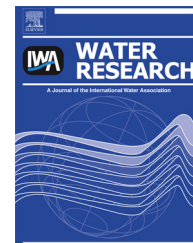


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Identifying polyvinylidene fluoride ultrafiltration membrane fouling behavior of different effluent organic matter fractions using colloidal probes

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ABSTRACT

The interaction forces between effluent organic matter (EfOM) fractions and membrane were measured by atomic force microscopy in conjunction with self-made membrane material colloidal probes. The inter-EfOM-fraction and intra-EfOM-fraction interactions were investigated using corresponding EfOM-fraction-coated colloidal probe. We combined this analysis with corresponding fouling experiments to identify the EfOM fractions responsible for polyvinylidene fluoride (PVDF) ultrafiltration membrane fouling. Results show that hydrophilic and hydrophobic fractions were the dominant fractions responsible for membrane fouling and flux decline in the initial and later filtration stages, respectively, which was mainly attributed to the stronger PVDF-hydrophilic fraction and intra-hydrophobic-fraction interaction forces. This phenomenon, in conjunction with the fact that each interaction force of PVDF–EfOM fraction was stronger than corresponding intra-EfOM-fraction force, suggests that the elimination of the PVDF-hydrophilic fraction interaction force is the best strategy for controlling EfOM fouling. Moreover, the inter-EfOM-fraction interaction force was mainly controlled by the corresponding intra-EfOM-fraction interaction forces. And, while the membrane–EfOM fraction and intra-EfOM-fraction interactions for each type of EfOM fraction are equivalent, the EfOM fractions with the molecular weight smaller than the molecular weight cutoff of the membranes used were mainly responsible for membrane fouling rather than the relatively high-molecular-weight fractions.

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1. Introduction

Secondary effluent reuse and reclamation are being increasingly emphasized as a strategy to resolve water shortages and prevent deterioration in the aquatic environment from

wastewater disposal (Shannon et al., 2008; Grant et al., 2012). Ultrafiltration (UF) membrane technology has been increasingly used in wastewater reuse and reclamation because it offers remarkable advantages relating to effluent quality and reduced footprint compared with conventional processes. However, membrane performance deteriorates with the

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accumulation and adsorption of effluent organic matter (EfOM) on the surface or in the pores of the membrane; that is, membrane fouling remains a major limitation for the wide application of membrane technologies (Wintgens et al., 2005; Henderson et al., 2011). Therefore, it is essential to unravel the membrane fouling mechanism of EfOM as this would be very useful for the widespread application of UF membranes in the field of secondary effluent reuse and reclamation and the control of membrane fouling.

A number of studies have focused on identifying the potential fractions of EfOM responsible for membrane fouling and understanding the fouling behavior. The most commonly used approach is the fractionation of EfOM into hydrophilic (HPI), hydrophobic (HPO), and transphilic (TPI) fractions based on the functionality of EfOM, and then the identification of the potential fractions responsible for membrane fouling by comparing their behavior, thereby obtaining a deeper understanding of membrane fouling mechanisms caused by EfOM (Wang et al., 2011). Several research groups have suggested that HPI fractions might be a more significant foulant for low-pressure membranes compared with HPO and TPI fractions (Zularisam et al., 2007; Zhu et al., 2010; Shen et al., 2010; Gray et al., 2007). However, Kim et al. provide evidence that HPI base/neutral fractions are not important membrane foulants (Kim and Dempsey, 2010). From a similar perspective, Shon et al. found that the flux decline with the HPO fraction was very high compared with the TPI and HPI fractions, which suggested the HPO fraction was the main component that caused severe fouling (Shon et al., 2006). However, Kim et al. also reported that almost all fouling of polyether sulfone UF membranes was due to hydrophobic plus hydrophilic (HPO/HPI) acids, and that although HPO/HPI base/neutral fractions were the dominant form of EfOM, they did not cause any significant fouling (Kim and Dempsey, 2008). Moreover, researchers have suggested that there is a close relationship between membrane fouling and the molecular weight distribution of foulants. For example, Shen et al. reported that foulants with large molecular weight tend to have higher fouling potential (Shen et al., 2010). On the other hand, several studies have demonstrated that relatively low-molecular-weight foulants were more easily adsorbed both inside pores and on the membrane surface (Teixeira and Sousa, 2013; Howe and Clark, 2002). Clearly, there are contradictory results on the fouling tendency of different EfOM fractions, which may be explained by differences in experimental conditions, membrane materials, and analytical methods used in these studies.

