



Influence of hydrophobic/hydrophilic fractions of extracellular organic matters of *Microcystis aeruginosa* on ultrafiltration membrane fouling



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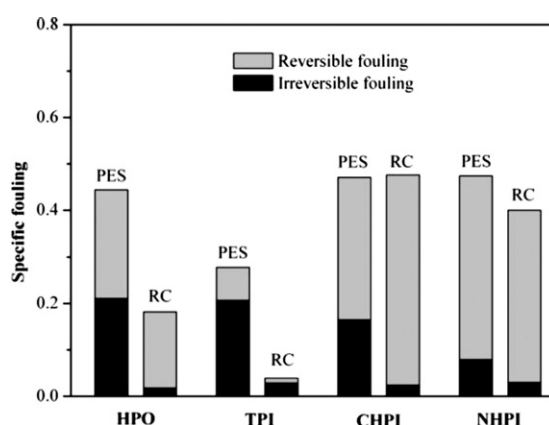
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HIGHLIGHTS

- EOM caused greater irreversible fouling on PES than RC membrane.
- CHPI and NHPI fractions caused greater flux decline over HPO and TPI fractions.
- Protein-like, polysaccharide-like and humic-like substances were the major components responsible for membrane fouling.

GRAPHICAL ABSTRACT



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ABSTRACT

Fouling is a major obstacle to maintain the efficiency of ultrafiltration-based drinking water treatment process. Algal extracellular organic matters (EOMs) are currently considered as one of the major sources of membrane fouling. The objective of this study was to investigate the influence of different hydrophobic/hydrophilic fractions of EOM extracted from *Microcystis aeruginosa* on ultrafiltration membrane fouling at lab scale. The experimental data indicated that EOM exhibited similar flux decline trends on polyethersulfone (PES) and regenerated cellulose (RC) membranes but caused greater irreversible fouling on PES membrane than RC membrane due to its hydrophobic property. It was also observed that charged hydrophilic (CHPI) and neutral hydrophilic (NHPI) fractions caused greater flux decline over hydrophobic (HPO) and transphilic (TPI) fractions. For PES membrane, the order of the irreversible fouling potentials for the four fractions was HPO > TPI > CHPI > NHPI, while the irreversible fouling potentials of RC membrane were tiny and could be ignored. Fluorescence excitation–emission matrix (EEM) spectra and Fourier transform infrared (FTIR) spectra suggested that protein-like, polysaccharide-like and humic-like substances were the major components responsible for membrane fouling. The results also indicated that the irreversible fouling increased as the pH decreased. The addition of calcium to feed solutions led to more severe flux decline and irreversible fouling.

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1. Introduction

Ultrafiltration (UF) has been increasingly used for drinking water treatment over recent decades. However, membrane fouling is one

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of the major obstacles to maintain the efficient performance on water clarification. With respect to fouling, it usually depends on the membrane properties such as hydrophobicity, charge, and morphology (Elimelech et al., 1997; Jucker and Clark, 1994), organic matter properties such as hydrophobicity, molecular weight, and charge density (Cho et al., 2000; Fan et al., 2001; Lin et al., 1999; Zularisam et al., 2011), as well as solution properties such as pH, ionic strength, and calcium concentration (Braghetta et al., 1998; Kulovaara et al., 1999; Lim and Mohammad, 2010). Many previous studies suggested that natural organic matter (NOM) was the dominant foulant for UF membrane (Huang et al., 2007; Jermann et al., 2007, 2008). In particular, the hydrophobic and humic substance fractions of NOM controlled the rate and extent of fouling (Combe et al., 1999; Yuan and Zydney, 1999). However, other researchers have claimed that the non-humic fraction of NOM (i.e., hydrophilic and neutrals) outcompeted the other fractions on membrane fouling (Carroll et al., 2000; Lin et al., 2000). Fan et al. (2001) reported the order of fouling potential of NOM fraction as hydrophilic neutral > hydrophobic acids > transphilic acids > hydrophobic charged. The characteristics of UF membranes also have a significant impact on membrane fouling. Previous studies have shown that strongly hydrophobic membranes tend to foul, causing a much greater flux reduction than hydrophilic membranes while hydrophilic membranes have better permeability (Nakatsuka et al., 1996). Less information is, however, available on the influences of hydrophobic/hydrophilic fractions of organic matters on different UF membrane types.

