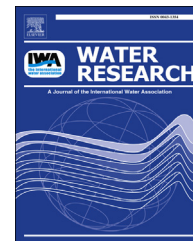




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Irreversible membrane fouling abatement through pre-deposited layer of hierarchical porous carbons

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ABSTRACT

In this work, dual-templated hierarchical porous carbons (HPCs), produced from a coupled ice-hard templating approach, are shown to be a highly effective solution to the commonly occurring problem of irreversible fouling of low-pressure membranes used for pre-treatment in wastewater reuse. For the first time, dual-templated HPCs, along with their respective counterparts – single-templated meso-porous carbon (MPCs) (without macropores) – are tested in terms of their fouling reduction capacity and ability to remove different effluent organic matter fractions present in wastewater and compared with a commercially available powdered activated carbon (PAC). The synthesized HPCs provided exceptional fouling abatement, a 4-fold higher fouling reduction as compared to the previously reported best performing commercial PAC and ~2.5-fold better fouling reduction than their respective mesoporous counterpart. Thus, it is shown that not only mesoporosity, but macroporosity is also necessary to achieve high fouling reduction, thus emphasizing the need for dual templating. In the case of HPCs, the pre-deposition technique is also found to outperform the traditional sorbent-feed mixing approach, mainly in terms of removal of fouling components. Based on their superior performance, a high permeability (ultra-low-pressure) membrane consisting of the synthesized HPC pre-deposited on a large pore size membrane support (0.45 μm membrane), is shown to give excellent pre-treatment performance for wastewater reuse application.

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1. Introduction

In the past few years, considerable effort has been devoted to understanding and addressing the problem of fouling of low-pressure membranes (LPMs) used for pre-treatment in

drinking water treatment and wastewater reuse (Lee et al., 2013; Ajmani et al., 2012; Wang et al., 2011; Ayache et al., 2013; Kim and Dempsey, 2013, 2012). Natural organic matter (NOM) (Howe and Clark, 2002; Lee et al., 2004; Huang et al., 2007), in the case of surface water and effluent organic matter (EfOM) (Huang et al., 2007; Laabs et al., 2006; Zheng et al.,

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2009), in the case of wastewater, are considered to be the major foulants of LPMs. Among the various components comprising the natural organic matter, small organic colloids, ranging from about 3 to 20 nm in diameter, have been demonstrated to be the important membrane fouling components in the case of surface waters (Howe and Clark, 2002). On the other hand, in the case of EfOM, organic colloids, polysaccharides, and proteins with sizes in the range of 10–100 nm were shown to be the major fouling components (Laabs et al., 2006). Among the various colloidal organic matter components, biopolymers have been characterized as the major, irreversible, LPM membrane fouling components based on liquid chromatography with on-line organic carbon detection (LC-OCD) (Kimura et al., 2014; Tian et al., 2013; Henderson et al., 2011; Filloux et al., 2012a; Chen et al., 2014; Kimura et al., 2004; Yamamura et al., 2008).

Many pre-treatment methods such as coagulation (Howe et al., 2006; Zheng et al., 2012; Huang et al., 2012a), biological filtration (Halle et al., 2009; Zheng et al., 2010), ion exchange (Huang et al., 2012a, 2012b), adsorption using porous carbon material (PCM) (Haberkamp et al., 2007; Matsui et al., 2009; Stoquart et al., 2012) or metal oxide particles (Cai et al., 2007; Kim et al., 2008a; Cui and Choo, 2013), and oxidation with ozone (Kim et al., 2008b), as well as modifying membrane surface (Diagne et al., 2012; Chen et al., 2011), and combinations thereof (Huang et al., 2012a; Humbert et al., 2007), have been investigated for reducing organic fouling of membranes. Among these, adsorption using PCM is of great interest because PCM, in addition to removing the fouling organic matter fraction, can also remove precursors for the formation of disinfection by-products (DBPs) as well as synthetic organic compounds (Stoquart et al., 2012). Some studies have indicated that the addition of PCM can increase membrane flux and enhance organic removal but other studies have suggested otherwise (Stoquart et al., 2012; Lin et al., 1999; Huang et al., 2009; Filloux et al., 2012b). Most of the studies involving the successful use of PCM in removing NOM, and thus mitigating membrane fouling, involve use of sub-micron powder activated carbon (PAC) particles (Matsui et al., 2009; Cai and Benjamin, 2011; Ando et al., 2010; Cai et al., 2013). Pre-deposited, crushed PAC particles with a mean size $\sim 1 \mu\text{m}$ were reported to give excellent fouling reduction as compared to non-crushed PAC particles which gave a similar trans-membrane pressure (TMP) profile as the membrane alone (Cai et al., 2013). On the other hand, hardly any studies have been reported on the use of templated carbons for addressing the membrane fouling issue. Moreover, the effect of PCM's porosity on NOM removal and its corresponding effect on reducing membrane fouling has not been explored. Since it has been previously established that the organic matter fraction that causes fouling of LPMs has size larger than 3 nm (Howe and Clark, 2002; Laabs et al., 2006), mesopore dominated (2–50 nm) or mesopore-macropore dominated (>2 nm) hierarchical carbons should thus be the obvious material of choice for removing such NOM components instead of the micropore dominated PCMs. However, to the best of our knowledge, no study has ever been reported on the use of any templated or hierarchically porous carbon for preventing fouling of LPMs. Only studies involving powder activated carbons, which pre-dominantly consist of small micropores

