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Removal of dyes from aqueous solution by activated carbon from sewage sludge of the municipal wastewater treatment plant

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ABSTRACT

Activated carbon from sewage sludge of the municipal wastewater treatment plant was obtained by chemical activation with $ZnCl_2$ as activator. Then, it was used as an adsorbent for the removal of Acid Scarlet GR and Methylene Blue from aqueous solution. The effects of contact time, temperature, and initial dye concentration were investigated. The experimental data were analyzed by the Langmuir and Freundlich adsorption models. Kinetic adsorption data were discussed using the pseudo-first-order model and pseudo-second-order model. Thermodynamic parameters, such as enthalpy (ΔH°), entropy (ΔS°), and Gibbs free energy (ΔG°), were calculated. The results showed that the equilibrium data fitted well with the Freundlich model for the adsorption of Methylene Blue on the activated carbon. However, adsorption data for Acid Scarlet GR were both fitted with the Langmuir isotherm and the Freundlich isotherm. The adsorption kinetic of Methylene Blue and Acid Scarlet GR on the activated carbon was both more accurately represented by pseudo-second-order model. The values of ΔH°, ΔS°, and ΔG° for adsorption of Methylene Blue were 53.01 kJ/mol, 190.12 J/mol and −2.78 kJ/mol, respectively. The absorption process of Methylene Blue was a spontaneous and endothermic reaction. The values of ΔH°, ΔS°, and ΔG° for adsorption of Acid Scarlet GR were 32.78 kJ/mol, 104.00 J/mol and 2.26 kJ/mol, respectively. The adsorption process of Acid Scarlet GR was less effective.

Keywords: Adsorption; Dyes; Activated carbon; Sewage sludge

1. Introduction

Dyes have been extensively used in many fields, such as textile, leather, tanning, paper production, food technology, and hair coloring [1]. Different kinds of dyes are being used every year [2]. The presence of these many dyes in wastewater is not only aesthetically displeasing, but also it impedes light penetration in the treatment plant; thus, upsetting the biological treatment processes within the treatment plant. And also the presence of these dyes in wastewater increases the biological oxygen demand and causes the lack of dissolved oxygen to sustain aquatic life. In addition, many dyes are toxic to some microorganisms, and may cause direct destruction or inhibition of their catalytic capabilities [3,4]. Therefore, it is necessary to reduce dye concentration in the wastewater before the biological treatment processes.

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Many treatment methods have been adopted to remove dyes from wastewater, which can be divided into physical, chemical, and biological methods [5]. Although chemical and biological methods are effective for removing dyes, they require special equipment and are usually quite energy-intensive. In addition, large amounts of by-products are often generated [2].

Generally, physical methods, which include adsorption, ion exchange, and membrane filtration, are effective for removing reactive dyes without producing unwanted by-products [6]. Among others, adsorption system has attracted extensive attention due to the high quality effluents with low concentration of dyes [7]. Using suitable adsorbent to remove colored and colorless organic pollutants from industrial wastewater is considered to be an important application of adsorption processes using suitable adsorbent. At present, there is growing interest in using low cost and commercially available materials for the adsorption of dyes. A wide variety of materials, such as fly ash, peat, wood, palm-fruit bunch particles, activated carbon from fertilizer waste, and activated slag, are being used [8–11].

Activated carbon is the most effective and commonly used as adsorbent for the treatment of dye wastewater. However, commercially available activated carbon is still an expensive material, and the high cost hampers the application in most cases [12]. This subsequently leads to a growing interest in the research on the production of activated carbons from cheap precursors [13]. In recent years, a number of studies have shown that some industrial and agricultural by-products have the potential to be the precursors to prepare activated carbons for the removal of dyes from wastewater, which include waste paper [14], waste ties [15], rice hull [16], olive cake [17], date stone [18], and apricot stone [19].

