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The adsorption of basic dyes from aqueous solution on modified peat–resin particle

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Abstract

Modified peat was prepared by mixing thoroughly raw peat with sulfuric acid, and modified peat–resin particle was obtained, by mixing modified peat with solutions of polyvinylalcohol (PVA) and formaldehyde. In this paper, the adsorption of Basic Magenta and Basic Brilliant Green onto modified peat–resin particle is examined. The adsorption isotherm showed that the adsorption of basic dyes on modified peat–resin particle deviated from the Langmuir and Freundlich equations. The pseudo-first order, pseudo-second order and intraparticle diffusion models were used to fit the experimental data. By comparing the standard deviation, it was found that the intraparticle diffusion model could be used to well describe the adsorption of two basic dyes on modified peat–resin particle. According to the change of intraparticle diffusion parameter, the adsorption processes could be divided into different stages. The kinetics experiment also indicated that initial dye concentrations, particle dose and particle size could affect the adsorption processes of basic dyes.

Keywords: Modified peat–resin particle; Basic dyes; Adsorption isotherm; Adsorption kinetics; Kinetic models

1. Introduction

Wastewater containing dyes from textile industry is very difficult to treat using conventional wastewater treatment methods, since the dyes are stable to light and oxidizing agents, and are resistant to aerobic digestion ([1,2]). Adsorption techniques to remove dyes in solution have been widely used. Most commercial systems currently use activated carbon as adsorbent to remove dyes in wastewater. Activated carbon is expensive, which means higher cost is in wastewater treatment. In order to decrease the cost of wastewater treatment, attempts have been made in finding inexpensive adsorbents. Studies showed that many materials, such as low rank coals, peat moss, chitosan and wood, could be used as adsorbents [2–5] which could effectively remove the dyes from solution.

Peat, as an adsorbent, is porous and rather complex material, containing lignin and cellulose. Recently, peat has been used to remove some pollutants (such as heavy metals, dyes and oil) from aqueous solution [6–9]. Many studies showed peat could effectively remove the dyes from aqueous solution ([1,10–15]). Poots et al. [1,2] investigated adsorption of acid dye (Acid blue 25) on peat by fixed bed. And results showed that peat was a good adsorbent for removal of acid blue 25. Using batch adsorption system, Allen et al. [10–12] studied adsorption processes of acid and basic dyes on peat. In the isotherm experiment of basic dyes, Allen et al. [10] found the isotherm of basic dyes exhibited deviation from the theory (Langmuir isotherm equation). They thought this deviation was attributed to the pressure of adsorbing ions creating a new surface by expanding the adsorbent particle. Allen et al. [11,12] investigated the diffusion of

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Nomenclature

C_t	dye concentration in solution at time t (mg l^{-1})
C_e	equilibrium concentration (mg l^{-1})
q_e	adsorption capacity in equilibrium (mg g^{-1})
C_0	initial dye concentration in solution (mg .l^{-1})
D_0	diffusion coefficient (m s^{-1})
F	fractional uptake of solute
k_f	rate constant of pseudo-first-order model (l min^{-1})
k_I	rate parameter of intraparticle diffusion model, (mg dye g^{-1} particle $min^{-0.5}$)
k_s	rate constant of pseudo-second-order model (g particle mg^{-1} dye min^{-1})
M	mass of modified peat–resin particle (g)
q_t	amount of adsorption at time t, (mg g^{-1})
r	particle radius (mm, cm, m)
t	time (min)
Q	maximum adsorption capacity (mg g^{-1})
V	volume of solution (l)
Δq	standard deviation

acid and basic dyes inside a peat particle, also. They used the intraparticle diffusion model to describe the adsorption processes of basic dyes on peat and divided adsorption processes into separate steps in terms of diffusion rate parameter.

Peat, as an adsorbent, can effectively remove the pollutants from solution and is inexpensive. However, when raw peat is directly used in wastewater treatment, there are many limitations, such as low chemical stability and mechanical strength, leach of fulvic acid from peat and difficult regeneration [16]. In order to overcome these limitations, we prepared the modified peat–resin particle by mixing modified-peat with polyvinylalcohol (PVA) and formaldehyde. The modified peat–resin particle contains polar functional groups, such as alcohols and acids. Both modified peat and resin in particle can adsorb the dyes from solution.

In this paper, the adsorption isotherm and kinetics experiments of basic dyes (Basic Magenta and basic Brilliant Green) were conducted and the different kinetic models were used to analyze adsorption processes of basic dyes on modified peat–resin particle.

