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Polyethersulfone-Activated carbon (PES-AC) hybrid particles are prepared using a liquid-liquid phase separation technique. The PES-AC hybrid particles are then used for the removal of phenobarbital (PB) from its aqueous solutions. The adsorption ability of the particles increased significantly when activated carbon was embedded into the particles, and with the increase of the activated carbon content in the hybrid particles, the PB adsorbed amount increased. Also when the PB concentration increased, the adsorbed amount increased. The adsorption of PB to the hybrid particles belongs to the Freundlich adsorption. To more effectively remove PB, PES-AC particle columns were prepared; and the particle column showed more high removal efficiency for PB. The results suggested that the PES-AC hybrid particles have a potential to be used for the removal of PB, and may be used in blood purification.

Keywords active carbon; particles; phenobarbital; polyethersulfone

INTRODUCTION

There is a long record of applications of carbon based adsorbents for the removal of different toxins from patients' blood (blood detoxication) mainly by forcing blood flow through a bed of adsorbent, packed inside a column (hemoperfusion) (1). However, when activated carbon (AC) was used directly, fine carbon particles together with soluble organic compounds in the carbons will be eluted and non-compatibility with blood was observed (2). Thus, the AC should be coated with a polymer film. The first clinical treatment with microencapsulated carbon to hemoperfusion was semipermeable synthetic membrane coated adsorbents, such as nitrocellulose-albumin coated activated charcoal (1,3). The membrane coating of the adsorbent prevented the generation of charcoal particles from the adsorbent surface and improved the blood compatibility.

Phenobarbital (PB), a sleeping potion, is also the most widely used antiepileptic drug in the world (4). Toxic levels of the drug can profoundly suppress the central nervous system and potentially result in death or respiratory failure. As a result, deliberate overdose with PB constitutes a medical emergency (5). Hemodialysis and hemoperfusion have been used in critically ill overdosed patients to enhance the elimination of PB. Because of the high degree of protein binding, hemoperfusion is generally viewed as a more effective means for the removal of PB than is hemodialysis (1).

In our earlier reports (6,7), DNA-loaded porous polyethersulfone particles were prepared through a liquid-liquid separation method. The same technique can also be used in embedding powdered AC. Polysulfone (PSF)-Activated Carbon particles were prepared by using this method in our recent report (8), and the particles could adsorb and remove bisphenol A (BPA). In another study Laszlo K investigated the heterogeneity of activated carbon adsorption isotherms of phenol from dilute aqueous solutions, and the Dubinin-Astakhov adsorption-isotherm equation was used to evaluate the adsorption parameters (9).

As we know, polyethersulfone (PES) shows outstanding thermal and hydrolytic stabilities as well as good mechanical and film-forming properties. The PES membranes also show good biocompatibility, and are widely used in advanced separation technology and biomedical fields for artificial organs and medical devices, such as blood purification (10,11). Therefore, it is reasonable to predict that by embedding AC particles within PES, novel and useful adsorbents may be obtained. In the present paper, PES-AC hybrid particles are prepared using a liquid-liquid phase separation technique, and then used to remove PB from its aqueous solutions.

EXPERIMENTAL

Preparation of the PES-AC Hybrid Particles

Polyethersulfone-PES (BASF Aktiengesellschaft, Germany)-was dissolved in N,N-dimethyl acetamide-DMAC (Chengdu Chemical Reagent, Inc, China)-to obtain the PES solution. The powdered AC (Chengdu

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Chemical Reagent, Inc, China) was then added into the PES solution at certain proportions, and stirred over 10 minutes to obtain the PES-DMAC-AC solution. The proportions of powered AC for the particles were 0%, 30%, 50%, and 75%, respectively. All the materials were used without further purification.

The PES-AC hybrid particles were prepared using a liquid-liquid separation technique, as described in detail in our earlier report (6). The PES-DMAC-AC solutions were injected into water using a 1.5 mm-diameter glass burette to obtain PES-AC 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 particles were then incubated in water for over 24 h to elute the solvent from the particles.