The sewage sludge from municipal wastewater treatment plant is an inevitable by-product during wastewater purification. Most disposal methods, such as sea dumping, landfill, individual combustion, and farmland utilization, have some limitations and lots of sewage sludge is not disposed properly causing serious waste and pollution. Thus, it is significant to search for innovative approaches using sewage sludge [20]. In recent years, some researchers have focused on converting sewage sludge into activated carbon based on its high content of organic components [21]. This would not only solve the disposal problem of sewage sludge but also turn solid waste into useful material in producing adsorbent for wastewater treatment. Several studies have demonstrated the feasibility of this approach including chemical and physical activation [22,23]. There are few reports about the adsorption process [24]. The detailed studies of the adsorption process parameters, the kinetics and thermodynamics of dyes adsorption would be necessary in order to improve the adsorption capacity.

Therefore, in this work, activated carbon from sewage sludge of the municipal wastewater treatment plant was obtained by chemical activation. Then, it was used for removal of Acid Scarlet GR and Methylene Blue from aqueous solution. The effects of temperature and initial dye concentration on dye adsorption by the activated carbon were investigated. The adsorption isotherms, kinetics, and thermodynamics of the model compound over the activated carbon were also determined and discussed in detail.

2. Experimental

2.1. Materials

The sewage sludge of the municipal wastewater treatment plant was obtained from the municipal wastewater treatment plant of Shaoxing City in Zhejiang Province of P.R. China. It contained around 33% inorganic matter mainly in the form of metal oxidation and salt, 63% of organic matter mainly in the form of death biosolid, and about 4% water content. The content of Cu, Cr, Pb, As, and Cd in the sewage sludge of the municipal wastewater treatment plant was 221, 165, 57, 3.6, and 2.58 mg/kg, respectively. The raw sewage sludge was dried at 105℃ for 8 h, to achieve constant weight, then comminuted and sieved into a uniform size of 80 mesh. Fifty grams of dehydrated sewage sludge was soaked stilly with 100 mL 25% $ZnCl_2$ solution in 250 mL-Erlenmeyer flasks for 24 h at room temperature. Then, it was dried again at 105℃ for 8 h to constant weight and was carbonized at 500℃ in a muffle furnace for 50 min. The product of 80 mesh activated carbon was thus obtained, which was designated as SSAC and then stored for later adsorption experiments.

The two dyes (Methylene Blue and Acid Scarlet GR) with commercial purity were used without further purification. Their chemical structures are shown in Fig. 1.

2.2. Adsorption experiments

Adsorption experiments were conducted in a set of 250 mL-Erlenmeyer flasks containing 0.10 g of SSAC and 100 mL of dye solutions with various initial concentrations (30, 40, 50, and 60 mg/L). The flasks were placed stilly in a shaker at 293, 303, and 313 K

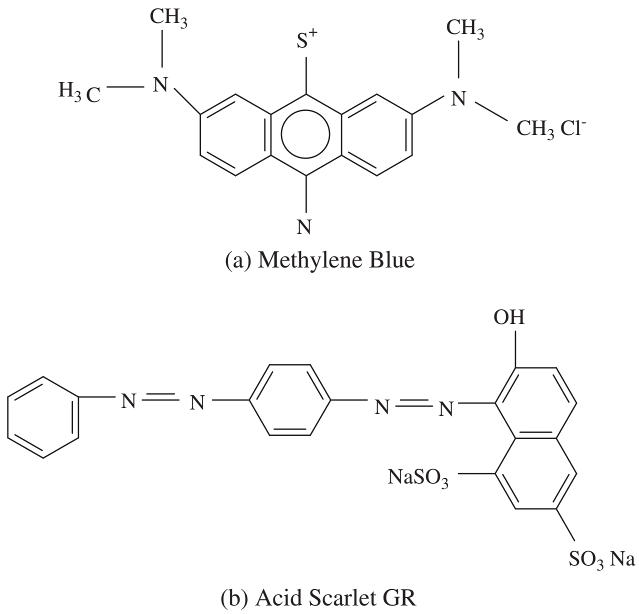

Fig. 1. Chemical structures of Methylene Blue and Acid Scarlet GR.

under pH 7.0 until equilibrium was reached. The samples were then filtered and the residual concentration of the dye was analyzed using a UV-1600 spectrophotometer at a wavelength corresponding to the maximum absorbance for each dye.

