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Polysulfone-Activated Carbon Hybrid Particles for the Removal of BPA

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Abstract: Polysulfone-activated carbon (PSF-AC) hybrid particles are prepared using a liquid–liquid phase separation technique. The PSF-AC hybrid particles are then used for the removal of bisphenol A (BPA) from its aqueous solutions. The PSF-AC hybrid particles have similar structure as the polysulfone beads; both of them have a skin layer outside and a porous structure inside. The adsorption ability increased significantly when activated carbons were embedded into the particles, and the hybrid particles showed relatively more adsorption ability when the activated carbon content was 50%. The adsorbed BPA to the hybrid particles could be effectively removed by ethanol, which indicates that the hybrid particles can be reused. It is easy to prepare the particle column using these kinds of hybrid particles and the hybrid particle column could more effectively remove BPA. The results suggested that the PSF-AC hybrid particles have a potential to be used for the removal of BPA in environmental application.

Keywords: Activated carbon, polysulfone, hybrid particles, bisphenol A, liquid–liquid phase separation

INTRODUCTION

There currently is an increasing interest in endocrine disruptors, since people have faced severe endocrine disruptor problems (1). Some methods to treat

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endocrine disruptors in polluted water and wastewater are essential. Recently, it has been reported that hydrophobic porous polymer membranes (2), hydrophobic porous polymer beads (3), water-insoluble DNA and DNA matrix materials (4–6) could selectively remove endocrine disruptors. Hydrophobic materials could bind endocrine disruptors having high octanol–water distribution coefficients, and DNA matrix materials selectively bind endocrine disruptors with a planar structure. Another method is the degradation of endocrine disruptors. Bacteria distributed in river water can degrade some kinds of the endocrine disruptors (7).

Adsorption is an effective and convenient method to remove endocrine disruptors. Activated carbons are well known adsorbents, and are widely used in industry for the separation, purification, and recovery processes, such as metal ions removal (8, 9) and phenol adsorption (10). Any inexpensive materials with high carbon content can be used as a raw material for the production of activated carbon. However, when the activated carbons are used directly, especially in liquid medium, fine carbon particles together with soluble organic compounds in the carbons will be eluted. Thus, when the activated carbons were used as absorbents to remove endocrine disruptors, particularly when used in juice purification and drinking water treatment, the activated carbons should be coated with a polymer film outside to prevent the elution of fine carbon particles and soluble organic compounds.

Bisphenol A (BPA; 4,4-isopropylidenediphenol; CAS Registry No. 80-05-7) is one of the endocrine disruptors and is made by combining acetone and phenol. It has been reported that BPA has an estrogenic activity (11) and an acute toxicity to aquatic organisms (12). BPA could be adsorbed onto carbonaceous materials produced from wood chips as organic waste (13), and could be removed by hydrophobic porous materials. Water-insoluble DNA could selectively accumulate endocrine disruptors; however it could not adsorb BPA due to the non-planar structure of BPA.

In the present study, we prepared polysulfone (PSF)-activated carbon hybrid particles, and investigated the adsorption characterization to bisphenol A onto the hybrid particles. We also prepared hybrid particle columns and tested the performance of the columns.

MATERIALS AND METHODS

Preparation of the Hybrid Particles

Polysulfone (PSF, BASF Aktiengesellschaft), was dissolved in N,N-dimethyl acetamide (DMAc, Chengdu Chemical Reagent, Inc., China) to obtain the PSF solution. Activated carbons (AC, Chengdu Chemical Reagent, Inc., China) were then added into the PSF solution at certain proportions, and stirred

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sufficiently to obtain the PSF-DMAC-AC suspension solution. All the materials were used without further purification.