Characterization of the PES-AC Hybrid Particles

For SEM observation, the particle was dried at room temperatures, 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).

A mercury porosimetry Model no. 1300 (Micromeritics, USA) was used to determine the specific surface area of the porous particles. The particles were dried at room temperature; about 0.1 g dried particles were used as samples. The data reported were obtained using Quantachrome Poremaster for Windows, Version 4.02.

The diameter and the porosity of the particles were calculated from the density of the polymer and the weight change before and after drying, using the following formulas:

Diameter(D) =

$$\left\{ \frac{6[W_A(1 - C\%) / \rho_P + W_A \times C\% / \rho_c + (W_B - W_A) / \rho_W]}{n\pi} \right\}^{1/3} \quad (1)$$

Porosity(P) =

$$\left\{ \frac{(W_B - W_A) / \rho_W}{W_A(1 - C\%) / \rho_P + W_A \times C\% / \rho_c + (W_B - W_A) / \rho_W} \right\}^{1/3} \times 100\% \quad (2)$$

$$\text{Pore volume (PV)} = \frac{n\pi D^3 \times P}{6W_A} \quad (3)$$

where W_B is the weight of the PES-AC hybrid beads before drying, g; W_A is the weight of the hybrid beads after drying, g; ρ_W is the density of water, $\rho_W = 1.0 \text{ g/cm}^3$; and ρ_P is the density of the PES, $\rho_P = 1.43 \text{ g/cm}^3$; ρ_c is the density of AC, $\rho_c = 2.2 \text{ g/cm}^3$; n is the number of the beads, $n = 60$; $C\%$ is the mass percent of the AC in the PES-AC system; D is the diameter of the particles, mm.

Accumulation and Removal of Phenobarbital by the Particles

The PES-AC hybrid particles were soaked in Tyrode buffer solution (pH=7.4), a solution mimicking the mineral composition and pH of blood, for over 24 hours before used in the PB adsorption experiments.

The accumulation of PB was examined by the following procedures: about 500 mg particles ($n = 60$) were put in 20 mL of PB aqueous solution $145 \mu\text{mol/L}$ and incubated at room temperature for 5 hrs. The amount of the PB in the solution was quantified by the absorption at 237 nm using a U-200A UV-vis spectrophotometer (Hitachi Co., Ltd., Tokyo, Japan).

The removal ratio (R_t) and the adsorbed amount (m_t) for the PB were calculated by using the following formulas:

$$R_t(\%) = \frac{C_0 - C_t}{C_0} \times 100\% \quad (4)$$

$$m_t = \frac{(C_0 - C_t) \times V \times M \times 10^{-6}}{m} \times 100\% \quad (5)$$

Where R_t is the removal ratio of the PB by the particles at time t , %; C_0 is the original PB concentration in the solution, $\mu\text{mol/L}$; C_t is the concentration at the time t , $\mu\text{mol/L}$; L ; m_t is the adsorbed amount of PB to the hybrid particles per gram at the time t , mg/g; V is the volume of the PB solutions, mL; $M = 232.24 \text{ g/mol}$, is the molecular weight of PB; m is the weight of the dried hybrid particles, g.

Accumulation and Removal of Phenobarbital by the Particle Column

Each kind of the PES-AC particles (about 4.0 g in dry weight) were placed in a 20 ml polypropylene syringe to prepare particle column, and the length of the mobile phase was approximately 80 mm. Two flow rates of PB solution were passed through the bed: 1.4 ml/min and 2.5 ml/min.

The removal of PB by the PES-AC particle column was examined by the following procedures. Method 1, the aqueous solution (10 ml, $145 \mu\text{mol/L}$) of PB was applied to the PES-AC particle column; 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. Method 2, the aqueous solution (10 ml, $145 \mu\text{mol/L}$) of PB was applied to the particle column; the concentration of the eluted solution was determined. Then, a fresh aqueous solution (10 ml, $145 \mu\text{mol/L}$) was applied to the same column, and the concentration of the eluted solution was also determined. This process was repeated four times.

