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Article

Comparative Adsorption of Acid Blue 25 and Reactive Orange 16 onto Coconut Shell Activated Carbon: Kinetics, Isotherm and Thermodynamic Studies

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Abstract: In this study, coconut shell activated carbon was used as the adsorbent. Through kinetic, isothermal model and adsorption thermodynamics methods, the adsorption process of acid blue 25 and active orange 16 by coconut shell activated carbon was deeply investigated. The results showed that this adsorbent had good adsorption performance for acid blue 25 and active orange 16. The internal pores of the adsorbent were abundant, and the equilibrium adsorption amount of the two dyes increased with the increase of the initial concentration of the dyes and the adsorption temperature. The pseudo-second-order rate equation could best describe the adsorption of the adsorbent for the two dyes. The activation energy of the adsorption of the adsorbent for the two dyes was small, mainly being physical adsorption. Under the conditions of T = 30℃ and pH = 7, the isothermal adsorption law of acid blue 25 and active orange 16 on coconut shell activated carbon conformed to the Langmuir model equation. The maximum saturated adsorption amounts were 258.48 and 248.05 mg/g, respectively, and the adsorption free energy was -8.875 and -4.436 $\mathrm{kJ/mol}$, respectively. The enthalpy changes ΔH of acid blue 25 and active orange 16 were 8.688 and 7.894 KJ/mol, respectively, indicating that the adsorption was an endothermic process and irreversible. The isothermal adsorption study, adsorption kinetics and thermodynamics study all showed that under the same conditions, the adsorption effect of the two dyes on coconut shell activated carbon was: acid blue 25 > active orange 16.

Keywords: Coconut shell activated carbon; Adsorption; Dyes; Adsorption kinetics; Isothermal line; Thermodynamics

1. Introduction

In today's society, many industries such as dye manufacturing plants, textile processing, food coloring, cosmetics, pulp mills, etc. all involve the use of dyes, which leads to a large amount of colored wastewater discharge. For example, acidic blue 25 and active orange 16. These colored wastewater, if not completely treated and discharged into the environment, will cause certain harm to the ecosystem and water sources [1-4]. Therefore, removing dyes from the environment is an important challenge in wastewater treatment and also an urgent problem to be solved.

Currently, the main technologies for removing dyes from wastewater include degradation methods P-, chemical precipitation methods, solvent extraction methods, ion exchange methods, and adsorption methods [5-6]. Among them, adsorption method is generally considered as a simple and effective method for removing dye wastewater, with advantages such as simple operation and rapidity [7-9]. Activated carbon, as the best adsorbent for wastewater treatment, has a large demand, which also leads to a large demand for coal and wood. With the increasingly severe depletion of earth resources, the development and utilization of activated carbon prepared from coal and wood are becoming less and less [10-13]. Out of the consideration for the sustainable development of the environment and protection, more and more researchers use agricultural waste as the raw material for preparing activated carbon. This not only alleviates the pressure on coal resources but also reduces the solution work for environmental waste. Coconut shell is one of the most important raw materials [14-17].

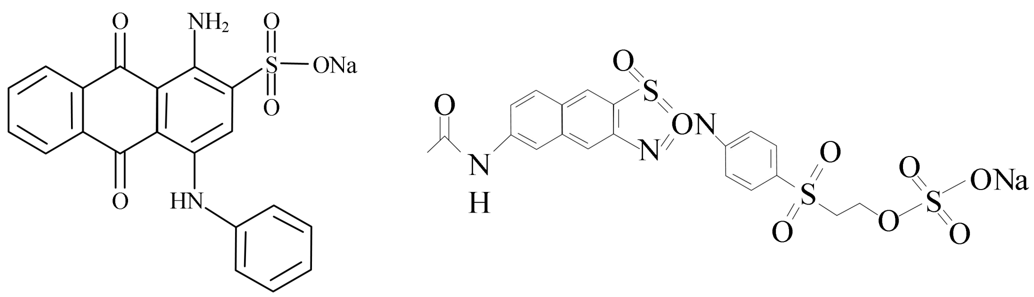
Coconut shell activated carbon is made from high-quality coconut shells through a series of processing techniques. Its appearance is black and it comes in two forms: granular and columnar. It has the advantages of well-developed pores, good adsorption performance, high strength, easy regeneration, and economic durability [18-20]. The product is mainly used for the purification, decolorization, dechlorination, and deodorization of drinking water, purified water, wine production, beverages, industrial wastewater, etc.; it can also be used in the refining oil industry for desulfurization of alcohols, etc. [21-22]. In this context, studying the comparative adsorption of coconut shell activated carbon for acidic blue 25 and active orange 16, analyzing the differences in kinetics, isotherms, and thermodynamics, has broad development prospects.

