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Sorption characteristics of peat from Brunei Darussalam for the removal of rhodamine B dye from aqueous solution: adsorption isotherms, thermodynamics, kinetics and regeneration studies

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ABSTRACT

This work reports the application of peat as cheap and effective adsorbent for the removal of rhodamine B dye (RhB) from aqueous solution. The effects of contact time, settling time and pH were studied to obtain the optimum conditions for adsorption studies. Optimum contact time was 4 h with 1 h settling time and ambient pH was used throughout the study. Adsorption isotherms, namely Langmuir, Freundlich, Temkin, Dubinin–Radushkevich, Redlich–Peterson and Sips, were employed in this study together with error functions to predict the most suitable isotherm model for the adsorption process. The Sips isotherm was found to be the best fit model. The maximum adsorption capacities ($q_{\max}$) obtained for the adsorption at 298, 313, 324, 334 and 344 K are 162.87, 166.22, 172.45, 186.82 and 201.19 mg g^{-1}, respectively. Thermodynamic parameters indicate that the adsorption was spontaneous, feasible and endothermic in nature. Pseudo second order kinetic model fitted well to the adsorption of RhB on peat. Regeneration of peat adsorbent was carried out by washing with water or using either acid or base treatment. For spent peat which was desorbed using base treatment, the removal efficiency was maintained even after five cycles, indicating that peat can be regenerated and reused effectively.

Keywords: Peat; Rhodamine B; Adsorption; Isotherms; Kinetics

1. Introduction

Applications of dyes and pigments in paper, textile, cosmetics, printing and food manufacturing industries have increased enormously, resulting the global dye consumption to reach about 10^7 kg per year [1]. Studies have revealed that about 12–20% of synthetic dyes are released into the aqueous environment through industrial wastewater, without complete treatment. These discharges may reduce light penetration in the aqueous medium, which in turn could reduce the photosynthetic activity inhibiting the growth of biota. There is also a tendency for dyes to chelate with metal ions in water which may result in

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micro-toxicity to fish and other micro-organisms [2]. For human, contamination of drinking water by dyes at even 1.0 mg L^{-1} could be toxic and is rated unsuitable for human consumption [3].

To solve these problems, various physical and chemical treatments have been investigated by researchers to remove the dye pollutants. These methods include coagulation by chemical agents [4], oxidation by Fenton process [5], photo-catalytic degradation [6], electrochemical oxidation [7] and sonochemical degradation [8], all of which have shown positive outcomes in dyes treatment. However, there are some drawbacks from these treatment methods. For example, high operational costs, expensive starting materials, long reaction time, possibility of forming secondary pollution from the solid wastes produced. Therefore, an alternative technology without the formation of sludge, known as adsorption, is a promising technique due to its ease of operation, cheap starting materials, low cost, energy saving and high efficiency in the application of discoloration process [9]. This technique has been successfully applied in the removal of various dyes. For example, methyl violet and methylene blue (MB) by Tarap skin [10], MB by peels of breadnut [11] and pineapple [12], methyl violet by *Casuarina equisetifolia* needle [13] and rhodamine B (RhB) by water chestnut peel [14].

This study is focused on the removal of RhB by peat using the adsorption technique. RhB is a highly water soluble cationic dye from the xanthene class that is widely used in textile and food industries, as well as water tracer fluorescent [15]. RhB is toxic if swallowed by human beings and animals, causing irritation to the skin, eyes and respiratory tract [16]. Carcinogenic aromatic amine is produced when RhB undergoes natural reductive anaerobic degradation [6]. Hence, it is worthwhile to make effort to remove RhB from the aqueous environment.

Peat is a dark brown soft soil with a distinctive smell, and is made up of plant remains from the partial decomposition of tress, leaves and mosses [17]. Major components of peat are cellulose, lignin and humic substances which bear polar functional groups of aldehydes, ketones, esters, alcohols, carboxylic acids and phenolic hydroxides, having the ability to interact with cation [18,19]. Peat is selected in this study because of its low cost and the presence of various functional groups including lignin phenols [20] which are expected to enhance the adsorption process. Generally, peat is capable of achieving high removal of most dyes. *Magellan* peat from Chile shows percentage removals of 29.1% for acid black 1, 2.3% for acid red 27, 1.3% for reactive black 5, 15.2% for reactive orange 16, 99.9% for the basic dyes (basic orange 2, basic blue 3, basic blue 24, basic green 4 and basic violet 4) [21]. *Sphagnum* moss peat from Northern Ireland was able to remove both acid blue 25 (Telon blue) and basic blue 69 (Astrazone blue), where $q_{\max}$ values of 251 mg g^{-1} (40˚C) and 14.4 (18˚C) mg g^{-1} were obtained for both dyes, respectively [22]. Peat from Brazil was used for the adsorption of MB, using 4.5 h as the optimum contact time [23]. Brazil peat was reported to be able to remove 40–99% of MB with an increase of dye concentration from 200 to 1,500 mg L^{-1} dye at 35˚C. The study was then extended and $q_{\max}$ values of 191.37–238.18 mg g^{-1} were obtained between 35 and 60˚C [24].

Peat adsorbent in this study was collected from Brunei Darussalam, where 18% (100,000 ha) of Brunei land is made up of peat soils that is mostly untouched [25,26]. Peat collected from different sites in Brunei Darussalam have shown its ability to remove heavy metals and dyes [27–29]. Peat from pristine and disturbed sites showed different adsorption ability for both dye and heavy metal. As compared to the disturbed site, pristine peat showed the best adsorption capability towards the removal of MB and copper(II) ions [30,31]. Further, pristine peat was investigated for its adsorption capability using crystal violet [32]. Here, as an extension from previous work, investigation for the removal of RhB was carried out using the pristine peat. To date, no work has been reported on the use of peat to remove RhB.

The aim for this study is to investigate the adsorption properties of peat for the removal of RhB dye from aqueous medium. Parameters such as contact time, settle time and pH were optimized. Surface characterization was analyzed by X-ray fluorescence (XRF), scanning electron microscope (SEM) and Fourier transform infrared (FTIR). Adsorption isotherms of Langmuir, Freundlich, Temkin, Dubinin–Radushkevich (D–R), Redlich–Peterson (R–P) and Sips models were applied in this work to determine the suitable isotherm models for the adsorption, together with the application of various error functions. Thermodynamics and kinetics studies were also investigated. Regeneration of peat adsorbent after adsorption was carried out to provide insight for its reusability and removal efficiency as a spent biosorbent.