RESULTS AND DISCUSSION

Characterization of the Hybrid Particles

The liquid-liquid phase separation technique employed for the preparation of the particles results in the porous

structure of the PES matrix. When the PES solution containing AC was added into water, the liquid-liquid phase separation caused by the rapid exchange of the solvent (DMAC) and water (non-solvent) occurred, and a skin layer formed due to the rapid phase separation, as shown in Fig. 1. With the completion of the exchange between the solvent and the non-solvent, the porous PES particles were prepared, and many pores existed in the spheres. The AC was embedded in the particles with the formation of the particles.

The skin layer outside the particles was formed by the PES matrix, thus restricted the AC to be eluted from the hybrid particles. During the phase separation it also formed porous structures inside, and the pore size gradually increased from the outer surface to the internal region of the particles. The porous structure led to large specific surface areas, which were essential for using as adsorbents. The PES-AC hybrid particles also have a large pore in the center of the particles. The presence of the large pore in the hybrid particle is caused during the phase-separation process because AC cannot be dissolved in the solvent DMAC. With the increase of the AC proportion, the size of the hole in the center of the particles decreased. When the AC proportion was 75%, the big hole disappeared due to the high content of AC, as shown in Fig. 1.

Table 1 shows the pore size and the specific surface area of the particles. As shown in the table, the porosity and the pore volume significantly decreased while the specific

surface area increased, when the proportion of AC increased. However, there is no change in the particle diameter with the increase in AC proportion. For the pure PES particle, the pore diameter mainly ranges from 100 to 1000 nm, much larger than the pore size of the powdered AC (less than 20 nm). The large pores had greatly contributed to the porosity and pore volume but not to the specific surface area. So the pure PES particle has the least specific surface area $28.13 \text{ m}^2/\text{g}$. While for the PES-AC hybrid particles, the large pores formed by the PES matrix were filled with the powdered AC, this will surely lead to the decrease in the porosity and the pore volume. At the same time, the increase of the AC proportion in the particles led to the significant increase in the specific surface area. The results above indicated that with the increase of AC content, the specific surface area increased, which may lead to a higher adsorption capacity. However, it is difficult to prepare PES-AC hybrid particles with the AC content more than 75%. This is because when the proportion of powdered AC for the particles was over 75%, the proportion of PES in the PES-DMAC-AC solution was less than 7.5%. Thus, the strength of the PES-AC hybrid particles was so weak that the AC could not be embedded in the particles completely.

Accumulation and Removal of Phenobarbital by the PES-AC Particles

Figure 2 shows the effect of the AC content in the particles on the removal ratio of PB. The PB removal ratios by the PES-AC hybrid particles were larger than that by the pure PES porous particles due to the higher specific surface areas as indicated in Table 1. It is also evident that the particles with higher AC proportion adsorbed PB more and faster than their counterparts with lower AC proportion. And the PES-AC hybrid particles possess a large quantity of pores (as indicated in Fig. 1) larger than the molecule size of PB (molecular weight 232.24, with the size $\sim \text{nm}$). For small molecules as PB, the adsorption properties mainly depended on the active sites where the adsorbate molecules are adsorbed (12). Therefore, when the AC content increased, a higher adsorbed amount of PB was obtained.

A comparison of PB adsorption performance between powdered AC, granular AC, and PES-AC75 with the same weight of AC was shown in Fig. 3. It can be clearly concluded that the powdered AC has the highest removal ratio to PB, while the removal amount of PB by the PES-AC75 was between the powdered AC and the granular AC. The specific surface area of the powdered AC is about $456.38 \text{ m}^2/\text{g}$, which is larger than that of the PES-AC75 as mentioned in Table 1 of only $192.84 \text{ m}^2/\text{g}$. All the PES-AC hybrid particles have a small specific surface area because the powdered AC was embedded under the skin layer. Also, because the powdered AC was embedded

