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Study on adsorption behavior of different adsorbents in rotating packed bed

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ABSTRACT

In order to explore the adsorption mechanism of different adsorbents for different adsorbents in a rotating packed bed (RPB), the experiments of adsorbing different adsorbents (resorcinol, phenol, and nitrobenzene) by different adsorbents (AC, silica gel, and molecular sieves) are studied. The adsorption performance of different adsorbents on different adsorbents in a high gravity environment is compared. The experimental results show that the adsorption capacity of AC for resorcinol was 7.02 and 4.45 times that of silica gel and molecular sieve in the same conditions. The adsorption rate and adsorption effect of AC on nitrobenzene are better than that of phenol and resorcinol. The adsorption capacity of AC to nitrobenzene is increased by 6.71% and 1.78% compared with that of phenol and resorcinol, respectively. RPB can more effectively improve the adsorption rate and adsorption capacity than the fixed bed. The pore structure and surface-active components of the adsorbent have a certain effect on different adsorbents, and the adsorption equipment also affects the adsorption performance.

Keywords: Adsorbates; Adsorbent; Adsorption kinetics; Adsorption rate; Rotating packed bed

1. Introduction

Industrial organic wastewater is produced by industrial production. Untreated wastewater not only seriously pollutes the environment and destroys natural ecology, but also has a serious impact on human health. Conventional organic wastewater has a relatively large molecular weight and is highly toxic. Microorganisms present in water alone cannot effectively break down these pollutants. From an ecological and environmental perspective, the treatment of organic wastewater is imminent and has environmental and economic benefits. At present, common methods for treating organic wastewater include ultrasonic degradation [1,2], photo catalysis [3,4], biological contact oxidation [5], and adsorption [6–8]. Ultrasonic degradation has the advantage of no secondary pollution, but it consumes a lot of energy and the degradation rate is slow. The process of photocatalytic degradation is fast and low investment, but it is not easy to separate ultraviolet and catalyst suspension during processing. The biodegradation method has a good effect, but biological cells are easily inactivated and cause secondary pollution. The adsorption method has high technology maturity, does not use toxic additives, and has no secondary pollution. It is the current mainstream process. Adsorbents can be reused and treated well, and have been widely used in the purification of organic wastewater [9].

The adsorption capacity is related to the operating parameters such as adsorbent, adsorbate, and adsorption equipment. Adsorption equipment is the core of the entire adsorption process, so the selection of adsorption equipment is particularly important for adsorption performance. Common adsorption equipment includes fixed bed [10,11], moving bed [12,13], and fluidized bed [14,15]. Conventional adsorption equipment has the disadvantages of large

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volume, small rate, and long adsorption time. In recent years, the rotating packed bed (RPB) has attracted attention in the field of adsorption due to its good mass transfer effect and low investment and operating costs [16,17]. RPB is a new type of mass transfer equipment, which can simulate the high gravity environment to enhance the chemical process through the high-speed rotation of packed bed. The adsorbent is placed in a packed layer of a RPB, and the adsorbent is in a high-speed rotating state during the adsorption process. The enhanced adsorption process is where the highly dispersed adsorbent of the liquid distributor contacts the adsorbent that rotates at high speed. The adsorbent is dispersed into fine liquid filaments and achieves good external diffusion with the adsorbent throughout the process. The strong external diffusion promotes the effective entry of the adsorbent into the inner pores of the adsorbent, thereby achieving deep-hole adsorption and effectively improving the adsorption capacity. The high gravity technology has been widely used in chemical process such as distillation [18], absorption [19], desorption [20], nano powder preparation [21], adsorption [22], and extraction [23]. It is well-known that the adsorption process mainly includes three processes, an external diffusion phase of the particles, a pore diffusion phase, and an adsorption reaction phase. The large shear force generated by the RPB accelerates the rate of interface renewal on the surface of the adsorbent, promoting the flow contact of the liquid phase in the porous medium and pores, thereby increasing adsorption and desorption rates. Therefore, RPB has certain feasibility and applicability for the field of adsorption.

