

where K_R (L g^{-1}) and a_R (L $mmol^{-1}$) are the Redlich–Peterson isotherm constants, while n is the exponent which lies between zero and one. This model has a linear dependence and an exponential function of concentration to represent the adsorption equilibrium [26] and can be applied either in homogeneous or heterogeneous system. It approaches Freundlich isotherm at high concentrations and Langmuir isotherm at low concentrations [7]. By taking logarithms, Eq. (4) can be converted into the linear form,

$$\ln\left(\frac{K_R C_e}{q_e} - 1\right) = n \ln C_e + \ln a_R \tag{5}$$

The Redlich–Peterson constants can be calculated from the plot of $\ln\left(\frac{K_R C_e}{q_e} - 1\right)$ vs. $\ln C_e$. As this model has three unknowns (K_R, n, and a_R), a minimization procedure is used to solve the linearized isotherm equation by maximizing the correlation coefficient [27].

Fig. 7 shows how the extent of adsorption at equilibrium (q_e) changes with the initial concentration of CR. This is a common situation for dye adsorption on natural adsorbents due to the large surface area of the adsorbate, which normally arranged in the flat–orientation on the adsorbent surface. However, the marginal increase in the extent of adsorption at higher initial concentrations is indicative of a small contribution to multilayer adsorption. Similar observations have already been reported elsewhere for different dyes [28].

Isotherm models plotted according to the respective linearized forms result in the adsorption parameters as shown in Table 2. The validity of the Langmuir and the Redlich–Peterson models, having R^2 values close to 1.00, further supports the claim that the CR–peat adsorption system shows mainly the monolayer adsorption with a small contribution to a multilayer process.

However, the efficiency of the removal of CR will probably be less than 100% in the real environment due to many factors, such as high effluent volumes, interferent effects, pH variations, and temperature fluctuations. Nevertheless, use of peat provides an environmental friendly approach in contrast to chemical treatment of industrial effluents.

Using non-linear forms of Langmuir, Freundlich, and Redlich–Peterson models, adsorption values at

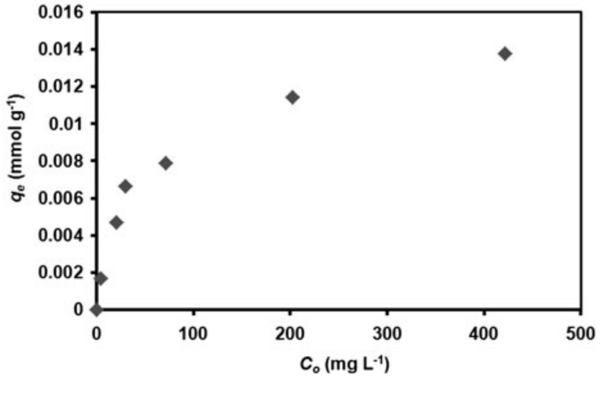

Fig. 7. Variation of the extent of removal of CR with initial concentration (0.050 g peat, 25.0 mL of 0–400 mg L^{-1} dye solutions).

Table 2
Results of the analysis of isotherm models for adsorption of CR on peat

Isotherms	Parameters of adsorption isotherms
Langmuir	$q_{\max}$ (mg g^{-1}) = 10.1
	K (L mmol^{-1}) = 0.0296
	R^2 = 0.992
Freundlich	K_F (mg$^{1-1/n}$g^{-1}L$^{1/n}$) = 0.0015
	n = 2.51
	R^2 = 0.935
Redlich–Peterson	K_R (L g^{-1}) = 0.00100
	n = 0.788
	a_R (L mmol^{-1}) = 0.2456
	R^2 = 0.994

equilibrium q_e (mmol g^{-1}) were calculated and plotted against equilibrium concentration C_e (mg L^{-1}) with experimental values of q_e and C_e (Fig. 8). It is clear that the Redlich–Peterson model is the closest to the experimental values of CR adsorption.

3.7. Error analysis

The Linear regression has been an important tool to determine the best isotherm model [29] because it avoids the inherent bias resulting from linearization. Error function analysis, in this regard, can be effectively used to evaluate the best fit of the isotherm to the experimental equilibrium data [30]. Equations used to calculate error functions are as follows [29]:
Average relative error (ARE):

$$\sum_{i=1}^{p}\left[\frac{(q_{e,\text{meas}} - q_{e,\text{calc}})}{q_{e,\text{meas}}}\right]_i \tag{6}$$

Hybrid fractional error function (HYBRID):