This paper uses coconut shell activated carbon as the adsorbent, acidic blue 25 and active orange 16 as the removal objects, and for the first time explores the removal ability of coconut shell activated carbon for acidic blue 25 and active orange 16 dyes from a kinetic, isotherm, and thermodynamic perspective. Using kinetic, isotherm models (Langmuir and Freundlich models) and adsorption thermodynamics to fit and analyze the experimental results, the adsorption activation energy, adsorption free energy, entropy change, and enthalpy change, etc. were calculated. The adsorption performance of coconut shell activated carbon for acidic blue 25 and active orange 16 was systematically compared, aiming to provide a theoretical basis for the application of coconut shell activated carbon in dye removal.

2. Method

2.1. Chemical Instruments and Materials

The structural formulas and main characteristics of the molecules of Acid Blue 25 ($C_{20}H_{13}N_2NaO_5S$) and Active Orange 16 ($C_{20}H_{17}N_3Na_2O_{11}S_3$) are shown in Figure 1. Both Acid Blue 25 and Active Orange 16 are produced by Shanghai Meilin Biochemical Technology Co., Ltd., with a purity of 45%. Hydrochloric acid, sodium hydroxide, and sodium chloride are all domestic analytical pure reagents. Powdered coconut shell activated carbon is produced by Shanghai Tangxin Activated Carbon Co., Ltd. The activated carbon is washed with deionized water, filtered to remove the dust of the activated carbon, then dried at 100°C for 1 day, cooled, ground through a 300-mesh sieve, and stored in a desiccator. According to relevant literature, the test results of the structural characteristics of this type of activated carbon are as follows [23]: specific surface area 1295 m^2/g , total pore volume 0.719 cm^3/g , large and medium pore volume 0.198 cm^3/g , micropore volume 0.522 cm^3/g , and large and medium pore volume ratio 27.3%.



(a) Acid Blue 25 (b) Active Orange 16

Figure 1. The structural formulas of Acid Blue 25 and Active Orange 16

Nova-1200 gas adsorption instrument, produced by Quantachrome Company of the United States; JA12002 electronic balance, Shanghai Precision Instrument Co., Ltd.; JB-3 constant temperature and speed adjustable magnetic stirrer, Shanghai Leiming Xingjing Instrument Co., Ltd.; 320-S pH meter, Mettler-Toledo Instruments (Shanghai) Co., Ltd.; UV-VIS8500 spectrophotometer, Shanghai Tianmei Scientific Instrument Co., Ltd.; T6 New Century ultraviolet-visible spectrophotometer, Beijing Puisha

General Instrument Co., Ltd.; XS224 analytical balance, Shanghai Youke Instrument and Meter Co., Ltd.; Reaction vessel, Gongyi Yu Hua Instrument Co., Ltd.; SHA-B water bath shaker, Shanghai Lichen Technology Co., Ltd.; WQF-510A Fourier transform infrared spectrometer, Beijing Oubel Scientific Instrument Co., Ltd.; Apreo 2S HiVac scanning electron microscope (Thermo Fisher Scientific).