2. Materials and methods

Raw peat was obtained from Jilin province, which was commercially available for agriculture purpose in China.

2.1. The preparation of modified peat–resin particle

Raw peat was dried at room temperature, and then broken using a hammer mill and finally screened through a 60 mesh sieve. The process of preparing the modified peat–resin particle is as follows:

1. *Oxidizing peat*: raw peat (200 g, d. w.) was thoroughly mixed with sulfuric acid (100 ml) in a flask (2l) at room temperature, which could prepare modified-peat.
2. *Gelling*: distilled water (300 ml) was mixed with modified-peat and heated it to a boil, then add 10% PVA solution (100 ml) and 37% formaldehyde solution (80 ml) were added with swift, agitation until “modified peat–resin gel” was formed. Then the “modified peat–resin gel” was broken with a hammar water was poured into the powdered mixture (200 ml) and heated it to a boil.
3. *Coating*: 5% PVA solution (100 ml) was added, agitating and heating. This step was repeated for five or six times.
4. *Washing and drying*: the particle was washed with deionized water until filtrate reached pH 5.5—6.5, then it was dried in an oven at 65±5°C for 24 h.
5. *Sieving*: the particle was crushed and sieved.

With regard to the modified peat–resin particle, the basic structure was that resin adhered modified-peat powder into microparticles and, then, many microparticles formed gradually particle with different size. The modified peat–resin particle had a macroreticular structure. The porosity and the ratio of macropore in the modified peat–resin particle changed with the particle size. Both modified-peat and resin could adsorb the basic dyes in the adsorption processes.

Fig. 1 shows the pore distribution of modified peat–resin particle. Fig. 1, shows that micropore (< 20 Å) and transitional pore (20—50 Å) are predominant in the modified peat–resin particle.

Table 1 shows the specific surface area of the particle (measured by nitrogen adsorption method, ASAP2000, MICROMERTICS). Table 1 shows that the specific surface area of modified peat–resin particle is very low at different size.

We think the specific surface area presented in Table 1 is lower than the true one. The low value of specific surface area is caused by high temperature in measurement. In order to dry the particle, a temperature of 150°C was used in measurement. The temperature of 150°C may seriously cause collapse of pore and/or

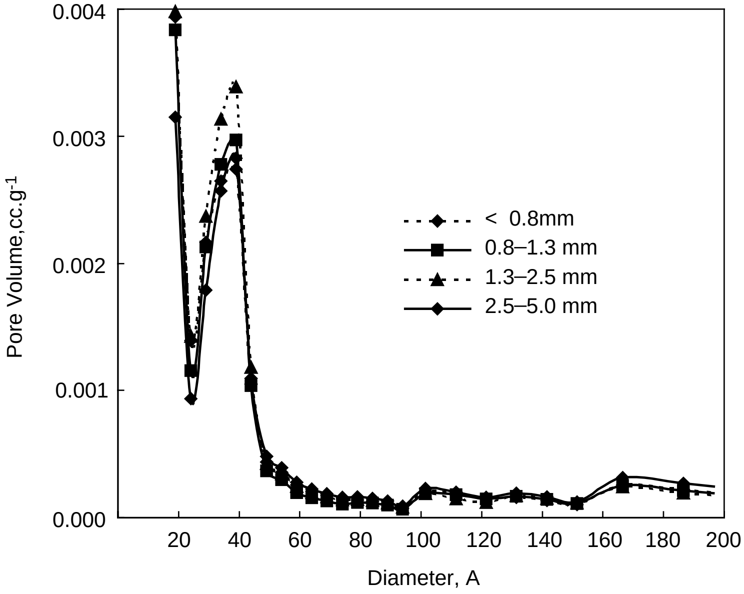


Fig. 1. Pore distribution of peat–resin particle.

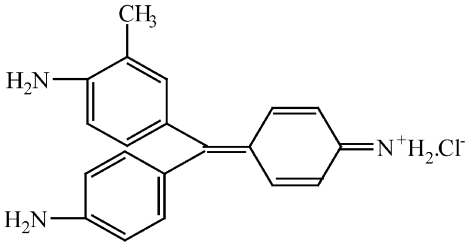
Table 1
Specific surface area of peat–resin particle ($m^2 g^{-1}$)

Size of particle (mm)	2.5–5.0	1.3–2.5	0.8–1.3	<0.8
Specific surface area	9.79	12.40	10.62	10.76

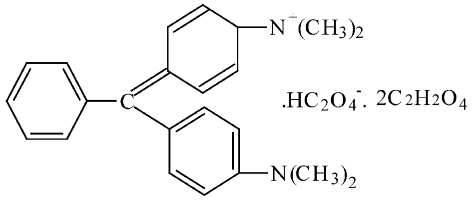
shrinkage of pore diameter. So Table 1 and Fig. 1 only present some information about specific surface area and pore distribution of modified peat–resin particle.