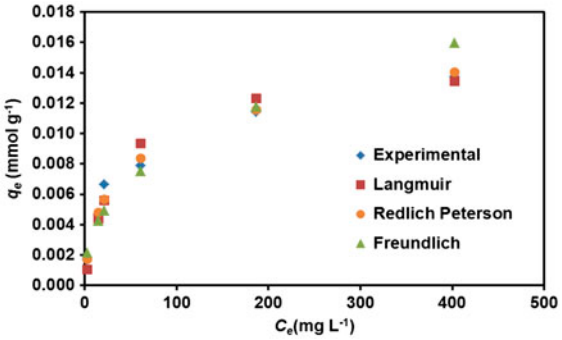

Fig. 8. Plot for equilibrium adsorption isotherms for experimental and calculated q_e values.

$$\frac{100}{n-p}\sum_{i=1}^{n}\left[\frac{(q_{e,\text{meas}} - q_{e,\text{calc}})^2}{q_{e,\text{meas}}}\right]_i \tag{7}$$

Sum of absolute error (EABS):

$$\sum_{i=1}^{n}\left|q_{e,\text{meas}} - q_{e,\text{calc}}\right| \tag{8}$$

Marquardt's percent standard deviation (MPSD):

$$\sum_{i=1}^{p}\left(\frac{q_{e,\text{meas}} - q_{e,\text{calc}}}{q_{e,\text{meas}}}\right)^2 \tag{9}$$

Nonlinear chi-square test (χ^2):

$$\sum_{i=1}^{n}\frac{(q_{e,\text{calc}} - q_{e,\text{meas}})^2}{q_{e,\text{meas}}} \tag{10}$$

In the above equations, $q_{e,\text{calc}}$ and $q_{e,\text{meas}}$ are calculated and measured adsorption capacities, respectively, n is the number of data points, and p is the number of parameters. The values obtained from these error calculations are given in Table 3. It shows that Redlich–Peterson isotherm produces the best fit on the measured errors, providing more evidence for monolayer adsorption with a contribution to a multilayer process. Similar results for CR removal using bagasse fly ash and activated carbon have been reported by Indra et al. [27].

3.8. Adsorption kinetics

Sorption kinetics is controlled by different steps including mass transfer, adsorption at a site on the surface, and diffusion of adsorbate molecules [31] and hence, adsorption kinetics should be studied for better understanding of the adsorption process. The pseudo-first-order model, the pseudo-second-order model, and the intra-particle diffusion model whose linearized equations are given below, were tested using the experimental data of CR adsorption to identify the most suitable kinetics model.

Table 3
Values of different error analyses for isotherm models

Model	ARE	HYBRID	EABS	MPSD	χ^2
Langmuir	14.77	0.019	0.005	22.97	0.0008
Freundlich	14.54	0.025	0.006	21.48	0.0010
Redlich–Peterson	5.06	0.006	0.002	9.70	0.0002

Table 4
Constants for the kinetics adsorption of CR (50.0 mg L^{-1}) on peat using different models

Model	Parameters
Pseudo-first-order model	k_1 (min^{-1}) = 0.0191 q_e (mg g^{-1}) = 4.43 R^2 = 0.899
Pseudo-second-order model	k_2 (g mg^{-1} min^{-1}) = 9.039×10^{-4} q_e (mg g^{-1}) = 4.30 R^2 = 0.935
Intra-particle diffusion model	k_3 (mg g^{-1} $min^{-1/2}$) = 2.229 C (mg g^{-1}) = 2.23 R^2 = 0.907

$$\text{Pseudo-first-order model: } \log q_e - q_t = \log q_e - \frac{k_1}{2.303}t \tag{11}$$

$$\text{Pseudo-second-order model: } \frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{12}$$

$$\text{Intra-particle diffusion model: } q_t = k_3 t^{1/2} + C \tag{13}$$

where k_1 is the rate constant of the pseudo-first-order adsorption (min^{-1}), k_2 (g mg^{-1} min^{-1}) is the rate constant of the pseudo-second-order adsorption, and k_3 (mg g^{-1} $min^{-0.5}$) is the intra-particle diffusion rate constant.

Weber and Morris equation for intra-particle diffusion (Eq. (13)) provides information on the transportation of species from the solution to the solid phase. The constant C (mg g^{-1}) is the intra-particle diffusion constant obtained as the intercept of the linear relationship (Eq. (13)). Having a non-zero intercept of this model indicates that intra-particle diffusion could not be the only mechanism involved in the diffusion process [32]. As the value of C is directly proportional to the boundary layer thickness [33], it is believed that negative value of C is due to the boundary layer thickness retarding adsorption and positive value means that there is rapid adsorption [34].