The solution was injected into distilled water by using a 1.5 mm-diameter glass burette to obtain polysulfone-activated carbon hybrid particles. The injection speed was controlled at 60–100 drops min. The air gap from the burette to the water was 5–10 cm. The hybrid particles were then incubated in water for over 24 h to elute the solvent from the particles

Characterization of the Hybrid Particles

The porosity of the hybrid particles was calculated from the density of the polymer and the activated carbon and the weight change before and after drying, using the following formula:

$$\text{Porosity (P)} = \frac{(W_B - W_A)/\rho_W}{W_A(1 - C\%)/\rho_P + W_A \times C\%/\rho_c + (W_B - W_A)/\rho_W} \times 100\%$$

where W_B is the weight of the hybrid particles before drying, g; W_A is the weight of the hybrid particles after drying, g; ρ_W is the density of water, $\rho_W = 1.0\,g/cm^3$; and ρ_P is the density of the polysulfone, $\rho_P = 1.24\,g/cm^3$; ρ_c is the density of activated carbon, $\rho_c = 2.2\,g/cm^3$. C% is the mass percents of the activated carbon in the PSF-AC system.

For SEM observation, the hybrid particle sample was dried at room temperature. Then cut with a single edged razor blade, attached to the sample supports and coated with a gold layer. The SEM images were recorded using an S-2500C microscope (Hitachi, Japan).

Accumulation and Removal of BPA

Bisphenol A (BPA, $(CH_3)_2C(C_6H_4OH)_2$, $M_w = 228.29$) was obtained from the Shanghai Chemical Reagent, Inc., China. As BPA show very low solubility in water. Therefore, we dissolved BPA in ethanol, and then diluted it in distilled water. The concentrations were controlled at 50, 100, 200, 300, and 500 μM. The accumulation of BPA was examined by the following procedures: the hybrid particles (about 40 mg in dry weight) were put into the respective solutions (7 ml) of different BPA concentrations and incubated at room temperature. The BPA concentrations were determined at different time intervals by using an UV-VIS spectrophotometer U-200A at the wavelength of 276 nm.

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The removal ratio and the adsorbed amount of the BPA were calculated by using the following formulas:

$$R_t(\%) = \frac{C_0 - C_t}{C_0} \times 100\%$$

$$m_t = \frac{(C_0 - C_t) \times V \times M \times 10^{-3}}{m} \times 100\%$$

where R_t is the removal ratio of BPA by the particles at the time t, %; C_0 is the original concentration of the BPA solution, μM; C_t is the concentration at the time t, μM; m_t is the adsorbed amount of BPA to the hybrid particles per gram at the time t, mg/g; V is the volume of the BPA solutions, ml; M = 228.29 g/mol, is the molecular weight of BPA; m is the weight of hybrid particles, g.

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Desorption Experiments

After the sorption experiments, the PSF-activated carbon hybrid particles were immersed into 10 ml of 2-propanol in a jar with a lid. Desorption experiments were carried out for 5 h using a shaker at a speed of 50 rpm and at room temperature. The hybrid particles were removed from the solutions, and the 2-propanol solutions were then diluted by distilled water. The concentration of BPA was determined using a spectrophotometer U-200A at 276 nm. The desoption ratio (R) of the BPA in desorption method was obtained as:

$$R_d(\%) = \frac{m_d}{m_a} \times 100\%$$

where m_d is the amount of BPA desorbed in the solution, m_a is the amount of BPA adsorbed onto the hybrid particles, which was calculated from the sorption experiments. Ethanol and 2% sodium dodecylsulfate (SDS) were also used in the desorption experiments.

Removal of BPA by the Hybrid Particle Column

The hybrid particles (8 ml) were placed in a 10 ml polypropylene syringe, and the length of the mobile phase was approximately 5 mm. The aqueous BPA solution (300 ml, 100 μM) was applied to the hybrid particle column at a flow rate of 2.5 ml/min; the concentration of the eluted solution was then determined by the spectra of the eluted solution. The eluted solution was reapplied, and this process was repeated four times. The removal ratio of each circle was calculated using the following formula:

$$R = (C_{I,n} - C_{F,n})/C_I$$

where $C_{I,n}$ is the initial concentration of BPA for the n times circulation, n = 1, 2, 3, 4 and 5, respectively; $C_{F,n}$ is the final concentration after the n times circulation, n = 1, 2, 3, 4 and 5; and C_I is the initial concentration of BPA, $C_{I,} = C_{I,1}$.