2.2. The agents

The Basic Magenta (C. I. Basic Violet 14) and Basic Brilliant Green (C. I. Basic Green 4) used was the commercial salt, which were widely used in textile industry. Their structure is as illustrated.



Basic Magenta (C. I. Basic Violet 14)



Basic Brilliant Green (C. I. Basic Green 4)

2.3. Experimental procedure

Adsorption isotherm: modified peat–resin particle, 0.5000 g was thoroughly mixed into 100 ml of 50—2400 $mg\,l^{-1}$, and then the container was sealed up to prevent change of volume of the solution during the experiments. The size of the particle was 2.5—5.0 mm. Suspensions were played for eight weeks at a 25±0.5°C constant temperature to ensure that adsorption equilibrium was established. The equilibrium solution was filtered using stainless steel sieve (300 mesh, the first filtrate of 5—10 ml was discarded).

Adsorption kinetics: the particle was thoroughly mixed with 2000 ml dye solution in the reaction vessel at constant temperature (25°C). 2 ml samples were drawn at suitable time intervals, and filtered through stainless-steel sieve (300 mesh).

The concentration of dyes in solution sample was determined using a spectrophotometer with a 1.0 cm glass light cell at a wavelength corresponding to the maximum absorbance.

2.4. Calculation

The amount of dye adsorbed at time t, q_t, was calculated from the mass balance equation

$$qt = \frac{(C_0 - C_t)V}{m}. \tag{1}$$

In order to quantitatively compare the applicability of different kinetic models in fitting to data, a normalized standard deviation, Δq, was calculated

$$\Delta q(\%) = 100 \times \sqrt{\frac{\sum[(q_{t\,\mathrm{exp}} - q_{t\,\mathrm{cal}})/q_{t\,\mathrm{exp}}]^2}{n-1}}, \tag{2}$$

where n is the number of data points; $q_{t\mathrm{exp}}$ the experimental values; and $q_{t\mathrm{cal}}$ the calculated values by model.

3. Theoretical

The adsorption of dye onto modified peat–resin particle can be divided into three consecutive stages. First, dye migrates through the solution to the exterior surface of modified peat–resin particle. Second, the dye moves within the particle. Then, third, the dye is adsorbed at sites on the interior surface of the modified peat–resin particle. Many factors can affect the adsorption rate of dye on modified peat–resin particle, such as the initial dye concentration, particle size, particle dose and turbulence state of solution. Generally the third stage is very rapid and does not form a rate-limiting stage in the adsorption.

3.1. Adsorption isotherm

In adsorption isotherm study, adsorption isotherm equations usually used include Langmuir equation, Freundlich equation, Redlich–Peterson equation and BET equation. For the liquid–solid system, the Langmuir and Freundlich isotherm equations usually are employed.

3.2. Kinetic models

In order to investigate the adsorption processes of two basic dyes on modified peat–resin particle, three kinetic models are used, including pseudo-first-order model, pseudo-second-order model and intraparticle diffusion model.

3.3. Pseudo-first-order model

The pseudo-first-order equation is

$$\frac{\mathrm{d}q_t}{\mathrm{d}t} = k_\mathrm{f}(q_\mathrm{e} - q_t), \tag{3}$$

where k_f is the rate constant of pseudo-first-order model. After definite integration by applying the initial conditions $q_t=0$ at $t=0$ and $q_t=q_t$ at $t=t$, the equation becomes [17]:

$$\log(q_\mathrm{e} - q_t) = \log q_\mathrm{e} - \frac{k_\mathrm{f}}{2.303}t. \tag{4}$$

Eq. (4) is a linear form. Plotting the $\log(q_\mathrm{e}-q_t)$ against t, a line can be obtained.

3.4. Pseudo-second-order model

The pseudo-second-order model can be represented in the following form [18]:

$$\frac{\mathrm{d}q_t}{\mathrm{d}t} = k_\mathrm{s}(q_\mathrm{e} - q_t)^2, \tag{5}$$

where k_s is the rate constant of pseudo-second-order model. After integrating Eq. (5) for boundary conditions $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_t$, the following form can be obtained:

$$\frac{t}{q_t} = \frac{1}{k_\mathrm{s}q_\mathrm{e}^2} + \frac{1}{q_\mathrm{e}}t. \tag{6}$$

Plotting the t/q_t against t, a line can be obtained and the q_e also can be calculated.