Kinetics of the adsorption reaction should be monitored before the system reaches equilibrium for results to be valid. Further, one of the reactants should be in excess to qualify for pseudo-order kinetics. Thus, a high concentration of CR (50 mg L^{-1}) was used in all kinetics studies, and measurements were taken at one minute intervals up to a 30 min period. Under these conditions, the parameters obtained from the linear plots of the above equations are given in Table 4, and the plot for the pseudo-second-order model and the intra-particle diffusion model, which result in higher regression coefficients, are shown in Fig. 9.

Adsorption mechanism can be described in three steps: surface diffusion, intra particle or pore diffusion, and adsorption on the interior side of the adsorbent. The third step is so rapid, it is assumed that overall rate of adsorption process will be controlled by either surface diffusion or intra-particle diffusion [35]. The Weber–Morris model is used to determine if intra-particle diffusion is the rate-limiting step by plotting q_t vs. $t^{1/2}$. A straight line is indicative of an adsorption process that is mainly governed by intra-particle diffusion, while a multilinear plot indicates two or more steps that are involved in adsorption process [36]. In this study, intra-particle model (Fig. 9) gave a straight line that does not pass through the origin indicating that surface diffusion is involved in rate-limiting step, further confirmed by higher R^2 value of pseudo-second-order model in this experiment. The validity of the pseudo-second-order model has been reported for the removal of CR using bael shell carbon [14], rice husk ash [37], fly ash [38], and activated carbon [39] supporting the findings of this research.

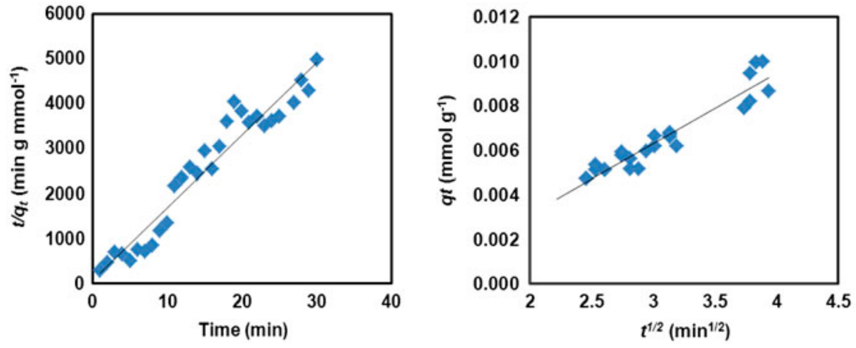

Fig. 9. Kinetics modeling for CR adsorption on peat: pseudo-second-order model (left) and intra-particle model (right).

Table 5
Comparison of adsorption capacities of various adsorbents for removal of CR

Adsorbent	$q_{\max}$ (mg g^{-1})	Reference
Bruneian peat	10.1	This work
Waste red mud	4.1	[2]
Bentonite	6.8	[14]
Kaolin	5.9	[17]
Rice husk ash	7.1	[37]
Fly ash from thermal power plant	4.9	[38]
Coal based mesoporous activated carbon	52–189	[40]
Coir pith carbon	6.7	[41]
Paddy straw	1.0	[42]
Peanut shell	15.1	[43]

Although, $q_{\max}$ can be calculated from pseudo-order kinetic models, the value obtained from the Langmuir adsorption model is probably more accurate, as kinetics is studied before the adsorption system reaches equilibrium. The values of adsorption capacities of CR on various adsorbents, including that of the adsorbent used in this study, are shown in Table 5 for comparative purposes.

4. Conclusion

The ambient pH of aqueous solutions of CR (pH 6.4) at concentration below 400 mg L^{-1} is suitable for adsorption of CR on peat as more acidic or more basic pH causes structural changes. The $\lambda_{\max}$ of CR, thus, contributes to significant error on quantitative determination of CR, unless medium pH is properly controlled. The optimum shaking time and settling time periods required to reach adsorption equilibrium of CR with peat are 1.5 and 0.5 h, respectively. The adsorption of CR is clearly evidenced by SEM images. Adsorption equilibrium experiments conducted under the optimized conditions within the initial concentration ranging from 5 to 400 mg L^{-1} result in monolayer adsorption with some contribution to multilayer adsorption fulfilling both the Langmuir and the Redlich–Peterson adsorption models with a capacity of 10.1 mg g^{-1}. Further, the adsorption of CR on peat follows pseudo-second-order kinetics with a rate constant of 9.04×10^{-4} g mg^{-1} min^{-1} according to the linearized equation. The relatively low $q_{\max}$ as compared to some other adsorbents is probably due to the very low pH$_{\mathrm{PZC}}$ of Bruneian peat. Based on these findings, it is clear that Bruneian peat can be used as an alternative adsorbent for treatment of wastewater containing CR, which is a novel approach.

Acknowledgments

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