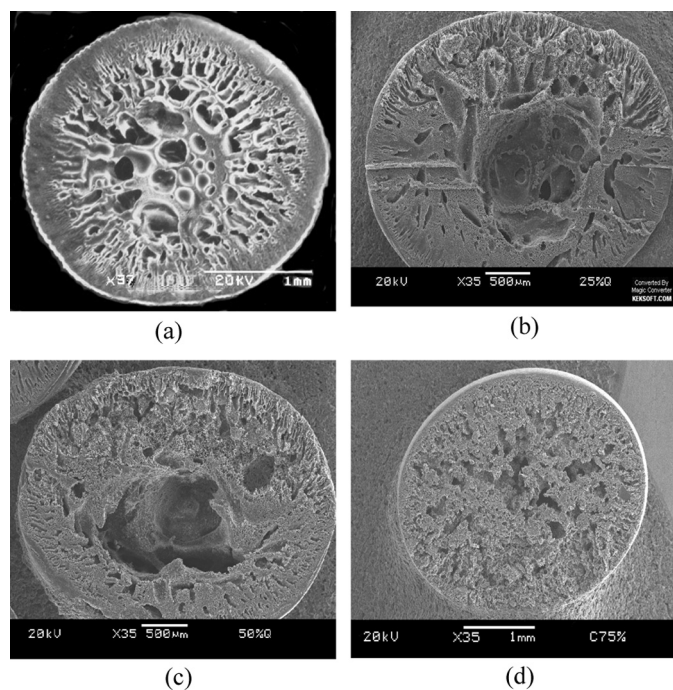


FIG. 1. SEM pictures of different hybrid particles; AC proportion for the particles in Fig. a, b, c, d are 0%, 25%, 50%, and 75%, respectively.

TABLE 1
Structural properties of different PES-AC hybrid particles

Sample	Specific surface area (m ² g ⁻¹)	Diameter (mm)	Porosity (%)	Pore Volume f(cm ³ g ⁻¹)
PES	28.13	2.95 ± 0.10	89.98 ± 0.06	5.47 ± 0.06
PES-AC30	89.63	2.95 ± 0.10	87.13 ± 0.04	3.80 ± 0.05
PES-AC50	134.72	2.94 ± 0.10	83.67 ± 0.04	2.65 ± 0.04
PES-AC75	192.84	2.97 ± 0.10	78.67 ± 0.03	1.57 ± 0.03
Granular AC	236.48	1.50–3.20	31.73	0.39
Powdered AC	456.38	0.03–0.05	32.21	0.38

PES-AC X, where X is the ratio of the mass AC to the mass of total PES and AC.

Data of the porosity, pore volume and diameter were calculated from Equations (1) to (3), $n = 60$.

under the skin layer of the hybrid particles, the removal ratios of PB by the PES-AC particles were lower than that by the powdered AC. Though the powdered AC has the best adsorption, however, when it was used directly, fine carbon particles together with soluble organic compounds in the carbons will be eluted and non-compatibility with blood was observed. And the solutions need centrifugation to separate the activated carbons. For further study and application, the haemolysis test was studied. The haemolysis ratio represents the extent of red blood cells broken by the sample contacting with blood. It is an important factor for the characterization of the haemocompatibility. The lower the haemolysis ratio, the better the haemocompatibility is. In our experiment, the haemolysis ratio of the powdered AC was 8.9%, while the haemolysis ratio of the PES-AC75 hybrid particles was 0.1%. These results indicated that the polyethersulfone -activated carbon hybrid particles may be a useful adsorbent for the removal of PB, and may be used in hemoperfusion.

Adsorption Isotherm of Phenobarbital

Analysis of the isotherm is important in order to develop an equation that accurately represents the results and which could be used for design purposes. There are several adsorption isotherm equations, two of them were used widely and termed the Freundlich and Langmuir equations.

