Study on the organics adsorption capacities of powdered activated carbon and activated coke in reclaimed water

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ABSTRACT

In this study, the adsorption capacities of powdered activated carbon (PAC) and activated coke (AC) for organic components with different characteristics from the secondary effluents of urban waste water plant were examined, and their adsorption mechanisms were discussed. The results showed that Freundlich isothermal model and pseudo-second-order model could describe the processes of PAC and AC to effluent organic matter (EfOM) from urban wastewater plant effectively. Soluble microbial metabolites and humus were the major components of organics in the secondary effluents, and most of them were with small molecular weight which is less than 3 kDa (the proportion is 28.3%). PAC effectively adsorbed the organics with small molecular weight (less than 3 kDa) and the removal rate of dissolved organic carbon (DOC) was 67.2%, whereas AC effectively adsorbed the organics with molecular weight of 3 kDa–10 kDa and the DOC removal rate was 45.5%. Compared with AC, PAC has characteristics of smaller particle size, larger specific surface area, rougher surface and lower Zeta potential, which leads to more effective adsorption sites and better results. AC can effectively adsorb the organics with variable molecular weight in water and can be used as replacement of PAC in the advanced purification of reclaimed water.

Keywords: EfOM; Adsorption; Molecular weight distribution; PAC; AC

1. Introduction

The shortage of water resources has been an important factor restricting the social and economic development in China [1, 2]. As a countermeasure, wastewater reclamation and reuse can effectively solve this problem. But after secondary biological treatment, the domestic wastewater still has a high concentration of organics (effluent organic matter, EfOM), and it is usually difficult to meet the quality requirement of reclaimed water used in urban landscape and industry [3, 4]. Currently, the major advanced treatment technologies for bio-treated effluent include coagulant sedimentation, activated carbon adsorption, chemical oxidation and membrane bioreactor [5]. And membrane bioreactor has been widely applied to the advanced treatment of reclaimed water with the characteristics of higher effluent quality and less sludge yield [6].

Studies have shown that adding powdered activated carbon (PAC) into the membrane bioreactor, the treatment effect of the system can be improved, and the membrane fouling can be effectively mitigated. Moreover, PAC may be transformed into biological powdered activated carbon (BPAC), which has

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the adsorption capacity still [7]. However, the cost of PAC is still high and this restricts its application in engineering consequently and widely. Activated coke (AC), which is a type of dark porous carbonaceous material with lignite as the major raw material, does not need to be fully dry distillation or fully activated and still with functions as activated carbon adsorbent and with good adsorption capacity [8]. If PAC can be replaced by AC in wastewater treatment engineering, the running cost can be reduced greatly. However, there are few relevant researches on this aspect at domestically and internationally.

On the other hand, when detecting the efficiency of organic pollutant removal in the advanced treatment of reclaimed water, the indicators have changed from the routine indicators such as COD_{Cr} to the detailed structural properties and composition of the organics. The activated carbon adsorption has become one of the effective methods to remove organic pollutants in water [9]. As indicated by Gur-Reznik that about 20% of dissolved organic carbon (DOC) in the bio-treated effluent failed to be absorbed by activated carbon, the removal rate of hydrophobic organics by activated carbon was approximately 65% [10]. It was found by Matilainen that the activated carbon could significantly remove the pollutants with medium molecular weight, and the adsorption capacity on organics was directly related to the molecular weight of pollutants and the pore size distribution of the activated carbon [11]. The analysis of the characteristics of organic pollutants in water facilitates have contributed to the evaluation of the treatment efficiency of a technique and the removal rate of the organic pollutants. For the adsorption process, the chemical and physical characteristics of adsorbent, types and molecular weight distribution of organic pollutant have great impact on the efficiency of pollutant removal.

In this article, the secondary effluent from a wastewater treatment plant in Beijing was studied in terms of determining the adsorption capacity of a variety of organic pollutant component in water by PAC and AC, the adsorption processes of the adsorbents and the adsorption mechanism.

2. Materials and methods

2.1. Experimental reagents

PAC (made from nutshell, 200–300 mesh, iodine value 700–1,000 mg/g, methylene blue value 100–150 mg/g, Heatton Co., China); AC (200–300 mesh, iodine value 620 mg/g, methylene blue value 60 mg/g, Clear Co., China); 1 mol/l HCl (AR) and 1 mol/l NaOH (AR) for pH value adjustment. Raw water for the experiment was the secondary effluent of a wastewater treatment plant in Beijing with the measures of TP 0.4 mg/l, SS 20 mg/l, DOC 6.04 mg/l, UV_{254} 0.112 cm^{-1} and pH 7.5–7.8.

