



ISSN 1001-0742
CN 11-2629/X

2012

Volume **24**
Number **8**

JOURNAL OF
**ENVIRONMENTAL
SCIENCES**



Sponsored by
Research Center for Eco-Environmental Sciences
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JOURNAL OF ENVIRONMENTAL SCIENCES

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Polyphenylene sulfide based anion exchange fiber: Synthesis, characterization and adsorption of Cr(VI)

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Received 03 October 2011; revised 16 November 2011; accepted 30 November 2011

Abstract

A fibrous strong base anion exchanger (QAPPS) was prepared for the first time via chloromethylation and quaternary amination reaction of polyphenylene sulfide fiber (PPS), and its physical-chemical structure and adsorption behavior for Cr(VI) were characterized by FT-IR, Energy Dispersive Spectrometry, TG-DTG, elemental analysis and batch adsorptive technique, respectively. The novel fibrous adsorbent could effectively adsorb Cr(VI) over the pH range 1–12, the maximum adsorption capacity was 166.39 mg/g at pH 3.5, and the adsorption behavior could be described well by Langmuir isotherm equation model. The adsorption kinetics was studied using pseudo first-order and pseudo second-order models, and the $t_{1/2}$ and equilibrium adsorption time were 5 and 20 min respectively when initial Cr(VI) concentration was 100 mg/L. The saturated fibers could be regenerated rapidly by a mixed solution of 0.5 mol/L NaOH and 0.5 mol/L NaCl, and the adsorption capacity was well maintained after six adsorption-desorption cycles.

Key words: strong base anion exchanger; polyphenylene sulfide fiber; Cr(VI); adsorption

DOI: 10.1016/S1001-0742(11)60948-0

Introduction

Hexavalent chromium (Cr(VI)) is a highly toxic metal ion and well-established carcinogen, which has a very bad impact on environmental and human health. Wastewaters containing Cr(VI) are generated by many industrial processes, including leather tanning, electroplating, paints and others. According to the criteria of the United States Environmental Protection Agency, the concentration of total chromium in drinking water has been regulated with a maximum allowable level of 0.1 mg/L. Thus, waste streams containing chromium must be treated before discharging to the environment to meet the growing environmental regulations.

Owing to high selectivity, less sludge volume produced, recoverability of valuable metal, and the ability to meet strict discharge specifications, the ion exchange method has been widely used for the removal of Cr(VI) from wastewaters (Almaguer-Busso et al., 2009; Lo et al., 2011; Hosseini-Bandegharai et al., 2010; Kang et al., 2011; Sarkar et al., 2010; Zeng et al., 2010; Zimmermann et al., 2010; Qiu et al., 2009; Wasag and Henryk, 2008; Shi et al., 2009; Pehlivan and Cetin, 2009; Gode and Pehlivan, 2005; Mustafa et al., 2010). Many commercial ion exchange materials have been used to remove Cr(VI) from aqueous solution, including weak base ion exchange materials (Wasag and Henryk, 2008; Shi et al., 2009; Neagu, 2009), and strong base ion exchange materials

(Zhu et al., 2009; Neagu et al., 2003; Atia et al., 2006). However, due to the low absorption/desorption rate and the physical form, the applications of these commercial ion exchange materials are limited, and require some strict treatment conditions.

The fibrous ion-exchanger (IEF) is a kind of new adsorption and separation polymer which has several advantages over granular ion exchange resins. IEF can be fabricated in various textures (fiber, thread, felts, nonwoven cloth, etc.), and has a much higher absorption/desorption rate due to its short transit distance (Dominguez et al., 2003). Thus, IEF can not only lead to the development of various novel ion exchange materials but also expand the application of ion exchange materials into new areas, for which granular resins are inefficient or impossible. Usually, weak base IEF can only adsorb Cr(VI) in an acidic medium, while strong base IEF can remove Cr(VI) in a broad pH range (Qiu et al., 2009; Neagu, 2009). However, the industrial preparation of strong base IEF is very difficult and expensive because of the demanding $^{60}\text{Co}_\gamma$ or high-energy electron irradiation grafting processes required (Dominguez et al., 2003; Soldatov et al., 1986; Zhang et al., 2008). Since the 1980s some studies have reported the synthesis of strong base IEF based on islands-in-a-sea type composite fiber (Yoshioka and Shimamura, 1983) and glass fiber (Dominguez et al., 2003), but their commercial preparation and application are yet to be realized.