2.2. Experimental Method

(1) Adsorption equilibrium experiment

Take 0.5g of coconut shell activated carbon and place it in 100 mL of different initial concentrations of acidic Blue 25 and Active Orange 16 dye solutions. Under a temperature of $30\,^{\circ}\mathrm{C}$, let it oscillate for adsorption for 1 Day. Use the T6 New Century ultraviolet-visible spectrophotometer to measure the absorbance of the dye solution after adsorption. Based on the change in dye concentration before and after adsorption, calculate the equilibrium adsorption amount.

(2) Adsorption kinetics study

During the adsorption process of acidic Blue 25 and Active Orange 16 dyes by coconut shell activated carbon, sample the solution at regular intervals to analyze the concentration of the dyes. The initial concentration is the equilibrium adsorption concentration. The other experimental conditions and the adsorption equilibrium experiment are the same.

To analyze the adsorption rate of coconut shell activated carbon for acidic Blue 25 and Active Orange 16 dyes, they are fitted with pseudo-first-order rate equation and pseudo-second-order rate equation respectively:

$$\lg(q_{\mathrm{e}} - q_t) = \lg q_{\mathrm{e}} - \frac{k_1}{2.303} t \tag{1}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_{\mathrm{e}}^2} + \frac{1}{q_{\mathrm{e}}} t \tag{2}$$

In the equation, q_{e} and q_t represent the equilibrium adsorption amount and the adsorption amount at time $t(\mathrm{min})$, respectively, in $\mathrm{mg/g}$;

k_1 - the pseudo-first-order rate constant, min^{-1};

k_2 - the pseudo-second-order rate constant, $\mathrm{g/(mg \cdot min)}$.

(3) Adsorption isotherm study

The Langmuir and Freundlich adsorption isotherm models were adopted. The Langmuir adsorption isotherm model represents a single-layer surface adsorption model, while the Freundlich adsorption isotherm model represents a non-uniform surface adsorption model. The equations of the Langmuir (L-type) and Freundlich (F-type) adsorption isotherm models are as follows:

$$\frac{C_e}{Q_e} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m} \tag{3}$$

$$Q = K_F C^{\frac{1}{n}} \tag{4}$$

In the equation, Q_m —— saturated adsorption capacity of activated carbon, $\mathrm{mg/g}$;

K_L —— Langmuir constant, $\mathrm{L/mg}$;

K_F —— Freundlich empirical constant, L/g;

n —— Freundlich empirical constant.

Take 0.5g of coconut shell activated carbon and place it in 100 mL of different initial concentrations of acidic Blue 25 and active Orange 16 dye solutions. Perform an adsorption experiment in a constant temperature oscillator at 100 r/min and 30°C under oscillation conditions until equilibrium is reached. After the solution is left to stand for 1.5 hours, take the upper clear liquid and measure its absorbance to calculate the adsorption amount and plot the adsorption isotherm.

(4) Adsorption thermodynamics study

This paper studies the thermodynamic parameters of free energy change ΔG, enthalpy change ΔH, and entropy change ΔS to understand the state characteristics of the adsorption reaction. The thermodynamic parameters can be calculated by the following formula:

$$K_C = \frac{C_A}{C_S} \tag{5}$$

$$\Delta G^0 = -RT \ln K_C \tag{6}$$

$$\ln K_C = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \tag{7}$$

In the equation, C_A represents the number of ions adsorbed by one unit of adsorbent at equilibrium, measured in $\mathrm{mg \cdot L^{-1}}$;

C_S represents the equilibrium concentration of ions in the solution, also measured in $\mathrm{mg \cdot L^{-1}}$;