The high-speed rotating adsorbent is in full contact with the strongly dispersed adsorbent to complete the adsorption process in a high gravity environment. Common adsorbents are porous materials [24–27]. AC (activated carbon) is the most commonly used sorbent and cheap. The surface state of the adsorbent directly affects the adsorption effect throughout the adsorption process. For example, the activation state, surface uniformity, and surface functional group active components on the surface are all factors that affect the adsorption rate. The surface chemistry of the adsorbent has an important influence on the adsorption capacity. The acidic functional groups on the surface of the adsorbent enhance polarity and hydrophilicity, which is beneficial to increase the adsorption of polar substances. Basic functional groups on the surface of the adsorbent can enhance non-polarity and hydrophobicity and help adsorb non-polar adsorbents. For example, AC is a non-polar adsorbent with hydrophobic and organic affinity, which is beneficial to the adsorption of non-polar organic substances. Therefore, reasonable selection and modification of the adsorbent are carried out according to the specific properties of the adsorbate. In addition, the ability of the adsorbate to diffuse on the surface and inside of the adsorbent is related to the activation energy and intermolecular forces. Different intermolecular forces achieve physical or chemical adsorption. The placement of the adsorbent also has an important effect on the adsorption capacity of the adsorbent. The acidic functional group on the surface of the adsorbent enhances its polarity and hydrophilicity, which facilitates the adsorption of polar substances. The basic functional groups on the surface of the adsorbent can enhance non-polarity and hydrophobicity, and help the adsorption of non-polar materials. AC is a hydrophobic and organic non-polar adsorbent, which is beneficial to the adsorption of non-polar organic substances. Therefore, reasonable selection and modification of the adsorbent should be based on the characteristics of the adsorbent. The surface reaction has to pass through the adsorption process of the reactant molecules on the solid surface. Different adsorbents and adsorbates have a great influence on the surface reaction kinetics. Therefore, it is of certain significance to use different adsorbents to adsorb different adsorbate kinetics.

This paper studies the adsorption capacity and kinetics of different packed adsorbents for different adsorbents with RPB as adsorption equipment. At the same time, the adsorption rate and the interaction between the adsorbent and the adsorbate are studied. The adsorption mechanism of the RPB is explored.

2. Experiments and methods

2.1. Adsorbent pretreatment

The purpose of pretreatment was to remove ash and impurities from the surface of the adsorbent. The silica gel (Tianjin Hengxing Chemical Co., Ltd.), 5 A molecular sieve (Tianjin Haochen Chemical Reagent Factory), and activated carbon (Shanxi Xinhua Chemical Co., Ltd.) were boiled for 40 min, respectively, and washed several times with ultrapure water, then placed in a blast dry box and dried at 110°C for 12 h and stored in a desiccator for later use.

2.2. Characterization

The surface area and pore size distribution were determined by the Brunauer–Emmett–Teller method (Quantachrome NOVA-4000e). The sample was degassed at 150°C for 300 min.

The resorcinol solution was filtered through a 0.45 μm filter and the concentration was determined by high-performance liquid chromatography (HPLC, Dai An u3000). The measurement conditions were as follows: the column type was C18 reverse phase column (250 mm × 4.6 mm, 5 μm), the detection wavelength was 277 nm, the mobile phase was methanol/water = 60/40, the injection volume was 20 μL, the temperature was 20°C, the flow rate is 1 mL/min. The detection wavelength of phenol detected by HPLC was 270 nm and the detection wavelength of nitrobenzene was 262 nm.

2.3. Adsorption experiment

Fig. 1 showed a process flow chart of adsorbing resorcinol, phenol, and nitrobenzene (Tianjin Xingfu Technology Development Co., Ltd.) by RPB (diameter is 150 mm, height is 100 mm, the rotating speed is 0–1,500 rpm, North University of China). The organic wastewater was prepared in the wastewater tank, and the solution was pumped by the liquid pump into the filler layer filled with the adsorbent. The liquid distributor in the center of the rotating bed was uniformly sprayed to the inner side of the packing zone, and the organic solution was sheared into a liquid filament, the liquid film and the droplets were fully contacted with

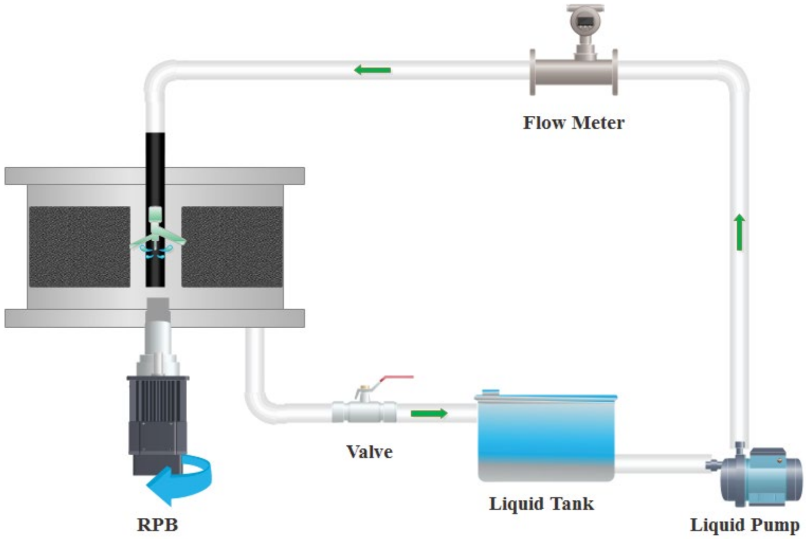

Fig. 1. Process flow chart for absorbing liquid in RPB.