In this article, a strong base anion exchange fiber has been prepared for the first time by chemical modification

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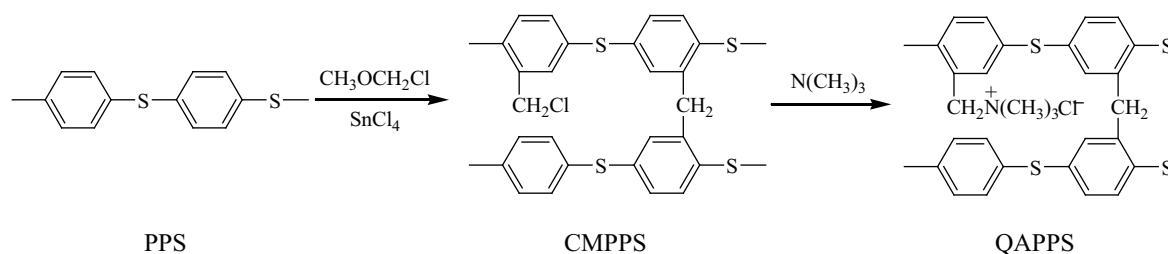


Fig. 1 Synthesis route for QAPPS.

of polyphenylene sulfide fiber (PPS). Compared with PP-ST-DVB-based strong base anion exchange fiber, the novel preparation process for strong base anion exchange fibers is simpler, lower cost and more amenable to commercialization. The novel functional fibers were able to adsorb Cr(VI) from aqueous solutions in the pH range 1–12, and exhibited high exchange capacity and a fast adsorption and desorption rate, as well as excellent thermal and chemical stability.

1 Experiment

1.1 Materials

PPS fiber was obtained from Si Chuan Deyang Chemical Co. Ltd., China. Chloromethyl methyl ether was purified by atmospheric pressure distillation with the distillation cut collected from 45 to 60°C. 1,2-Dichloroethane (DCE) and SnCl_4 were AR grade, trimethylamine aqueous solution (CR, 33 wt.%) was purchased from Sinopharm Chemical Reagent Co. Ltd., China. The stock solution of Cr(VI) was prepared by the dissolution of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$, AR) in double-distilled water; other chemicals were AR grade reagents.

1.2 Methods

The adsorption capacity of the novel IEF materials, the functional group distribution in the fibrous matrix, as well as its chemical and thermal stability, were determined and characterized by chemical titration means, Energy Dispersive Spectrometer (JSM-7500F JEOL, Japan), FT-IR (NEXUS-470 NICOLET, USA) and elemental analyzer (EA 1112 Thermo Flash, USA), and thermogravimetric (TG) and differential thermogravimetric (DTG) analysis (STA 409 PC NETZSCH, Germany), respectively. The Cr concentration of the solutions was determined using a Shimadzu Uvmini-1240 spectrophotometer from the absorption at 540 nm, using diphenylcarbazide as the complexing agent.

1.3 Synthesis of the strong base anion exchange fiber

PPS fiber (5.0 g) (C: 66.40 wt.%; H: 3.85 wt.%; S: 22.96 wt.%) was subjected to swelling with 30 mL 1,2-dichloroethane for 4 hr in a 100 mL three-necked flask with reflux condenser connected to an anhydrous CaCl_2 tube, then 30 mL chloromethyl methyl ether and 2 mL SnCl_4 were added. The chloromethylation and attendant crosslinking reaction were carried out at 50°C for 10 hr, and 60°C for another 5 hr. After the resulting fibers were washed with methanol and deionized water, and dried at

50°C under vacuum, the chloromethylated fiber (CMPPS) was obtained (C: 54.30 wt.%; H: 3.10 wt.%; S: 22.30 wt.%).

The above fibers were immersed in liquid trimethylamine (wt.%; 33%) for 8 hr, and then the amination reaction was carried at 30–40°C for 15 hr. After being contacted with 1 mol/L HCl, washed with deionized water and dried at 50°C under vacuum, the strong base anion exchange fiber (QAPPS) was obtained (C: 48.70 wt.%; H: 6.40 wt.%; S: 13.50 wt.%; N: 4.60 wt.%). The synthesis route for QAPPS is shown in Fig. 1.