2. Materials and methods

2.1. Adsorbent

Peat was collected from Damit (04˚N, 114˚E), located in the Belait district in Brunei Darussalam. The collected sample was dried in an oven at 60˚C until constant mass was obtained. After drying, the adsorbent was sieved to obtain particle sizes between 355 and 850 μm and store in a sealed plastic bag for further use.

2.2. Adsorbate and chemicals

RhB dye was purchased from Sigma–Aldrich and used without purification. Stock solution (1,000 mg L^{-1}) was prepared by dissolving the dye in double distilled water and diluted to the required concentrations. The structure of the dye is shown in Fig. 1. The solutions at different pHs were adjusted by using 0.1 M HNO_3 and 0.1 M NaOH, both purchased from Fluka.

2.3. Surface characterization of peat

Morphological characteristic of peat adsorbent was characterized by using Tescan Vega XMU Scanning electron microscopy (SEM) and was coated by SPI-MODULE™ Sputter Coater. Surface functional groups of the peat adsorbent were determined by FTIR spectrophotometer (Shimadzu Model IRPrestige-21) recorded in the scanning range of 4,000–400 cm^{-1} using KBr method. XRF spectrophotometer (PANalytical Axios^{max}) was used to determine the percentage of elements before and after RhB treatment.

2.4. Adsorption studies

Optimization parameters such as contact time, settle time and pH of dye solution were carried out. All experiments were conducted in a 125 mL Erlenmeyer flask containing 0.050 g of peat adsorbent and 25.0 mL of RhB solution with initial dye concentration of 10.0 mg L^{-1} at room temperature. Shaking was carried out using an orbital shaker set at a constant speed of 250 rpm, unless otherwise stated. The mixture of peat adsorbent and RhB was separated using a fine metal sieve. The extent of dye removal was determined through absorbance measurements of filtrates using UV–vis spectrophotometer (Shimadzu/Model UV-1601PC) at λ_{max} of 554.0 nm. All experiment was conducted in duplicate/triplicate.

Contact time was investigated from 30 to 240 min and the absorbance of the supernatant was recorded at 30 min interval to determine the optimum equilibrium time between peat adsorbent and RhB dye. The solutions were then allowed to settle at different time periods up to 240 min for the optimum settling time. Effect of pH of dye solution was performed by adjusting the pH of RhB solution from 2.0 to 10.0, using the optimized contact and settling time. The uptake of RhB at equilibrium, q_e (mg g^{-1}) and the percentage removal was calculated by using:

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

$$\text{Removal (\%)} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

where C_0 and C_e (mg L^{-1}) are the initial and equilibrium concentration of RhB, respectively, while V is the volume of RhB solution (L) and W is the mass of peat adsorbent used (g).

Effect of temperature was carried out by agitating the peat adsorbent with RhB dye (100 mg L^{-1}) using temperature controlled water-bath shaker set at 298, 314, 324, 334 and 344 K. Thermodynamics parameters, such as Gibbs free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°), were calculated for the dye sorption process. Effect of ionic strength was done by preparing different concentrations of KNO_3 solutions (0.1–1.0 M) and mixed with peat adsorbent using the pre-determined shaking and settling time. The peat adsorbent and dye mixture was separated and the filtrate was analyzed. Adsorption isotherms of peat adsorbent and RhB mixture were carried out by preparing a series of RhB initial concentrations ranging from 0 to 1,000 mg L^{-1}. Kinetics study was done by mixing peat adsorbent (0.05 g) with RhB dye solution (500 mg L^{-1}). The dye solution was withdrawn from each solution at 1 min interval until equilibrium has reached. The amount of adsorption at time (t) was calculated using the equation:

$$q_t = \frac{(C_0 - C_e)V}{W} \quad (3)$$

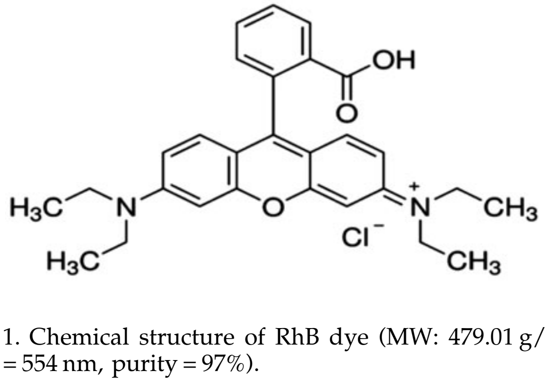

Fig. 1. Chemical structure of RhB dye (MW: 479.01 g/mol, λ_{max} = 554 nm, purity = 97%).

2.5. Regeneration of adsorbent

Regeneration was carried out by agitating peat adsorbent (0.50 g) with 100 mg L^{-1} RhB solution (250 mL) using the optimized contact time to ensure that the active sites were fully saturated. The dye-loaded adsorbent was washed with distilled water to eliminate the excess RhB dye on the surface and dried overnight at 60°C to remove the moisture. Dye-loaded adsorbent was regenerated by desorption using 250 mL of 0.1 M HNO_3, 0.001 M NaOH and water. A control (without washing) was kept to compare the adsorption capacity with the desorbed adsorbent. The amount of RhB adsorbed for five successive adsorption-regeneration cycles was analyzed using UV–vis spectrophotometer.

3. Results and discussion

3.1. Surface characterization of peat

SEM was used to study the morphology of peat adsorbent before and after adsorption. SEM image in Fig. 2(a) clearly shows that the surface of peat adsorbent before RhB adsorption is porous and irregular, which provides suitable binding sites for RhB. After adsorption, the SEM image (Fig. 2(b)) clearly showed that the pores and irregular surface of peat adsorbent were completely filled by RhB molecules.