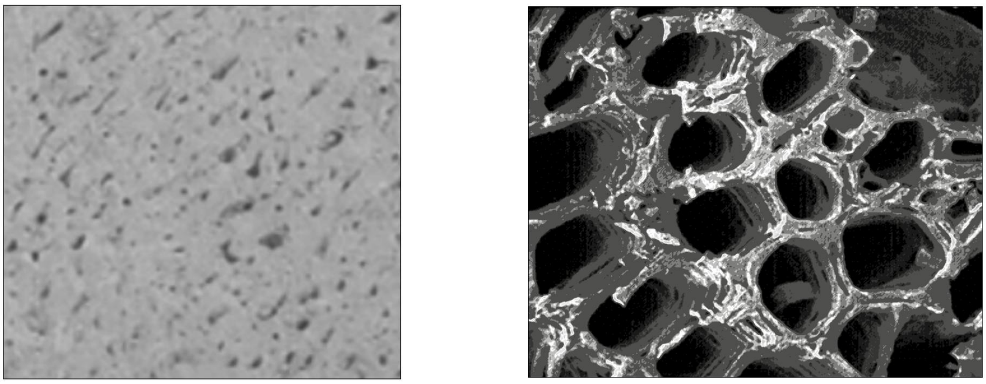
R is the gas constant, equal to 8.314 J/mol·K;

T represents the temperature, measured in K.

3. Results and Discussion

3.1. Properties of Adsorbents

After testing, it was found that coconut shell activated carbon has excellent adsorption performance for acid blue 25 and active orange 16, indicating that it has a well-developed microporous and mesoporous structure. The surface characteristics of coconut shell activated carbon under 100 and 10 μm magnification electron scanning microscopy (SEM) are shown in Figure 2. As seen in Figure 2(a), the surface of coconut shell activated carbon contains many pore structures, with large pore diameters and irregular shapes. In some parts of the coconut shell with defects, more small pores can be observed at the defect cross-section, and the pore structure is more abundant than the surface. This indicates that during the activation process, the defect positions are more likely to be activated, and at the junction of the defect channels and the activated carbon particles, the pore structure is particularly developed. Some skeleton structures formed during the activation process can be clearly observed.



(a) 10 μm (b) 100 μm

Figure 2. Surface characteristics of coconut shell activated carbon

As shown in Figure 2(b), the pore distribution of the activated carbon structure is relatively wide, with well-developed micropores, mesopores and macropores. Generally, the pore walls of the macropores have small pores, forming a structure where the larger pores enclose the smaller ones. There are even smaller pores on the pore walls of the macropores, and the pore diameters of both the macropores and the small pores are relatively narrow. In some parts of the activated carbon material, there is a well-developed pore structure with uniform pore diameters. The pore walls of the activated carbon show a basic net-like arrangement, with a large number of pores in both the horizontal and vertical directions, with a diameter of approximately 5-20 μm.

3.2. Adsorption Kinetic Analysis

Due to the slow adsorption process of acid blue 25 and active orange 16 on coconut shell activated carbon, and the long time required to reach equilibrium, when the system's adsorption capacity does not further increase, it is considered that the system has reached the adsorption equilibrium state. This approach has a certain degree of subjectivity, but it is often used in research and analysis. The author appropriately extended the sampling interval and adsorption time to reduce experimental errors.

When the initial mass concentrations of the two dyes, C_0, were 20, 50, and 100 mg/L, and the temperature T was 30℃, the curves of the adsorption amount q_t of the two dyes on coconut shell activated carbon with time t are shown in Figure 3. It can be seen that when the temperature remains constant, the equilibrium adsorption amount q_e of acid blue 25 and active orange 16 by coconut shell activated carbon both increase with the increase of the initial concentration, and the larger the initial concentration, the longer the time required to reach equilibrium. When the initial concentrations of the two dyes are the same, the q_e of acid blue 25 is higher than that of active orange 16. This is because the molecular weight of active orange 16 (617.54) is larger than that of acid blue 25 (416.38), and diffusion is relatively difficult in the smaller pore channels.