1.4 Batch adsorption of Cr(VI)

The effect of pH on chromium adsorption was investigated by contacting 0.05 g of dried QAPPS fiber and 100 mL of Cr(VI) solution (100 mg/L) of different pH at 20°C for 4 hr, where 1 mol/L HCl and 1 mol/L NaOH solution were used to adjust pH. The sorption kinetics was studied by contacting 0.05 g of dried fibers and 100 mL Cr(VI) solution (100 mg/L) for different time intervals at 20°C. The adsorption isotherm experiments were carried out by placing 0.05 g dried fibers in 100 mL of Cr(VI) solution with initial concentration varying from 20 to 200 mg/L at 20°C for 4 hr.

1.5 Regeneration of QAPPS fiber

A QAPPS fiber sample of 0.05 g was shaken with 100 mL of Cr(VI) solution (100 mg/L) for 4 hr at 20°C to reach equilibrium. The Cr(VI)-loaded fiber was separated by filtration, and washed with deionized water to remove unadsorbed Cr(VI). All Cr(VI)-loaded fiber was transferred to a conical flask, and regenerated by shaking with 25 mL of a mixed solution of 0.5 mol/L NaOH and 0.5 mol/L NaCl for 15 min at 20°C. The amount of released Cr(VI) was determined by the diphenylcarbazide (DPC) method. The regenerated QAPPS fiber was shaken with 100 mL of Cr(VI) solution (100 mg/L) at 20°C for the next adsorption-desorption cycle.

2 Results and discussion

2.1 Characterization of QAPPS fiber

The weight gain of PPS fiber was 33.6% after chloromethylation, and the C, H, S and Cl contents of the resulting fiber were 54.30 wt.%, 3.10 wt.%, 22.30 wt.% and 18.41 wt.% (deduction), respectively. The analysis data indicated that chloromethyl groups had been successfully introduced into the PPS matrix (about 5 mmol/g). Besides, a slight crosslinking reaction was observed at high

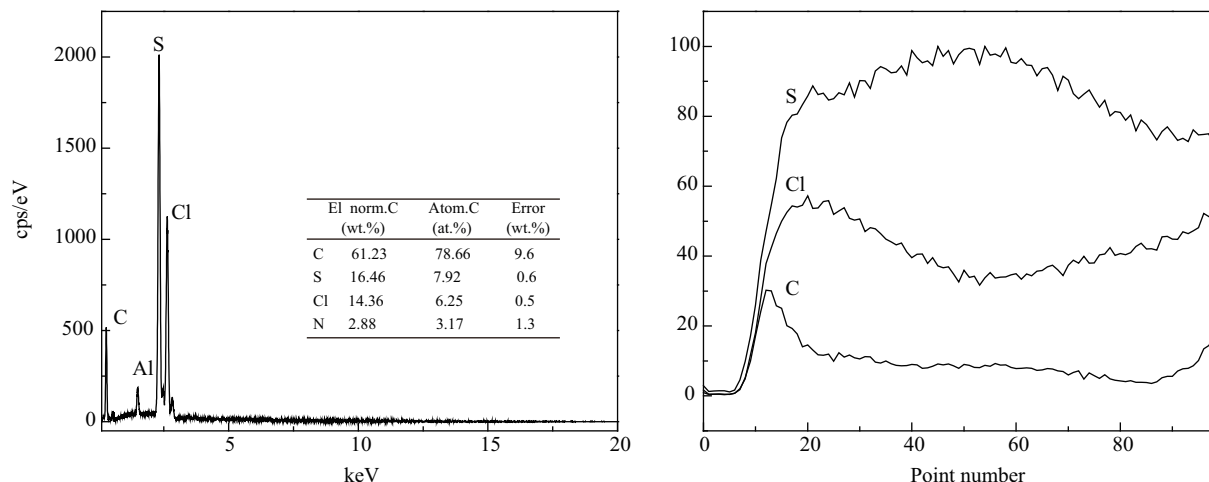


Fig. 2 Energy Dispersive Spectrometer quantitative analysis of the cross section of QAPPS fiber.

levels of chloromethylation in our investigation. Andrei (2007) reported that the intercrosslinking of linear polymers could have a beneficial influence on the chemical and thermal stabilities and operating performance of ion exchange resins. The influence of a crosslinked network on QAPPS fiber needs to be further studied.