the adsorbent in the packed zone under the action of high-speed rotation to complete the adsorption. The adsorbed organic wastewater flowed into the wastewater tank and was circulated for adsorption.

3. Results and discussion

3.1. Characterization of different adsorbents

According to the IUPAC classification, the adsorption isotherms of adsorption of nitrogen on silica gel, 5 A molecular sieves, and AC are classified into type I spectra, which indicates that they belong to microporous adsorbents and monolayer adsorption is dominant in the adsorption process. The physical properties of the specific surface area, pore size, and pore volume of the three adsorbents are presented in Table 1. The specific surface area of three adsorbents is AC > silica gel > 5A molecular sieve. The average pore size of silica gel and 5 A molecular sieve is greater than that of AC. The surface area of silica gel is large because it contains more macropores and mesopores, while AC is mainly composed of micropores and has a strong adsorption force.

3.2. Comparison of fixed bed and RPB adsorption capacity

Resorcinol is adsorbed by AC in the same specifications of RPB and fixed bed. The liquid flow rate is 50 L/h, the initial resorcinol concentration is 900 mg/L, the AC mass is 100 g, and the resorcinol solution volume is 3 L. The resorcinol removal rate equation is shown in Eq. (1), and the equilibrium adsorption capacity equation is shown in Eq. (2).

$$\eta=\frac{\left(c_0-c_e\right)}{c_0}\times 100\% \tag{1}$$

where η is the removal rate (%); c_0 is the initial concentration (mg/L), and c_e is the equilibrium concentration (mg/L).

$$q_e=\frac{\left(c_0-c_e\right)V}{w} \tag{2}$$

where q_e is the equilibrium adsorption capacity (mg/g), V is the resorcinol volume (L), and w is the AC mass (g).

The β (high gravity factor) indicates the rotational speed of RPB. It is expressed by the following Eq. (3).

$$\beta=\frac{\omega^2 r}{g} \tag{3}$$

where ω was the rotor angular velocity(1/s), r was the radius of filler rotor(m).

Fig. 2 shows the removal rate and adsorption capacity of resorcinol in fixed bed and RPB (β = 41.3). As a result, it is found that the RPB removal rate and adsorption

Table 1
Specific surface area, pore size, and pore volume of different adsorbents

Adsorbent	Specific surface area (m^2/g)	Average aperture (nm)	Pore volume (cc/g)
Silica gel	675.99	3.41	0.49
5 A molecular sieve	467.34	3.84	0.30
AC	671.50	0.52	0.46