The FTIR spectra of the peat adsorbent before and after adsorption are shown in Fig. 3. The band at 3,396 cm^{-1} is attributed to the presence of –OH and –NH functional groups. The bands at 2,920 and 2,851 cm^{-1} represent the asymmetric stretches of –CH groups and symmetric stretching vibrations of –CH group, respectively. The dual bands at 1,613 and 1,460 cm^{-1} indicates the presence of –COO, –C=O and –NH groups for the peat adsorbent before and after adsorption. After adsorption, the bands have become sharper and shifted to 1,590 cm^{-1}, with a shoulder band formed at 1,684 cm^{-1}. Another sharp band formed after treatment with RhB was observed at 1,181 cm^{-1} and this can be attributed to the C–O stretching. New band appeared at 822 cm^{-1} indicates the presence of aromatic heterocyclic molecules, suggesting the possibility of adsorption RhB molecules on peat adsorbent.

Characterization of elements for peat adsorbent before and after adsorption was carried out by XRF and the results are listed in Table 1. The elements present in peat adsorbent are Al, Br, Ca, Cl, Fe, K, Na, Mg, P, Ru, S, Si, Sr and Zn. Of these, Fe (20.16%) was present in the highest amount, followed by Zn (19.01%) and Si (10.74%). After adsorption with RhB, the amounts of Al, Ca, K, Mg and Na have reduced. For Al, the amount was reduced from 2.03% to 0.82%, for Ca from 6.36 to 3.45%, for K from 0.87 to 0.09%, for Mg from 0.50 to 0.19% and for Na from 0.47 to 0%. Hence, the reduction of elements after adsorption with RhB indicates that there is a possibility of metal replacement due to the fact that RhB dye is cationic in nature. This finding is similar to report on Tarap and breadnut peel treated with cationic dyes [10,11].

3.2. Adsorption optimization parameters

3.2.1. Effect of contact and settling time

Effect of contact time, settling time and pH of dye solution were investigated. Contact time was carried out by mixing peat adsorbent with RhB dye solution at different time intervals up to 240 min, followed by settling time to achieve full equilibrium in the peat adsorbent/dye system. As shown in Fig. 4, the uptake of RhB adsorbed onto peat increases rapidly from 30 to 120 min and gradually decreases from 150 to 240 min. This is due to the availability of large number of

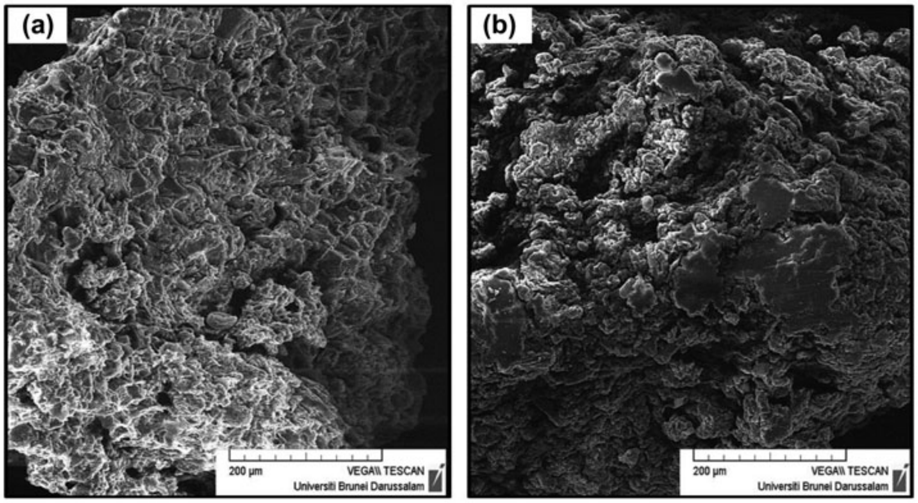

Fig. 2. SEM images of peat adsorbent (a) before adsorption and (b) after adsorption at 500× magnification.

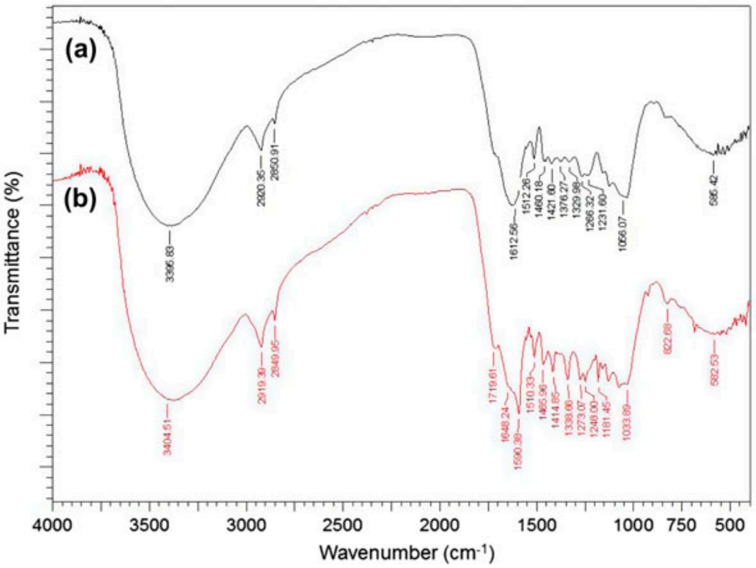

Fig. 3. FTIR spectra of (a) peat adsorbent before adsorption and (b) after adsorption with 1,000 mg L^{-1} of RhB.

Table 1
Characterization of elements present in peat adsorbent before and after adsorption

Elements	Normalized percentage (%)	
	Before adsorption	After adsorption
Al	2.03	0.82
Br	0.09	0.18
Ca	6.36	3.45
Cl	0.65	0.39
Fe	20.16	28.72
K	0.87	0.09
Mg	0.50	0.19
Na	0.47	–
O	34.95	29.45
P	0.76	0.58
Ru	1.41	1.70
S	1.96	1.93
Si	10.74	3.00
Sr	0.05	–
Zn	19.01	29.35
Zr	–	0.07

vacant sites on peat adsorbent at the initial stage of adsorption (30–120 min) and thereafter, from 150 to 240 min, the percentage removal of RhB remains constant at approximately 93% indicating the equilibrium between peat adsorbent and RhB molecules has been reached at this stage. Therefore, the optimum contact time was set at 240 min (4.0 h) for all experiments.