The adsorption capacity of QAPPS fiber was 3.51 mmol/g (theoretical value: 3.91 mmol/g) in our investigation. The Energy Dispersive Spectrometer analysis indicated that the density of quaternary ammonium groups had a slightly decreasing distribution from the surface to the center along the cross section of the QAPPS fiber (Fig. 2). The IR (Fig. 3) and elemental analysis results demonstrated that there were some residual chloromethyl and carbonyl groups in the QAPPS matrix, which could be ascribed to oxidation and insufficient amination of CMPPS fiber under our experimental conditions.

The structures of CMPPS and QAPPS fibers were characterized by FT-IR (Fig. 3). For CMPPS fiber the IR absorption at 1257 and 707 cm^{-1} could be ascribed to the C-H stretching vibration and C-Cl in-plane bending vibration of the chloromethyl group, while the appearance of the C-N stretching vibration peak at 1321 cm^{-1} and the disappearance of the C-Cl bending vibration peak at 707

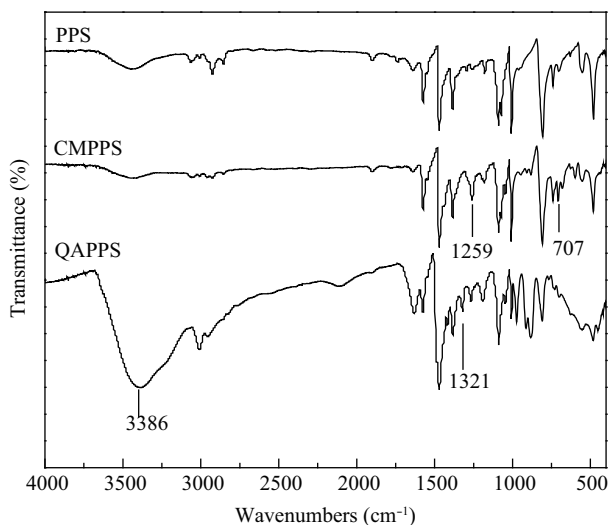


Fig. 3 FT-IR spectra of PPS, CMPPS and QAPPS fibers.

cm^{-1} demonstrated that the chlorine in QAPPS had been successfully substituted by trimethylamine groups.

The thermostability of QAPPS and commercial Fiban A-1 strong base fibers (Wasag and Henryk, 2008) were determined using TG and DTG (Fig. 4). In our investigation both strong base fibers had two mass decrement peaks which could be ascribed to the loss of adsorbed water and quaternary salt groups. For Fiban A-1 fiber (ST-DVB matrix), however, the mass decrement peaks were observed at 81 and 233 $^{\circ}\text{C}$, while for QAPPS fiber (PPS matrix) these peaks moved to 96 and 272 $^{\circ}\text{C}$, respectively. Moreover there was a sharp pyrolysis for Fiban A-1 fiber after 400 $^{\circ}\text{C}$, but the same process was not observed for QAPPS materials. The excellent thermal stability of QAPPS fibers could be ascribed to the special aryl thioether matrix of the fibers.

The chemical stability of QAPPS fiber was studied in various acid, alkali and oxidizing mediums. In comparison to literature reports (Neagu et al., 2010), the oxidation resistance of QAPPS fiber was much better than that of Amberlite IRA-400 strong base anion exchange resin (ST-DVB matrix). The experimental results showed (Table 1) that the QAPPS fiber had good chemical stability in HCl solution (5 mol/L) at 25–50 $^{\circ}\text{C}$, although a slight loss of exchange capacities was observed in H_2O_2 solution (1–3 mol/L). There was a clear decrease (about 21%) of the

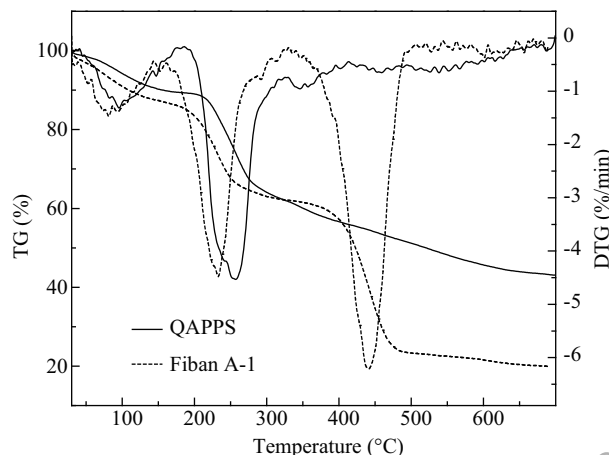


Fig. 4 TG and DTG curves of QAPPS and Fiban A-1.