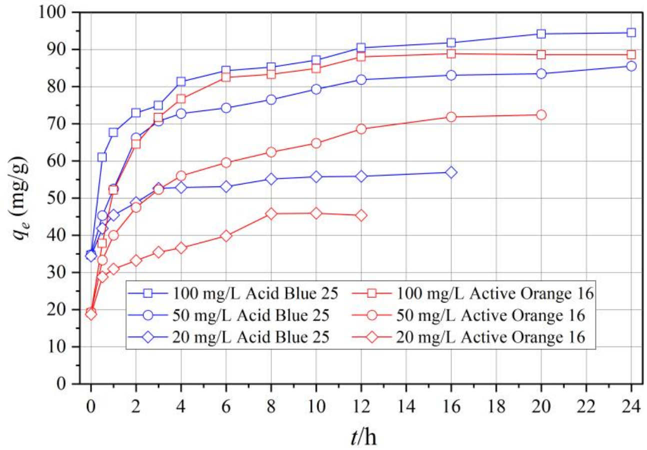


Figure 3. The q_t-t curves of the two dyes at different initial concentrations

When the initial dye concentration C_0 is 100 mg/L and the temperature T is 30, 40, and 50℃, the curve of q_t changing with time t is shown in Figure 4. When the initial concentration remains unchanged, the equilibrium adsorption amount q_e of Reactive Orange 16 increases with the increase in temperature. From 30℃ to 40℃, the increase in q_e is relatively large, and from 40℃ to 50℃, the increase in q_e decreases. Although the equilibrium adsorption amount of Acid Blue 25 also increases with the increase in temperature, the increase is very small. Therefore, appropriately increasing the temperature is beneficial for the adsorption of Reactive Orange 16, but it has little effect on Acid Blue 25.

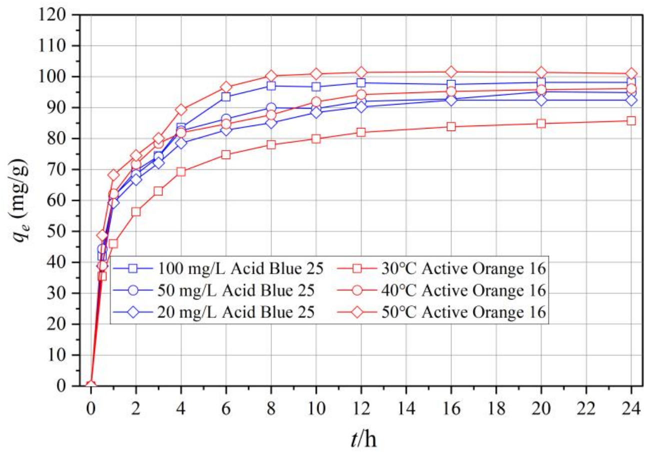


Figure 4. The q_t-t curves of the two dyes at different temperatures

It is worth noting that when the temperature exceeds 40℃, the equilibrium adsorption amount of active orange 16 by coconut shell activated carbon (96.2 mg/g) exceeds that of acid blue 25 (94.9 mg/g).

The reasons for this are as follows: On the one hand, as the temperature rises, the random movement of active orange 16 molecules increases, resulting in more frequent contact with coconut shell activated carbon, and more active orange 16 molecules enter the pores and are adsorbed. Additionally, the larger molecular mass of active orange 16 inevitably leads to an increase in the adsorption amount. On the other hand, coconut shell activated carbon may have surface adsorption of active orange 16. Active orange 16 is an anionic dye, and its molecular structure contains $-SO_3^-$. Due to the protonation effect of $-SO_3^-$ groups, the dye molecules exhibit strong negative charge, which leads to a strong electrostatic attraction between the negative charge of the dye and the residual activators or functional groups on the surface of coconut shell activated carbon. At the same time, the protonation of $-SO_3^-$ groups forms $-SO_3H$, which exchanges with Cl^- in coconut shell activated carbon, thereby increasing the adsorption amount.