For the Freundlich equation, the adsorbed amount Q_e can be predicted using the following equation at the equilibrium conditions:

$$Q_e = kC_e^{1/n} \quad (6)$$

where C_e is the equilibrium concentration of the adsorbate in solution after adsorption; Q_e is the adsorbed amount per unit weight of the adsorbent; the parameters k and n are the adsorption constants, k is an indication of the adsorbent capacity, and $1/n$ is a measure of the surface

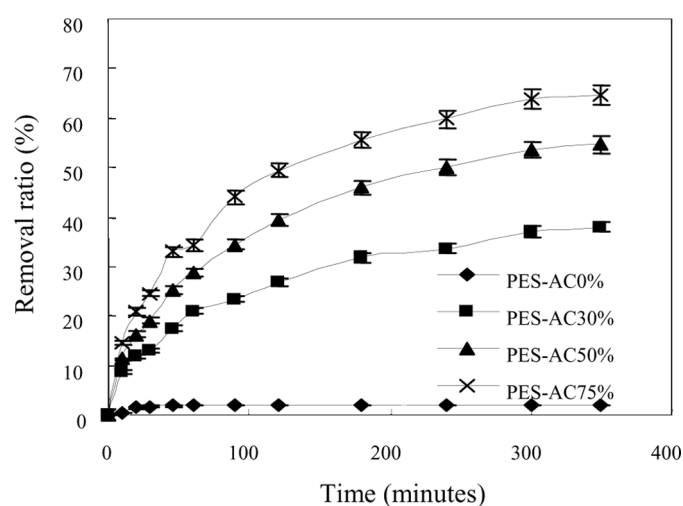


FIG. 2. Phenobarbital adsorption on the particles with different proportion of AC; PB solution: 10 ml, 145 μmol/l; particles: 500 mg; Data are expressed as the mean ± SD of three independent measurements.

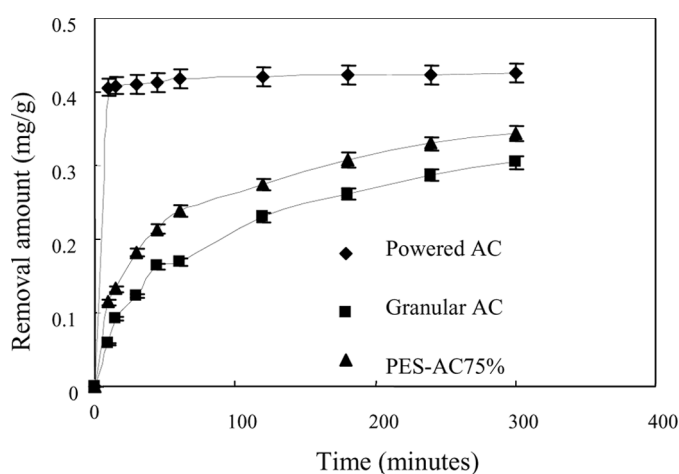


FIG. 3. Comparison of PB adsorption between powdered AC, granular activated carbon and PES-AC75 with the same weight of activated carbon; PB solution: 10 ml, 145 μmol/l; particles: 500 mg; Data are expressed as the mean ± SD of three independent measurements.

heterogeneity, ranging between 0 and 1, becoming more heterogeneous as its value gets closer to zero.

Equation (6) can be rewritten as follows:

$$\log Q_e = \log k + 1/n \log C_e \quad (7)$$

Figure 4 shows the relationships of the $\log Q_e$ and $\log C_e$, which are also the adsorption isotherms of PB onto the hybrid particles containing various percent of activated carbons. The plots of the adsorption isotherms for PB to the hybrid particles were linear. So the adsorption data fit the Freundlich adsorption isotherm (R^2), as shown in Table 2. The coefficient and the constants, $1/n$ and k of the Freundlich plot of PB adsorption by different particles are also shown in Table 2. When the percent of AC increased, the constants k for the hybrid particles increased while the constants $1/n$ decreased. The constants k for the PES-AC75 particles was 0.4574 mg/g, which were larger than that for the PES-AC50 (0.2923 mg/g) and PES-AC30 (0.14 mg/g) particles. The values of $1/n$ were also found to be less than 1, which suggests favorable adsorption behavior of PB onto the hybrid particles. When the data in our study were applied in the Langmuir equation, they were not suitable.