2.3. Analytical methods

The textural characteristics of SSAC including surface area, pore volume, and pore size distribution were determined using standard N_2-adsorption techniques [25]. The surface physical morphology of SSAC was observed by a scanning electron microscope.

The value of pH was measured with a pH probe according to APHA Standard Method. The concentration of dyes was measured with a UV-1600 spectrophotometer at a wavelength corresponding to the maximum absorbance for each dye: 665 nm for Methylene Blue and 510 nm for Acid Scarlet GR, respectively. The Cu, Cr, Pb, As, and Cd content were measured by atomic absorption spectrophotometry.

The amount of adsorbed dye q_t (mg/g) at different time was calculated as follows:

$$q_t = \frac{(C_0 - C_t) \times V}{m} \qquad (1)$$

where C_0 and C_t (mg/L) are the initial and equilibrium liquid-phase concentrations of dye, respectively. V (L) is the solution volume and m (g) is the mass of adsorbent used.

2.4. Statistical analyses of data

All experiments were repeated in duplicate and the data of results were the mean and the standard deviation (SD). The value of the SD was calculated by Excel Software. All the data were analyzed by the Langmuir and Freundlich adsorption models to test for the effects of the contact time, temperature, and initial dye concentration. The kinetic adsorption data were discussed using the pseudo-first-order model and pseudo-second-order model. All error estimates given in the text and error bars in figures are SD of means (mean ± SD). All statistical significance was noted at $\alpha = 0.05$ unless otherwise noted.

3. Results and discussion

3.1. Characteristics of SSAC

The textural characteristics of SSAC are obtained from the standard N_2-adsorption techniques. The BET surface area is 302.45 m^2/g, the total pore volume is 0.31 cm^3/g, and the nominal pore size is 0.63 nm. It shows that SSAC has a large specific surface area typical for commercial activated carbons [26]. In addition, the content of Cu, Cr, Pb, As, and Cd in SSAC was 462, 512, 130, 8.2, and 6.13 mg/kg, respectively. The chemical activation of the sewage sludge of the municipal wastewater plant before the pyrolysis resulted in an increase of the adsorption capacity [27].

Fig. 2 shows the TEM image of SSAC. Through this image irregular and porous structures can be

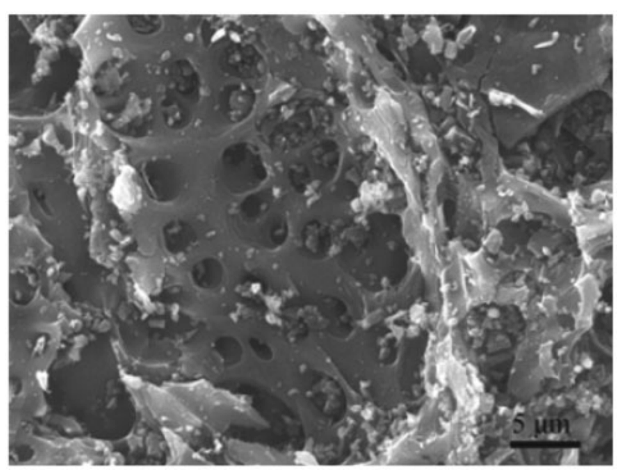

Fig. 2. SEM image of SSAC.

observed, indicating that SSAC presents an adequate morphology for dyes adsorption.

3.2. Determination of contact time

The contact time needed to reach adsorption equilibrium should be determined in studying the adsorption equilibrium and adsorption kinetics of SSAC. Fig. 3 shows the effect of contact time on the adsorption of dyes onto SSAC.

The adsorption rates of Methylene Blue and Acid Scarlet GR both increased dramatically in the initial period and reached equilibrium gradually at 360 and 420 min, respectively. The fast adsorption at the initial stage may be due to the higher driving force making fast transfer to dye ions to the surface of SSAC particles and the availability of the uncovered surface area and the remaining active sites on the adsorbent [28]. Thus, under the test conditions, the contact time of 360 and 420 min can be considered as the optimum contact time for Methylene Blue and Acid Scarlet GR onto SSAC, respectively.