In 1998, Bowen and co-workers found that using atomic force microscopy (AFM) in conjunction with a colloidal probe to quantify the force of interaction between a single particle and a membrane surface was a relatively rapid procedure for assessing the potential of membrane fouling by particles (Bowen et al., 1998). Subsequently, an increasing number of systematic studies have attempted to unravel membrane fouling behavior using this method. Almost all studies in this field have demonstrated that the measurement of the membrane–foulant and foulant–foulant interaction forces is a powerful method for predicting membrane fouling behavior (Yamamura et al., 2008; Hashino et al., 2011; Lee and Elimelech, 2006). This phenomenon can be explained by the

common notion that, in the initial filtration stage, when the foulant is in the vicinity of the membrane–bulk foulant interface, the interaction force between membrane surface and foulant will determine the attachment of the foulant onto the membrane surface. With the accumulation of foulants on the membrane surface, the interaction forces between the bulk foulants and foulants deposited on the membrane surface will determine the attachment of the foulant onto the fouled-membrane surface (Basri et al., 2012; Tang et al., 2011). Recently, a membrane material colloidal probe was developed by our group that was used to unravel the fouling behavior of typical organic foulants in PVDF ultrafiltration membranes (Wang et al., 2013). However, in the current literature on using AFM force measurements to investigate membrane fouling behavior, most studies have focused on understanding fouling behavior of model foulants (such as humic acid, protein, and polysaccharides), while there have been few reports relating to membrane fouling of complex actual wastewaters.

In this study, one of the widely used membrane materials, polyvinylidene fluoride (PVDF), was chosen as a membrane material to prepare flat sheet ultrafiltration membranes. A membrane material (PVDF) colloidal probe was prepared by sintering PVDF microspheres onto the free end of a cantilever. The EfOM-fraction-coated colloidal probes were prepared by adsorbing corresponding EfOM fractions on the surface of PVDF microspheres that had been sintered onto the cantilever. The EfOM was isolated into hydrophobic, hydrophilic, and transphilic fractions and several fractions with different molecular weight distributions. In conjunction with PVDF colloidal probes, AFM was used to quantitatively measure the interaction forces between PVDF and different EfOM fractions. The interactions between the same EfOM fractions (intra-EfOM-fraction) and between the different EfOM fractions (inter-EfOM-fraction) were determined by AFM in conjunction with the corresponding EfOM-fraction-coated colloidal probe. Fouling experiments of different fractions were conducted and combined with corresponding AFM force measurements to compare and provide a visual insight into the fouling mechanism of different EfOM fractions and the interaction characteristics of different fractions. The ultimate goal is to identify the dominant EfOM fractions responsible for membrane fouling and to understand the fouling behavior of each EfOM fraction in different filtration periods, which would provide a theoretical basis for the application of membranes in the reuse field of secondary effluent and the prevention/control of membrane fouling.

2. Materials and methods

2.1. Wastewater samples and fractionation methods

The secondary effluent samples were obtained from the fourth municipal wastewater treatment plant in Xi'an (China), which is the largest wastewater treatment plant in Xi'an, and a conventional A²/O process is used in this plant. Samples were filtered with 0.45 μm microfiltration membranes to remove any suspended solids and insoluble matter. The effluent of 0.45 μm microfiltration membranes (prefiltered

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