To fully illustrate the adsorption kinetic characteristics of coconut shell activated carbon for active orange 16 and acid blue 25, and to find the most suitable kinetic model, the author selected three kinetic models: pseudo-first-order rate equation, pseudo-first-order rate equation, and particle internal diffusion equation to fit the above data. The suitability of the kinetic model was determined based on the linear correlation coefficient R^2. The fitting parameters are shown in Table 1. It can be seen that the pseudo-second-order equation has R^2 values greater than 0.99 for both dyes at different temperatures and initial concentrations, indicating that the pseudo-second-order equation has a high fitting degree. This result is consistent with previous discussions [24]. The particle internal diffusion equation has a significant difference in simulation for both dyes. The R^2 value range for acid blue 25 is 0.6060 to 0.8814, and for active orange 16, it is 0.9380 to 0.9851. This indicates that the particle internal diffusion process has little influence on the adsorption of acid blue 25, and particle internal diffusion is not the controlling step of acid blue 25 adsorption. However, the adsorption process of active orange 16 is controlled by particle internal diffusion, but the fitting curve of the particle internal diffusion equation for active orange 16 does not pass through the origin, indicating that particle internal diffusion is not the only rate-controlling step, and is also controlled by the particle external diffusion process, such as surface adsorption and liquid film diffusion.

Table 1. Kinetic Simulation Parameters of Two Dyes under Different Conditions

Dye	T /°C	C_0 / (mg/g)	**Pseudo-first-order equation**		**Pseudo-second-order equation**		**Diffusion equation within particles**	
			k_1/h^{-1}	R^2	k_2/ (g · mg^{-1} · h^{-1})	R^2	k_2/ (g · mg^{-1} · h^{-1})	R^2
Acid Blue 25	30	20	1.12490	0.9866	0.18815	0.9926	18.53463	0.6851
	30	50	0.29109	0.9736	0.03460	0.9919	14.33987	0.8488
	30	100	0.24066	0.9301	0.03113	0.9939	16.63187	0.8026
	100	30	0.27611	0.9138	0.03745	0.9964	13.25157	0.8441
	100	40	0.30379	0.9065	0.04033	0.9970	17.30462	0.6060
	100	50	1.44919	0.9212	0.09462	0.9969	10.31740	0.8814
Active Orange 16	30	20	1.40138	0.9342	0.03334	0.9989	15.51588	0.9380
	30	50	0.55049	0.9926	0.03081	0.9980	16.87267	0.9611
	30	100	0.74109	0.8441	0.03577	0.9982	22.09849	0.9467
	100	30	0.73370	0.9370	0.03693	0.9974	21.14410	0.9608
	100	40	0.79219	0.9526	0.03515	0.9972	22.18224	0.9851
	100	50	0.83282	0.9374	0.03260	0.9991	23.00834	0.9488

3.3. Analysis of Adsorption Isotherms

The classic nonlinear isothermal adsorption model equations include the Freundlich type (F type, Equation (3)) and the Langmuir type (F type, Equation (4)). Under the conditions of T = 30°C and pH = 7, the isothermal adsorption curves $Q_e \sim C_e$ of the two dyes on coconut shell activated carbon are shown in Figure 5. It can be seen that under this experimental condition, the isothermal adsorption of the two dyes by coconut shell activated carbon does not conform to a linear relationship.