Table 1 Chemical stability of QAPPS fiber

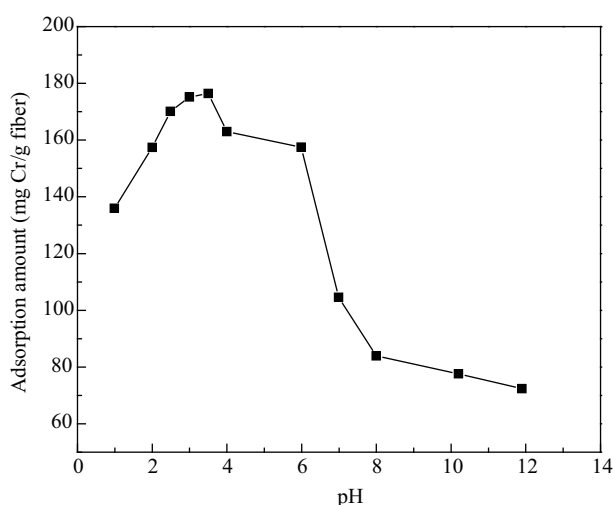
Media	Strong base exchange capacity (mmol/g)		Loss of exchange capacity (%)	
	25°C	50°C	25°C	50°C
1 mol/L HCl	3.51	3.51	0	0
5 mol/L HCl	3.51	3.51	0	0
1 mol/L NaOH	3.51	3.12	0	11.11
5 mol/L NaOH	3.09	2.74	11.97	21.94
5% H ₂ O ₂	3.38	3.35	3.70	4.56
10% H ₂ O ₂	3.34	3.26	4.84	7.12

exchange capacity when the fibers were subjected to 1 mol/L NaOH at 50°C and 5 mol/L NaOH at 25°C, which could be due to Hofmann degradation of the quaternary amines.

2.2 Effect of pH on Cr(VI) adsorption

The solution pH could influence the structure, surface charge and speciation of QAPPS fiber and Cr(VI) ions. Usually Cr(VI) ions exist in aqueous solution in five main ionic forms, and their relative abundance is dependent on both total chromium concentration and pH value. According to the speciation diagram of Cr(VI) at different pH and initial concentration of 100 mg/L (Zhu et al., 2009), HCrO₄⁻ was the dominant Cr(VI) species in the pH range from 1.0 to 6.5, with a small amount of Cr₂O₇²⁻ and HCr₂O₇⁻ also present. The H₂CrO₄ and CrO₄²⁻ nearly disappeared at pH 3.0 to 4.0. When pH ≤ 0.8, H₂CrO₄ was the dominant species. Cr(VI) was present in the form of CrO₄²⁻ when pH was above 6.5.

As shown in Fig. 5, solution pH had a great effect on the adsorption of Cr(VI) on the QAPPS fiber. In our experimental conditions, the maximum adsorption was observed at pH 3.5 because of the disappearance of H₂CrO₄ and CrO₄²⁻, and the active sites of the fibers could exchange with Cr(VI) at the rate of 1:1. When the pH was lower than 3.5, the adsorption amount was found to slightly decrease, which could be due to the increase of the non-anionic species H₂CrO₄ and the protonation of amine groups (Vincent and Guibal, 2001). On the other hand, the adsorption capacity was found to significantly decrease when the pH was above 7.0. The most important reason

**Fig. 5** Effect of pH on adsorption of Cr(VI) on QAPPS.

was that CrO₄²⁻ became the only chromate species for pH greater than 7.5, and one CrO₄²⁻ could take up two active sites of the QAPPS fiber. However, the adsorption capacity of the QAPPS at pH 12 still attained 72.6 mg/g which was higher than other strong base anion exchangers, such as Lewatit MP500 (28.6 mg/g at pH 5.0) (Pehlivan and Cetin, 2009), silica-based adsorbent (68 mg/g at pH 2.5) (Qiu et al., 2009), and N-methylimidazolium strong base resin (58 mg/g at pH 12) (Mustafa et al., 2010).