2.2. Experimental methods

In the adsorption isotherm experiment, 20.0, 40.0, 60.0, 80.0 and 100.0 mg of PAC and AC were added into 1,000 ml of raw water, respectively, and kept stirring by thermostatic oscillator (THZ-82, Guohua Co., China) at room temperature (24–25.0°C) for 48 h at 160 rpm. Each sample was then filtered through the 0.45-μm membrane, respectively, and the organics UV_{254} (cm^{-1}) of the solutions, and the equilibrium adsorption (q_e, mg/g) of the adsorbent were calculated so as to plot the static adsorption curves.

Six-Joint Mixer (ZR4-6, Jar Tester Co., China) was employed in the adsorption kinetic experiment. PAC and AC (40.0 mg) were added into 1,000 ml of raw water, respectively, at room temperature (24–25.0°C), and the solutions were stirred at the rate of 160 rpm. After 0.5, 1, 2, 3, 4, 5, 10, 15, 30, 35, 45, 60, 90 and 120 min, the sampling was performed at 4 cm below the fluid level, respectively. After the solutions were filtered through 0.45-μm membrane, the concentration C_e (mg/l) of DOC of the solutions was detected one by one. The equilibrium adsorption q_t (mg/g) in different reaction time was calculated in order to get the variation trends of organics adsorption quantity in water by the two adsorbents with time.

Before and after adsorption, the water samples were filtered through 0.45-μm membrane. The regenerated fibrous membranes with different molecular weight cutoffs (100 kDa, 30 kDa, 10 kDa and 3 kDa, Millipore Co., USA) were separately placed in the centrifugal ultrafiltration cells (Millipore Co., USA). The experiments were carried out by molecular weight classification at filtration pressure of 0.10 Mpa.

2.3. Analytical instruments and methods

After the samples filtered through 0.45-μm membrane, Ultraviolet absorbance at 254 nm (UV_{254}) of the samples was detected by UV/VIS Spectrophotometer (UV6000, Metash Co., China). The quality of reagents was weighed by Electronic Balance (AUW120D, ±0.01 mg, Shimadzu Co., Japan). DOC was detected by TOC Analyzer (Liqui TOC II, Elementar Co., Germany). The 3-D fluorescence was detected by Fluorescence Spectrophotometer (F7000, Hitachi Co., Japan). The pH was detected by pH handheld meter (206-pH$_1$, Testo Co., Germany). The particle size of PAC/AC was measured by particle size analyzer (MS2000 Hydro 2000 MU, Malvern Co., UK). Zeta potential was detected by Zeta sizer (Nano ZS, Malvern Co., UK). After the samples dried and tableted at 300°C, Fourier transform infrared spectrometry (FTIR) was carried out by FTIR spectrometer (NICOLET 8700, Thermo Co., USA).

3. Results and discussion

3.1. Adsorption of organics in secondary effluents by PAC and AC

3.1.1. Adsorption isotherm

The adsorption isotherm experiments were carried out in different dosages of PAC and AC, respectively. Langmuir, Freundlich and Temkin adsorption models were built through the experimental data fitting [12]. The fitted curves and parameters were shown in Fig. 1 and Table 1, respectively.

Langmuir:

$$q_e = \frac{bq_mC_e}{1 + bC_e} \tag{1}$$

Freundlich:

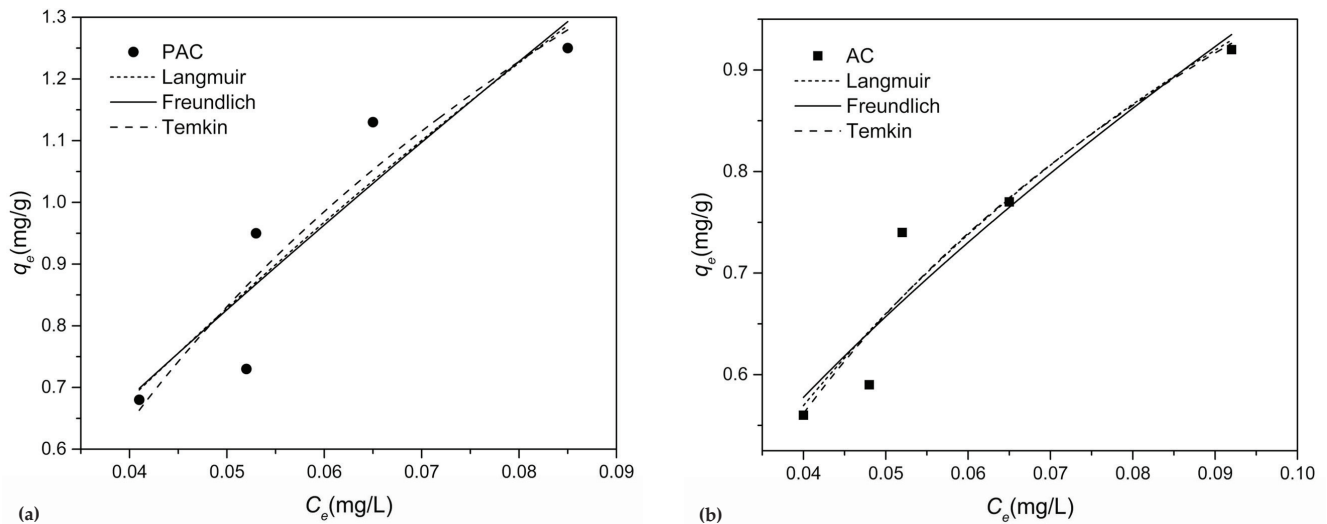$$q_e = K_FC_e^{1/n} \tag{2}$$

Temkin:

$$q_e = A + B\ln C_e \tag{3}$$

Fig. 1. Adsorption isotherm of PAC (a) and AC (b) on organics (T = 24~25.0°C, pH = 8.5, t = 48 h, r = 160 rad/min).

Table 1
Adsorption isotherm parameters of PAC and AC on organics

Adsorbent	Langmuir model			Freundlich model			Temkin model		
	q_m	b	R^2	K_F	1/n	R^2	A	B	R^2
PAC	5.83	3.33	0.94	10.10	0.50	0.96	3.33	0.83	0.94
AC	1.83	11.36	0.95	3.77	0.58	0.99	1.99	0.44	0.97

where q_e is equilibrium adsorption (mg/mg), C_e is equilibrium concentration (mg/l), q_m is the maximum adsorption capacity (mg/mg), b is a constant characterizing the strength of adsorption surface, which is related to temperature and adsorption heat (l/mg); K_F is the adsorption coefficient related to the sorption energy; 1/n is the increasing degree of adsorption capacity with the increasing of concentration, and its value represents the difficulty level of adsorption; A and B are constants.

The results showed that the equilibrium adsorption of PAC and AC increased to the maximum with the rising of organics concentrations. Three adsorption isotherm equations reflected different energy relationship in the adsorption process. Langmuir model is only suitable for the system with known adsorbent concentration and can be applied to the homogeneous adsorption surface with monomolecular layer; Freundlich model is suitable for the bilayer adsorption on the heterogeneous surface; and Temkin model is suitable for the adsorption process on heterogeneous surfaces [12–14]. Although the above adsorption isotherm equations were developed for pure substance, it was shown by comparing the correlation coefficients in different models that Freundlich model described the adsorption process of EfOM with PAC and AC best. This indicated that the adsorption of EfOM by PAC and AC was bilayer adsorption on the heterogeneous surface.

It can be known from the adsorption coefficients of Freundlich model that K_F value directly reflected the adsorption capacity of adsorbents. The K_F values of PAC and AC were 10.10 and 3.77, respectively, which meant that PAC had a stronger adsorption capacity. Moreover, the magnitude of the exponent, 1/n, gave an indication of the favorability of the adsorption [15]. Values of 1/n between 0.5 and 1.0 means a favorable adsorption condition, and 1/n with value larger than 2.0 means it is difficult to absorb the organics [16]. Because the values of 1/n of the two adsorbents were both below 1.0, they were favorable to adsorption. As presented in Table 1, the 1/n value of PAC is smaller, which means that the adsorption by PAC is less difficult than that by AC.

3.1.2. Adsorption kinetics

Fig. 2 shows the variation of adsorption capacity of PAC and AC on organics in water with time. The results indicated that the organics adsorption processes of the two adsorbents could be divided into rapid diffusion stage and slow diffusion stage. In 0–5 min of rapid diffusion stage, the organics adsorption capacities of PAC and AC reached 40.5% and 32.2%, respectively. After about 20 min, the slow diffusion stage worked.

The experimental results were fitted to the pseudo-first-order kinetic model (Eq. (4)) and pseudo-second-order kinetic model (Eq. (5)), as shown in Table 2.

$$q_t = q_e(1 - e^{-k_1^t}) \tag{4}$$

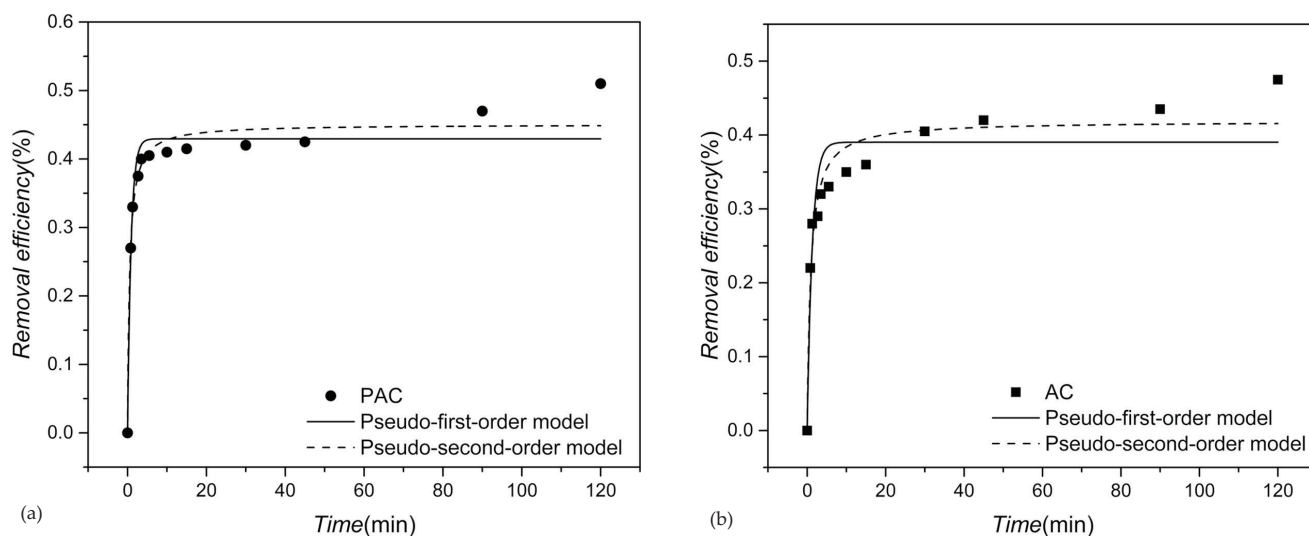$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \tag{5}$$

Fig. 2. Model fitting results of PAC (a) and AC (b) adsorption kinetics on organics ($T = 24$~$25.0°C$, $t = 2$ h, $r = 160$ rad/min).

Table 2
First order and Secondary dynamic model after PAC and AC adsorption on organics

Adsorbent	Pseudo-first order $q_t = q_e(1 - e^{-k_1 t})$			Pseudo-second order $\dfrac{t}{q_t} = \dfrac{t}{q_e} = \dfrac{t}{k_t q_e^2}$		
	q_e (mg/g)	k_1	R^2	q_e (mg/g)	k_2	R^2
PAC	0.43	1.64	0.94	0.44	6.27	0.97
AC	0.38	1.26	0.84	0.41	4.21	0.92

where q_e (mg/g) and q_t (mg/g) are equilibrium adsorption and the adsorption quantity at t (min), respectively; k_1 (1/min) is the rate constant of pseudo-first-order, and k_2 (mg/(mg·min)) is the rate constant of pseudo-second-order.

As shown in Table 2, the value of correlation coefficients (R^2) of the pseudo-second-order kinetic models for the two adsorbents were very close to 1, which indicated that the organics adsorption processes of the two adsorbents matched the pseudo-second-order kinetic model better than pseudo-first-order kinetic model. Generally speaking, the adsorption processes conforming to the pseudo-first-order kinetic model were mainly physical adsorption, while those conforming to pseudo-second-order kinetic model were mainly chemical adsorption [13]. Therefore, the organics adsorption processes of PAC and AC might be chemical adsorption processes. Moreover, in the two adsorption kinetic models, the value of k was the rate constant of adsorption, and the larger the k value, the higher the adsorption rate will be. Compared with AC, the adsorption rates of PAC were higher in both pseudo-first-order and pseudo-second-order.

3.2. Adsorption efficiency of organics with different molecular weight in secondary effluent by PAC and AC

3.2.1. Distribution of organic molecular weight in secondary effluent before and after adsorption

It was indicated in previous researches that UV_{254} could indicate the existence of humic substances and aromatic compounds with unsaturated double bond to some extent, and SUVA, defined as the ratio of UV absorbance at 254 nm (UV_{254}) divided by the DOC concentration, was a quantitative measurement for these substances. Water with high SUVA values contained high contents of aromatic components [17]. During the experiments, the distribution of organic molecular weight was detected (UV_{254} and DOC) for the secondary effluent and that after the adsorption of PAC and AC (shown in Fig. 3 and Table 3).

It was shown in Fig. 3 and Table 3 that the content of organics in secondary effluent with the molecular weight less than 3 kDa was the highest, accounting for 28.3% of the total content of organics in secondary effluent. It was mainly because that some organics with large molecular weight in the wastewater were removed by sedimentation, while those with small molecular weight were transformed into dissolved organics after biological treatments. Most of the dissolved organics with small molecular weight could be biodegraded but some still remained in water [18].

The comparison of the adsorption effect of organic compounds in water by PAC and AC was presented in Table 3. The results showed that PAC adsorbed the organics with small molecular weight of less than 3 kDa effectively and the removal rate of DOC reached 67.2% and the SUVA value declined from 2.13 l/(mg·m) to 0.89 l/(mg·m). While AC could effectively adsorb the organics with the molecular weight range of 3–10 kDa, and the removal rate of DOC was 45.5%, the SUVA value declined from 1.35 l/(mg·m) to 1.18 l/(mg·m).
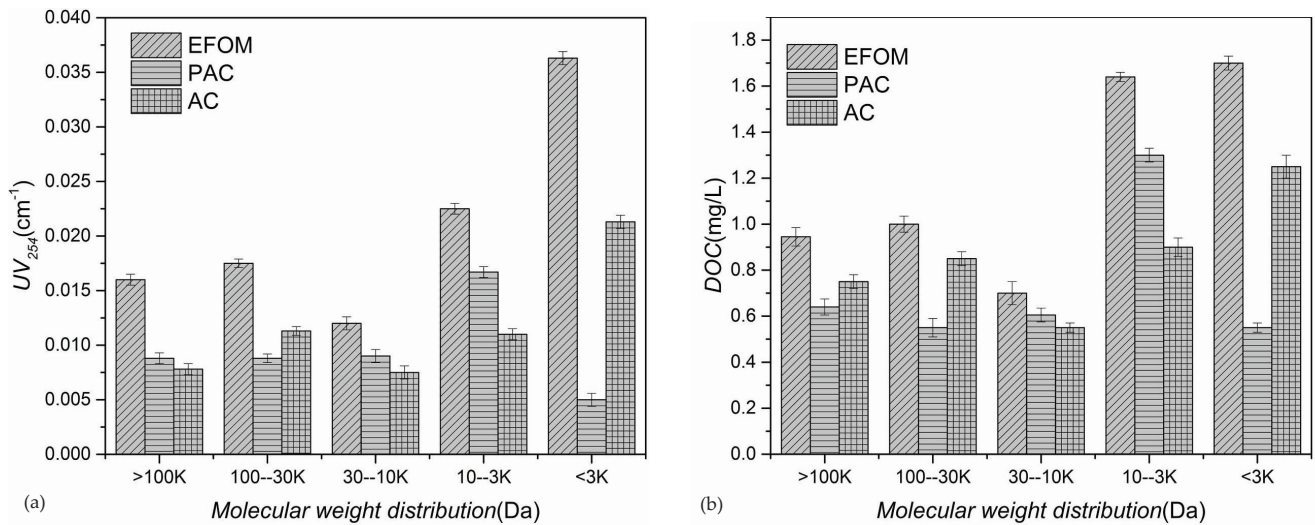
Fig. 3. Distribution of organics with different molecular weight in EfOM, after PAC and AC adsorption: (a) UV_{254}, (b) DOC.

Table 3
The adsorption effects of PAC and AC on different molecular weight organics in secondary effluent

Items		Secondary effluent	After adsorption				
			>100 K	100~30 K	30~10 K	10~3 K	<3 K
UV_{254}	EfOM (cm⁻¹)	0.11	0.02	0.02	0.01	0.02	0.04
	PAC (%)	51.9	42.9	47.6	13.1	24.3	86.3
	AC (%)	42.1	49.7	33.3	30.1	52.0	40.5
DOC	EfOM (mg/L)	6.04	0.95	1.00	0.71	1.67	1.71
	PAC (%)	38.6	32.1	42.8	12.5	21.7	67.2
	AC (%)	27.7	21.1	14.2	18.8	45.5	25.7
SUVA (L/(mg·m))	EfOM	1.70	1.65	1.73	1.52	1.35	2.13
	After PAC	1.33	1.39	1.59	1.51	1.30	0.89
	After AC	1.36	1.05	1.34	1.31	1.18	1.70

3.2.2. Analysis of 3-D excitation-emission matrix (3D-EEM) fluorescence of organics before and after adsorption

Three-dimensional fluorescence spectrum was used to analyze the types and contents of organic pollutants in water [19, 20]. The secondary effluents usually contains aromatic organic compounds such as humic acids, oils and phenols, and most of them have conjugated double bond or conjugated system such as carbonyl and carboxyl groups [21, 22]. When these compounds are irradiated and excited by light with specific wavelengths in the ultraviolet region, the fluorescence with different wavelength will be emitted [23]. The 3D-EEM analysis was carried out on the organics in water before and after the adsorption using the two adsorbents under the dosage of 40 mg/l. The EEM spectra were divided into five regions shown in Fig. 4, such as Region I and II (aromatic proteins, λ_{ex} < 250 nm, λ_{em} < 380 nm), Region III (fulvic acids, λ_{ex} < 250 nm, λ_{em} > 380 nm), Region IV (dissolved microbial metabolites, λ_{ex} >250 nm) and Region V (humic acids, λ_{ex} > 250 nm, λ_{em} > 380 nm). The results in Fig. 5 demonstrated that the integration of each fluorescent region in water sample before and after

adsorption and the proportion of fluorescence value of each region to total fluorescence value [24].

As shown in Figs. 4 and 5, the 3-D fluorescence contours of secondary effluent from the wastewater treatment plant (EfOM in Fig. 4) was denser in Region IV and V. This means that the contents of organics with small molecular weight in water such as soluble microbial metabolites and humus were higher, and the proportion of organic compounds with molecular weight less than 3 kDa account for the largest. After the adsorption of PAC and AC, the contours of different regions in the 3-D fluorescence spectra became sparse. It indicated that the contents of aromatic proteins, fulvic acids, microbial metabolites and humus all decreased, and the proportions of components with different molecular weight changed. PAC effectively adsorbed the organics with the molecular weight less than 3 kDa in Region V, where the removal rate reached the highest proportion of 65.7%. After the adsorption by PAC and AC, the proportion of the microbial metabolites increased from 27.8% to 46.4~46.8%, which means that the microbial metabolites in water were difficult to be absorbed by the two adsorbents.
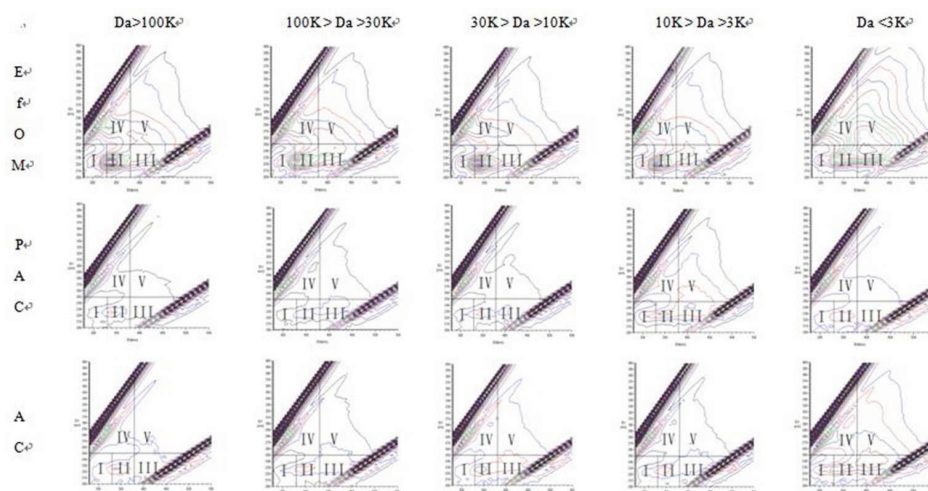
Fig. 4. Distribution of organics with different molecular weight in secondary effluent, after PAC and AC adsorption.

3.3. Discussion on the adsorption mechanism of PAC and AC

The particle-size distribution of PAC and AC (Fig. 6) showed that the particle sizes of PAC and AC were mainly in the range of 10–30 μm. The results in Fig. 7 demonstrated that the micropore content (smaller than 2 nm) of each adsorbent was dominant. Because the micropore content of PAC is slightly more than AC, for the adsorption effect of PAC to small molecular weight organics was better than AC. And for the range of 2~50 nm or large than 50 nm, the pore size content of AC was larger than PAC, which was the reason why AC could adsorb large molecular weight organic matter more effectively than PAC. The chemical and physical properties of PAC and AC are presented in Table 4. Compared with AC, PAC has a smaller particle size and a larger specific surface area, which results in more effective adsorption sites and higher adsorption efficiency.

The surface characteristics were detected by FTIR before and after PAC/AC adsorption, and the results are shown in Fig. 8. Some studies had indicated that the oxygen-containing functional groups on the PAC surface mainly include carboxyl, phenolic hydroxyl and carbonyl [25]. As shown in Fig. 8, before the adsorption, the absorption peak of AC at the wave number of 3,424 cm^{-1} was caused by the association of water, carboxyl and phenolic hydroxyl groups, while after the adsorption, the absorption peaks of PAC and AC occur at the wave number of 3,442 cm^{-1} and 3,467 cm^{-1}, respectively, corresponding to the associated hydroxyl group. Before the adsorption, the peaks caused by the stretching vibration of C=O of carboxyl group on aromatic ring occurred at the wave number of 1,430 cm^{-1} and 1,444 cm^{-1} [26], whereas after the adsorption, the carboxylic functional groups of the two adsorbents disappeared. Before the adsorption, the peaks caused by the stretching vibration of C-O-C of ether bond occurred at the wave number of 1,029 cm^{-1} and 1,043 cm^{-1}, whereas after the adsorption, the PAC and AC resulted in a redshift of 56 cm^{-1} and 38 cm^{-1}, respectively. The two adsorbents showed the absorption peak at the wave number of 1,085 cm^{-1} and 1,081 cm^{-1}, respectively, which proved the existence of carbonyl group. Therefore, the adsorption capacity of PAC and AC may be related to the carbonyl group on their surfaces, and the greater the number of the carbonyl group, the stronger the adsorption capacity will be. In addition, the absorption peaks in the range of wave number less than 1,000 cm^{-1} might be due to the heterocyclic aromatic hydrocarbons of the adsorbents [27]. The oxygen-containing functional groups on the surfaces of PAC and AC before and after adsorption were shown in Fig. 9 [25]. Before the adsorption, there were carboxyl and ether bond on the surfaces of PAC and AC, whereas after the adsorption, they were substituted by hydroxyl and carbonyl groups.

4. Conclusion

(1) Freundlich isothermal model and pseudo-second-order kinetic model could effectively describe the processes of organics adsorption in the secondary effluents by PAC and AC. The EfOM adsorption process by both PAC and AC was chemical adsorption process, which can be divided into stages of rapid diffusion and slow diffusion. In the rapid diffusion stage (within 0–5 min), the organics adsorption capacity of PAC and AC reached 40.5% and 32.2%, respectively. After about 20 min, the slow diffusion stage began.

(2) Soluble microbial metabolites and humus were the major components of organics in the secondary effluents, and the proportion of organics with the molecular weight less than 3 kDa was the highest. PAC effectively adsorbed the organics with small molecular weight (less than 3 kDa),
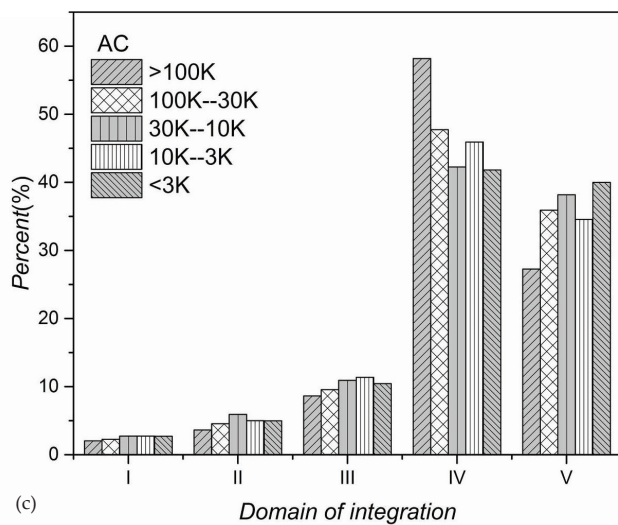
Table 4
Physical and chemical properties of PAC and AC

Adsorbent	Average particle size (μm)	Specific surface areas (m²/g)	Zeta potentials (mv)	Average pore diameter (nm)
PAC	19.1	821	−8.7	3.351
AC	21.9	697	−8.2	3.532
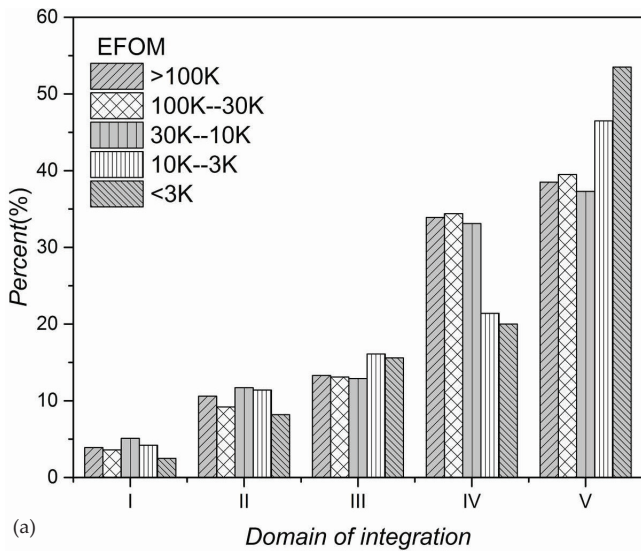
(a)
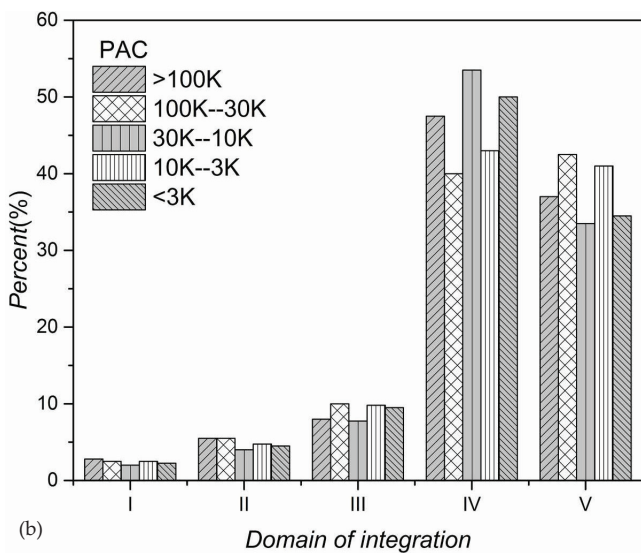
(b)

(c)

Fig. 5. Proportion of different Excitation-emission matrix area integral: (a) EfOM, (b) after the adsorption by PAC, (c) after the adsorption by AC.
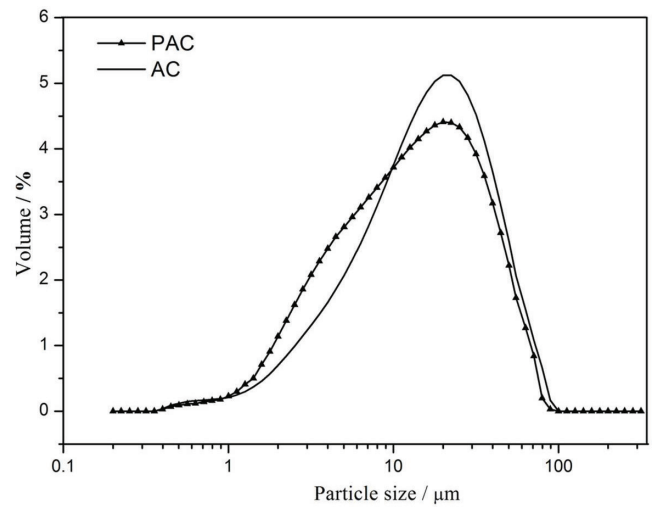

Fig. 6. The particle size distribution of PAC and AC.
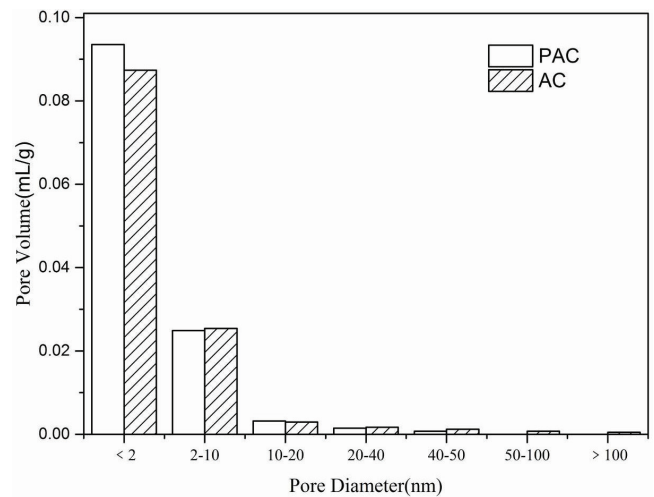

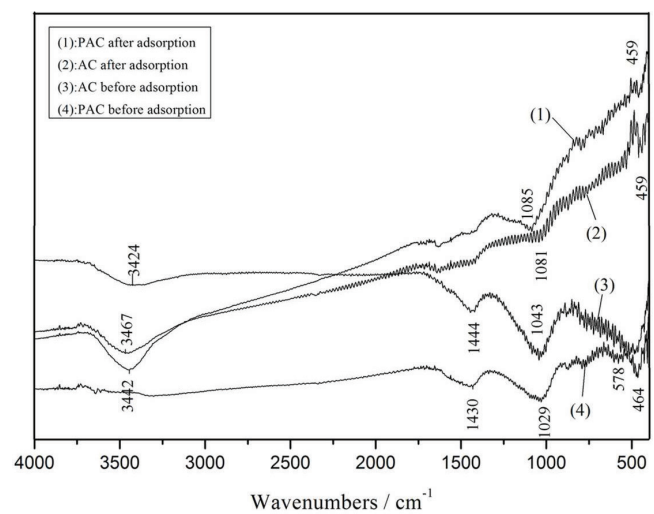Fig. 7. The pore size distribution of PAC and AC.

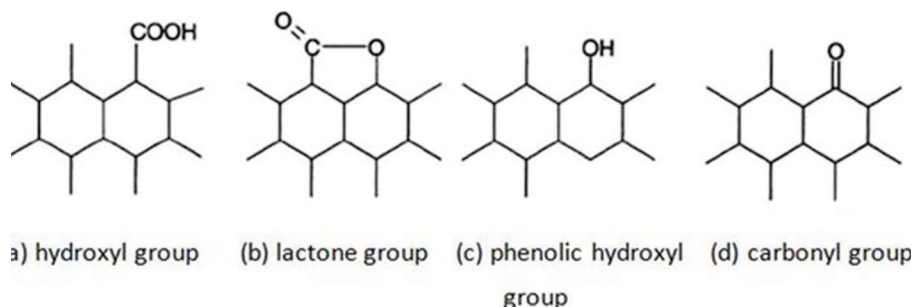Fig. 8. The FTIR analysis of PAC and AC.

Fig. 9. The oxygen-containing functional groups in PAC and AC.

whereas AC effectively adsorbed the organics with the molecular weight of 3 kDa-10 kDa.

(3) Compared with AC, PAC has smaller particle size, larger specific surface area, rougher surface and lower Zeta potential, which resulted in more effective adsorption sites and better efficiency of organics adsorption in water.

(4) AC can not only effectively adsorb the organics with different molecular weight in water but can also cost less when compared with PAC. Therefore, AC can replace PAC in the advanced purification of reclaimed water.

Acknowledgments

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