Cyanobacterial blooming in lakes and reservoirs has increasingly been a worldwide environmental concern (Teixeira and Rosa, 2006, 2007; Yang et al., 2008). Certain harmful cyanobacterial species in the algal blooming (e.g. *Microcystis aeruginosa*) can produce lethal toxins and soluble extracellular organic matters (EOMs) into water during cell growth and lysis (Codd, 2000; Henderson et al., 2008; Ma et al., 2012). Aqueous EOM derived from algae metabolites entering downstream water treatment systems can cause water treatment operational problems such as a rising coagulant demand, membrane fouling, and numerous disinfection by-products (DBPs) during chlor(am)ination (Fang et al., 2010; Henderson et al., 2010; Li et al., 2012; Qu et al., 2012). Recently, UF has been proved an ideal approach to efficiently remove algal cells than conventional clarification processes (Liang et al., 2008; Zhang et al., 2012). However, membrane fouling caused by EOM is still one of the major challenges that limit the widespread application of this technology (Campinas and Rosa, 2010). It has been demonstrated that the hydrophobic proteins and hydrophilic polysaccharides which are the major constituents of algal EOM were widely regarded as responsible for the significant fouling during UF processes (Chiou et al., 2010). Moreover, Qu et al. (2012) investigated the influence of interfacial characteristics of EOM and suggested that the three fouling mechanisms including cake layer formation, pore plugging and hydrophobic adhesion were the main mechanisms for membrane fouling caused by algal EOM. In another study, Zhang et al. (2013) compared the flux decline and fouling potentials from different algal growth phases, with fouling for the stationary > late exponential phase > early exponential phase. However, few studies refer to the hydrophobic/hydrophilic fractionation of organic matters prior to entry into the UF system. It is therefore not clear whether hydrophobic/hydrophilic fractions can influence membrane fouling of UF, particularly when using different types of membranes.

On the other side, solution properties such as ionic strength, pH and divalent cations are able to govern the extent of membrane fouling. Divalent cations affect flux decline during membrane filtration by charge neutralization of both negative membrane surface and negative charge functional groups (carboxylic, phenolics and methoxy carbonyls) of organic matters. Particularly, calcium ions (Ca^{2+}) can promote cation-organic matter complexation and aggregation, thus later resulting in the formation of a densely packed fouling layer and intensifying fouling potential (Katsoufidou et al., 2007; Liang et al., 2008). Solution pH also plays an important role in altering the physical and chemical properties.

Low pH was found to shift the electrostatic charge, allow the organic matter molecules to accumulate more densely at the membrane surface and decrease the natural organic matter to a smaller molecular size range (Huang et al., 2007; Zhang et al., 2012). Therefore, an investigation is needed to clarify the impact of different solution properties on the UF membrane process.

In this paper, a bench-scale UF system was used and algal EOM extracted from *M. aeruginosa* was fractionated into four hydrophobic/hydrophilic fractions. The objectives of this research were to comprehensively investigate the influence of different hydrophobic/hydrophilic fractions of EOM on the membrane fouling of two different membrane types (PES and RC membranes). Furthermore, the critical control factors including calcium ion and pH on the influence of flux decline and fouling reversibility were evaluated in this study. In addition, advanced and novel analytical technologies including fluorescence excitation-emission matrix spectra and Fourier transform infrared spectra were also employed to identifying the organic foulants in UF membrane.

2. Materials and method

2.1. Algae cultivation and EOM extraction

A toxic strain of *M. aeruginosa* (FACHB-912) was purchased from the Institute of Hydrobiology, Chinese Academy of Sciences (Wuhan, China) and cultivated in 1 L conical flasks using BG11 medium. The conical flasks were placed in an incubator at a controlled temperature of 25 °C. To simulate the natural sunlight, the conical flasks were also illuminated by ~30–40 $\mu\text{mol photons m}^{-2} \text{s}^{-1}$ tubular fluorescent lamps with a 12-h diurnal cycle. The algae solution was harvested during its stationary period and then centrifuged at 6000 $\times g$ for 10 min. The algal EOM was extracted by subsequently filtering the supernatants through 0.45 μm cellulose acetate membranes.

2.2. XAD and IRA resin fractionation

The XAD-8/XAD-4/IRA-958 (Amberlite, USA) column was used to fractionate the algal EOM into hydrophobic (HPO), transphilic (TPI), charged hydrophilic (CHPI) and neutral hydrophilic (NHPI) fractions according to the method developed by Carroll et al. (2000). The EOM sample, acidified to pH 2, was passed consecutively through the XAD-8 and XAD-4 resins. The XAD-8 and XAD-4 resin columns were back-eluted with NaOH (0.1 M) and HPO and TPI fractions were obtained, respectively. The remaining sample, neutralized to pH 8, was subsequently passed through the IRA-958 resin. The CHPI fraction was eluted from the IRA-958 resin with 1 M NaOH/NaCl mixture. The non-retained sample comprised the NHPI fraction. The experimental recovery rate of fractionation was 87%. The fractions of HPO, TPI, CHPI and NHPI accounted for 25.6%, 9.9%, 19.8% and 44.7% of dissolved organic carbon (DOC) in EOM, respectively. Each EOM fraction solution was adjusted to neutral pH with an initial DOC concentration of ~4.5 mg C L^{-1} prior to tests.

2.3. Membranes and UF tests

The flat sheet of polyethersulfone (PES) membrane and regenerated cellulose (RC) membrane (Sartorius, Germany) was used in the experiment. Table 1 summarized the two different UF membranes, based on hydrophobic or hydrophilic properties and contact angle measurements. The two types of membranes were anticipated to exhibit different trends of flux decline and membrane fouling depending on EOM characteristics and membrane properties.

As shown in Supplementary Fig. S.1, the UF tests were carried out by using a dead-ended filtration cell with approximately 20 cm^2 membrane area. Nitrogen gas at the constant pressure of 0.1 MPa was used to drive the feed solution through the membrane. Prior to each experiment, the stabilized pure water flux of each membrane was

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