RESULTS AND DISCUSSION

Characterization of the Hybrid Particles

To prepare polysulfone (PSF) porous particles, a liquid–liquid phase separation method was used as described in our earlier report (5). When PSF solution was injected into water, liquid–liquid phase separation caused by the rapid exchange of the solvent and water occurred, a skin layer formed due to the rapid phase separation, under which was a finger-like structure, as shown in Fig. 1a. For preparing polysulfone-activated carbon (PSF-AC) particles, we also used the liquid–liquid phase separation technique. When the PSF-AC solution was dropped into water, liquid–liquid phase separation also occurred due to the exchange of solvent and water. A skin layer was found outside the PSF-AC particle, under which followed the finger-like structure, and many pores presented in the spheres, and a very big hole at the center of the particle. With the completion of the exchange between the solvent and the non-solvent, the porous hybrid particles were prepared, and the activated carbons were embedded in the particles, as shown in Fig. 1b.

As shown in Fig. 1, both of the PSF and PSF-AC hybrid particles have skin layers outside and porous structures inside. These porous structures led to very large specific surface areas, which were essential for using as absorbents. The skin layer of the PSF-AC hybrid particle was formed by the PSF matrix, thus restricted the AC to elute from the hybrid particles. The size of

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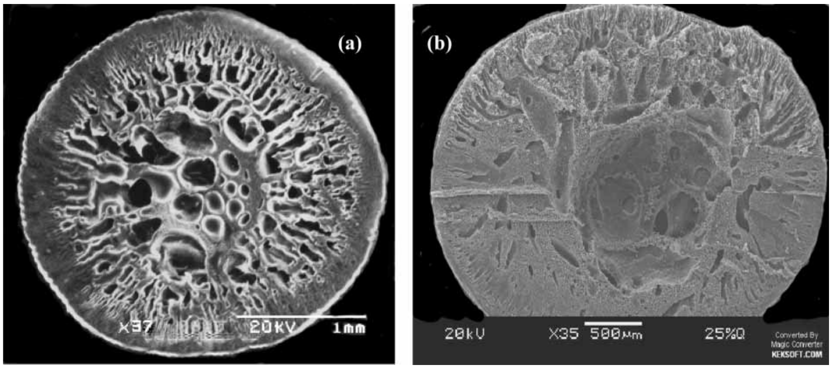

Figure 1. Cross-section of the porous PSF bead (left) and polysulfone-activated carbon hybrid particle (right, 25% active carbon). (a) Voltage: 20 kv; Magnification: ×37; (b) Voltage: 6.0 kv; Magnification: ×35.

the activated carbon particles is about 10 μm in our study. Figure 1 does not show any carbon particles, since the carbon particles were embedded in the polymer, and also the magnification for the SEM picture is small. We could find many small particles when the magnification in the SEM pictures was larger than 1000 times.

The porosity of the particles was calculated from the formula 1 as shown in Table 1.

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Effect of PSF Concentration and Activated Carbon Content on the BPA Removal

Firstly, we examined the effect of PSF concentration used to prepare the hybrid particles on the BPA removal, as shown in Fig. 2, the ratio of PSF to AC was controlled at 1 : 1. The removal ratio increased with time, and reached an equilibrium value after about 24 hours. The equilibrium removal ratios were 84%, 78%, and 58% for the particles prepared from the PSF concentration of 10%, 12.5%, and 15%, respectively. It is clear that with the increase of PSF concentration, the removal ratio decreased. Both the BPA removal ratio and the removal amount increased when a low PSF concentration were used, it is presumably caused by the large pore size in the hybrid particles and the thinner skin layer, thus the BPA could enter the hybrid particles with greater ease. However, it is difficult to prepare the hybrid particles using a very low PSF concentration (lower than 8%). When 8% PSF solution was used to prepare hybrid particles, the solution could not form particles, but became membranous. On the other hand, when high concentration was used (higher than 18%), the particles had long tails, and the BPA removal ratios were very small. Hereafter, the porous hybrid particles were prepared from 10% PSF solution without further indication.

