



Original article

Preparation and characterization of polyphenylene sulfide-based chelating fibers

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ABSTRACT

A series of novel chelating fibers containing sulfur, nitrogen, oxygen heteroatoms were prepared via the functionalization of chloromethylated polyphenylene sulfide (CMPPS). The structures, micromorphology and physicochemical properties of these fibrous adsorptive materials were characterized by FT-IR, elementary analysis, TG and SEM-EDS. The results show that chelating fibers had high functional group contents (3.94 mmol/g for thiourea, 3.85 mmol/g for mercapto, 5.00 mmol/g for methylamine and 6.07 mmol/g for ethylenediamine, respectively). Owing to the unique matrix of polyphenylene sulfide fiber, these fibrous adsorbents possess excellent thermostability. This synthetic method proved a simple and efficient way for the preparation of chelating fibers.

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1. Introduction

As well known, chelating material is a kind of materials with functional groups containing one or more (multiple) atoms of oxygen, nitrogen, sulfur, phosphorus, which can be covalently bonded to metal ions. Commercially available chelating/ion exchange materials with amino or iminodiacetic acid groups are widely used to remove heavy metal from aqueous solution [1–3]. Compared to granular materials, fiber is known to have large apparent specific surface, excellent osmotic stability and good kinetic properties. As a result of these properties, the intensive research has been carried out to develop the preparation methods for different types of fibrous chelating materials.

Many methods are used for prepare chelating fibers, such as chelating fibers with chemical grafting [4,5], radiation-inducing, electron-beam-induced grafting [6,7], and chemical modifications [8,9]. However, these preparation techniques are cumbersome and ineffective because two or more steps are required for the preparation of chelating fibers, and demand ⁶⁰Co_γ or high-energy electron irradiation for the grafting processes. Polyphenylene sulfide (PPS) fiber is a new type performance material with excellent mechanical strength, chemical and thermal stability. Due to its rich benzene rings, our laboratory has prepared strong acid/base ion exchange fibers on PPS fiber [10,11] with high exchange

capacity and excellent chemical stability. The advantage of PPS fiber-based preparations is that the irradiation by ⁶⁰Co_γ or high-energy electron was avoided.

In the present work, we prepared a series of chelating functional fibers through chloromethylation and further functionalization on the PPS fiber. The structure and properties were proven by FT-IR, elementary analysis, thermogravimetric analysis and SEM-EDS.

2. Experimental

Chloromethylated polyphenylene sulfide (CMPPS) fiber was prepared according to the method previously reported [12]. As shown in Scheme 1, the chelating fiber was prepared.

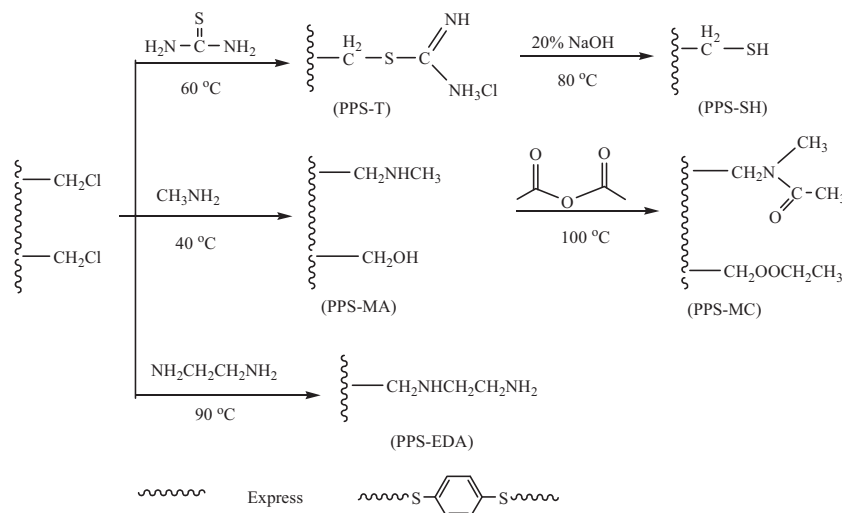
2.1. Preparation of thiourea and thiol chelating fibers

About 1 g of CMPPS fiber (C, H, S and Cl content were 52.05%, 3.09%, 20.07% and 24.79%, respectively) was swelled in 1,4-dioxane for 12 h. And then 30 mL of ethanol, 2 g of thiourea and a phase transfer catalyst tetrabutyl ammonium bromide (TBAB, 0.02 g) were added. The mixture was heated in an oil bath at 60 °C for 12 h. The fiber was filtered and rinsed thoroughly with distilled water to remove residual impurities. The products were left to dry completely in vacuum to produce the thiourea chelating fiber PPS-T (C, 35.90%; H, 3.96%; S, 22.65%; N, 11.03%).