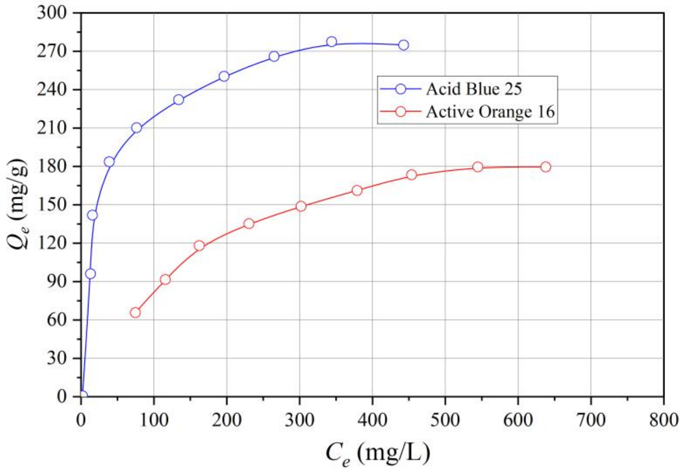
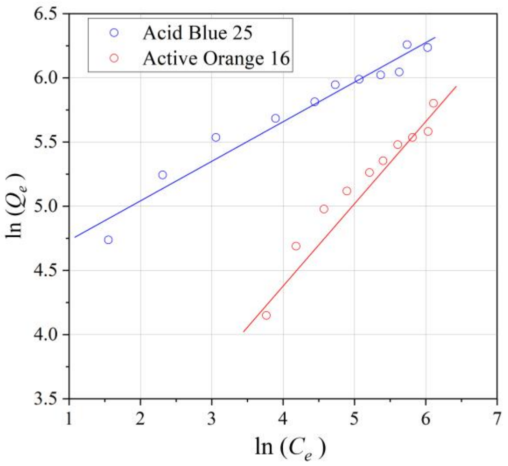
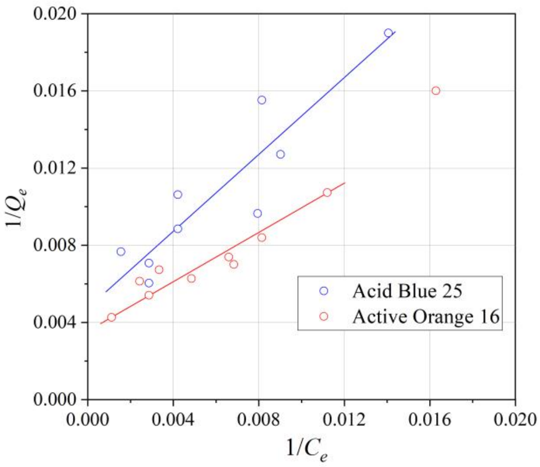


Figure 5. Isothermal Adsorption Q_e-C_e Curve

The isothermal adsorption results, transformed and fitted by the F-type equation, are shown in Figure 6(a), and by the L-type equation in Figure 6(b). The F-type and L-type isothermal adsorption equations and model parameters of Acid Blue 25 and Active Orange 16 obtained through the fitting are presented in Table 2. Judging from the correlation coefficients of the F-type and L-type isothermal adsorption equations of each dye, $R_L > R_F$, it can be concluded that the adsorption of the two dyes on activated carbon is more in line with the Langmuir isothermal adsorption model. According to the Gibbs equation, the adsorption free energy is $\Delta G = -RT\ln K$, where K represents the adsorption equilibrium coefficient. In this experiment, K can be taken as the adsorption coefficient k_L in the Langmuir isothermal equation, T is the absolute temperature (K), and R is the gas constant 8.314 (J/mol·K).



(a) F-type isothermal fitting curve



(a) F-type isothermal fitting curve

Figure 6. Isothermal adsorption of Acid blue 25 and active Orange 16 on activated carbon

Table 2. Isothermal adsorption Model Equation on activated carbon

Model		Acid Blue 25	Active Orange 16
Type F	Equation	$Q_e = 9.18 \times C_e^{0.448}$	$Q_e = 6.06 \times C_e^{0.523}$
	Correlation coefficient R_F	0.963	0.966
Type L	Equation	$Q_e = \dfrac{1.187 \times C_e}{(1+0.0054C_e)}$	$Q_e = \dfrac{0.7885 \times C_e}{(1+0.0026C_e)}$
	Correlation coefficient R_L	0.995	0.991
	Q_{Max} (mg/g)	258.48	248.05
	K_L (g/mg)	0.0054	0.0026