2.3 Adsorption isotherms of Cr(VI)

The adsorption isotherms of QAPPS and Fiban A-1 fibers for Cr(VI) were investigated, and the data were analyzed with the Langmuir adsorption isotherm model Eq. (1).

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_{\max}} + \frac{1}{bQ_{\max}} \quad (1)$$

where, C_e (mg/L) is the equilibrium concentration, Q_e (mg/g dry fibers) is the amount of Cr(VI) adsorbed at equilibrium, Q_{\max} (mg/g dry fibers) and b (L/mg) are Langmuir constants related to the maximum adsorption capacity and energy of sorption, respectively. The Langmuir curve fitting of the experimental data is shown in Fig. 6 with a high regression coefficient (Table 2). This revealed that the adsorption of Cr(VI) was increased with increasing equilibrium concentration, and both of the fibers reached saturation very quickly, but the maximum adsorption capacity of Cr(VI) on QAPPS (166.39 mg/g) calculated by the Langmuir isotherm model was higher than that of Fiban A-1.

Theoretically the adsorption capacity of QAPPS for Cr(VI) was 182.5 mg/g (3.51 mmol/g), and under our experimental conditions the adsorption capacity for Cr(VI) was 166.4 mg/g. Thus, 91.2% of the active sites of the QAPPS matrix participated in the adsorption of Cr(VI).

Table 2 Langmuir adsorption isotherms constants of QAPPS and Fiban A-1

Fiber	Q_{\max} (mg/g)	b (L/mg)	R^2
QAPPS	166.39	0.63	0.997
Fiban A-1	142.65	1.32	0.999

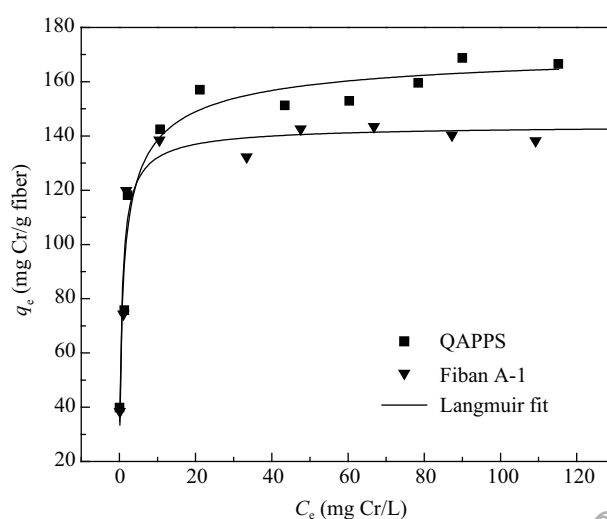
**Fig. 6** Adsorption isotherms of Cr(VI) by QAPPS and Fiban A-1.

Table 3 Pseudo first- and second-order constants for the adsorption of Cr(VI) on QAPPS

Fiber	$q_{e,exp}$ (mg/g)	Pseudo first-order kinetics			Pseudo second-order kinetics				
		k_1 (min ⁻¹)	$q_{e,cal}$ (mg/g)	$t_{1/2}$ (min)	R_1^2	k_2 (g/mg·min)	$q_{e,cal}$ (mg/g)	$t_{1/2}$ (min)	R_2^2
QAPPS	174.41	0.1985	161.89	3.50	0.982	0.00157	179.27	3.56	0.994
Fiban A-1	137.86	0.1495	134.79	4.63	0.992	0.00134	151.23	4.93	0.991

Table 4 Regeneration and desorption of QAPPS fiber

Adsorption and regeneration cycle	Adsorption amount (mg Cr/g fiber)	Desorption rate (%)
1	169.28	99.31
2	165.07	99.45
3	165.60	98.14
4	165.01	99.26
5	162.31	97.92
6	162.33	98.75

This ratio is much higher than previous literature data (Shi et al., 2009; Pehlivan and Cetin, 2009; Neagu, 2009).

2.4 Adsorption kinetics of Cr(VI)

The adsorption kinetics experiments were carried out at initial Cr(VI) concentration 200 mg/L, pH 3.5 and 20°C (Fig. 7). The adsorption rates of QAPPS and Fiban A-1 fibers were very rapid in the first 10 min. and the adsorption equilibrium could be reached during 20–30 min.