As mentioned above, the skin layer affected the BPA removal ration. Of course, the hybrid beads with a more open structure would have better adsorption without loosing the carbon. We could prepare hybrid particles with a more open structure by adding additives and changing the non-solvents. When 1,2-propylene glycol was added into the polymer solution, and a mixture solution of DMAC and water solution was used as the precipitation medium instead of distilled water, a more open structure was obtained, and the BPA removal ratio increased.

Table 1. Porosity and diameter of the PSF-AC hybrid particle

Proportion	25%AC	50%AC	75%AC
Porosity (%)	83.3	79.7	71.7
Diameter (mm)	2.7 ± 0.5	2.6 ± 0.5	2.5 ± 0.4

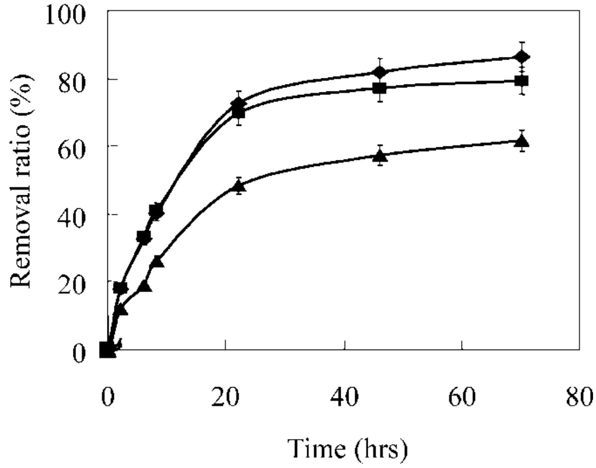

Figure 2. Effect of PSF concentrations on the BPA removal. Particles: 40 mg; AC% = 25%; BPA solution: 7 ml, 500 μM; PSF concentration used: –●– 10%; –■– 12.5%; –◆– 15%. Data are expressed as the mean ± SD of three independent measurements.

Figure 3 shows the effect of the AC content in the particles on the removal ratio of BPA. The BPA removal ratios by the polysulfone-activated carbon hybrid particles were much higher than that by the pure PSF porous particles, but lower than that by the pure activated carbons. Polysulfone is a highly hydrophobic polymer material; the adsorption of BPA by the PSF particles is caused by the hydrophobic interaction between the PSF and the BPA (3), the porosity and the high specific surface area of the PSF particle, which is about 50 m^2/g (14). The specific surface of the activated carbons is about 800m^2/g, which is far larger than that of the PSF porous particles; also the particle size of the AC is smaller. Therefore, the removal ratio by the pure AC was the largest.

For the polysulfone-activated carbon hybrid particles, the activated carbons were embedded under the skin layer of hybrid particles. Thus, the removal ratios of BPA by the PSF-AC particles were among the PSF porous particles and the activated carbon. And with the increase of the AC content to 62.5% in the hybrid particles, the removal ratio of BPA increased. Furthermore, there is no significant difference between the particles when the AC contents were 50% and 62.5%. With the increase of AC% content in the particles, the total specific surface areas increased, but the porosity of hybrid particles decreased as shown in Table 1. Hereafter, the porous hybrid particles used in this study were prepared from the 10% PSF solution and ratio of polysulfone and activated carbon was controlled at 50 : 50.