3.3. Effect of temperature

In order to observe the effect of temperature on the adsorption capacity of dyes by SSAC, the tests were carried out at three different temperatures (293, 303, and 313 K). Fig. 4 shows the adsorption capacity of dyes at different temperatures. It was found that the adsorption equilibrium of dyes increased with the temperature rise, indicating the endothermic nature of the adsorption reaction. The increase in adsorption capacity was attributed to the enlargement of pore size and activation of the sorbent surface with temperature. Further rise in temperature increased the mobility of the large dye ions and reduced the swelling effect, and thus enabling the large dye molecule to penetrate further [29].

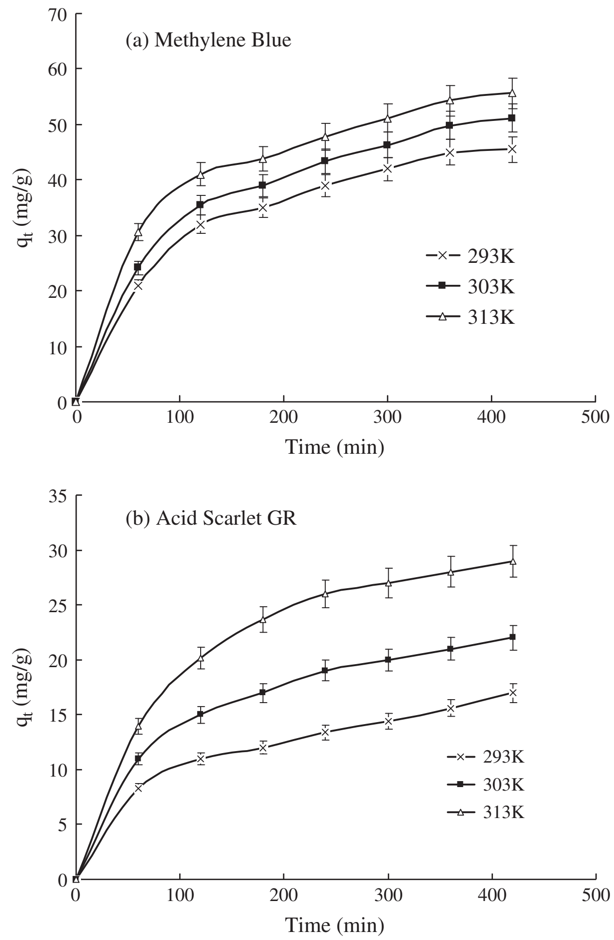

Fig. 4. Effect of temperature on adsorption of Methylene Blue and Acid Scarlet GR onto SSAC. Experimental conditions: 0.10 g of SSAC, the initial dye concentration of 60 mg/L, contact time of 420 min, 80 meshes of particle size, and pH 7.0.

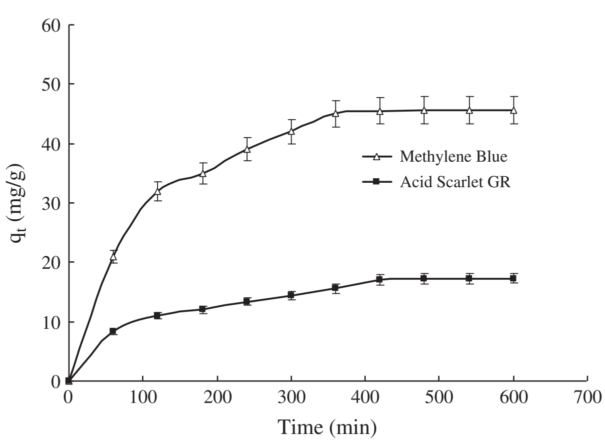

Fig. 3. Effect of contact time on adsorption of Methylene Blue and Acid Scarlet GR onto SSAC. Experimental conditions: 0.10 g of SSAC, 60 mg/L of initial dye concentration, 80 meshes of particle size, 293 K, and pH 7.0.