(<2 nm) that dramatically limit their capability for the removal of large-sized molecules (Ji et al., 2010; Teng et al., 2013; Han et al., 2000), have been reported so far and that too with conflicting findings (Stoquart et al., 2012; Lin et al., 1999; Huang et al., 2009; Filloux et al., 2012b). Although lack of mesoporosity has been reported as one of the reasons for the poor performance of PCMs in reducing membrane fouling (Filloux et al., 2012b), no systematic study probing the effects of the presence and extent of mesoporosity in PCMs on their ability to prevent membrane fouling has been reported yet. Only one non-carbonized polymeric mesoporous adsorbent (poly-sulfone colloids with pore size ~ 25 nm) has been reported for reducing membrane fouling (Clark et al., 2005; Koh et al., 2006). Similarly, the effect of the presence or absence of macroporosity in PCMs on membrane fouling abatement, has not been investigated at all.

Over the past few years, a large number of dual templating strategies have been reported for the synthesis of hierarchical (mesopore-macropore dominated) carbons (Petkovich and Stein, 2013; Nishihara and Kyotani, 2012; Lu et al., 2012; Dua et al., 2013; Yoon et al., 2005; Zhang et al., 2010; Liang and Dai, 2009; Górkka and Jaroniec, 2011; Taguchi et al., 2003; Chai et al., 2004; Deng et al., 2007; Petkovich and Stein, 2011). Such techniques involve the use of soft templates (such as surfactant, block co-polymers) and/or hard templates (such as colloidal silica, colloidal polymers), where two templates with dimensions at two different length scales are used together to generate macropores and mesopores. Recently, our group developed a combination of ice and hard templating for producing such hierarchical porous carbons (HPCs) (Dua et al., 2013). The technique involves freeze casting of an aqueous solution of colloidal silica and glucose. The removal of ice template upon freeze drying generates macropores and the removal of hard template (colloidal silica) through NaOH etching generates mesopores. In this study, first of all, a successful application of the synthesized dual templated hierarchical porous carbons (HPCs) is reported for reducing fouling of LPMs. Secondly, to explore the effects of different types of porosities in PCMs – mesoporosity and macroporosity – on irreversible fouling abatement, a set of four different carbons with varying types and levels of pore size distributions were synthesized. Two HPCs, having similar size macropores, but different size mesopore peaks, were synthesized to explore the effect of mesoporosity. Also, two more carbons having similar mesoporosity as the first two, but without macroporosity (synthesized using hard templating alone, without ice-templating) were also synthesized to test the favorable or unfavorable effect of presence of macropores. It is important to note that the differences in fouling mitigation performance of synthesized carbons can be safely assigned to differences in their respective porosities, as all the carbons were synthesized using the same precursor, same template and same synthesis protocol and thus have similar surface chemistry. A commercial PAC, Norit SA-Super, previously shown to be the best performing carbon for biopolymer removal (Haberkamp et al., 2007), was also tested for comparison purposes.

Recently, pre-deposition of adsorbent particles on a membrane surface (Ellerie et al., 2013) or microgranular adsorption (Cai et al., 2007; Cai and Benjamin, 2011; Cai et al., 2013) has been proposed as a novel technology for combating

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