After the optimum contact time was determined, peat:RhB mixture was allowed to settle in order to ensure that full equilibrium has reached, as there is a possibility that the system would undergo various mass transfer or diffusion steps, which is a slow process [33,34]. Therefore, effect of settling is also an important parameter to be considered. In this case, the peat:RhB mixture was settled from 30 to 240 min. From Fig. 4, it was observed that not much change occurred after 1.0 h of settling time. Therefore, optimum settling time was fixed at 1.0 h throughout the experiment.

3.2.2. Effect of solution pH

The pH of dye solution is also an important parameter in adsorption studies. The change of pH

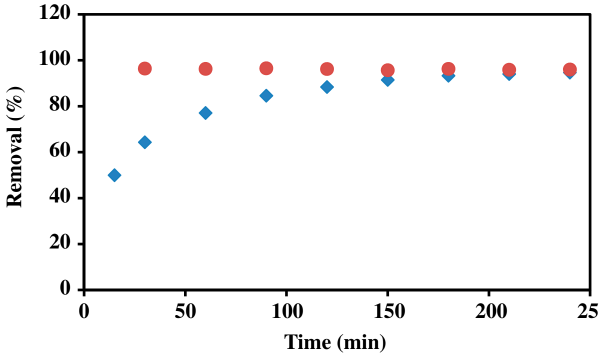

Fig. 4. Effect of contact time (◆) and settling time (●) on the adsorption of RhB onto peat adsorbent (mass of peat: 0.05 g/25.0 mL; dye concentration: 10.0 mg L^{-1}; room temperature).

could affect the surface charge, dye binding site on the cell surface and degree of ionization of the dye molecule [35]. RhB is an aromatic amino which when dissociates in aqueous solution forms a positive charge on one of the nitrogen. At the ambient pH of RhB (pH 4.40), the percentage removal is 91% as shown in Fig. 5. The percentage removal increases from pH 2–3 (87–93%) and reduced from pH 5–10 (91–88%), which is similar to the adsorption of RhB by modified tannery waste [36].

At low pH (pH 2), the surface of peat adsorbent is positive and has weak interaction with the RhB cation, due to the presence of more H^+ ions. On the other hand, at higher pH (pH 5–10), the surface of peat adsorbent is negative due to the availability of more OH^- group, allowing RhB cation to interact with the peat adsorbent. Nevertheless, when the pH of RhB is larger than 5, the zwitterionic form of RhB in water decreases the removal of RhB [2], hence the removal of RhB reduces from pH 5 to 10. Higher removal was observed at pH 3 because RhB molecules, in its monomeric form at this pH, can easily enter the pore structure of peat adsorbent [37]. Therefore, optimum pH condition for the removal of RhB was set at its ambient pH.

3.2. Effect of ionic strength

Presence of salts in industrial wastewater often leads to high ionic strength which would interact with the adsorbent and influence the ability of adsorbent to remove dyes. In this study, peat adsorbent and RhB (100 mg L^{-1}) was mixed with KNO_3 solution prepared at different concentrations ranging from 0.1 to 1.0 M. No significant effect was seen in Fig. 6 on the extent of dye removal from 0.1 to 1.0 M KNO_3, indicating the strong affinity of peat as an adsorbent for dye removal. The percentage removal of RhB at 0 M KNO_3 was 54% and increased to 75% at 0.1 M KNO_3, suggesting peat has the ability to overcome the ionic atmosphere created by K^+ and NO_3^- ions on the surface of peat particles. When the concentration of KNO_3 increases from 0.2 to 1.0 M, the extent of removal reduced slightly from 70 to 68%, which is still higher than the percentage removal of peat at its ambient condition. This could probably be due to the aggregation of dye molecules which could promote the adsorption. Hence, adsorption of RhB on peat is effective in high ionic strength condition, which is an advantage in real life application of wastewater treatment.

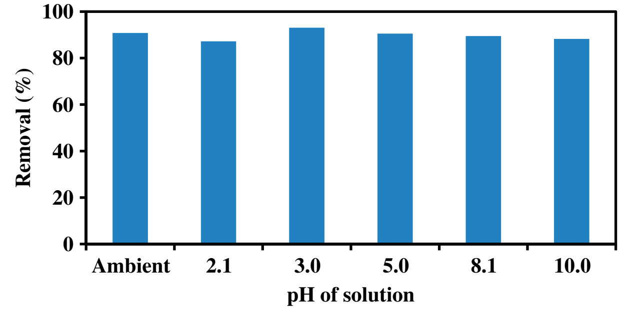

Fig. 5. Effect of initial pH on adsorption of RhB by peat (mass of peat: 0.05 g/25.0 mL; dye concentration: 10.0 mg L^{-1}; room temperature).

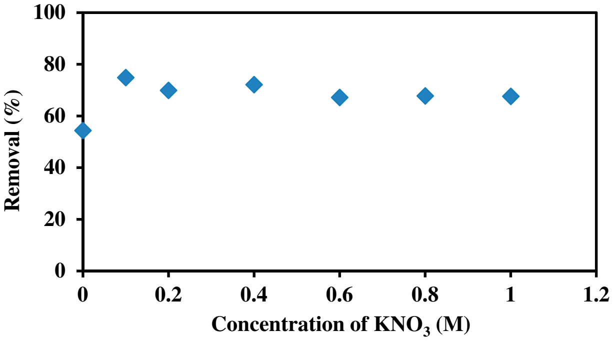

Fig. 6. Effect of ionic strength on adsorption of RhB by peat (concentration of dye: 100 mg L^{-1}; mass of peat: 0.050 g; volume of solution: 25.0 mL).

3.3. Effect of initial dye concentration and temperature

The extent of removal of RhB on peat was determined at different initial concentrations ranging from 10 to 1,000 mg L^{-1} at different temperatures (298–344 K). According to Gile’s classification [38], the adsorption between peat and RhB follow the L-type adsorption (Fig. 7), where the surface of peat adsorbent becomes saturated as the concentration increases from 500 to 1,000 mg L^{-1}. At this point, the adsorption sites are being filled; hence provide less chance of RhB molecules to accommodate onto the peat adsorbent. L-curve also indicates that when adsorption occurs, the RhB molecules would either lies with flat orientation on the surface of peat or there is competition for the surface sites of adsorbent.