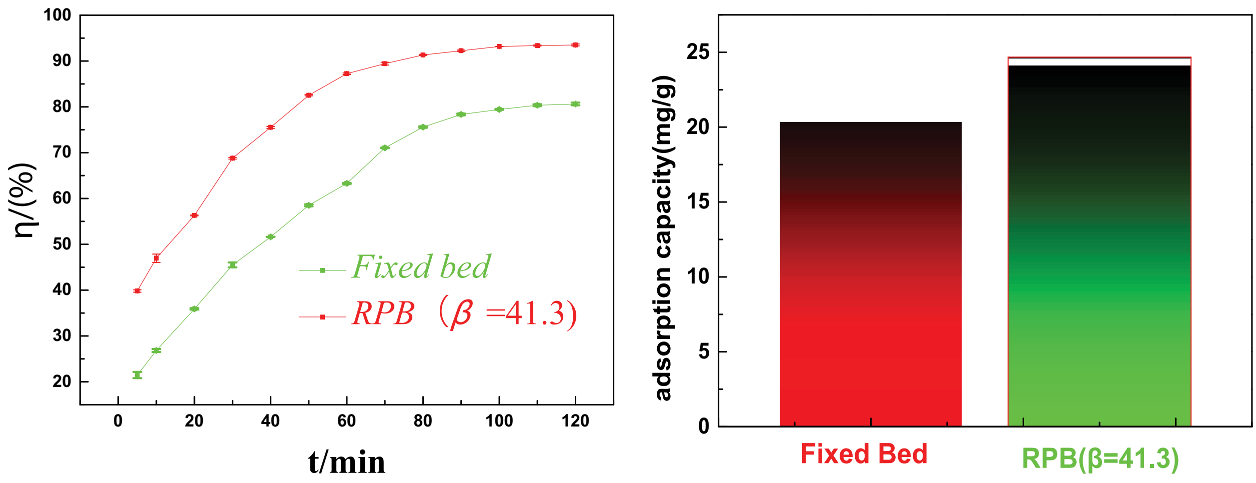

Fig. 2. Comparison of resorcinol removal rate and adsorption capacity between fixed bed and RPB.

capacity are higher than fixed bed. It is because RPB can disperse resorcinol into extremely small droplets. The large contact area quickly increases the resorcinol renewal rate on the AC surface. [28]. RPB can increase the external diffusion of resorcinol on the surface of AC and resorcinol is more likely to enter the interior of AC. The liquid film at the solid–liquid phase interface is sheared thinner, the mass transfer resistance is reduced, and the adsorption of resorcinol by AC is promoted [29]. Fig. 3 shows the removal rate and adsorption capacity of resorcinol by RPB with different high gravity factors. The results show that the larger the high gravity factor, the greater the removal rate, and adsorption capacity. RPB exhibits good adsorption capacity.

3.3. Effect of different adsorbents on the adsorption of resorcinol

The adsorption capacity of silica gel, 5-molecular sieve, and AC to resorcinol is studied by RPB as the adsorption device. The process conditions throughout the adsorption process are the liquid flow rate of 50 L/h, a rotational speed of 1,100 r/min, a pH of 5, a temperature of 20°C, an initial concentration of resorcinol of 500 mg/L, and a loading of the adsorbent of 100 g.

Fig. 4 shows the effect of different adsorbents on adsorption of resorcinol. The results indicate that AC had the highest removal rate of resorcinol, which is higher than 7.78% and 15.92% compared with 5 A molecular sieve and silica gel, respectively. The removal rate of resorcinol is related to the specific surface area, pore size, and surface physicochemical properties of the three adsorbents. Silica gel a porous adsorbent with silicon–oxygen crosslinked structure and plurality of silanol groups on the surface. The silanol group can form a hydrogen bond with a polar compound or an unsaturated compound to make the silica gel adsorbable. The active hydroxyl group exists in the small pores on the surface of the silica gel, and thus the silica with a small surface pore is highly adsorbed. Resorcinol and water are more polar and contact with silica gel to form hydrogen bonds on the surface of the silica gel. However, the water content of the solution is high and the formed

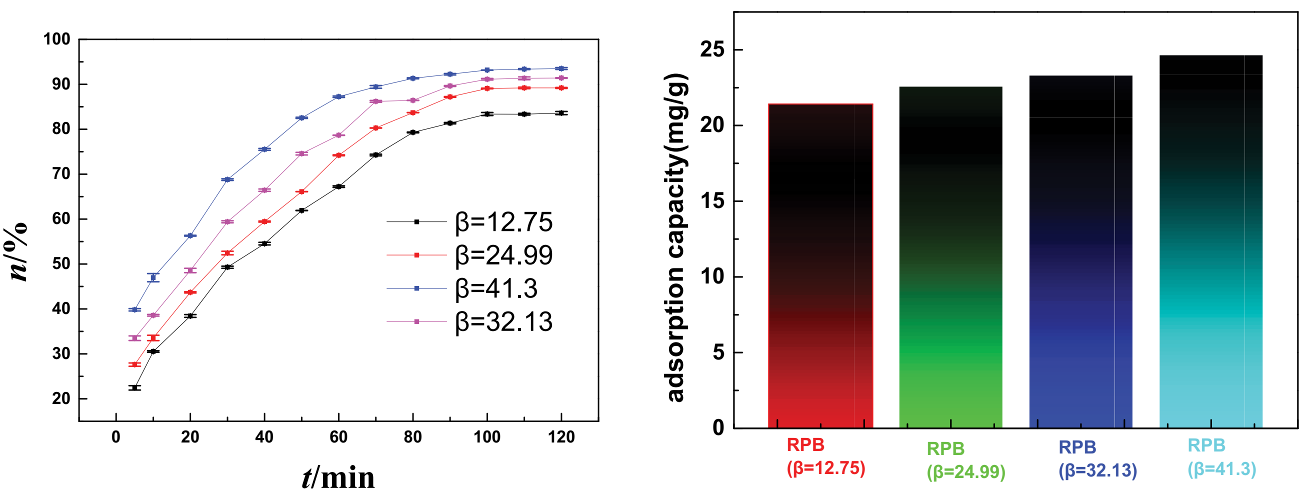

Fig. 3. Removal rate and adsorption capacity of resorcinol by different β in RPB.