3.5. Intraparticle diffusion model

Theoretically, adsorption rate of the dye onto the particle depend on the rate of mass transport processes of the dye within the modified peat–resin particle. For the porous modified peat–resin particle, the mass transport processes may mainly be conformed in the form of diffusion.

In a liquid–solid system, the fractional uptake of the solute on particle, F, will vary with the function [12]:

$$\frac{D_0 t^{0.5}}{r^2}.$$

Hence there is a linear relationship between F and $t^{0.5}$ for much of the adsorption processes. F can be defined as

$$F = (C_0 - C_t)/C_0. \tag{7}$$

The diffusion rate of the dye molecule in particle, k_i, can be calculated by the following equation:

$$k_\mathrm{i} = q_t/t^{0.5}. \tag{8}$$

Its linear form is

$$q_t = k_\mathrm{i}^{0.5} \tag{9}$$

where, k_i is the intraparticle diffusion rate.

According to previous studies, the plot of q_t against $t^{0.5}$ may present a multi-linearity [12], which indicates that two or more steps occur in the adsorption processes. The first sharper portion is the external surface adsorption or instantaneous adsorption stage. The second portion is the gradual adsorption stage, where the intraparticle diffusion is rate-controlled. The third portion is the final equilibrium stage, where the intraparticle diffusion starts to slow down due to the extremely low solute concentration in solution [5,19].

4. Results and discussion

4.1. Adsorption isotherm

Fig. 2 is the adsorption isotherm of Basic Magenta and Basic Brilliant Green. With the initial dye concentration increase, the amount of adsorption of dye on modified peat–resin particle increased swiftly. No maximum adsorption value was observed as shown in Fig. 2.

The data of adsorption isotherm of Basic Magenta and Basic Brilliant Green could not be described by isotherm equations of Langmuir and Freundlich. Plots of C_e/q_e vs. C_e and $\ln q_e$ vs. $\ln C_e$ were not line. The fitness between experimental data and theoretical models was not good ($r^2 < 0.95$). This result is similar to that of Allen et al. [10].

Allen et al. [10] studied adsorption isotherm of basic dyes using sphagnum peat, the results show that the adsorption isotherm of basic dyes deviate from Langmuir and Freundlich adsorption isotherm equations. They thought that the deviation could be attributed to the pressure of adsorbing ions creating new surface by expanding the adsorbent particle.

The adsorption isotherms indicated that the adsorption of Basic Magenta and Basic Brilliant Green on modified peat–resin particle might be the "multi-molecular layer in thickness".

The experiments of the adsorption isotherm showed that modified peat–resin particle could effectively remove both Basic Magenta and Brilliant Green from the aqueous solution (especially, at a high dye concentration). The modified peat contained an amount of negatively charged groups and exchangeable ions (such as H^+) associated with humic acid. The basic dyes could be adsorbed by electrostatic interactions and ion exchange. Of course, the resin, which contained an amount of hydroxide (–OH), could also adsorb the basic dyes by chemical reaction.

4.2. Kinetics of adsorption

4.2.1. Effect of experimental conditions on adsorption processes

Figs. 3–6 show the effect of the initial dye concentration, particle size, agitation speed and particle mass on adsorption of Basic Magenta. Figs. 3–6 indicate that adsorption rate of the dye was very fast at the beginning stage and, then, slow down gradually. The initial dye concentration, particle size and mass could significantly affect the adsorption rate. In Fig. 5, the adsorption rate of less than 0.8 mm was between that of 2.5–5.0 and 1.3–2.5 mm, the reason needs to be investigated further. The difference of adsorption rate at different agitation speed was insignificant. Similar phenomena were observed in the kinetic experiment of Basic Brilliant Green.

4.3. Test of kinetic models

In order to explore the kinetic processes of adsorption, it is a good method that using different models to fit the experimental data and comparing the normalized standard deviation. Table 2 presents the result of fitting experimental data with pseudo-first and pseudo-second-order models and intraparticle diffusion model.

