



Chitosan membrane adsorber for low concentration copper ion removal



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ABSTRACT

Thin chitosan membranes with symmetric and interconnected pore structure were prepared using silica as porogen, and their physical properties including pore structure, pore size distribution, porosity and water affinity were analyzed. The membrane showed a maximum Cu(II) adsorption capacity of 87.5 mg/g in static adsorption, and the adsorption fitted pseudo-second order kinetics and Toth adsorption isotherm. The membranes were then stacked in layers as an adsorber to remove small concentration Cu(II) from water dynamically. At feed concentration of 5 mg/L, the adsorber could retain Cu(II) effectively when its thickness reached over 200 μm , and the performance was further improved by using more membranes layers. Within a certain limit, the adsorber showed a 'flow-independent' loading behavior, an indication of fast mass transfer inside the membrane. The adsorption process was correlated well with bed depth service time (BDST) model, Thomas model and Yoon and Nelson model, and the adsorber was also found to be regenerable and re-usable.

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1. Introduction

Heavy metal pollution is a serious threat to environment and human health. Pushed by increasingly grim situation, emission standard for heavy metals is becoming more stringent. As a result, 'clean' water in the past with very small concentration of metal ions still needs to be 'cleaned' further at present. In such a scenario, traditional treatment technologies like precipitation and flocculation, which were once effective, are not sufficient anymore (Bratskaya, Pestov, Yatluk, & Avramenko, 2009). One of the most promising alternatives is adsorption, particularly for low concentration pollutant removal from wastewater (Li, Zhou et al., 2015). Activated carbon (Lo, Wang, Tsai, & Lin, 2012; Jung et al., 2013), sludge (Nowak, Aschenbrenner, & Winter, 2013), zeolite (Motsi, Rowson, & Simmons, 2011), plant wastes (Kelly-Vargas, Cerro-Lopez, Reyna-Tellez, Bandala, & Sanchez-Salas, 2012; Wan Ngah & Hanafiah, 2008) and resins (Kalaivani et al., 2014), have all been used as

effective adsorbents for heavy metal sequestration. As a natural biosorbent, chitosan has attracted much attention for heavy metal removal in recent years (Wu, Tseng, & Juang, 2010; Zeng et al., 2015). Due to the inherent availability of abundant amino ($-\text{NH}_2$) and hydroxyl ($-\text{OH}$) groups, pristine chitosan binds heavy metal ions through either chelation or electrostatic attraction (Wan Ngah, Teong, & Hanafiah, 2011). As reported in literature, major heavy metals in environmental water system including Cu(II) (Dinu & Dragan, 2010; Dragan, Dinu, & Timpu, 2010; He, Lu, & Luo, 2014; Dalida et al., 2011; Igberase, Osifo, & Ofomaja, 2014), Cd(II) (He et al., 2014), Cr(VI) (Jung et al., 2013; Li, Zhang, Wang, & Liu, 2014; Li, Li, Cao, & Yang, 2015; Li, Li, & Yang, 2016), Hg(II) (Wang, Deng, Xie, & Wang, 2013), Zn(II) (Adamczuk & Kołodyńska, 2015), Pb(II) (Cui et al., 2013; Lu, He, & Luo, 2013) and Ni(II) (Dinu & Dragan, 2010) could all be effectively adsorbed by chitosan or its derivatives. In addition, chitosan is nontoxic and biodegradable, and is therefore considered as a real 'green' and 'environment-friendly' adsorbent. Moreover, compared with conventional adsorbents such as resins, the extensive and cheap sources of chitosan make it economically competitive.

In a dynamic adsorption process, packed column is the most common applied form in industry. However, such column often causes high pressure drops because of the tightly packed small resin particles in a long bed. Packed column also has low adsorption rates because the majority of the binding sites are located inside the

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pores of the resinous adsorbents that requires intra-particle diffusion. Considering the high throughput of macroporous chitosan membranes, stacking these membranes as a membrane bed, which is also referred to here as a membrane adsorber, can be an alternative to resin column for dynamic adsorption. First, it combines the merits of both adsorption process and membrane process, including high efficiency, no phase change, continuous operation and so on. Second, mass transfer of solute in a membrane bed is fast, mainly achieved by convective flow through the pores of the membranes (Knudsen, Fahrner, Xu, Norling, & Blank, 2001). This differs from a resin packed column, where mass transfer is hindered by the slow solute diffusion to the intra-pores of the resin particles. Third, membrane bed has small height-to-diameter ratio, resulting in small operating pressure and making high throughput more readily achievable (Yang, Fane, & Pin, 2002).

Some successful applications of chitosan membrane adsorber have already emerged, but only in the separation of high value-added biological products (Orr, Zhong, Young, & Chou, 2013; Liu, Feng, Shao, & Chen, 2012). The reason that it is barely used for wastewater treatment is believed to be the low performance/cost ratio. This technology is not intended for separation of concentrated product; it aims for recovery of extremely low concentration yet highly valuable materials. Therefore, to make it adopted in wastewater treatment, one of the two criteria must be met: (1) the targeted material to be removed or recovered should be of great value; (2) the targeted material must be in very low concentration that other technologies cannot afford the separation.