3.3. Effect of initial dye concentration

The effect of initial dye concentration on adsorption of the Methylene Blue and Acid Scarlet GR was investigated with various initial concentrations (30, 40, 50, and 60 mg/L). The results of tests are shown in Fig. 5.

As observed in Fig. 5, for both dyes (Methylene Blue and Acid Scarlet GR), the increase in initial dye concentration caused the uptake amount of dye onto

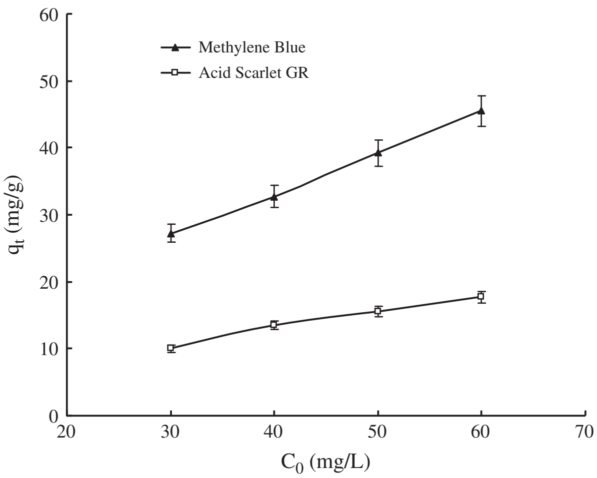

Fig. 5. Effect of initial dye concentration on adsorption of Methylene Blue and Acid Scarlet GR onto SSAC. Experimental conditions: 0.10 g of SSAC, contact time of 420 min, 80 meshes of particle size, 293 K, and pH 7.0.

the resulting sample to increase remarkably. This is because with the increase in dye concentration, the driving force for mass transfer also increased. At low concentrations, there would be unoccupied active sites on the adsorbent surface.

3.4. Adsorption isotherms

The Langmuir and Freundlich adsorption models are commonly adopted to investigate the adsorption behavior of materials and the correlation among adsorption parameters. Accordingly, equilibrium data were simulated by the Langmuir [30] and Freundlich models [31].

Generally, based on the assumption that each molecule possesses constant enthalpies and sorption activation energy, the Langmuir isotherm model suggests that adsorption occurs on homogeneous sites within an adsorbent. This model is expressed by following equation:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L} \times \frac{1}{C_e} \tag{2}$$

where C_e is the equilibrium concentration (mg/L), q_e is the amount of adsorbate (mg/g), q_m is q_e for complete monolayer adsorption capacity (mg/g), and K_L is the equilibrium adsorption constant (L/mg). The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless separation factor, R_L, which indicates the possibility of the adsorption process being irreversible ($R_L = 0$), favorable ($0 < R_L < 1$), linear ($R_L = 1$), or unfavorable ($R_L > 1$) [32,33]:

$$R_L = \frac{1}{1 + K_L C_0} \tag{3}$$

where K_L is the Langmuir constant and C_0 is the initial adsorbate concentration (mg/L).

The Freundlich isotherm can be expressed as follows:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \tag{4}$$

where K_f (mg/g) is the Freundlich adsorption constant and $\frac{1}{n}$ is a measure of the adsorption intensity that determines whether the adsorption intensity and type of isotherm are favorable $\left(0.1 < \frac{1}{n} < 1\right)$ or unfavorable $\left(\frac{1}{n} > 1\right)$.

The corresponding values of Freundlich and Langmuir isotherms for Methylene Blue and Acid Scarlet GR adsorption on SSAC were listed in Table 1.

Table 1 showed that the values of Langmuir and Freundlich constant and the correlation coefficients R^2 were obtained from the linear regression. It was observed that the Freundlich isotherm better described the adsorption of Methylene Blue with the higher correlation coefficient R^2, suggesting that some heterogeneity on the surfaces or pores of SSAC played an important role in Methylene Blue adsorption and that different sites with several adsorption energies were involved. However, adsorption data for Acid Scarlet GR were both fitted with the Langmuir isotherm and the Freundlich isotherm.