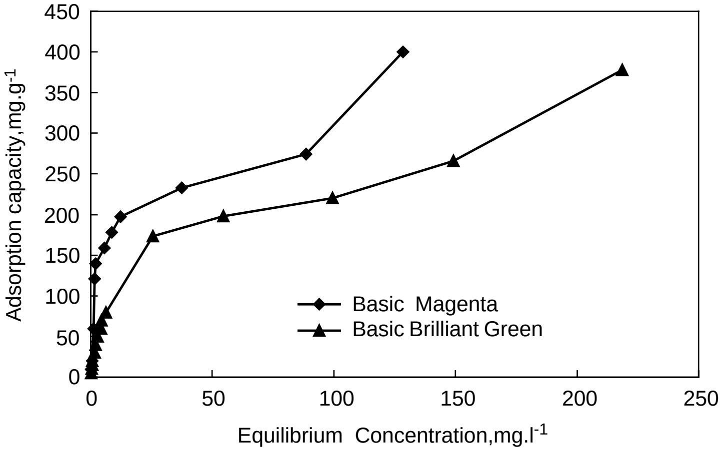


Fig. 2. Adsorption Isotherm of Basic Magenta and Basic Brilliant Green.

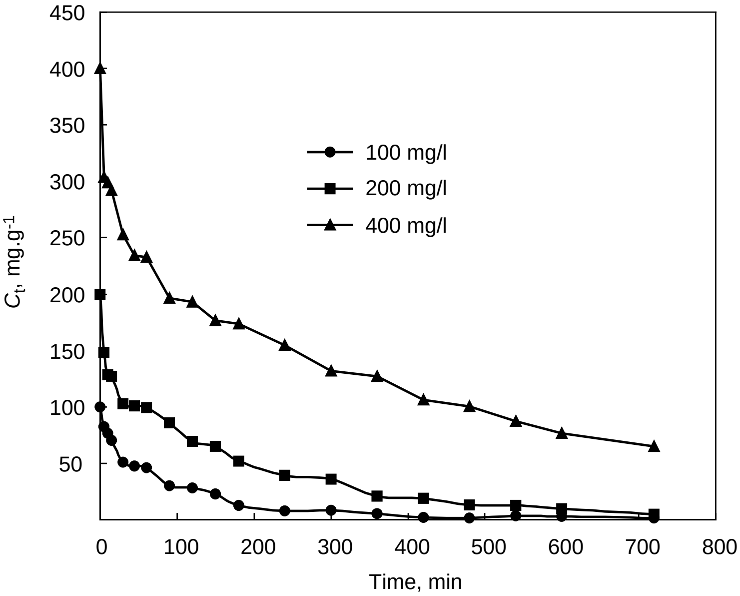


Fig. 3. Plot of C_t vs. time at different initial concentrations (25°C, 300 rpm).

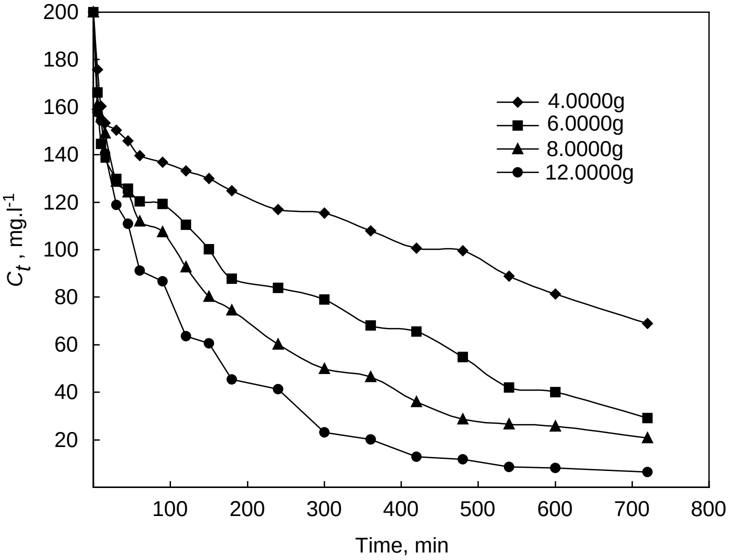


Fig. 4. Plot of C_t vs. time at different particle mass ($C_i = 200\,\mathrm{mg\,l^{-1}}$, 25°C, 300 rpm).

From Table 2, the order of Δq(%) was pseudo-first > pseudo-second > intraparticle diffusion model in all of the experimental conditions, which indicated that the intraparticle diffusion model was the best one in describing the adsorption kinetics of basic dyes on modified peat–resin particle. Fig. 7 typically illustrates the comparison between the calculated and measured results for adsorption of Basic Magenta. From Fig. 7, it was found that the pseudo-first and pseudo-second-models underestimate at the initial stage of adsorption. The measured results indicated that the adsorption rate was very fast at the beginning stage of adsorption.