In this work, thin chitosan membranes with large pores were prepared by an immersion-precipitation process involving the use of silica particles as porogen. Such membranes, intended as the building blocks of a membrane adsorber, are not easy to get using conventional membrane preparation methods such as non-solvent or thermally induced phase separation. As one of the most common heavy metals studied, copper was selected to evaluate the adsorption performance of the membrane and the membrane adsorber

with static and dynamic adsorption experiments respectively. To our knowledge, this is the first time that such membranes are systematically studied through dynamic filtration although research reports on heavy metal ions removal by chitosan are not rare (Cestari, Vieira, de Oliveira, & Bruns, 2007; Kannamba, Reddy, & Apparao, 2010; Wan, Kan, Rogel, & Dalida, 2010). As a result, we found three models, namely bed depth service time (BDST) model, Thomas model and Yoon and Nelson model, which were initially developed for resin column bed, can also be used to describe the performance of the membrane adsorber. We believe this study is of important significance in the exploration of chitosan membrane technology for wastewater treatment.

2. Experimental

2.1. Materials

Chitosan powder ($M_w = 700\text{--}1000$ kDa, 97.3% deacetylated) was purchased from Zhejiang golden-shell pharmaceutical Co., Ltd. (China) and used as received. Silica with particle size of $7\text{--}15\text{ }\mu\text{m}$ was supplied by Qingdao Meigao Chemicals Co., Ltd. (China). Other reagents including acetic acid, glutaraldehyde solution (50 wt%), sodium hydroxide, ammonium citrate, ammonia, ammonia chloride, acetone, diphenylcarbazine and copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) were analytically pure; bicyclohexone oxalyldidrazone (BCO), sulfuric acid, phosphoric acid and potassium dichromate were guaranteed reagents with purity higher than 99.99%.

2.2. Membrane preparation

Porous chitosan membranes were prepared by using the method introduced in literature (Zeng & Ruckenstein, 1996). First, chitosan powders and glutaraldehyde amounting to 0.3% moles of amino groups of chitosan to be crosslinked were dissolved in 2 wt% acetic acid to prepare a solution containing 2.5 wt% of chitosan. Next, silica

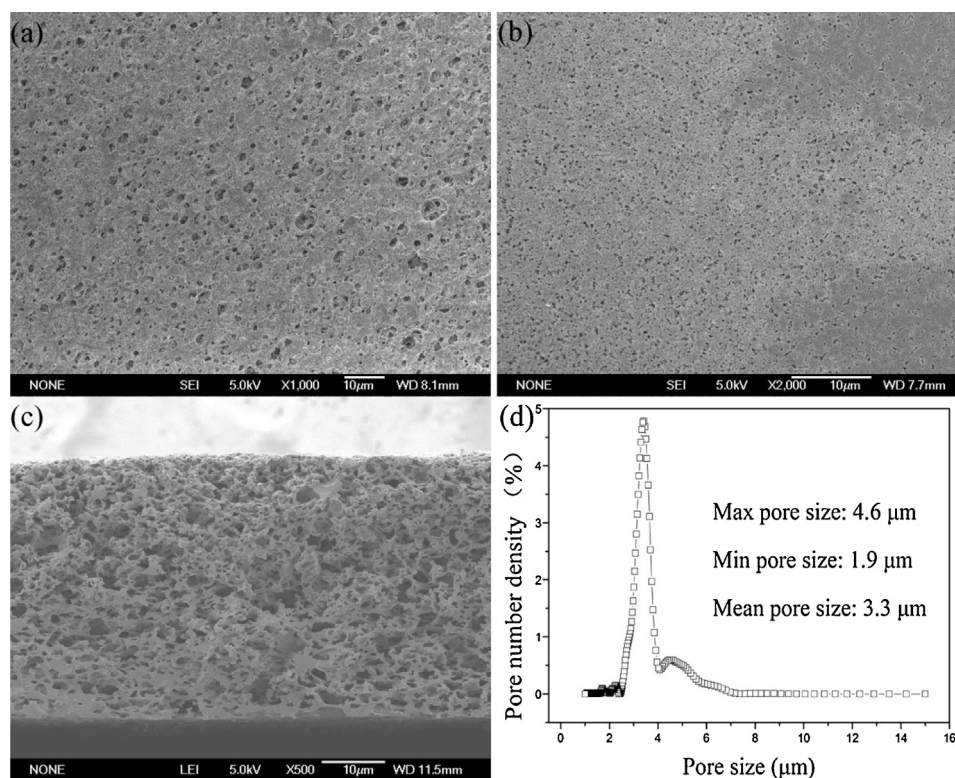


Fig. 1. Morphology and pore size of the as-prepared chitosan membrane: (a) top surface; (b) bottom surface; (c) cross-section; (d) pore size distribution.

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