Adsorption capacity increases when the temperature of the solution medium increases, suggesting an endothermic behavior of adsorption between peat and RhB. This indicates that high temperature improves the adsorption process by enlarging the pores of the adsorption sites on peat, at the same time increases the number of active sites on peat for adsorption and reduces the viscosity of RhB solution. Thus, this

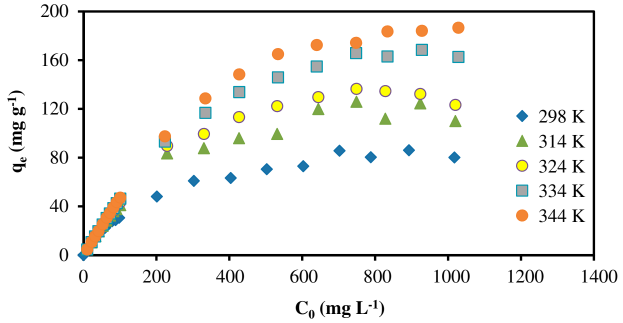

Fig. 7. Effect of initial dye concentration for the removal of RhB by peat at 298–344 K (concentration of dye: 10–1,000 mg L^{-1}; mass of peat: 0.050 g; volume of solution: 25.0 mL).

enhances the ability of functional groups present on the surface of peat adsorbent to be able to attach to the active site of RhB. Similar finding was reported for the adsorption of degreased coffee bean [38] and acid-heat rectorite [39].

3.4. Adsorption isotherms

Adsorption isotherms provide better understanding between the interaction of peat and dye molecules in aqueous condition, especially in designing and analyzing the amount of adsorbent needed to adsorb on dye molecules [40]. Initial concentrations of RhB from 0 to 1,000 mg L^{-1} were used in the present study and carried out at different temperatures from 298 to 344 K (Fig. 7). The adsorption data was fitted to six different isotherm models namely Langmuir [41], Freundlich [42], Temkin [43], D–R [44], R–P [45] and Sips [46] isotherms for the prediction of the suitable isotherm to describe the adsorption process. The non-linear and linear forms of isotherm models are summarized in Table 2. The isotherm parameters for the adsorption of RhB at 298–344 K are tabulated in Table 4. The applicability of the isotherm equations is determined through the comparison of the correlation coefficient (R^2) and error functions (Table 5) between different isotherm models.

Langmuir isotherm [41] assumes monolayer adsorption, where adsorption can only takes place at the homogeneous sites and the adsorbent becomes saturated after monolayer adsorption. The maximum adsorption capacity ($q_{\max}$) for the adsorption of RhB from 298 to 344 K was found to be 85.45, 118.99, 132.52 168.17 and 187.98 mg g^{-1}, respectively (Table 4). Langmuir constant (K_L) was 0.058 L mg^{-1} (344 K) which

Table 2
Non-linear and linearized form various adsorption isotherm models

Isotherm models	Non-linear	Linear	Plot
Langmuir	$q_e = \frac{K_L C_e q_{\max}}{1+K_L C_e}$	$\frac{C_e}{q_e} = \frac{1}{K_L q_{\max}} + \frac{C_e}{q_{\max}}$	C_e/q_e vs. C_e
Freundlich	$q_e = K_F C_e^{\frac{1}{n}}$	$\ln q_e = \frac{1}{n} \ln C_e + \ln K_F$	$\ln q_e$ vs. $\ln C_e$
Temkin	$q_e = \frac{RT}{b_T} \ln K_T C_e$ where $B = \frac{RT}{b_T}$	$q_e = B \ln K_T + B \ln C_e$	q_e vs. $\ln C_e$
D–R	$q_e = q_{\max} \exp(-\beta\varepsilon^2)$ $\varepsilon = \mathrm{RT} \ln\left[1 + \frac{1}{C_e}\right]$ $E = \frac{1}{\sqrt{2\beta}}$	$\ln q_e = \ln q_{\max} - \beta\varepsilon^2$	$\ln q_e$ vs. ε^2
R–P	$q_e = \dfrac{K_R C_e}{1 + a_R C_e^{\alpha}}$	$\ln\left(K_R \frac{C_e}{q_e} - 1\right) = \alpha \ln C_e + \ln a_R$ where $0 \le \alpha \le 1$	$\ln\left(K_R \frac{C_e}{q_e} - 1\right)$ vs. $\ln C_e$
Sips	$q_e = \dfrac{q_{\max} K_s C_e^{1/n}}{1 + K_s C_e^{1/n}}$	$\ln\left(\frac{q_e}{q_{\max} - q_e}\right) = (1/n) \ln\ C_e + \ln K_s$	$\ln\left(\frac{q_e}{q_{\max} - q_e}\right)$ vs. $\ln C_e$

Notes: q_e is the amount of dye adsorbed, C_e is the equilibrium concentration of the dye, $q_{\max}$ is the maximum adsorption capacity (mg g^{-1}), K_L (L mg^{-1}) and K_F (mg g^{-1}) are the Langmuir and Freundlich constant, n is the empirical parameter which is related to the adsorption intensity, K_T is the equilibrium binding constant (L mg^{-1}) corresponding to the maximum binding energy, constant B is related to the heat of adsorption, R is gas constant and T is absolute temperature, β gives the mean free energy, E of sorption per molecule of sorbate, K_R (L g^{-1}) and a_R (L mg^{-1}) are R–P constants and α is the exponent which lies between 0 and 1, K_s is Sips constant (L mg^{-1}) and $1/n$ is the Sips model exponent.

Table 3
R_L values based on Langmuir equation of RhB dye adsorption onto peat at 298 K

Dye concentration (mg/L)	RhB
50	0.42
100	0.31
500	0.08
1,000	0.04

is higher than $0.023\,\text{L mg}^{-1}$ at 298 K, indicating that better affinity of peat and RhB adsorption process is observed at high temperature.

Langmuir isotherm can be evaluated by dimensionless adsorption intensity (R_L) [47],

$$R_L = \frac{1}{1 + K_L C_0} \tag{4}$$

where C_0 (mg L^{-1}) is the highest initial concentration of dye and K_L (L mg^{-1}) is the Langmuir constant. The parameter R_L indicates that the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). The R_L values calculated for RhB are tabulated in Table 3. The value of R_L for RhB dye was in between 0 and 1, suggesting that the adsorption of RhB on the surface of peat was favorable. The R_L values decreases from 0.42 to 0.04 when the initial concentration of the dye increases. Study has shown that low R_L value implied that the interaction between dye molecules with peat could be relatively strong.