Table 1
Langmuir and Freundlich parameters for the adsorption of Methylene Blue and Acid Scarlet GR onto SSAC. Experimental conditions: 0.10 g of SSAC, contact time of 420 min, 80 meshes of particle size, 293 K, and pH 7.0

	Langmuir model			Freundlich model		
Dye	q_m (mg/g)	K_L (L/mg)	R^2	K_f (mg/g)	n	R^2
Methylene blue	46.95	0.47	0.87	19.31	3.32	0.94
Acid Scarlet GR	59.52	0.01	0.98	1.02	1.35	0.97

3.5. Adsorption kinetic

Various kinetic models have been reported in the literature to describe the adsorption process [34]. In this study, the pseudo-first-order model and pseudo-second-order models were used to estimate the rate constants, initial adsorption rates, and adsorption capacities of the SSAC.

The linear pseudo-first-order model of Lagergren is given as follows [35]:

$$\ln(q_e - q_t) = \ln q_e - k_1 \times t \tag{5}$$

where q_e and q_t are the amounts of dye absorbed onto the adsorbent (mg/g) at equilibrium and at t, respectively. k_1 is the rate constant of first-order adsorption (min^{-1}).

The pseudo-second-order kinetic model developed by Ho and McKay [36] is based on the experimental information of solid-phase sorption. The linear pseudo-second-order model can be expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{6}$$

where k_2 is the rate constant of second-order adsorption ($\text{g mg}^{-1}\,\text{min}^{-1}$).

The kinetic models of pseudo-first-order and pseudo-second-order for the adsorption of Methylene Blue and Acid Scarlet GR on SSAC at 293 K were shown in Fig. 6. The rate constant (k_1 and k_2), correlation coefficient R^2, and equilibrium adsorption density of q_e could be derived from this line. As shown in Fig. 6, the plot of t/q_t vs. t provides a very straight line. Noticeably, R^2 of the pseudo-second-order model for Methylene Blue and Acid Scarlet GR were 0.997 and 0.981, respectively, which were both much higher than R^2 of the pseudo-first-order model (R^2 of the pseudo-first-order model for Methylene Blue and Acid Scarlet GR were 0.893 and 0.873, respectively). This result confirmed that the adsorption of Methylene Blue and Acid Scarlet GR onto the SSAC both better fitted to pseudo-second-order kinetic model compared to pseudo-first-order kinetic model.

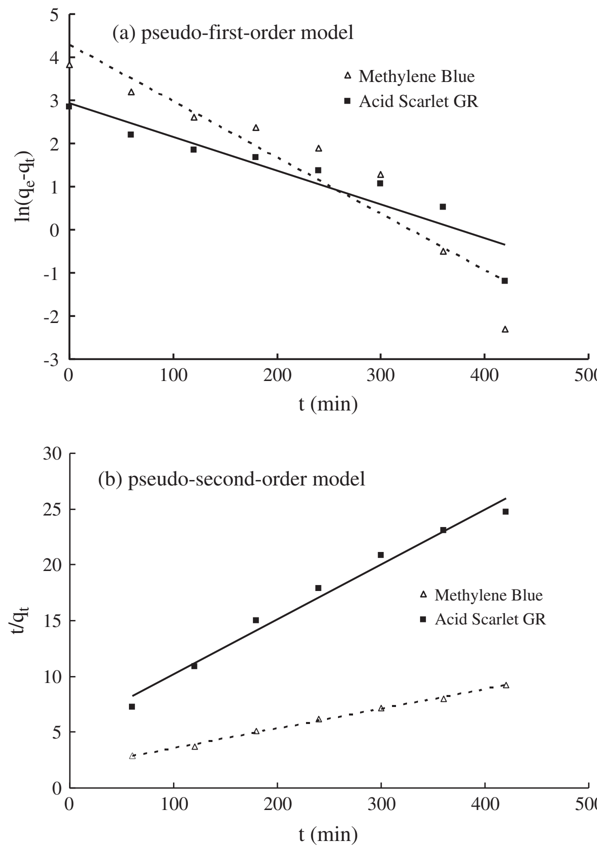

Fig. 6. (a) Pseudo-first-order model for adsorption of Methylene Blue and Acid Scarlet GR onto SSAC at 293 K. (b) Pseudo-second-order model for adsorption of Methylene Blue and Acid Scarlet GR onto SSAC at 293 K. Experimental conditions: 0.10 g of SSAC, the initial dye concentration of 60 mg/L, contact time of 420 min, 80 meshes of particle size, 293 K, and pH 7.0.