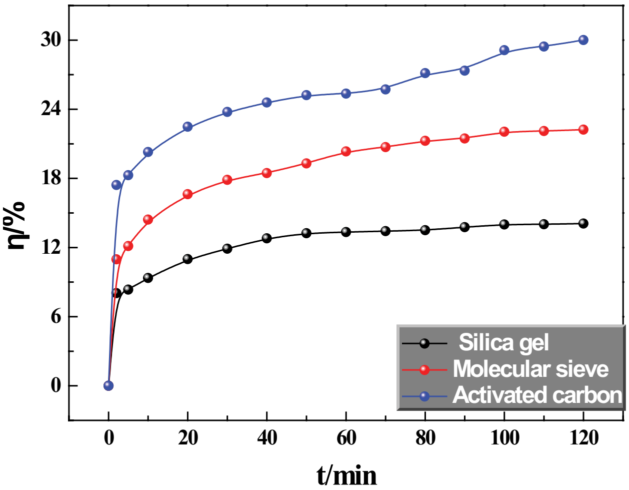

Fig. 4. Effect of different adsorbents on the adsorption of resorcinol.

hydrated silanol groups deactivate the silica gel. Therefore, the specific surface area of silica gel is high, but the removal rate of resorcinol is low. The pore size of the 5 A molecular sieve is mesoporous, and the micropores play a major adsorption role in the process of adsorbing resorcinol. 5 A Molecular sieves have strong water absorption similar to silica gel, which is one of the reasons for the poor adsorption of 5 A molecular sieves. AC is a non-polar adsorbent with hydrophobic and organophilic properties and is beneficial for the adsorption of resorcinol. AC has a pore size of 0.59 nm and is a microporous adsorbent with more active sites. Therefore, AC has the highest adsorption rate for resorcinol.

Figs. 5 and 6 show the resorcinol adsorption capacity of different adsorbents. The results verify that the equilibrium adsorption capacity of AC is 14.988 mg/g, which is 7.025 and 4.45 times that of silica gel and 5 A molecular sieve, respectively. The removal rate of resorcinol can reach 99.92%, which is 85.84% and 77.69% higher than that of silica gel and 5 A molecular sieve, respectively. The good equilibrium adsorption capacity of AC is determined by its specific surface area and micropore volume, and AC provides more adsorption sites during the adsorption process.

3.4. Kinetics of adsorption of resorcinol by different adsorbents

The adsorption kinetics of resorcinol by different adsorbents is studied by the pseudo-first-order and second-order kinetic models. The pseudo-first-order kinetic model and the pseudo-second-order kinetic model are respectively represented by the following equations:

$$\ln\left(q_e - q_t\right) = \ln q_e - k_1 t \tag{4}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{5}$$

where q_t (mg/g) and q_e (mg/g) are the adsorption capacity at time t and equilibrium; k_1 (1/min) is the first-order adsorption

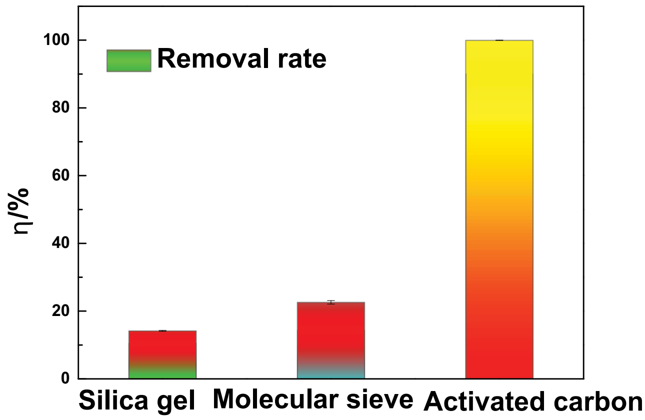

Fig. 5. Removal rate of resorcinol by different adsorbents.

