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Environmentally friendly preparation of a strong basic anion exchange fibers and its application in sugar decolorization

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ABSTRACT

An efficient route for the synthesis of a strong basic anion exchange fibers is described. In this synthesis route, the commercially available p-chloromethylstyrene was directly grafted onto a polypropylene fiber substrate, which eliminated the need of the highly carcinogenic chloromethyl methyl ether in the chloromethylation of grafting fiber. Several interdependent parameters such as monomer concentration, bath ratio and the influence of solvents on the grafting copolymerization were investigated. The removal efficiency of sugar colorants by the anion exchange fibers was evaluated. The results show that simultaneous irradiation and grafting p-chloromethylstyrene onto polypropylene fiber can obtain a good grafting level under appropriate conditions. The optimal monomer concentration and bath ratio are 40% and 1:30 for a moderate grafting degree and the suitable solvent is toluene. The grafted fiber changed from flexible to rigid. And the prepared material maintains stable before 134.5 °C. The static ion exchange capacity of the synthesized anion exchange fiber is up to 4.72 mmol g^{-1} and the fiber possesses better ability to remove sugar colorants. The experimental results indicate that the decolorization ratio of the product anion exchange fibers was greater than that of commercial material.

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1. Introduction

Ion exchange fiber (IEF) is characterized by higher effective specific surface area and short transit distance which contributes to a higher speed of both adsorption and desorption for metal ions, poisonous and deleterious compounds. The useful properties of ion exchange fibers, mainly containing acid or base groups, have been described in a great number of publications. In these publications numerous advantages of these materials compared to granular sorbents of the same chemical nature, as well as their potential and real applications, have been discussed [1–7]. However, the complicated and environmentally unfriendly synthesis route restricts the commercial applications of IEF.

Generally, IEF was prepared according to the approach that conventional ion exchange resins have been used. Conventional strong basic anion exchange resins are prepared by the functionalization of a polymeric backbone, for example crosslinked polystyrene, in which the polymer beads are swollen with organic solvents and chloromethylated with chloromethyl methyl ether, subsequently followed by amination of the chloromethylated fibers with trimethylamine aqueous solution [8]. And the strong basic fibrous ion exchangers were designed with similar procedure. It is the only different point that the functional groups were bound to the fiber which act as a precursor.

There are a number of drawbacks associated with the above approach. Firstly, swelling agents must be used to reduce the effects of osmotic shock and maintain the spherical form of the bead. Examples of these include but are not limited to toluene, methylene chloride, perchloroethylene and carbon tetrachloride [8,9]. These solvents will remain in the finished product, and also have some degree of toxicity associated with them. Secondly, during the chloromethylation stage, chloromethyl methyl ether and their side production are poisonous compounds. These environmentally unfriendly solvents carry an added expense not only in the operating costs but also in the additional environmental protection agency (EPA) requirements for handling and disposal them. Worst of all, the most frequently stressed disadvantage of the conventional method is the serious damage to the mechanical properties of parent fiber [9,10].

Color is one of the most important parameters used for sugar quality evaluation. Therefore, it is vital to have a suitable decolorization technology [11,12]. Bone carbon and activated carbon are classic decolorizing agents [13,14]. However, they have been

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replaced by the highly developed ion exchangers for their heavy equipment investment and material cost [15–20]. In some case, ion exchange resins were also used for decolorization. However, when used directly in sugar decolorization, ion exchange resin can be easily polluted by gel. Moreover, the accumulation of calcium ions is easy to plug the resin, which results in ion exchange resin's short using life and difficulty in regeneration [21]. Ion exchange fiber (IEF) is a fibrous ion exchange and adsorption material. It shows potential adsorption performance due to the larger effective specific surface area, higher exchange rate, shorter transit distance, stronger adsorption ability, and longer using life. In addition, its more easily to be regenerated, compared with the traditional ion exchange resin [21].

Many kinds of synthetic fibers, such as polyester, polyacrylonitrile, polyethylene, have been used as the parent fiber of the conventional IEF. However, the bad mechanical properties, unqualified thermal stability and poor anticorrosion performance still limit their applications [22,23]. It is well recognized that PP fiber is a hydrocarbon structure of the lowest specific gravity among the fiber forming polymers. The fiber has good mechanical properties and many advantages such as plentiful of material, resistance to the corrosion of acid and alkali, low price, well flexibility and so forth, which makes it attractive for a large number of real applications [24].