Substituting k_L into the above formula, the calculated results show that at 303 K, the adsorption free energies ΔG_{303K} of coconut shell activated carbon for acid blue 25 and active orange 16 are -8.875 and -4.436 kJ / mol respectively. The change in free energy ΔG_{303K} is less than 0, indicating that the adsorbate tends to spontaneously adsorb from the aqueous solution onto the surface of the activated carbon. Generally, the free energy change of physical adsorption is smaller than that of chemical adsorption. The former varies within the range of -20 to 0 kJ / mol, while the latter is within the range of -400 to -80 kJ / mol. Therefore, it can be concluded that under this experimental condition, the adsorption of acid blue 25 and active orange 16 on coconut shell activated carbon is all of physical spontaneous adsorption. From the maximum saturated adsorption amount Q_{Max} and ΔG_{303K} values, it is also easy to find that the isothermal adsorption effect of the two dyes on coconut shell activated carbon is: acid blue 25 > active orange 16.

3.4. Thermodynamic Parameter Analysis

In order to further understand the characteristics of coconut shell activated carbon in adsorbing two types of dyes, namely acid blue 25 and active orange 16, the thermodynamic parameters of the adsorption and activation states of these two dyes were analyzed. The results are shown in Table 3.

Table 3. Thermodynamic Parameter Analysis

T (°C)	Acid Blue 25			Active Orange 16		
	ΔG (KJ/mol)	ΔH (KJ/mol)	ΔS (J/K· mol^{-1})	ΔG (KJ/mol)	ΔH (KJ/mol)	ΔS (J/K· mol^{-1})
30	-8.483			-7.530		
35	-9.566			-7.859		
40	-10.287	8.688	63.721	-8.056	7.894	56.738
45	-10.896			-8.233		
50	-11.256			-8.357		

From the table, it can be concluded that the negative value of ΔG indicates that the adsorption of the two dyes, Acid Blue 25 and Reactive Orange 16, by coconut shell activated carbon is a spontaneous process, and high temperature is beneficial to the reaction. The ΔG value ranges from -20 to 0 kJ / mol, which again verifies that this process is physical adsorption, which is consistent with the conclusion drawn from the isothermal line analysis. ΔH is all positive values, and the ΔH value of Acid Blue 25 is greater than that of Reactive Orange 16, indicating that the adsorption is an endothermic process, and the overall adsorption effect on Acid Blue 25 is greater than that on Reactive Orange 16, which is the same as the conclusion derived from the adsorption isotherm line. The positive value of ΔS indicates that the reaction is irreversible and desorption is not easy to occur.

4. Conclusion

This paper uses dynamic models, isothermal line models and adsorption thermodynamics to study the adsorption process of acid blue 25 and active orange 16 by coconut shell activated carbon. The conclusions are summarized as follows:

(1) The pores of coconut shell activated carbon are well-developed and abundant, and it shows good adsorption effects on both acid blue 25 and active orange 16 dyes.

(2) The equilibrium adsorption amounts of acid blue 25 and active orange 16 by coconut shell activated carbon increase with the increase of the initial concentration of the dyes; with the increase of temperature, the equilibrium adsorption amount of acid blue 25 increases slightly, while the adsorption amount of active orange 16 increases significantly.

(3) The pseudo-second-order rate equation best describes the entire adsorption process of acid blue 25 and active orange 16 on coconut shell activated carbon, while the pseudo-first-order model is slightly worse and is only suitable for the initial stage of adsorption.

(4) The adsorption activation energies of acid blue 25 and active orange 16 by coconut shell activated carbon are both small, -8.875 and -4.436 kJ / mol respectively, mainly being physical adsorption.

(5) Based on the results of comprehensive kinetics, isothermal line and thermodynamics studies, it can be concluded that under the same conditions, the adsorption effect of coconut shell activated carbon on acid blue 25 is overall better than that of active orange 16.

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