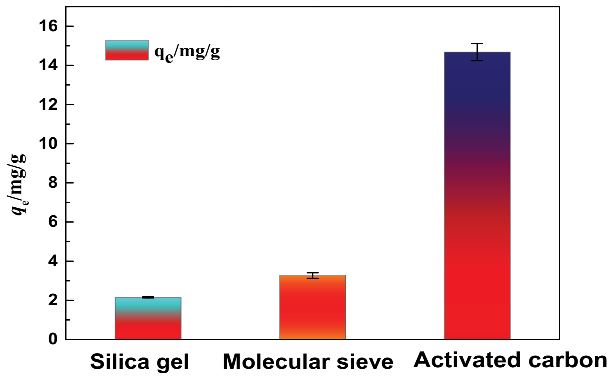

Fig. 6. Equilibrium adsorption of resorcinol by different adsorbents.

rate constant; k_2 (g/(mg min)) is the second-order adsorption rate constant.

Figs. 7 and 8 show a linear fit of the quasi-first and quasi-secondary kinetic models. Table 2 shows the fitting results. The results indicate that the linear correlation coefficient R^2 of the pseudo-secondary model of the three adsorbents is higher than the pseudo-first, which proves that the experimental and pseudo-second-kinetic models can be well-matched. The pseudo-second-order kinetic equation shows that the adsorption rate constants of silica gel, 5 A molecular sieve, and AC are 0.0519 g/(mg min), 0.02335 g/(mg min), and 0.01814 g/(mg min), respectively. The pseudo-second-kinetic model shows that the adsorption of resorcinol by three adsorbents is caused by external liquid membrane diffusion, surface adsorption, and intraparticle diffusion, which fully reflects the adsorption process of resorcinol on the adsorbent.

3.5. Adsorption effect of AC on different adsorbates

AC exhibits good adsorption properties in the three adsorbents. In order to investigate the adsorption effect of AC on different adsorbates in RPB, the adsorption performance of phenol, resorcinol, and nitrobenzene is compared by using AC. Table 3 shows the physical properties of the three adsorbates.

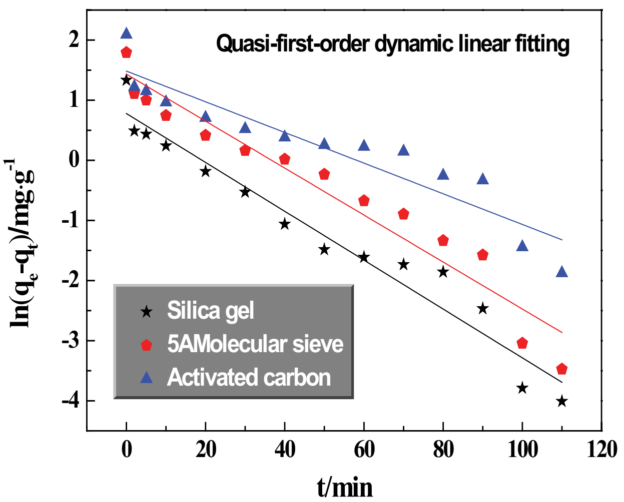

Fig. 7. Linear fitting of quasi-first-order kinetic models of different adsorbents.

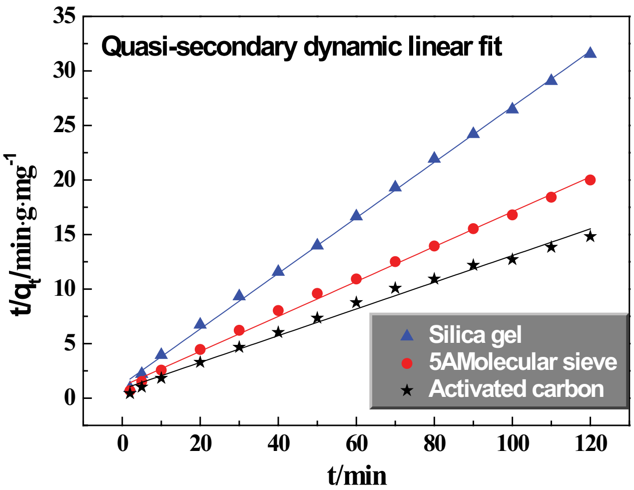

Fig. 8. Linear fitting of quasi-secondary kinetic models of different adsorbents.