4.4. The intraparticle diffusion of basic dye in particle

As mentioned above, the intraparticle diffusion model could describe the adsorption kinetics of basic dyes well. Fig. 8 is the plot of q_t vs. $t^{0.5}$ at different initial dye concentration, which indicated that the plot of q_t vs. $t^{0.5}$ was multi-linear. The intraparticle diffusion constants

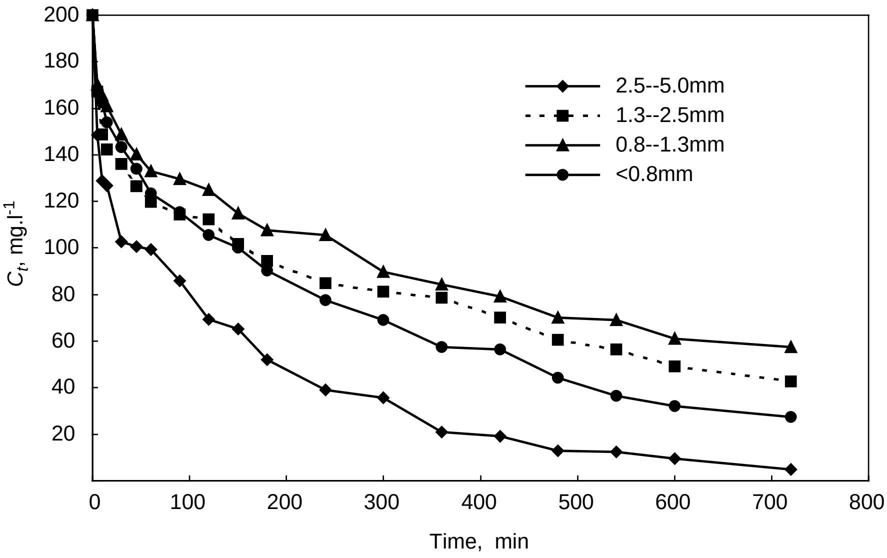


Fig. 5. Plot of C_t vs. time at different particle sizes (25°C, $C_\mathrm{i} = 200\,\mathrm{mg\,l^{-1}}$, 300 rpm)

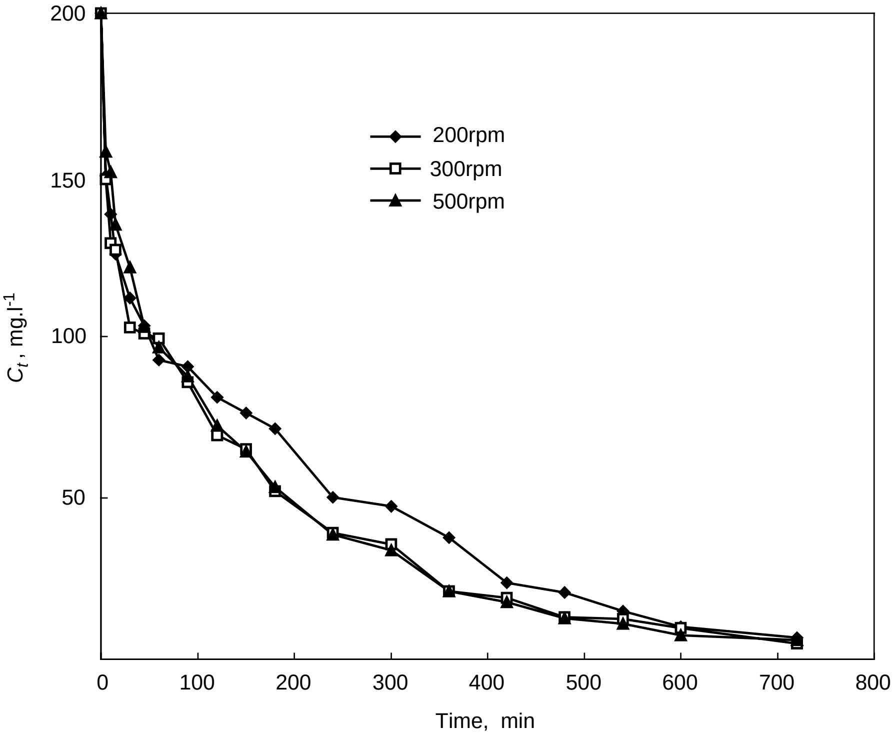


Fig. 6. Plot of C_t vs. time at different agitation speed ($C_\mathrm{i} = 200\,\mathrm{mg\,l^{-1}}$, 25°C)

could be calculated using Eq. (8) or Eq. (9). Table 2 presents intraparticle diffusion constants (k_{i1}, k_{i2} and k_{i3}). The k_{i1}, k_{i2} and k_{i3} express diffusion rates of the different stages in the adsorption.