The adsorption capability of the hybrid particles is lower than that of activated carbons. However, the hybrid particles can be regarded as good

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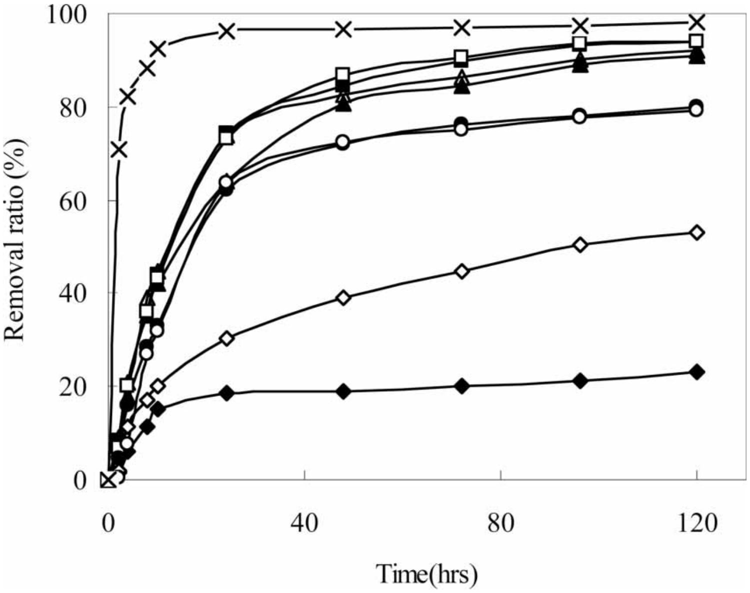

Figure 3. Effect of AC content on the BPA removal PSF concentration: 10%; BPA solution: 7 ml, 500 μM; Particles: 40 mg; AC%: –☐– 0%; –☐– 12.5%; –●– 25%; –○– 37.5 %; –■– 50%; –□– 62.5%; –☐– 75%; –▲– 87.5%; – × – 100%. Duplicated experiment showed similar results.

adsorbents. When activated carbon powders are used directly, the solutions need centrifugation to separate the activated carbons. Also, fine carbon particles and soluble organic compounds in the carbons will be eluted into the purified solutions, and this is very harmful when the activated carbons are used to purify juice and drinking water. As we known, polyphenols, brown color, and other harmful compounds are present in juice (15). In this study, the hybrid particle has a skin layer to prevent fine carbon particles elute from the particle, and the polysulfone show outstanding oxidative, thermal, and hydrolytic stabilities, which allow the hybrid particles could be used at many conditions as adsorbents. Furthermore, it is easy to prepare hybrid particle column for use.

BPA Adsorbed Amounts by the Hybrid Particles

Figure 4 shows the effect of BPA concentration on the adsorbed BPA amount by the 50%AC hybrid particles. The BPA adsorbed amount increased with time, and reach an adsorption equilibrium value gradually. Also, with the increase of BPA concentration, the adsorbed amount increased.

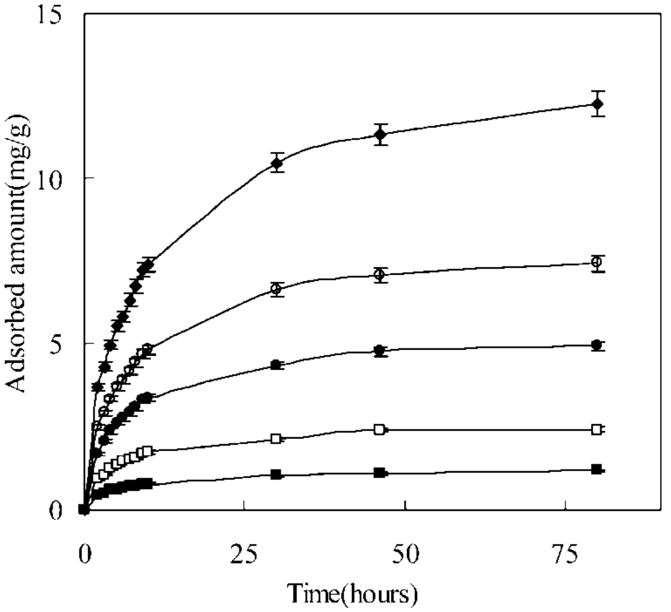

Figure 4. Effect of BPA concentrations on the adsorbed BPA amount. Particles: 40 mg; BPA solution: 7 ml; –■– 50 μM; –□– 100 μM; –●– 200 μM; –○– 300 μM; –◆– 500 μM. Data are expressed as the mean ± SD of three independent measurements.