3.6. Thermodynamic modeling

Three basic adsorption thermodynamic parameters, enthalpy (ΔH°), entropy (ΔS°), and Gibbs free energy (ΔG°) were calculated using the following equations [37]:

$$\Delta G^\circ = -RT \ln K_a \tag{7}$$

$$\ln K_a = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \tag{8}$$

$$K_a = \frac{q_e}{C_e} \tag{9}$$

where T is the solution temperature (K), K_a is the adsorption equilibrium constant, R is the gas constant ($8.314\,\text{J mol}^{-1}\,\text{K}^{-1}$), q_e is the amount of adsorbate adsorbed per unit mass of adsorbate at equilibrium (mg/g), and C_e is the equilibrium concentration of the adsorbate (mg/L).

Thermodynamic parameters (ΔH°, ΔS°, and ΔG°) for dye adsorption were evaluated using Eqs. (7)–(9). The values of ΔH° and ΔS° were determined from the slope and intercept of the plot of ln K_a vs. $1/T$ (Fig. 7). The positive values of ΔH° (the values of enthalpy for Methylene Blue and Acid Scarlet GR are 53.01 and 32.78 kJ/mol, respectively) indicate that the adsorption

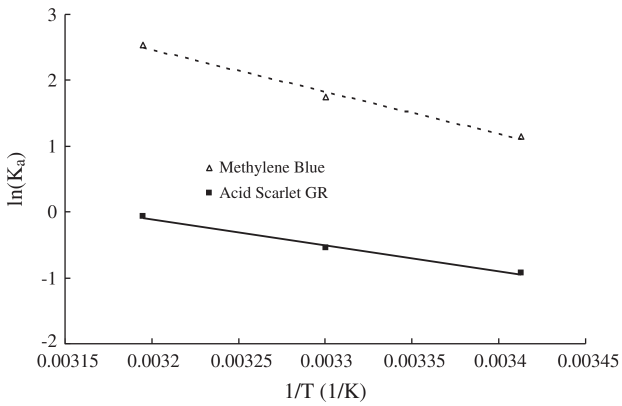

Fig. 7. Enthalpy and entropy determination for adsorption of Methylene Blue and Acid Scarlet GR onto SSAC. Experimental conditions: 0.10 g of SSAC, the initial dye concentration of 60 mg/L, contact time of 420 min, 80 meshes of particle size, and pH 7.0.

of Methylene Blue and Acid Scarlet GR onto SSAC was an endothermic reaction. Meanwhile, the positive value of ΔS° (the values of entropy for Methylene Blue and Acid Scarlet GR are 190.12 and 104.00 J/mol, respectively) reflect the affinity of SSAC for Methylene Blue and Acid Scarlet GR. The calculated value of ΔG° for Methylene Blue (−2.78 kJ/mol) at 293 K indicates the adsorption of process was a spontaneous and endothermic reaction. The calculated value of ΔG° for Acid Scarlet GR (2.26 kJ/mol) at 293 K indicated the adsorption process was unfavorable.

4. Conclusions

In summary, the adsorption isotherm model and kinetic model analyses by the activated carbon revealed that the adsorption of Methylene Blue fitted well with the Freundlich model. However, adsorption data for Acid Scarlet GR were both fitted with the Langmuir isotherm and the Freundlich isotherm. The adsorption kinetic of Methylene Blue and Acid Scarlet GR was both more accurately represented by pseudo-second-order model. The thermodynamic parameters (ΔH°, ΔS°, and ΔG°) showed that the absorption process of Methylene Blue was a spontaneous and endothermic reaction and the adsorption process of Acid Scarlet GR was unfavorable.

Acknowledgements

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