Freundlich isotherm [42] is derived from the assumption that the adsorption sites are distributed exponentially based on multilayer adsorption. The parameters are given in Table 4. Freundlich parameter $1/n$ relates to the surface heterogeneity. Adsorption is considered favorable when $0 < 1/n < 1$; the adsorption is homogeneous and no interaction between the adsorbed species when $1/n = 1$ and unfavorable adsorption when $1/n > 1$. In this case, the $1/n$ value for RhB lies between 0.37 and 0.40, indicating a favorable adsorption process.

Table 4
Adsorption isotherm parameters for the adsorption of RhB onto peat at 298–344 K

	Temperature (K)				
Model	298	314	324	334	344
Langmuir					
q_{max} (mg g^{-1})	85.45	118.99	132.52	168.17	187.98
K_L (L mg^{-1})	0.023	0.044	0.061	0.054	0.058
R^2	0.9890	0.9902	0.9957	0.9972	0.9980
Freundlich					
K_F (mg g^{-1})	8.07	12.65	14.93	16.89	18.31
n	2.71	2.70	2.71	2.56	2.48
R^2	0.9839	0.9357	0.9198	0.9192	0.9141
Temkin					
K_T (L mg^{-1})	0.79	1.29	1.66	1.70	1.81
b_T (J mol^{-1})	203.12	151.94	141.90	116.96	107.63
R^2	0.9363	0.9609	0.9751	0.9868	0.9884
D–R					
q_{max} (mg g^{-1})	46.74	77.31	91.42	115.61	128.17
β (J mol^{-1})	2.05×10^{-7}	2.10×10^{-7}	1.88×10^{-7}	1.83×10^{-7}	1.66×10^{-7}
E (J mol^{-1})	1560.20	1542.10	1631.92	1653.57	1736.84
R^2	0.6596	0.8196	0.8511	0.8858	0.8770
R–P					
K_R (L g^{-1})	0.045	0.061	0.097	0.100	0.200
α	0.68	0.69	0.68	0.67	0.63
a_R (L mg^{-1})	1.98	1.60	2.33	2.00	4.35
R^2	0.9988	0.9816	0.9741	0.9677	0.9610
Sips					
q_{max} (mg g^{-1})	162.87	166.22	172.45	186.82	201.19
K_s (L mg^{-1})	0.05	0.07	0.08	0.09	0.09
$1/n$	0.48	0.56	0.58	0.70	0.75
n	2.10	1.79	1.71	1.43	1.34
R^2	0.9938	0.9729	0.9741	0.9838	0.9860

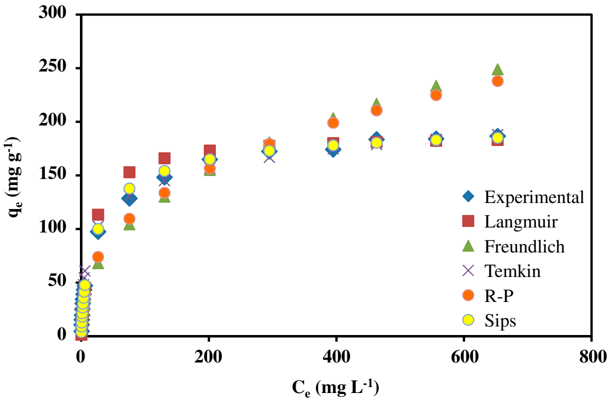

Fig. 8. Different isotherm models simulated for adsorption of RhB on peat at 344 K.

Temkin isotherm [43] assumes the heat of adsorption decreases linearly with the surface coverage due to the uniform distribution of the binding energies. D–R isotherm [44] assumes no homogeneous surface of the adsorbent. The constant β is the mean free energy E of the sorption per molecule of sorbate.

R–P isotherm [45] incorporates both Langmuir and Freundlich isotherms into a single equation. At high concentrations, R–P isotherm reduces to Freundlich isotherm; when $\alpha = 1$, R–P isotherm reduces to Langmuir equation and when $\alpha = 0$, R–P equation reduces to Henry's equation. R–P isotherm contains three parameters; hence a minimization procedure was required by maximizing the R^2 of the linear plot by using Microsoft Excel software.

Sips isotherm [46] is another combined form of Langmuir and Freundlich isotherms used for predicting the heterogeneous adsorption systems. Sips isotherm reduces to Freundlich isotherm at low dye concentration and predicts a monolayer adsorption capacity characteristic of a Langmuir isotherm at higher dye concentration.

Through comparison of R^2 values in Table 4, the Langmuir isotherm is the best-fit isotherm model for RhB adsorption on peat with the R^2 values closer to 1. However, it is sometimes difficult and inaccurate to identify the best isotherm model based on the comparison of the R^2 values. Hence, simulation of non-linear plots (Fig. 8) and various error functions (Table 5) were used in this study to provide better accuracy.

From both simulated plot (Fig. 8) and error values in Table 6, D–R isotherm model has the lowest R^2 and highest error values for the adsorption of RhB on peat, which is not suitable to describe the adsorption of RhB for all temperatures. Based on the error values and simulated plots, the Sips isotherm model is the best fit isotherm model to describe the adsorption process for all temperatures due to its high R^2 values and lowest error values compared to the other isotherm models. Both Freundlich and R–P isotherm models do not fit well onto the experimental points. However even though Langmuir and Temkin isotherm models seem to fit well onto the experimental points in Fig. 8 but the error values are higher than the Sips isotherm. Therefore, Sips isotherm model was the most suitable isotherm model to explain the adsorption process of peat adsorbent and RhB, which is valid based on the monolayer formed at high concentration of RhB as shown in Fig. 8.

The comparisons of adsorption capacities ($q_{\max}$) of peat adsorbent were made with other adsorbents as shown in Table 7 based on the best experimental condition of each work. From Table 7, it can be seen that peat has higher $q_{\max}$ than some natural adsorbents, as well as the modified adsorbents.