The relationship of the adsorbed amount and adsorption time could be expressed using the following formula:

$$A_t = A_0 e^{a/t^2 - b/t}$$

where A_t is adsorbed amount at time t, mg/g; A_0 is equilibrium adsorbed amount, mg/g; a and b are constants, related to the BPA concentrations and particles; t is the adsorption time.

We take reciprocal of adsorption time (t), and the natural logarithm of adsorbed amount (mg/g), and then the constants a and b were obtained, as shown in Table 2. The adsorbed amount and time accorded with this formula very well at different BPA concentrations. It is clear that the constants a and b increased for all the three kinds of particles with the increase of BPA concentration, and then the equilibrium adsorbed amounts increased. The constants a and b measured the interaction between the surface of the hybrid particles and the adsorbents, and the adsorption capacity, and further study is proceeding.

Figure 5 shows the effect of AC content in the particles on the adsorbed amount of BPA. With the increase of AC content, the BPA adsorbed amount to the hybrid particles increased. The equilibrium adsorbed amounts were

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Table 2. Constant values for different concentrations

AC content	Constant	50 μM	100 μM	200 μM	300 μM	500 μM
25%	a	3.01	3.22	3.53	4.02	4.67
	b	3.22	3.53	4.06	3.54	4.53
50%	a	3.67	3.76	4.27	5.27	6.36
	b	3.85	3.85	4.30	4.87	5.60
75%	a	2.54	2.67	3.13	3.30	3.51
	b	3.05	3.11	3.50	4.22	5.01

about 5.5 mg/g and 150 mg/g for the polysulfone beads and the activated carbons, respectively. For the 50%AC and 75%AC hybrid particles, the amounts were 65 mg/g and 69 mg/g, respectively, and there is no significant difference between them.

The relationship between AC% and equilibrium adsorbed amount is not a linear relation. Both the porous polysulfone beads and the activated carbons could adsorb BPA, when we prepared the hybrid particles, along with the proportion of AC content increased, the total specific surface area of the hybrid particles increased, however the porosity of the hybrid particles decreased. These induced above the results.

The desorption of BPA from the hybrid particles were also examined after the sorption experiments. The BPA could be easily removed from the pure polysulfone beads, and the removal ratio was nearly to 100%. However, for the hybrid particles, the removal ratio was about 80–85%; for the activated

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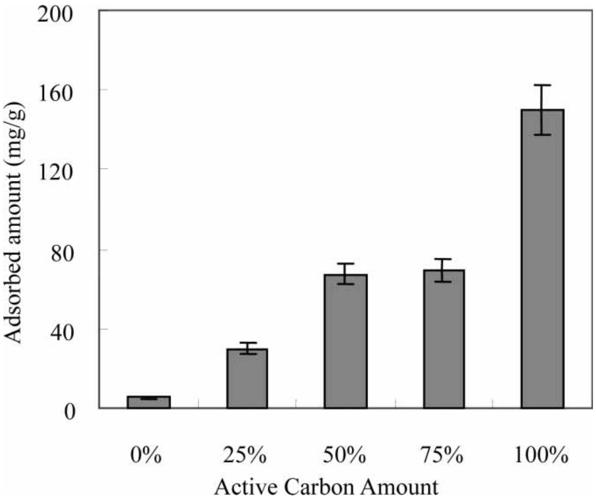

Figure 5. Equilibrium amounts of BPA adsorbed to AC-PSF microspheres. Data are expressed as the means ± SD of independent measurements. n = 3.

carbon the removal ratio was only about 76%. These suggested that the binding ability to the hybrid particles is higher than that to PSF. Most of the adsorbed BPA could be removed by immersing the particles into ethanol, which indicates that the particles could be reused. When 2% sodium dodecylsulfate (SDS) solution was used as the desorption medium, the removal ratio was only about 15%.

PSF is one of the most important polymeric materials and is widely used; PSF-based materials show outstanding oxidative, thermal and hydrolytic stability as well as good mechanical and film-forming properties. Thus the PSF-AC hybrid particles can be used in many extreme conditions, such as in boiling water, acid and alkaline conditions.

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Adsorption Isotherms of BPA

The adsorption isotherms of BPA at 298 K onto the hybrid particles containing various percent of activated carbons are shown in Fig. 6. The amount of BPA adsorbed onto the 50% AC particles was larger than that onto the other two AC content particles mentioned above.

The adsorption isotherms of bisphenol A were approximated by the Freundlich equation: $\log X = \log K + (1/N) \log C$, we approximated the adsorption isotherms of BPA by the Freundlich equation: where K and $1/N$

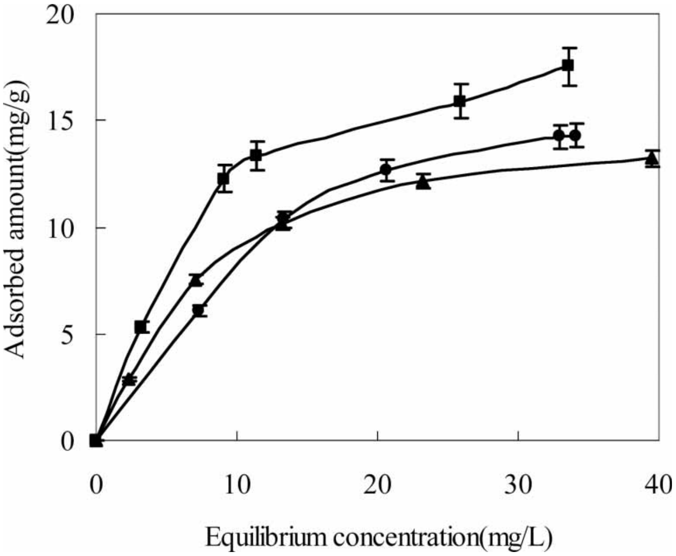

Figure 6. Adsorption isotherms of BPA onto the hybrid particles. Particles: 40 mg; AC%: –●– 25%; –■– 50%; –▲– 75%. Data are expressed as the mean ± SD of three independent measurements.

are the adsorption constants. The reciprocal of the slope ($1/N$) of the Freundlich plot measured the interaction between the surface of the hybrid particles and the adsorbents, while the intercept (K) of that was both the interaction and the adsorption capacity (11). The correlation coefficient and the constants, $1/N$ and K, of the Freundlich plot of BPA are shown in Table 3.

The Freundlich plots of the adsorption isotherms for BPA to the hybrid particles were not linear. The constants K for the hybrid particles and the activated carbons were far greater than the pure polysulfone beads. The constant $1/N$ of the pure polysulfone beads was similar to that of the 25%AC hybrid particles and the constant $1/N$ of the 50%AC hybrid particles was similar to that of activated carbons.