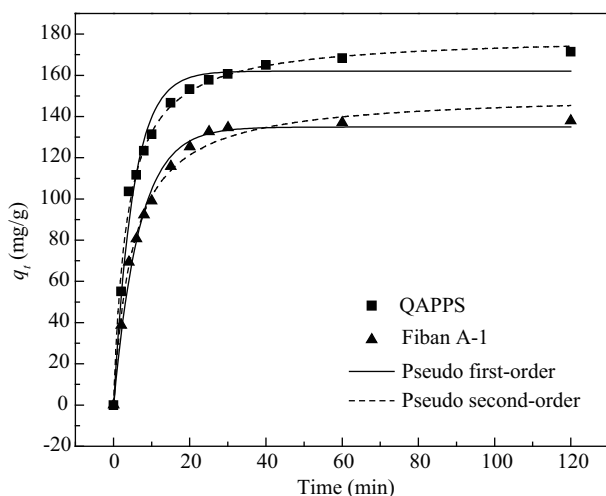
Pseudo first- and second-order models were employed in the investigation of the adsorption kinetic mechanism of QAPPS. The pseudo first-order model is expressed as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (2)$$

where, q_t and q_e are the amounts of adsorbed Cr(VI) at time t and equilibrium, respectively. k_1 is the rate constant of pseudo first-order adsorption. Eq. (2) can be rearranged as:

$$q_t = q_e - e^{-k_1 t} \quad (3)$$

where, q_e and k_1 are calculated by plotting q_t versus t .

**Fig. 7** Effect of contact time on QAPPS and Fiban A-1 fibers.

The pseudo second-order model is given as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

where, k_2 is the rate constant for pseudo second-order adsorption. Equation (4) can be rearranged as:

$$q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \quad (5)$$

where, q_e and k_2 were attained by plotting q_t versus t .

The fitted results according to the pseudo first- and second-order models are shown in Table 3. It can be seen that both models fitted the data well with high regression coefficient (R^2) values. However the equilibrium concentration calculated from the pseudo second-order model fitted the experimental data better. From the data in Table 3, it can be seen that the maximum adsorption capacity and rate constant of QAPPS fiber are higher than those of commercial Fiban A-1 fiber. This indicated that QAPPS has minimal transfer resistance and easily accessible active sites.

2.5 Desorption and regeneration of QAPPS fiber

The QAPPS fiber with adsorbed Cr(VI) could be desorbed effectively by 0.5 mol/L NaOH and 0.5 mol/L NaCl mixed solution (Table 4), the desorption rate could exceed 98% in 15 min, and the adsorption capacity was not changed significantly after six adsorption-desorption cycles.

3 Conclusions

A strong base anion exchanger based on PPS fiber had been synthesized. The novel adsorption material could effectively adsorb Cr(VI) over a wide pH range, and the maximum adsorption capacity of Cr(VI) was 166.39 mg/g in 20 min. Owing to excellent physicochemical stability and regeneration capability, the functional fiber had a great application potential for the treatment of wastewater and exhaust gas containing Cr(VI).

Acknowledgment

The authors are grateful for the support of the National Natural Science Foundation of China (No. 20574063, 21004055).

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Journal of Environmental Sciences (Established in 1989)

Vol. 24 No. 8 2012

Supervised by	Chinese Academy of Sciences	Published by	Science Press, Beijing, China
Sponsored by	Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences		Elsevier Limited, The Netherlands
Edited by	Editorial Office of Journal of Environmental Sciences (JES) P. O. Box 2871, Beijing 100085, China Tel: 86-10-62920553; http://www.jesc.ac.cn E-mail: jesc@263.net , jesc@rcees.ac.cn	Distributed by	Domestic Science Press, 16 Donghuangchenggen North Street, Beijing 100717, China Local Post Offices through China Foreign Elsevier Limited http://www.elsevier.com/locate/jes
Editor-in-chief	Hongxiao Tang	Printed by	Beijing Beilin Printing House, 100083, China
CN 11-2629/X	Domestic postcode: 2-580		Domestic price per issue RMB ¥ 110.00

ISSN 1001-0742



9 771001 074123