



Probing molecular interaction mechanisms of organic fouling on polyamide membrane using a surface forces apparatus: Implication for wastewater treatment

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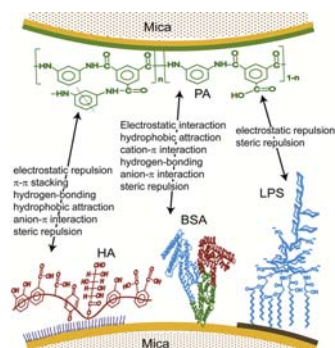
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HIGHLIGHTS

- Interaction mechanisms of organic fouling on polyamide (PA) membrane
- Adhesion between PA and humic acid on 3-aminopropyltriethoxysilane-modified mica
- Adhesion between PA and mica-supported bovine serum albumin
- Repulsion between PA and lipopolysaccharides on polystyrene-coated mica
- Time-dependent adsorption of HA, BSA, or LPS in solution on PA

GRAPHICAL ABSTRACT



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ABSTRACT

Surface fouling is the dominant fouling mechanism of thin-film composite (TFC) membranes used in reverse osmosis (RO) technology. Understanding the complex interactions between foulant-membrane which drive the attachment and growth of foulants on membrane surface is of both fundamental and practical importance. This work aims to understand the molecular interaction mechanisms of organic fouling on RO-TFC membranes. A surface forces apparatus (SFA) was employed to directly measure the interaction forces and time-dependent adsorption behaviors between model organic foulants: humic acid (HA), bovine serum albumin (BSA), and lipopolysaccharides (LPS) and a polyamide (PA) thin film. PA thin film was prepared by interfacial condensation polymerization on mica substrates using *m*-phenylenediamine and 1,3,5-benzenetricarbonyltrichloride. The interaction forces between PA films and different foulant (HA, BSA, LPS) were directly measured under controlled solution chemistries in an asymmetric configuration (foulant-deposited PA films vs. PA films). The adsorption behaviors of these foulants on PA films were directly monitored in a symmetric configuration (PA films vs. PA films). These interactions were examined as a function of both contact time and solution chemistry (ionic strength). In asymmetrical configuration, both HA and BSA show repulsion with PA surface during approach and adhesion during separation, but LPS demonstrates repulsion only. In symmetrical configuration, all the foulants show adsorption on PA surface and the initial adsorption rate of foulant to PA surface follows the order of BSA > LPS > HA. The interaction mechanisms between PA films and foulants were discussed based on the interaction forces and interaction dynamics measurement.

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1. Introduction

Reverse osmosis (RO) technology using thin-film composite (TFC) membranes has been effectively applied for water/wastewater treatment (e.g., seawater desalination, drinking water treatment, and wastewater reclamation) (Elimelech and Phillip, 2011; Giwa et al., 2016; Kim et al., 2011; Lee et al., 2011; Palma et al., 2016; Rodríguez-Calvo et al., 2015; Shen and Schäfer, 2015; Yin et al., 2013). Most commercial TFC membranes are supported by a highly “porous” mechanical layer that is coated with a cross-linked insoluble “nonporous” aromatic polyamide (PA) thin-film layer. The selectivity and permeability of RO-TFC membranes are determined by the structural properties of the membranes. A challenging and unresolved problem limiting the wide application of RO-TFC membranes in water/wastewater treatment is the accumulation and fouling of substances and particles on the membrane surfaces and within membrane pores (Jiang et al., 2017; Ochando-Pulido, 2016). Such fouling results in membrane pore blockage, pore narrowing, and cake layer formation that can deteriorate the membrane performance and increase operation expenses.

The extent of membrane fouling is dictated by the properties of both the membrane surfaces and foulants as well as interactions between them (Al-Amoudi, 2010; Goosen et al., 2004). Because of the very small pores of RO-TFC membranes, surface fouling is the dominant fouling mechanism: foulants are initially deposited on a membrane surface driven by foulant-membrane interactions while the growth of foulants on the membrane is determined by foulant-foulant interactions. Understanding these complex interactions at molecular level and nanoscale is of both fundamental and practical importance, and can aid in the prevention and control of membrane fouling. Interfacial force measurements between foulant and membrane and between foulant-foulant can provide a quantitative evaluation of these interactions. In recent years, atomic force microscopy (AFM), as a highly sensitive force probing technique, has been applied in studying membrane fouling, e.g., membrane scaling, abiotic and biotic fouling, protein fouling, etc. (Johnson and Hilal, 2015). To understand the fouling of organic materials on PA membrane, interaction studies have been done between an AFM colloid probe glued with a carboxylate-modified-latex (CML) particle and a membrane surface in aqueous solutions of different water chemistries with or without foulants (Ang and Elimelech, 2007, 2008; Ang et al., 2006; Herzberg et al., 2009; Kang et al., 2007; Lee and Elimelech, 2006; Li and Elimelech, 2004). The CML particle was used to mimic any organic foulants that carry carboxyl groups such as humic acid (HA), alginate, Suwansnee River natural organic matter, extracellular polymeric substances (EPS), bovine serum albumin (BSA), or fatty acid. These studies demonstrated that adhesion force measurements obtained by AFM allowed quantitative understanding of foulant-membrane and foulant-foulant interactions during membrane fouling (Ang and Elimelech, 2007, 2008; Ang et al., 2006; Herzberg et al., 2009; Kang et al., 2007; Lee and Elimelech, 2006; Li and Elimelech, 2004).

The above mentioned AFM studies using CML particle as a surrogate for foulants are capable of providing interaction information originating from carboxyl groups in the foulants. Although carboxyl groups are common in organic foulants, there are many other functional groups in organic foulants. For example, HA, a major organic foulant in natural waters, contains a wide variety of functional groups, such as carboxyl groups, quinones, phenols, catechols, and sugars, etc. Considering the complex nature of organic foulants such as molecular weight, polarity, functional groups, conformation etc., a wide range of forces may exist between foulants and membrane surfaces such as electrostatic interaction, van der Waals, H-bonding, hydration, hydrophobic interaction, steric interaction, etc. These interaction forces between the foulants and the membrane surfaces influence the initial interactions and adsorption of foulants onto membrane surfaces, which is a key step controlling the following fouling process. Thus understanding the initial fouling in terms of direct interaction forces and interaction dynamics

between foulants and membrane surfaces (e.g., PA films) is critical for a complete understanding of the fouling process and antifouling strategies.

In this work, for the first time, the interaction forces between foulants and PA films under controlled solution chemistries have been directly measured using a surface forces apparatus (SFA). SFA has been widely used to measure the surface forces (F) as a function of absolute separation distance (D) in many biological and non-biological systems, with high force sensitivity and distance resolution (Helm et al., 1991; Israelachvili et al., 2010; Israelachvili and Adams, 1976, 1978; