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Bifunctional activated carbon with dual photocatalysis and adsorption capabilities for efficient phenol removal

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Abstract Bifunctional activated carbons (AC) with the abilities of both photocatalysis and adsorption were fabricated *via* the sol–gel route combined with hydrothermal treatment and N_2 reactivation method. TiO_2 was located mainly at the entrance of the surface macropores of AC. Under UV light irradiation, efficient removal of phenol was realized by combination of adsorption and photocatalytic degradation for the obtained bifunctional materials. In insufficient light or dark, phenol removal occurred mainly through adsorption. The prepared bifunctional carbon with a mass ratio of 50 TiO_2 per AC ratio exhibited high efficiency for phenol removal. The total phenol removal capacity of $50TiO_2/AC$ was almost 5 times of that of pure AC and 6 times of that pure TiO_2 after 10 cycles. The prepared bifunctional carbons possess the advantages of high pollutant removal capability and good recyclability, making them promising for the efficient treatment of lightly polluted aqueous solutions.

Keywords Photocatalysis · Adsorption · Activated Carbon · Titanium dioxide · Bifunctional carbon

1 Introduction

Activated carbon (AC) adsorption has been widely used as an efficient method for the treatment of water or air polluted with organic compounds. However, adsorption by AC is mainly through the filling of micropores, removal of pollutant realized by the physical phase transfer of organic pollutants from the aqueous to the solid phase (Shin et al. 2007; Liu et al. 2007; An and Zeng 2003). Generally, the common used AC regeneration method is expensive for the fact of energy consumption, time consume and carbon loss of 5–15% due to oxidation and attrition. Then bring the problems short life time, low efficiency and high cost of AC (Jung et al. 2008). With the aim of overwhelming these shortcomings, most of research works have been paid to the functionalization of traditional porous carbonaceous materials (Schmidt et al. 2008; Li et al. 2007).

Heterogeneous photocatalysis by TiO_2 semiconductors is promising for elimination of hazard environmental pollutants especially for the degradation of bio-recalcitrant organic contaminants (Lettmann et al. 2011; Choi et al. 2001; Zang et al. 1998). Combination of TiO_2 with adsorptive materials such as AC and zeolite particles can enhance the photocatalytic activity effectively (Colon et al. 2004; Chen et al. 2006; Matos et al. 1998; Ingaki et al. 2003; Liu et al. 2007; Araña et al. 2003a, 2003b; Cordero et al. 2007; Lee et al. 2004; Ao and Lee 2003; Przepiórski et al. 2001; Tryba et al. 2006; Ravichandran et al. 2010; Zhu and Zou 2009; Velasco et al. 2010). Compared with other adsorptive materials, AC emerges as one of the most attractive TiO_2 support (Yap et al. 2010). Wang et al. (2009) prepared a high performance composite of TiO_2 deposited on activated carbon by dip-hydrothermal method. It was found that the composite catalyst TiO_2/AC was better than the physical mixture of AC and TiO_2. Cao and Shiraishi (Cao and Shiraishi 2010) investigates the mechanism of the photocatalytic and adsorptive treatment of an aqueous DNP (2, 4-dinitrophenol) by TiO_2/AC-PET film (a polyethylene terephthalate film adhesively fixing activated carbon particles covered with a thin and porous film of titanium dioxide). They found that the diffusion of DNP from a bulk solution to a TiO_2 film is based on the gradient of DNP concentration, generated by a rapid adsorption of DNP onto

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AC and photocatalytic reaction, in the very neighborhood of TiO_2 film, and this diffusion increases the DNP concentration at the surface of TiO_2 film, thereby enhancing the rate of photocatalytic decomposition. Ravichandran et al. (2010) observed increased photocatalytic activity of TiO_2-P25 by the addition of activated carbon by weight of 4, 8, 10 and 12% by weight; their result revealed that the defluoridation is more effective in 10AC-TiO_2-P25 than bare TiO_2-P25. The result of our group showed that the AC in TiO_2/xAC catalysts can act as the site where organic molecules are adsorbed (Liu et al. 2007). The organic molecules then transfer to the illuminated TiO_2 decomposition center located on the AC surface, because of the concentration difference. The activity of the TiO_2/xAC catalysts may be regulated by the content of AC. TiO_2/5AC catalyst exhibited better separation from the reaction mixture and less deactivation after several runs, as well as being less sensitive to pH changes than the other catalysts studied (Chen et al. 2006; Liu et al. 2007).

Also, our previous result revealed that it is possible to regenerate exhausted carbon saturated with phenol in a commercial TiO_2 suspension system (Liu et al. 2004). For the activated carbon saturated with phenol mixed with TiO_2 in an aqueous phase and then irradiated with UV light, TiO_2 can act as a degradation center and degrade the adsorbed phenol which was desorbed from the saturated carbon gradually, then producing organics concentration difference within pores of AC, lead to the adsorbed organics transferred to the illuminated TiO_2 gradually centre and realized regeneration of AC (Liu and Sun 2006; Liu et al. 2003, 2004). Cao and Shiraishi (2010) found that the TiO_2/AC-PET film can lower the burden of the adsorption of DNP onto AC compared because a part of DNP molecules are photocatalytically decomposed.

Considering the fact that AC can enhance activity of TiO_2, irradiated TiO_2 can lower the burden of the adsorption of organics onto AC and then realize in-situ regeneration, it is may be useful to fabricate a novel material combined the advantage of AC adsorption and TiO_2 photocatalysis effectively. However, to the best of our knowledge, most of conducted works concerning the combination of AC with TiO_2 were focused on the development of a composite photocatalyst, AC was just as a porous support and provide a relatively high concentration of organic compound for TiO_2, the adsorption ability of AC can not be utilized efficiently and the role of adsorption was neglected. Unlike most of the previous reports, in the present study both the roles of adsorption and photocatalysis for the removal of organics were strengthened. A novel bifunctional carbon with the dual abilities of photocatalysis and adsorption was fabricated and prepared. The removal capacity of pollutants using phenol as model compound was tested both in the dark and under UV irradiation.

2 Experimental

2.1 Preparation of materials

Ethanol (35 mL) was mixed with deionized water (32 mL), then HCl (1 $mol \cdot L^{-1}$) was added until pH of 3.0 was achieved (Solution A). AC powder (details listed in Table 1) was added to a mixture of $Ti(OBu)_4$ (35 mL), acetic acid (23 mL), ethanol (70 mL) and 5 mL of PEG (molecular weight of 400), then stirred vigorously to give solution B. Solution A was added drop-wise into solution B at a rate of 1 $drop \cdot s^{-1}$ with stirring. After the addition was complete, the mixture was stirred at 26 °C for 100 min. The obtained TiO_2/AC precursor was aged at room temperature for 36 h, dried at 105 °C for 24 h. Then, the product was put into a Teflon-lined autoclave with certain amounts of deionized water. The autoclave was sealed and maintained at 150 °C for 2 hours. Finally, the hydrothermally treated product was heated to 200 °C and maintained for 1 h. Then heated to 550 °C in an N_2 atmosphere and maintained at 550 °C for 2 h. The sample was cooled to room temperature in an N_2 atmosphere. The obtained bifunctional carbon (denoted as $xTiO_2$/AC, where x is the mass ratio of TiO_2, $0 < x < 70$). TiO_2 was prepared under the similar conditions but without AC addition.

2.2 Characterization

The BET surface area (S_{BET}), total pore volume and pore size distribution (PSD) of the samples were obtained from N_2 adsorption-desorption isotherms at 77 K using an automatic adsorption apparatus (Autosorb-1, Quanta Chrome). Prior to obtaining measurements, the samples were outgassed at 100 °C under an N_2 flow for 2 h at a pressure between 10^{-5} and 10^{-6} Torr. The S_{BET} values of the samples were calculated by the Brunauer, Emmett and Teller (BET) method using adsorption isotherms in the range of $0.05 \leq$ relative pressure $(p/p_0) \leq 0.30$. The total pore volume of the samples was calculated from the volume of adsorbed N_2 at $p/p_0 = 0.99$. The PSDs of the samples were calculated by the Barrett, Joyner and Halenda (BJH) method. The t-plot method was used to calculate the micropore volume and external surface area. The surface structure and particle size of the synthesized bifunctional carbonaceous materials were observed initially using scanning electron microscopy (SEM, QUANTA 200).

2.3 Phenol removal in the dark

0.1 g of the prepared composite carbon sample was added to each phenol solution (100 ml), which had various concentrations from 20 to 1000 $mg \cdot L^{-1}$. The bottles were sealed with

Table 1 Surface area and pore structure parameters of the prepared bifunctional carbonaceous materials $x\mathrm{TiO_2/AC}$ (x, mass ratio of $\mathrm{TiO_2}$)

Item	AC	$10\mathrm{TiO_2/AC}$	$30\mathrm{TiO_2/AC}$	$50\mathrm{TiO_2/AC}$	$70\mathrm{TiO_2/AC}$
Measured $S_{\mathrm{BET}}/\mathrm{m^2 \cdot g^{-1}}$	803.8	737.5	584.24	449.96	277.93
$x \cdot S_{\mathrm{BET\text{-}AC}}/\mathrm{m^2 \cdot g^{-1}}$	–	723.4	562.7	401.9	241.4
$y \cdot S_{\mathrm{BET\text{-}TiO_2}}/\mathrm{m^2 \cdot g^{-1}}$	–	4.4	13.2	22.0	31.2
[a]Theoretical $S_{\mathrm{BET}}/\mathrm{m^2 \cdot g^{-1}}$	–	726.8	575.9	423.9	272.6
Micropore area/$\mathrm{m^2 \cdot g^{-1}}$	525.6	476.75	369.17	149.46	147.42
External surface area/$\mathrm{m^2 \cdot g^{-1}}$	278.2	260.78	215.07	100.50	120.51
Total pore volume/$\mathrm{cm^3 \cdot g^{-1}}$	0.59	0.56	0.54	0.47	0.41
Micropore volume/$\mathrm{cm^3 \cdot g^{-1}}$	0.29	0.25	0.19	0.07	0.07
Mesopore volume/$\mathrm{cm^3 \cdot g^{-1}}$	0.30	0.31	0.35	0.40	0.34
Average pore size/nm	2.95	3.06	3.72	4.37	6.10

[a]Theoretical $S_{\mathrm{BET}} = xS_{\mathrm{BET\text{-}AC}} + y \cdot S_{\mathrm{BET\text{-}TiO_2}}$, x and y are the mass ratios of AC and $\mathrm{TiO_2}$, respectively

paraffin film and then shaken at 25 °C for 48 h. The concentration of phenol after adsorption was determined spectrophotometrically using a TU-1900 UV spectrometer. The adsorbed amount of phenol was calculated according to the change in phenol concentration.

2.4 Phenol removal under UV irradiation

Phenol removal under UV irradiation was carried out in a cylindrical quartz photoreactor (275 ml) using phenol as the model pollutant. An 8 W high pressure mercury lamp ($\lambda_{\max} = 365$ nm) was positioned inside the reactor as the irradiation source. The carbon sample or pure $\mathrm{TiO_2}$ (1.0 g) was added to an aqueous solution of phenol (250 mL, 50 $\mathrm{mg \cdot L^{-1}}$). The suspension was magnetically stirred in the dark for 30 min and then the lamp was turned on. Samples of 5 ml were collected every 20 min from the suspension which immediately centrifuged and measured spectrophotometrically to determine the phenol concentration.

2.5 Recycling performance test

When the removal test under UV irradiation was finished, the reaction mixture containing the prepared carbon was allowed to settle, then the supernatant solution was removed by decantation and the remaining carbon was reused for further phenol removal test under UV illumination.

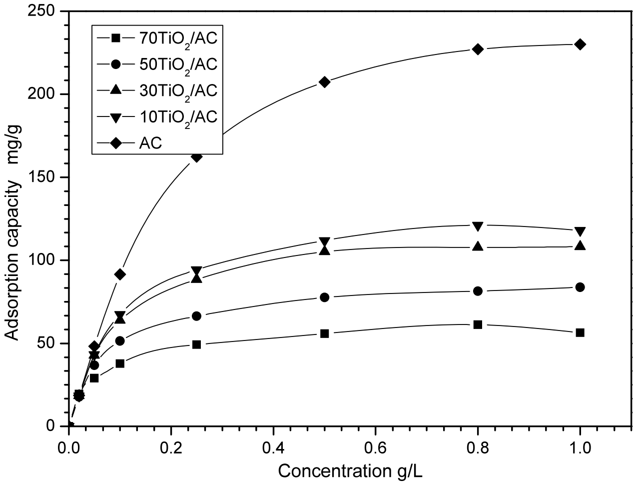


Fig. 1 Phenol removal by the bifunctional carbons and pure AC in the dark

3 Results and discussion

3.1 Removal of phenol in the dark

For the prepared functional carbon composites, AC has a well-developed pore structure and strong adsorption capacity, so it acts as a center where organic molecules adsorb. Illuminated $\mathrm{TiO_2}$, which decomposes the organic molecules and act as a decomposition center (Liu and Sun 2006; Liu et al. 2003, 2007; Haick and Paz 2003). Therefore, the removal of organic pollutants over the prepared functional carbon was realized by the combination of adsorption photocatalytic degradation (Yu et al. 2002).

Considering the photoactive nature and lower porosity of $\mathrm{TiO_2}$, the removal of phenol in the dark using the prepared functionalized carbon was realized through adsorption by AC (Torimoto and Okawal 1997). The corresponding isotherms are presented in Fig. 1. The phenol adsorption data for all of the carbon samples fit the Langmuir isotherm well and exhibited type I behavior according to the IUPAC classification (Fig. 2). This may be an indicator that a single layer of phenol molecules was chemically adsorbed on the AC. The Langmuir isotherm constants were calculated using the following equation and the val-

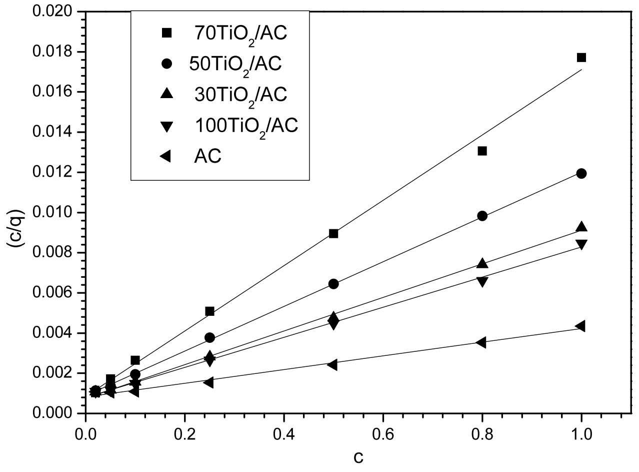


Fig. 2 Adsorption of phenol on the bifunctional carbons and pure AC treated by Langmuir equation

ues obtained are presented in Table 1 (Shin et al. 2007; An and Zeng 2003).

$$\frac{c}{q}=\frac{c}{q_\mathrm{m}}+\frac{1}{q_\mathrm{m}k_\mathrm{L}} \tag{1}$$

where q_m and k_L are the Langmuir constants determined from the slope and intercept of the plot, respectively, and indicate the maximum adsorption capacity (mg·g^{-1}) of carbon and energy of adsorption, respectively. c and q are the equilibrium concentration and equilibrium amount of adsorbed material, respectively. The adsorption capacity of the prepared functionalized carbons was lower than that of AC$_\mathrm{raw}$ (293.255 mg·g^{-1}), as shown in Table 1. As expectation, with the TiO$_2$ content increased, the maximum adsorption capacity decreased gradually due to the less porous characteristic of titania.

3.2 Removal of phenol under UV irradiation

The results for phenol removal under UV illumination are presented in Fig. 3. It is clear that the prepared functional carbons with various TiO$_2$ to AC mass ratios exhibited different phenol removal percentage. It can be seen from Fig. 3, phenol removal capacity increased was proportional to the mass ratio of AC content during the dark adsorption (prior to the light was turn on). However, the case was conversed when the UV light was turn on. Kinetic analysis showed that phenol removal fitted pseudo-first-order kinetics well (Fig. 4). 50TiO$_2$/AC exhibited the highest phenol removal efficiency. For 70TiO$_2$/AC, due to the low AC content and high initial phenol concentration, its phenol removal capability was much lower than other functionalization carbon samples. For the same reason, it clear that the activity of pure TiO$_2$ was even lower than 70TiO$_2$/AC, after 180 min runs, only 70.3% of phenol was removed. In contrast, 10TiO$_2$/AC removed the highest amount of phenol; however, this was mainly through physical adsorption rather than decomposition by TiO$_2$, the result of further recycling performance demonstrated this.

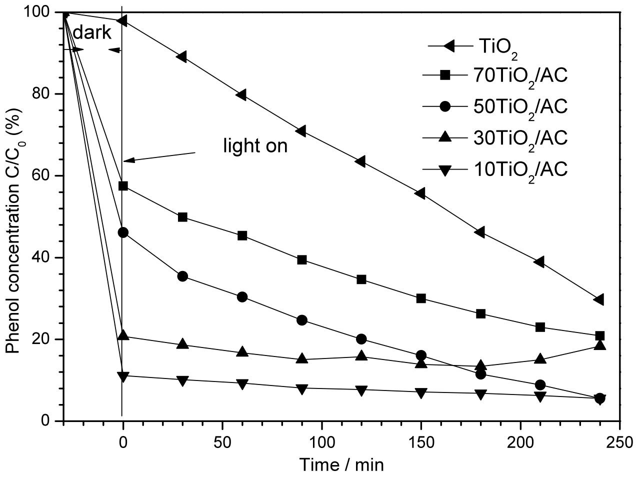


Fig. 3 Phenol removal by the bifunctional carbons under UV irradiation

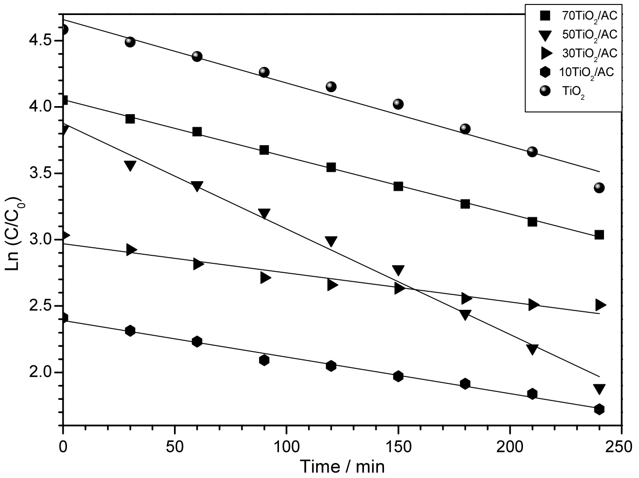


Fig. 4 Kinetics of phenol removal by the bifunctional carbons under UV irradiation

3.3 Recycling performance of 50TiO$_2$/AC

To determine the recycling performance of the prepared carbon, it was used several times to remove phenol under UV illumination. Between each phenol removal cycle, the carbon sample was separated and then used for further cycles without any treatment. The results for the recycling performance revealed that suitable mass ratio of AC to TiO$_2$ is essential for the organics removal capability of the prepared carbons. The recycling performance curve of 50TiO$_2$/AC and pure AC are shown in Table 2. It can be seen that the phenol removal capacity of 50TiO$_2$/AC decreased slowly as the number of cycle increased; 81.98% removal of phenol could still be achieved within 120 min after 10 cycles. In contrast,

Table 2 Parameters obtained from curve-fitting of the Langmuir equations to the experimental data obtained for the adsorption of phenol by the bifunctional carbon samples

Adsorbent	Langmuir		
	q_m	k	r^2
AC_{raw}	293.255	0.41	0.9473
$10TiO_2/AC$	133.51	0.94	0.9581
$30TiO_2/AC$	119.62	1.1	0.9937
$50TiO_2/AC$	89.92	1.26	0.9926
$70TiO_2/AC$	61.46	1.91	0.9914

Table 3 Total phenol removal capacity of $50TiO_2/AC$, AC and TiO_2 after 10 cycles

No. of cycles	Phenol removal capacity mg/g		
	$50TiO_2/AC$	AC	TiO_2
1	49.84	46.62	35.15
2	47.2	24.88	20.27
3	46.48	14.00	16.32
4	43.96	1.52	2.40
5	43.72	–	
6	43.24	–	
7	42.96	–	
8	42.56	–	
9	42.44	–	
10	40.96	–	
Total	443.36	87.02	74.14

pure AC removed only 49.12% of the phenol when recycled twice, and just 3.35% after 4 cycles, almost completely losing its removal capacity. Also, the result shown as Table 2 revealed that the activity of TiO_2 lost greatly, only 4.8% phenol could be removed within 180 min after 4 photocatalytic runs. This indicating that some deactivation of TiO_2 was significantly. An adsorption capacity test of $50TiO_2/AC$ in the dark showed that it adsorbed 40.76% as much as the original material after 10 runs. This may be caused by the generation of some strongly adsorbed intermediates occupying AC adsorption sites.

The amount of phenol removed by $50TiO_2/AC$ and AC from 250 mL aqueous phenol solutions with a concentration of 50 $mg{\cdot}L^{-1}$ over 10 cycles is summarized in Table 2. The total removal capacity by $50TiO_2/AC$ was 443.36 $mg{\cdot}g^{-1}$, which is almost 5 times of that pure AC and 6 times of that pure TiO_2. The high pollutant removal capacity of the bifunctional carbonaceous material makes it promising for the effective treatment of water polluted with a small amount of organic compounds.

3.4 SEM analysis

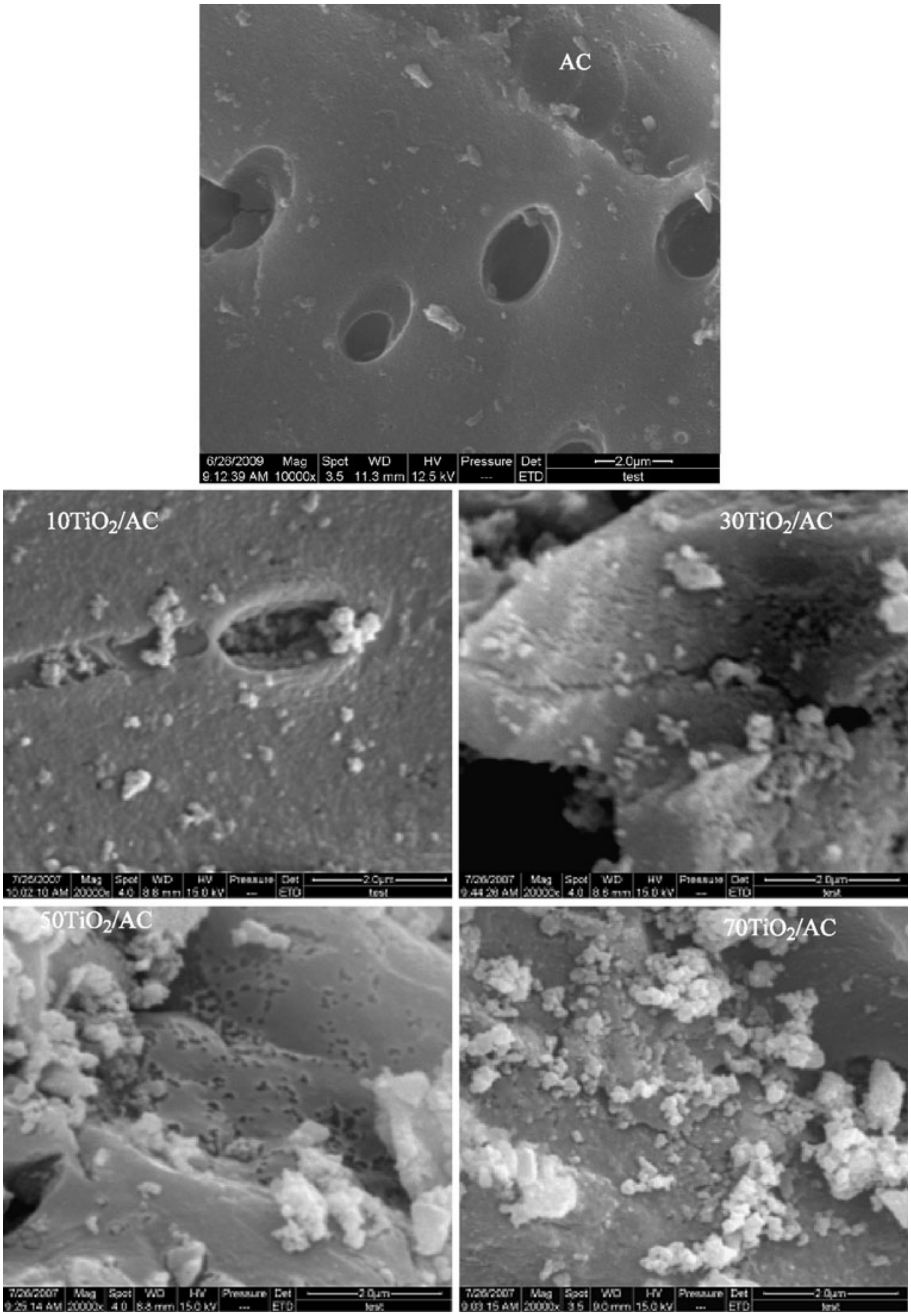
Typical SEM images of the prepared functionalized carbons and pure AC are shown in Fig. 5. The particle size of TiO_2 prepared by sol-gel method ranged from 13 to 16 nm (Liu and Sun 2006; Matos et al. 1999), which is much larger than the micropores of AC (<2 nm). Therefore, according to the pore size sieving effect of physical adsorption, TiO_2 particles cannot access the micropores of AC. It can be seen from Fig. 5 that the original morphology of AC was maintained after functionalization. From $10TiO_2/AC$ to $70TiO_2/AC$, as the TiO_2 content increased, the coverage area and agglomerate size of TiO_2 increased from 200 nm to 1000 nm. For all samples, TiO_2 particles were not uniformly distributed on the AC surface; instead they were mainly located at the entrance of the surface macropores. Also, part of titania entered into these pores. However, it can be seen that most of macropores were not blocked completely.

3.5 Pore structure analysis

Figure 6 shows the adsorption–desorption isotherms of N_2 at 77 K for the bifunctional carbons and pure AC. The volume of adsorbed nitrogen decreases in the order AC > $10TiO_2/AC > 30TiO_2/AC > 50TiO_2/AC > 70TiO_2/AC$. Textural properties obtained from the experimental isotherms are presented in Table 3. For pure AC, the nitrogen adsorption–desorption isotherms are of Type I, the slope and height of the isotherms at low relative pressures of nitrogen indicate the presence of micropores, which was confirmed by the micropore volume of 0.29 $cm^3{\cdot}g^{-1}$, as shown in Table 1. With respect to the high relative pressure range, different kinds of behavior were observed depending on the mass ratio of TiO_2. Different with the type I of pure AC, $50TiO_2/AC$ and $70TiO_2/AC$ displayed Type IV isotherms with a broad hysteresis loop, which indicates that the pores were mainly "bottleneck" mesopores, which corresponds to pores with narrow entrances and wider internal areas (Matos et al. 1999). This result also consistent with SEM observation, that part of TiO_2 particles entered the macro and mesopores.

As the TiO_2 content increased, the micropores disappeared gradually from AC, as confirmed by the decrease in the micropore volume of 0.29 $cm^3{\cdot}g^{-1}$ of pure AC to 0.07 $cm^3{\cdot}g^{-1}$ of $70TiO_2/AC$ (Table 1). This may be the main reason for the significant decrease in the S_{BET}. However, it is interesting to note that compared with pure AC, the total pore volume of the prepared composites decreased slightly while the mesopore volume increased (except in $70TiO_2/AC$). Also, the measured S_{BET} was much larger than the theoretically calculated S_{BET}. This may due to the reactivation at 550 °C produced some new pores, which helped to counteract the blocking effect of TiO_2. Overall, the total pore volume is affected less than might be expected.

Fig. 5 SEM images of the bifunctional carbons and pure AC



Pore size distribution (PSD) of pure AC and $10TiO_2$/AC, $30TiO_2$/AC, $50TiO_2$/AC and $70TiO_2$/AC were calculated using the BJH method (Fig. 7). It is clear that the size distributions of the prepared functionalized carbon shifted gradually toward higher size. As the content of TiO_2 increased, the proportion of mesopores in the composites increased. For $70TiO_2$/AC, a maximum value of 59 Å was observed *versus* that of pure AC of 17 Å.

4 Conclusions

In conclusion, bifunctional carbon containing TiO_2 particles adsorbed on AC that possess the dual abilities of adsorption and photocatalysis can be prepared by the sol-gel technique combined with hydrothermal treatment and reactivation method. The TiO_2 particles were located mainly at the entrance of the surface macropores of AC. As the TiO_2

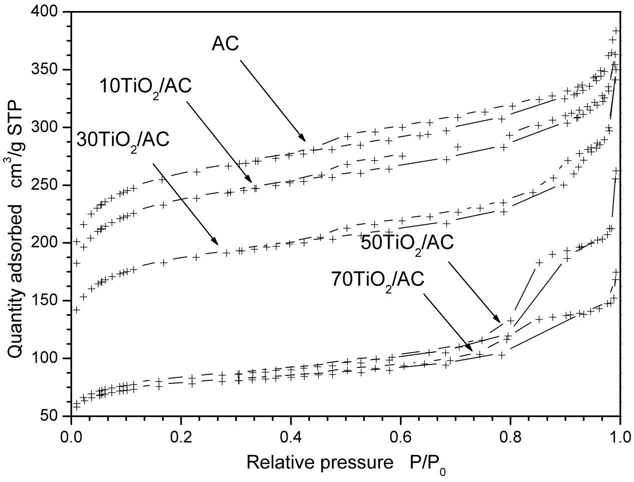


Fig. 6 N_2 adsorption-desorption isotherms of the bifunctional carbons and pure AC at 77 K

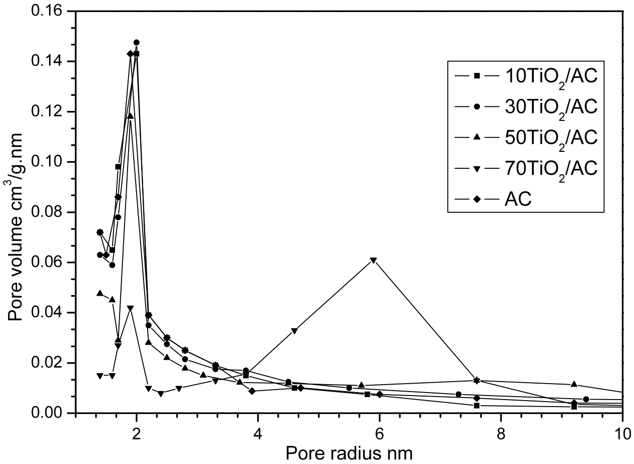


Fig. 7 Pore size distribution of bifunctional carbons and pure AC

content increased, the coverage area of TiO_2 on the AC surface and the size of the TiO_2 aggregates increased. The results of low temperature nitrogen adsorption isotherm experiments showed that the specific surface area decreased and the number of mesopores increased as the TiO_2 content increased in the functional carbonaceous material. A bifunctional carbonaceous material with a suitable AC to TiO_2 ratio exhibited high phenol removal capability. Under UV light irradiation, both adsorption and photocatalytic degradation of the pollutant occurred. When there was insufficient light, phenol removal by the prepared bifunctional carbonaceous material was mainly through adsorption by AC. 50TiO_2/AC exhibited the highest phenol removal capability under UV light irradiation. The total phenol removal capacity of 50TiO_2/AC was 443.36 mg·g^{-1} after 10 cycles, which is almost 5 times that of AC and 6 times of that pure TiO_2. The prepared bifunctional carbons possess the advantages of high pollutant removal capability and good recyclability, making them promising for the efficient treatment of lightly polluted aqueous solutions.

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