From Table 2, it is seen that the order of adsorption rate was the first stage (k_{i1}) > second stage (k_{i2}) > third stage (k_{i3}). At the beginning, the dye was adsorbed by the exterior surface of the modified peat–resin particle,

Table 2
Kinetic parameters and normalized standard deviation for adsorption of basic dyes on modified peat–resin particle (25°C)

Solute	Pseudo-first order		Pseudo-second order			Intraparticle diffusion			
	k_{f} ($\times 10^{-3}$)	Δq (%	k_{s}($\times 10^{-4}$)	q_{e}	Δq(%)	$k_{i.1}$	$k_{i.2}$	$k_{i.3}$	Δq (%)
Basic Magenta									
particle mass (g)									
4.0000	1.84	53.62	0.30	90.90	48.38	6.11	1.76	1.54	5.78
6.0000	3.22	48.72	1.00	62.50	39.31	5.47	1.61	1.23	6.05
8.0000	4.14	44.79	3.00	47.62	22.86	3.16	1.56	0.39	3.54
12.0000	5.99	40.46	12.00	33.22	22.20	2.42	1.33	0.31	4.52
Initial concentration (mg l^{-1})									
100	8.75	33.33	13.60	19.95	9.86	1.74	0.97	0.12	4.84
200	6.45	39.29	5.82	39.29	23.19	3.99	1.29	0.42	3.19
400	2.99	51.65	2.94	67.56	25.97	5.78	1.80	0.99	5.07
Particle sizes (mm)									
2.5–5.0	6.45	39.29	5.82	39.29	21.70	3.99	1.29	0.42	3.19
1.3–2.5	2.53	53.00	11.60	29.33	28.81	3.06	0.86	0.54	4.43
0.8–1.3	2.07	52.81	10.20	31.35	27.30	1.64	0.89	0.32	3.46
<0.8	3.22	46.85	7.79	35.84	22.83	2.06	1.17	0.49	2.91
Agitation speed (rpm)									
200	5.30	44.65	5.12	39.53	23.86	3.26	1.11	0.43	2.02
300	5.76	44.16	5.82	39.29	21.70	3.99	1.29	0.42	3.19
500	6.45	39.60	5.18	39.78	17.86	3.29	1.48	0.40	3.28
Basic Brilliant Green									
Initial concentration (mg l^{-1})									
100	6.68	47.76	25.20	19.92	15.79	1.47	0.80	0.10	5.18
200	5.76	45.81	6.50	39.22	27.00	2.90	1.26	0.38	17.21
400	5.76	47.68	2.10	68.97	39.32	5.91	1.65	0.82	19.34
Particle mass (g)									
5.0000	4.61	42.08	3.39	58.35	22.74	4.20	2.39	1.12	9.89
10.0000	5.30	43.04	6.30	39.84	16.06	2.90	1.26	0.38	10.23
15.0000	6.22	45.51	13.84	26.88	6.15	2.75	1.09	0.17	4.38
20.0000	7.14	46.52	24.41	20.24	6.78	2.23	0.73	0.11	5.70

both the modified peat and resin could adsorb the dye, so the adsorption rate was very fast. When the adsorption of the exterior surface reached saturation, the molecular dye entered into the modified peat–resin particle by the pore within the particle and was adsorbed by the interior surface of the particle. When the molecular dye diffused in the pore of the particle, the diffusion resistance increased, which caused the diffusion rate to decrease. With decrease of the dye concentration in the solution, the diffusion rate became lower and lower, the diffusion processes reached the final equilibrium stage. Therefore the changes of k_{i1}, k_{i2} and k_{i3} could be attributed to the adsorption stages of the exterior surface, interior surface and equilibrium, respectively. Allen et al. [12] thought that there were four separate regions depicting the mass transfer onto peat, i.e. external mass transfer effect, macropore diffusion, transitional pore diffusion and micropore diffusion.