3.4. Thermodynamics

The thermodynamic parameters such as standard Gibb's free energy (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°) were determined by the following equations:

$$\Delta G^\circ = -RT \ln K_c \quad (5)$$

Table 5
List of different error functions

Error functions	Equations
Sum square errors (SSE)	$\sum_{i=1}^{n}(q_{e,calc} - q_{e,meas})_i^2$
Hybrid fractional error function (HYBRID)	$\frac{100}{n-p}\sum_{i=1}^{n}\left[\frac{(q_{e,meas}-q_{e,calc})^2}{q_{e,meas}}\right]_i$
Sum of absolute error (EABS)	$\sum_{i=1}^{n} \lvert q_{e,meas} - q_{e,calc} \rvert$
Non-linear chi-square test (χ^2)	$\sum_{i=1}^{n}\frac{(q_{e,calc}-q_{e,meas})^2}{q_{e,meas}}$

Notes: $q_{e,calc}$ and $q_{e,meas}$ are the calculated and measured adsorption capacity; n is the number of data points; p is the number of parameters.

Table 6
Error values for various isotherms at different temperature

Error functions	Temperature (K)	Isotherm models					
		Langmuir	Freundlich	Temkin	D–R	R–P	Sips
SSE	298	0.0032	0.0021	0.0041	0.0523	0.0010	0.0007
	314	0.0059	0.0147	0.0059	0.1052	0.0086	0.0034
	324	0.0057	0.0221	0.0049	0.1274	0.0143	0.0024
	334	0.0085	0.0307	0.0042	0.1931	0.0182	0.0015
	344	0.0083	0.0468	0.0047	0.2361	0.0369	0.0013
HYBRID	298	0.3213	0.3213	0.9898	4.7637	0.0422	0.0313
	314	0.3569	0.5114	0.7003	8.2162	0.2881	0.1451
	324	0.3582	0.7165	0.5132	9.8017	0.4686	0.1562
	334	0.4418	0.8500	0.5691	13.9391	0.5104	0.1281
	344	0.4227	1.1208	0.7011	16.4570	0.9183	0.1407
EABS	298	0.2154	0.1297	0.2209	0.9159	0.0817	0.0752
	314	0.2910	0.3393	0.2980	1.3063	0.2497	0.1942
	324	0.2811	0.4315	0.2591	1.4155	0.3276	0.1608
	334	0.3121	0.5315	0.2370	1.7230	0.3942	0.1342
	344	0.3220	0.6765	0.2327	1.8935	0.5958	0.1273
χ^2	298	0.0546	0.0157	0.1683	0.8098	0.0067	0.0050
	314	0.0607	0.0869	0.1190	1.3967	0.0461	0.0232
	324	0.0609	0.1218	0.0872	1.6663	0.0750	0.0250
	334	0.0751	0.1445	0.0967	2.3696	0.0817	0.0205
	344	0.0719	0.1905	0.1192	2.7977	0.1469	0.0225

Table 7
Comparison of maximum adsorption capacity (q_{max}) for peat adsorbent on RhB removal (based on the best experimental condition of each work)

Adsorbent	q_{max} (mg g^{-1})	References
Peat—298 K	163	This work
Peat—314 K	166	
Peat—324 K	172	
Peat—334 K	187	
Peat—344 K	201	
Fe_3O_4 nanoparticles with humic acid	162	[2]
Jute stick powder	88	[48]
Duolite C-20 cation resin	29	[49]
Polymer modified biomass	268	[50]
Ferromagnetic $BiFeO_3$	12	[51]
Turbinaria conoides (Phaeophyta)	80	[52]
Sodium montmorillonite	42	[53]
Iron-pillared bentonite	99	[54]
Kaolinite	46	[55]
Acacia nilotica leaf—microwave treated	24	[56]
Acacia nilotica leaf—chemical treated	22	
Acid-heat activated rectorite	59	[39]
Sago waste carbon	16	[57]
Cetyltrimethylammomium bromide modified sepiolite	26	[58]
Carbonaceous industrial waste	91	[59]

Table 8
Thermodynamic parameters for the adsorption of RhB on peat at various temperatures

T (K)	ΔG° (kJ mol^{-1})	ΔS° (J mol^{-1} K^{-1})	ΔH° (kJ mol^{-1})
298	−1.35	151.08	43.66
314	−3.89		
324	−5.02		
334	−7.15		
344	−8.16		

$$\ln K_c = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{6}$$

where ΔG° is the Gibbs free energy (kJ mol^{-1}), R is the universal gas constant (8.314 J mol^{-1} K^{-1}), T is the absolute temperature (K) and K_c is the distribution coefficient (C_s/C_e). C_s is concentration of dye on adsorbent (mg L^{-1}) and C_e is the equilibrium concentration of dye in solution phase (mg L^{-1}). The enthalpy (ΔH°) and entropy (ΔS°) values can be calculated from the Van't Hoff equation (Eq. (6)). Enthalpy (ΔH°) and entropy (ΔS°) values of RhB dye on peat was obtained from the slope ($-\Delta H^\circ/R$) and the intercept ($\Delta S^\circ/R$) of the ln K_c vs. $1/T$ plot (figure not shown). The thermodynamic parameters are presented in Table 8.

The negative ΔG° values suggested that the adsorption of RhB on peat was spontaneous and become more favorable at higher temperature. The ΔG° value up to −20 kJ mol^{-1} indicates a physisorption process while more negative than −40 kJ mol^{-1} implies a chemisorption process. When the value of ΔG° lies between −20 and −40 kJ mol^{-1}, both chemisorption and physisorption are taking place in the adsorption process [60]. In this study, the ΔG° value is less than −20 kJ mol^{-1}; suggesting that the adsorption is a physisorption process [61].

Positive value of ΔH° (43.66 kJ mol^{-1}) confirms the endothermic nature of dye adsorption, where the adsorption capacity increases from 163 to 201 mg g^{-1} from 298 to 344 K (Table 4). The positive value of ΔS° shows the increased disorder at solid-solution interface during the adsorption process, indicating a possible structural change and favors ion exchange.

3.5. Kinetics

Kinetic models such as Lagergren-pseudo first order model [62], pseudo second order model [63] and intraparticle diffusion model [64] were used to investigate the adsorption kinetics and rate limiting step for the adsorption of RhB.