Table 2
Pseudo-first-order and second-order fitting model for adsorption of resorcinol by different adsorbents in RPB

Association	Adsorbent	Linear fitting equation	R^2	Adsorption rate constant (1/min)
$\ln(q_e - q_t) - t$	AC	$y = -0.0255x + 1.4839$	0.86887	0.0255
	Silica gel	$y = -0.04064x + 0.7780$	0.94913	0.04064
	5 A molecular sieve	$y = -0.03906x + 1.4332$	0.93637	0.03906
$t/q_t - t$	AC	$y = 0.12244x + 0.82624$	0.99042	0.01814
	Silica gel	$y = 0.25471x + 1.2489$	0.99877	0.0519
	5 A molecular sieve	$y = 0.15995x + 1.09578$	0.9965	0.02335

Table 3
Physical properties of different adsorbates

Adsorbate	Molecular weight	Molecular diameter (nm)
Phenol	94.11	0.62
Resorcinol	110.11	0.56
Nitrobenzene	123.06	0.33 ± 0.02

Fig. 9 shows the removal rate of different adsorbates by AC. The results show that the removal rate of different adsorbates after 2 h is: phenol < resorcinol < nitrobenzene, the removal rate are 92%, 96.86%, and 98.62%, respectively. The removal rates of the three adsorbates are similar, because AC adsorption is physical adsorption and no selectivity. However, AC is a non-polar adsorbent. The smaller the polarity of the adsorbate, the easier it is to be adsorbed. The polarity of a compound depends on the functional group and molecular structure contained in the molecule. The polarity of the functional groups of various compounds is: $-CH_3 < -CH_2 < -CH < -O-R < -S-R < -NO_2 < -N(R)_2 < -OCOR$, $NH_2 < -OH < -COOH < -CN < -SO_3H$. The polarity of nitrobenzene is less than the polarity of phenols. Compared with a hydroxyl group of phenol, resorcinol has two hydroxyl groups, and the hydroxyl group is an electron donating group, so that the electron pair on the hydroxyl group is biased toward H, which is not easy to lose and has a small polarity. The difference in the position of the two electron-donating groups on resorcinol only determines the difference in the size of the electron donating effect and does not affect the polarity of resorcinol. In summary, the phenol is more polar than resorcinol. The polarity of the three adsorbates is: nitrobenzene < resorcinol < phenol. AC is an adsorbent which is a non-polar adsorbent. Therefore, the removal rate of nitrobenzene is the largest and the removal rate of phenol is the smallest.

3.6. Kinetics of adsorption of different organic compounds by AC

In order to further study the adsorption mechanism of AC on different adsorbates in the pseudo-primary and pseudo-secondary kinetic models are used to linearly fit the adsorption data. Table 4 shows the fitting results of the quasi-first and quasi-secondary kinetic models. Fig. 10 shows the quasi-secondary kinetic fit of AC adsorption of different organic matter.

The results show that the fitting curve of the pseudo-second-order kinetic equation can be well-coincident with the data points, and the correlation coefficient is

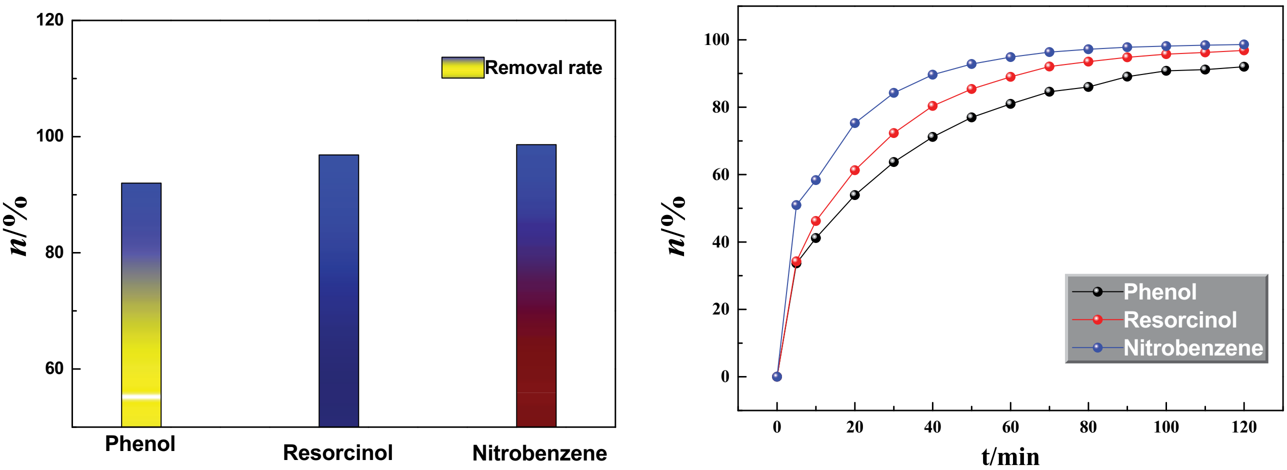

Fig. 9. Removal rate of different adsorbates by AC.