Table 2 shows that all of k_{i1}, k_{i2} and k_{i3} increased with initial dye concentration. The driving force of diffusion was very important for adsorption processes. Generally the driving force changes with the dye concentration in bulk solution. The increases of dye concentration result in increase of the driving force, which will increase the diffusion rate of the molecular dye in pore.

k_{i1}, k_{i2} and k_{i3} decreased with increase of particle mass. The increase of particle mass usually results in increase of the exterior surface, which causes swift decrease of the dye concentration in bulk solution and driving force.

Generally, the diffusion rate increases with decreasing particle size [12]. From Table 2, it is found that the effect of diffusion rate on particle size was irregular, which could be related to structure and chemical components of modified peat–resin particle.

Table 2 also indicates that the effect of diffusion rate on agitation speed was not significant. With increase of agitation speed from 200 to 500 rpm, the change of diffusion rates, k_{i1}, k_{i2} and k_{i3}, was little, which means that the resistance of particle surface was very low for molecular dye from bulk solution entering into a

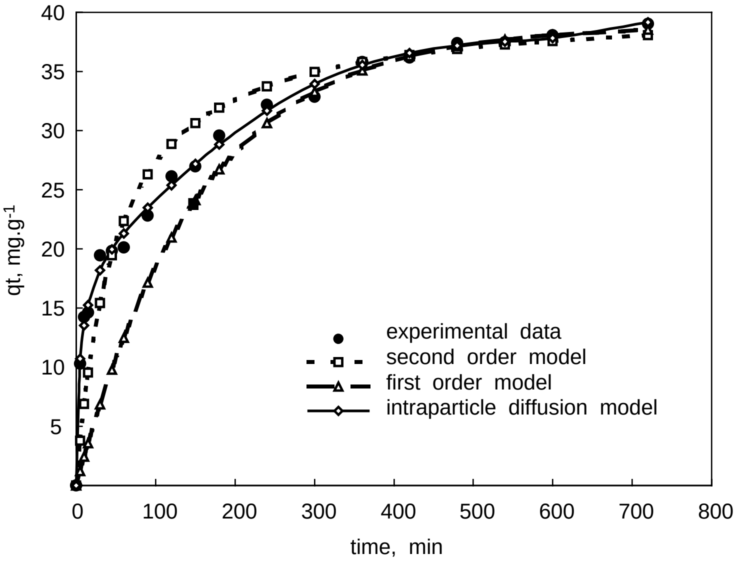


Fig. 7. Plot of between the measured and modeled time profiles for adsorption of Basic Magenta at initial concentration 200 mg l^{-1} (25°C)

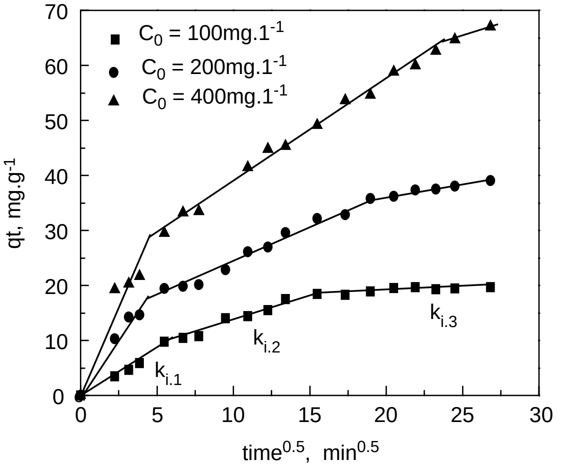


Fig. 8. Plot of q_t vs. $t^{0.5}$ at different concentrations.

particle. This result was different from that obtained by Allen et al. [12].

5. Conclusions

The adsorption isotherm and kinetics of two basic dyes were studied in batch experimental system. The following results were obtained:

1. The adsorption isotherm of Basic Magenta and Basic Brilliant Green deviated from the Langmuir and Freundlich equations. The adsorption capacity of Basic Magenta and Basic Brilliant Green on a modified peat–resin particle was very high.
2. The adsorption processes of Basic Magenta and Basic Brilliant Green on a modified peat–resin particle could be well described by intraparticle diffusion model, and the adsorption rate of two basic dyes on a modified peat–resin particle was mainly controlled by the diffusion rate of the molecular dye within a particle.
3. The initial dye concentration could significantly affect the diffusion rate of molecular dye in a particle. The diffusion rate of basic dyes increased with initial dye concentration, and decreased with increase of particle mass.
4. The change of agitation speed did not cause significant difference of intraparticle diffusion parameter in experimental conditions.

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