Accumulation and Removal of Phenobarbital by Particle Column

Since the PES-AC hybrid particles could remove about 70% of the PB from its aqueous solution, we prepared PES-AC particle columns to more effectively remove PB. The PES-AC hybrid particles were packed into a polypropylene syringe; then the PB aqueous solution was applied to the column. The removal of PB was determined by the absorption spectra of the eluted solution. When the aqueous PB solution was applied to the PES-AC particle column, the PB was bound to the PES-AC particle column. The eluted solution was reapplied four times, each time the concentration was determined by the spectra of the eluted solution. The results are shown in Fig. 5.

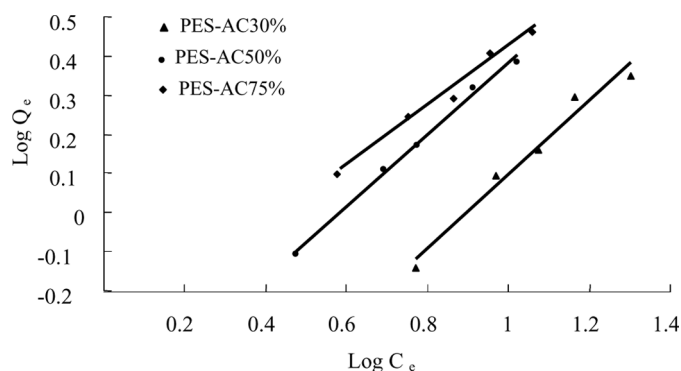


FIG. 4. Freundlich adsorption isotherm for PB.

TABLE 2
Freundlich constants of adsorption for PB

Samples	1/n	k(mg/g)	R ²
PES-AC30	0.9508	0.1400	0.9722
PES-AC50	0.9155	0.2923	0.9942
PES-AC75	0.7612	0.4574	0.9824

As shown in Fig. 5, when the PB solution was applied to the PES-AC particle column for the first time, the concentration sharply decreased from about 145 $\mu\text{mol/L}$ to about 68 $\mu\text{mol/L}$ when the flow rate was 2.5 ml/min. A similar result was obtained when the flow rate was changed to 1.4 ml/min and the concentration decreased from about 145 $\mu\text{mol/L}$ to about 55 $\mu\text{mol/L}$. These mean that about 53% and 63% of the PB in their respective solutions were removed when the solutions were applied to the particle column, respectively. When the eluate was reapplied for the fourth time, the concentrations for PB decreased to about 18.8 $\mu\text{mol/L}$ and 8.6 $\mu\text{mol/L}$ for the flow rate of 2.5 ml/min and 1.4 ml/min, respectively. It indicated that over 89.6% and 95% of the PB were removed by the PES-AC particle column. And the total removal amounts of the PB by the column were 0.073 mg/g and 0.079 mg/g at the flow rate of 2.5 ml/min and 1.4 ml/min, respectively.

Next, we determined the accumulation and removal of PB when the eluted solutions were not reapplied. Aqueous PB solutions (10 ml, 145 $\mu\text{mol/L}$) were applied to the PES-AC particle column, the concentration of the eluate was then determined by the spectra of the eluted solution.

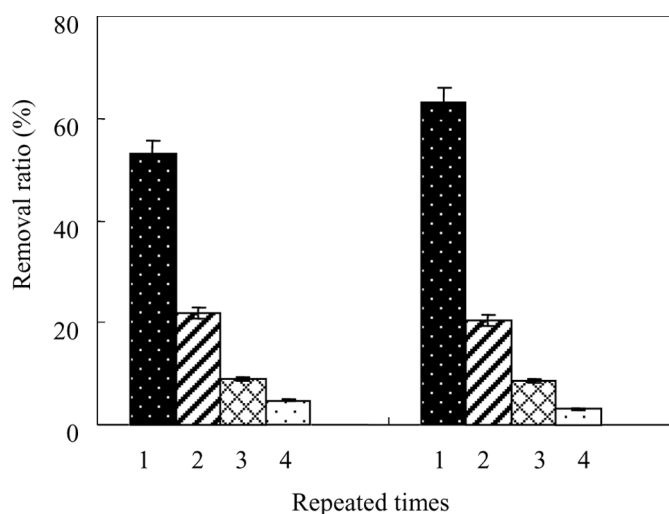


FIG. 5. Concentration change with repeated times when the eluted solutions were reapplied to the PES-AC particle column; Flow rate: 2.5 ml/min (left), 1.4 ml/min (right). Initial PB solution: 10 ml, 145 $\mu\text{mol/L}$; hybrid particles: 4g; Data are expressed as the mean \pm SD of three independent measurements.

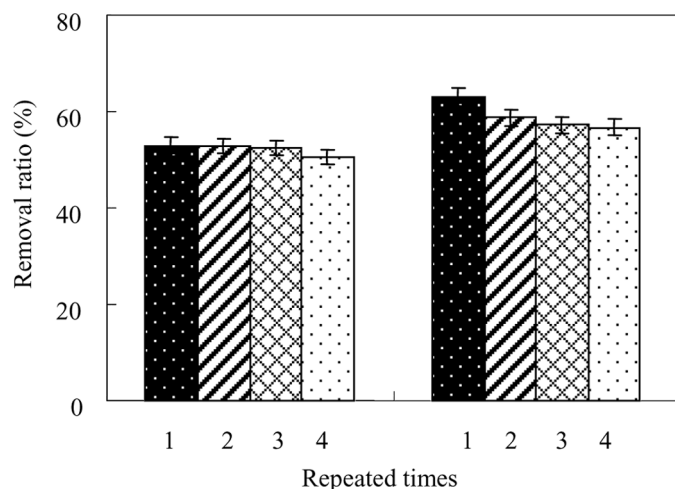


FIG. 6. Concentration change with repeated times when the new solutions were applied to the PES-AC hybrid particle column; Flow rate: 2.5 ml/min (left), 1.4 ml/min (right); PB solution for each time: 10 ml, 145 μ mol/l; hybrid particles: 4 g; Data are expressed as the mean \pm SD of three independent measurements.

Then, a fresh aqueous solution (10 ml, 145 μ mol/L) was applied to the same column, and the concentration of the eluted solution was also determined. This process was repeated four times. The results are shown in Fig. 6.

As shown in Fig. 6, when the aqueous PB solution (10 ml, 145 μ mol/L) was applied to the PES-AC porous particle column at a flow rate of 2.5 ml/min for the first time, the concentration decreased to about 68 μ mol/L. For the second to the fourth time, the concentrations decreased from 145 μ mol/L to about 68.3 μ mol/L, 69.2 μ mol/L and 71.9 μ mol/L, respectively. The removal ratios were slightly decreased to 52.9%, 52.3%, and 50.4%. A similar result was obtained when the flow rate was 1.4 ml/min and the concentrations decreased from 145 μ mol/L to about 55 μ mol/L for the first time, and 59.8 μ mol/L, 62.1 μ mol/L and 62.8 μ mol/L for the following times, respectively. The removal ratios were 63%, 58.8%, 57.2%, and 56.7%, respectively. The total removal amounts for the four times were 0.176 mg/g and 0.200 mg/g at the flow rate of 2.5 ml/min and 1.4 ml/min, respectively. These mean that the PES-AC hybrid particle columns could treat a very large amount of PB, while there is no significant decrease in the removal ratios. From these data we also find that when the flow rate decreased, the concentration of PB in the eluted solutions decreased and the removal ratio increased. This suggested that high removal ratio could be obtained not only by reapplying the eluted solution but also by decreasing the flow rate.

CONCLUSION

Polyethersulfone-activated carbon hybrid particles could be prepared by a liquid-liquid phase separation technique and its adsorption properties to PB was evaluated. The hybrid particles could be easily prepared to column, both the hybrid particles and the particle column can accumulate PB, and the PES-AC hybrid particle column could more effectively remove PB. It is reasonable to predict that by embedding activated carbon within PES, a novel and useful adsorbents may be obtained in the haemoperfusion column for blood purification.

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