Table 4
Quasi-secondary kinetics fitting results of AC adsorption on different adsorbates in an RPB

	Adsorbate	Fitting equation	R^2	Adsorption rate constant (1/min)
$\ln(q_e - q_t) - t$	Phenol	$y = -0.0391x + 3.1943$	0.96972	0.0391
	Resorcinol	$y = -0.04288x + 3.1786$	0.99335	0.04288
	Nitrobenzene	$y = -0.05071x + 2.97406$	0.99345	0.05071
$t/q_t - t$	Phenol	$y = 0.03555x + 0.5789$	0.99629	0.001258
	Resorcinol	$y = 0.0239x + 0.45493$	0.999	0.002182
	Nitrobenzene	$y = 0.03535x + 0.2392$	0.9996	0.00522

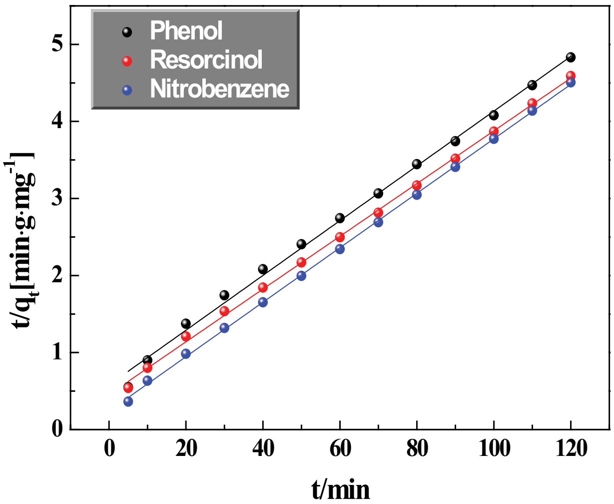

Fig. 10. Fitting diagram of pseudo-second-kinetic model for adsorption of different adsorbates by AC in RPB.

higher than the pseudo-first-order kinetic equation, which indicates that the pseudo-second-order kinetic equation can better describe the adsorption process of phenol, resorcinol, and nitrobenzene on AC. The adsorption rate of different adsorbates on AC is: phenol < resorcinol < nitrobenzene. The adsorption rate of the adsorbate on the adsorbent is related to the pore structure of the adsorbent. The transition pores (mesopores) in the adsorbent can promote the diffusion of the adsorbate to the adsorption site. On the other hand, the number of micropores in the adsorbent also plays an important role in the adsorption rate, and the developed microporous can promote the adsorption of the adsorbate. The pore diameter of the AC was 0.524 nm. The molecular diameters of phenol and resorcinol are 0.62 and 0.56 nm, and their molecular diameters are slightly larger than the micropore diameter of AC. The molecular diameter of nitrobenzene is 0.33 nm, which is smaller than the micropore diameter of AC. If only the size exclusion effect is considered, the organic molecules can only enter the pores whose pore diameter is larger than the molecular diameter. The well-developed mesopores of the AC are favorable for the adsorption of phenol. The molecular diameter of nitrobenzene is smaller than that of AC, and the diffusion is not hindered by the size of the micropores, and the size rejection effect can be neglected. The adsorption rate of nitrobenzene is mainly determined by the number of micropores. The adsorption rate of phenol and resorcinol is determined by the size exclusion effect and the number of micropores. Therefore, the adsorption rate of nitrobenzene is higher than that of resorcinol and phenol.

4. Conclusions

The adsorption capacity and adsorption kinetics of different adsorbents for different adsorbents were studied by using a RPB as the adsorption equipment. The results showed that the adsorption effect of AC on resorcinol was higher than that of 5 A molecular sieve and silica gel in the same conditions, which increased by 75.6% and 81.2%, respectively. It is mainly due to the large specific surface area and rich microporous structure of AC. The RPB disperses the resorcinol liquid into a fine state, which matches the micropores of the AC, thereby achieving good adsorption. In addition, the adsorption of AC on different adsorbates showed that the adsorption of AC on nitrobenzene was superior to that of resorcinol and phenol. The pseudo-second-order kinetic equation can better describe the adsorption behavior in an RPB. The experimental results also show that the adsorption rate of AC on different adsorbates is the largest. The adsorption rate of nitrobenzene on AC is higher than that of phenol and resorcinol.

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