In this study, we effectively prepared a strong basic anion exchange fiber (AIEF) through grafting of p-chloromethylstyrene onto polypropylene fiber. Several interdependent synthesis parameters were investigated. The reagents used to prepare AIEF were easily available and the reaction conditions were relatively mild while no chloromethylation agent was needed. The grafted fibers were characterized through a variety of techniques such as scanning electron microscopy (SEM), diffuse reflectance infrared spectroscopy (FT-IR), differential scanning calorimeter (DSC), thermogravimetric analysis (TGA), monofilament elongation and acid–base neutralization titrations. The removal efficiency of sugar colorants by the anion exchange fibers was evaluated.

2. Experimental

2.1. Reagents and materials

All reagents were analytical grade (AR) and each was used without further purification.

Polypropylene (PP) fiber (1.52 Denier) used for this study was manufactured by Zhongshan Xinshun Special Fiber Co., Ltd. The filament had a diameter of 80 μm. p-chloromethylstyrene (CMS) was purchased from Changzhou Wujin Linchuan chemical co., Ltd., Jiangsu, China. A commercial strong basic anion exchange resin 201 × 7(717) was used as reference.

2.2. Preparation of strong basic anion exchange fiber

The PP fibers were washed with acetone, and dried in a vacuum oven at 50 °C for 48 h. The treated PP fiber and a certain amount of CMS solution were placed in a 100 mL flask. The flask was sealed after the solution was bubbled with nitrogen for 20 min to remove oxygen. The PP fibers with CMS solution in the flask were irradiated in γ-rays with a ^{60}Co source at a radiation dose rate of 0.837 KGy h^{-1} for about 58 h. After irradiation, the simultaneous irradiated fibers was taken out, extracted in a sohxlet apparatus with toluene to remove the residual monomer and homopolymers. The grafted fibers (PP-g-CMS) were dried in a vacuum oven at 50 °C for 48 h and then weighed. The degree of grafting was obtained using the following formula:

$$G\% = \frac{W_g - W_o}{W_o} \times 100 \quad (1)$$

where W_o and W_g are the weight of the original and grafted fiber, respectively.

Subsequently, the grafted fiber was dunked in trimethylamine aqueous solution under stirring at 25–40 °C for 10 h. After the amination reaction, the product fibers, PP based anion exchange fibers (AIEF), were washed with deionized water and dried at 50 °C under vacuum.

2.3. Determination of decolorization performance

International Commission for Uniform Methods of Sugar Analysis (ICUMSA) is the only international organization solely concerned with analytical methods for the sugar industry. According to the ICUMSA Method GS9/1/2/3-8 (2011), determination of sugar solution color at pH 7.0 was used to evaluate the decolorization performance of the prepared anion exchange fiber. ICUMSA color was calculated by the following equation:

$$IU_X = \frac{A_X}{bc} \times 1000 \quad (2)$$

where IU_X and A_X are the ICUMSA color and absorbency of sugar solution at 420 nm or 560 nm, b and c are the thickness of colorimetric utensil and the concentration of sugar solution, respectively.

The decolorization performance of the prepared anion exchange fiber and the reference material were determined and compared with the same standard (the decolorization ratio and decolorization capacity) under the same conditions (static or dynamic method).

The ICUMSA color of the initial and treated sugar solutions was measured using a TU-1900 model UV–Vis spectrophotometer with a 10 mm cell. The absorbencies of the effluent solutions were determined at different intervals time to evaluate the color removal efficiency. Absorbencies were measured at 560 nm, against a blank solution. The decolorization ratio and decolorization capacity were calculated by the following equation:

$$E = \frac{IU_o - IU_e}{IU_o} \times 100\% \quad (3)$$

$$Q = \frac{IU_o - IU_e}{IU_o \times m} \quad (4)$$

where E and Q are the decolorization ratio and decolorization capacity of the prepared anion exchange fiber or the reference material, IU_o and IU_e are the ICUMSA color of the initial and treated sugar solution, and m is the weight of dry fiber material, respectively.

2.4. Characterization and test

Infrared spectra were obtained with FT-IR analyzer (Nicolet/Nexus 670). FT-IR–ATR measurements were carried out at a range of 4000–650 cm^{-1}, equipped with a continuum microscope and ATR objective.

SEM micrographs were obtained with a field emission scanning electron microscope (JSM-6330F, Japan). All samples were sputter-coated with Au.

DSC measurements were evaluated on a DSC822 (METTLER TOLEDO) differential scanning calorimeter ranged from 30 to 250 °C at heating rates of 10 °C min^{-1} under a constant flow of a nitrogen atmosphere of 50 mL min^{-1}.

Thermogravimetric analysis (TGA) was done with a thermo gravimetric analyzer (TGA, Netzsch TG-20) under a nitrogen atmosphere from 30 °C to 600 °C at a heating rate of 20 °C min^{-1}.

A monofilament mightiness instrument (YG001A Textile Mill, Taicang, China) was used in order to evaluate the mechanical properties of parent PP fiber and prepared materials. All tensile testing

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