The adsorption disciplinarian of the hybrid particles is complicated when two or more materials are in the particles. When the constant K was larger, an increase in the affinity didn't cause a decrease in the $1/N$ constant. This result indicates that the affinity between BPA and hybrid particles is not similar to that between BPA and activated carbon (11). For the 75%AC hybrid particles, the interaction between BPA and the hybrid particles is greater than that of the 25% and 50%AC hybrid particles. Adsorption onto carbon whose surface is hydrophobic is mainly based on the London dispersion force, which is part of the van der Waals force. The more closely the adsorbent molecules in the pores are located to the surrounding pore walls, the higher will be the adsorption force (11). But the more AC content caused the lower porosity, thus the adsorption capability of the 75%AC hybrid particles is lower than that of the 50%AC hybrid particles. However, the adsorption capacity to BPA by the hybrid particles was greater than that by pure polysulfone beads but lower than that by activated carbons.

Removal of BPA by the Hybrid Particles Column

Since the hybrid particles could remove BPA from its aqueous solutions, we prepared the hybrid particles column to more effectively remove the BPA. Figure 7 shows the removal ratio of BPA by the hybrid particle column for each circulating time. At the first circulation, about 38% of the BPA were removed by the hybrid particle column; at the second circulation, about 18% of the BPA were removed. With the increase of circulating time, the

Table 3. Freundlich constants of adsorption for BPA

Samples	1/N	K (mg/g)
0%AC	0.60	0.35
25%AC	0.57	1.95
50%AC	0.80	2.11
75%AC	0.72	1.56
100%AC	0.85	13.40

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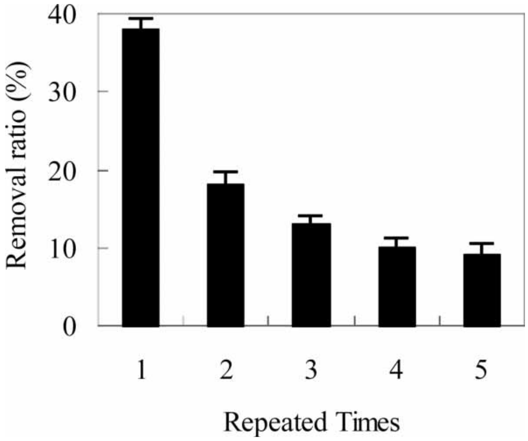

Figure 7. Removal ratio of BPA by the hybrid particles column each circulating time. Data are expressed as the mean ± SD of three independent measurements.

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removal ratio decreased rapidly. It is easy to understand from the formula $R = (C_{I,n} - C_{F,n})/C_I$ that each time the hybrid particles column remove part of the BPA from the solution, and the denominator C_I is the initial concentration of BPA, which is a constant in the experiments. Thus, the removal ratio of the BPA declines rapidly. In the formula, $C_{F,n} = C_{I,n+1}$, so the final removal ratio, which is the sum of the removal ratio for each circulating time, could be calculated as 90%. This result indicated that the hybrid particle columns could more effectively remove BPA compared to the hybrid particles themselves.

Furthermore, we investigated the effect of the flow rate on the removal of BPA. The concentration of the BPA in the eluted solution decreased when the flow rate of the particle column decreased. When the flow rate decreased from 2.5 ml/min to 0.5 ml/min, the BPA concentration in the eluate for the first circulation decreased from about 62 μM to about 36 μM, which mean that the removal ratio increased from about 38% to about 64%. This suggested that the high removal ratio could be obtained not only by reapplying the eluted solution but also by decreasing the flow rate.

CONCLUSION

It is concluded that hybrid particles containing polysulfone and activated carbon could be prepared by using a phase separation technique, which was induced by the liquid–liquid separation of the polysulfone solution, and the activated carbon was embedded in the particles. The hybrid particles have good adsorption capability to BPA. Though the adsorption efficiency is lower than that of the pure activated carbons, the hybrid particles have wide application for the purification of juice and drinking water. The adsorption capability and efficiency varied when the proportion of activated carbons changed. The adsorbed BPA could be removed by ethanol, and the particles could be reused. It is easy to prepare particle column using these kinds of hybrid particles and the hybrid particle column has an extremely effective capability for the removal of the BPA. All these suggested that the PSF-AC hybrid particles have a potential to be used for the removal of BPA in environmental applications.

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