The PPS-T fiber was subjected to a hydrolysis reaction with 40 mL, 20% aq. NaOH at 80 °C for 12 h. The product was washed with distilled water till filtrate was neutral, then left to dry

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Scheme 1. Schematic illustration of the preparation of PPS chelating fiber.

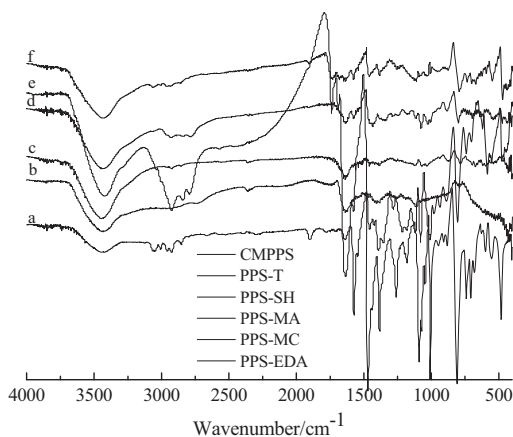


Fig. 1. FT-IR spectra of chelating fibers: (a) CMPPS; (b) PPS-T; (c) PPS-SH; (d) PPS-MA; (e) PPS-MC; (f) PPS-EDA.

completely in vacuum for 24 h to obtain the thiol chelating fiber PPS-SH (C, 50.82%; H, 3.43%; S, 40.16%; N, 0.19%).

2.2. Preparation of the amino and acylamide chelating fibers

About 1 g of CMPPS (weight gain was 38.69% and C, H, S and Cl content were 52.16%, 3.24%, 21.60% and 23.00%, respectively) was immersed in 40 mL of 25% aqueous methylamine for 8 h, and then the reaction was carried out at 40 °C for 24 h. The fiber was filtered

and soaked in 1 mol/L NaOH for at least 8 h. The product was rinsed thoroughly to neutral pH with distilled water and dried at 80 °C under vacuum to a constant weight to give PPS-MA (C, 58.45%; H, 5.51%; S, 16.85%; N, 7.00%).

The above fiber was put into a flask containing 25 mL of acetic anhydride and the reaction mixture was heated in an oil bath for 8 h. The resulting product was filtered and rinsed thoroughly with distilled water until the filtrate was neutral, then left to dry completely in vacuum for several days to produce PPS-MC (C, 54.76%; H, 4.97%; S, 14.94%; N, 4.63%).

2.3. Preparation of the ethylenediamine chelating fiber

About 1 g of CMPPS fiber (C, H, S and Cl contents were 52.16%, 3.24%, 21.60% and 23.00%, respectively) was swelled in 1,2-dichloroethane for 12 h, and then 25 mL of ethylenediamine was added quickly, the reaction mixture was heated in an oil bath at 90 °C for 24 h. After the reaction was complete, the fiber was filtered and soaked in 1 mol/L NaOH for at least 8 h, and then fiber was washed to neutral pH with distilled water and dried at 80 °C under vacuum to constant weight to give PPS-EDA (C, 60.06%; H, 5.08%; S, 19.66%; N, 9.20%).

2.4. Characterization of the chelating fiber

The chelating fibers were characterized by Fourier transform infrared spectroscopy (FT-IR 20009 AmericanThermo Nicolet Co., Ltd.), elementary analysis (EA 1112 Thermo Flash, USA), scanning

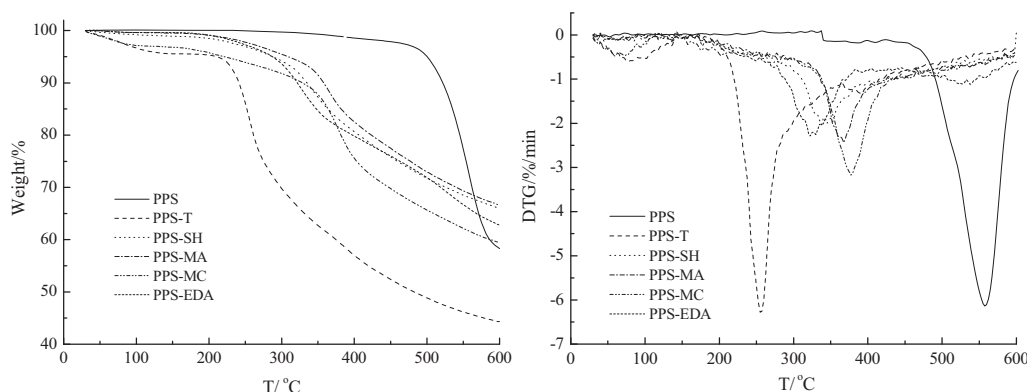


Fig. 2. Thermal stability of chelating fiber.

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