The equation for Lagergren-pseudo first order model is given as:

Table 9
Kinetic parameters for the adsorption of RhB onto peat

Parameters	Values
$q_{e,exp}$ (mg g^{-1})	85.19
Pseudo first order	
k_1 (min^{-1})	0.008
$q_{e,cal}$ (mg g^{-1})	56.95
R^2	0.6927
Psuedo second order	
k_2 (g mg^{-1} min^{-1})	0.0005
$q_{e,cal}$ (mg g^{-1})	80.65
R^2	0.9362
Intraparticle diffusion model	
k_{id} (mg g^{-1} min$^{-1/2}$)	4.05
R^2	0.7104

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t \tag{7}$$

where q_e (mg g^{-1}) and q_t (mg g^{-1}) are the amount of RhB adsorbed at equilibrium and at any time t, respectively. k_1 (min^{-1}) is the rate constant for pseudo first order. Linear plot of log($q_e - q_t$) vs. t was plotted to calculate the k_1 and $q_{e,calc}$ values. The kinetic parameters are tabulated in Table 9. Correlation coefficient (R^2) of the pseudo first order plot is 0.6927 which is very low and the $q_{e,calc}$ value (56.95 mg g^{-1}) does not fit well onto the $q_{e,exp}$ value (85.19 mg g^{-1}). This shows that the adsorption of RhB on peat adsorbent does not follow the pseudo first order kinetics.

The pseudo second order model is calculated by:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{8}$$

where k_2 is the pseudo second order rate constant (g mg^{-1} min^{-1}). The linear plot for this model is t/q_t vs. t. From Table 9, the $q_{e,calc}$ (80.65 mg g^{-1}) and $q_{e,exp}$ (85.19 mg g^{-1}) values are very close to each other, compared to pseudo first order model. In addition, the R^2 of pseudo second model is closer to 1. This suggests that pseudo second order is the more suitable kinetic model to explain the interaction of RhB on peat adsorbent.

The equation for the intraparticle diffusion model is:

$$q_t = k_{id} t^{1/2} + C \tag{9}$$

where k_{id} (mg g^{-1} min$^{-1/2}$) is the intraparticle diffusion rate constant and C is a constant directly proportional to the boundary layer thickness. The k_{id} value is obtained from the linear plot of q_t vs. $t^{1/2}$ and C is the intercept on the y-axis. From Table 9, the low R^2 value indicates that the adsorption of RhB is not controlled by the intraparticle diffusion. Adsorption that is controlled by intraparticle diffusion must have a straight line that passes through the origin and with an intercept that is equal to zero [65].

3.6. Regeneration

Generally, dye-saturated adsorbents are usually discarded or dumped in landfill since they are of no use after adsorption process. Hence, it is more economical if the dye-saturated adsorbent can be regenerated, especially after adsorption process. This will minimize the cost for adsorbent treatment and subsequently reduce the cost of waste disposal. In this study, dye-loaded adsorbent was regenerated by using 0.1 M HNO_3, 0.001 M NaOH and water for five adsorption-regeneration cycles using the predetermined contact time. In Fig. 9, the regeneration efficiency of peat adsorbent by acid is similar for cycles 1 (45%) and 2 (43%). Thereafter, the adsorption uptake of RhB from cycles 3–5 gradually reduced from 33 to 24%. Similar trends were observed for regeneration with washing in water and the control (no washing). The regeneration efficiencies of peat adsorbent from cycles 1–5 have reduced from 45 to 22 and 20% for water and control respectively. The reduction might be attributed to the collapse of porosity on the surface of peat adsorbent after various adsorption-regeneration cycles, causing the pore blockage which restricts RhB molecules to adsorb onto the surface. Regeneration efficiency remains almost constant from cycles 1 (45%) to 3 (44%) for base regeneration and reduces to 40% at cycle 5. This indicates that amongst the methods used in this study, base regeneration is considered to be the most suitable regeneration method for the peat adsorbent, which enabled the reusability of adsorbent for a longer period while maintaining its high removal efficiency.

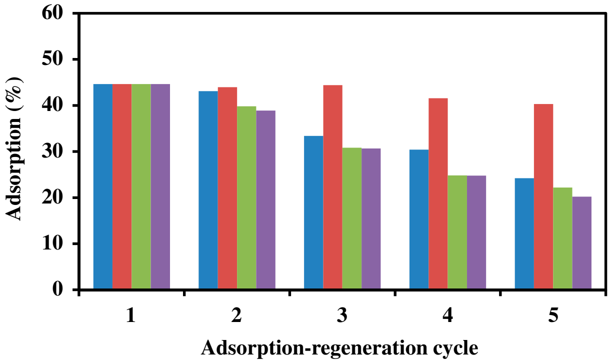

Fig. 9. Regeneration of peat adsorbent by (●) 0.1 M HNO_3, (●) 0.001 M NaOH, (●) water and (●) control for five consecutive cycles.

4. Conclusion

The present study shows that peat adsorbent is an effective and cheap adsorbent for the removal of RhB dye from aqueous solution. The adsorption was fixed at 4 h for the contact time and 1 h for the settling time to ensure total equilibrium has achieved. No pH adjustment was needed throughout the adsorption process. Percentage removal of dye increases from 54% to 68% when concentration of KNO_3 increases. Adsorption capacity increases when the temperature of the solution medium increases. Negative ΔG° indicates that the adsorption is spontaneous and more feasible as the temperature increases. The values of ΔH° and ΔS° are 43.66 kJ mol^{-1} and 151.08 J mol^{-1} K^{-1}, respectively. Adsorption isotherm for RhB fits well to the Sips isotherm model which has the lowest error functions compared to the other isotherm models. The adsorption capacity (q_{max}) of RhB for 298, 314, 324, 334 and 344 K are 162.87, 166.22, 172.45, 186.82 and 201.19 mg g^{-1}, respectively, which is comparable with other natural and activated adsorbents. Kinetics of adsorption followed the pseudo second order model. Dye-loaded peat adsorbent could be regenerated and recycled by base and the regeneration efficiency remains almost constant for five consecutive adsorption-regeneration cycles.

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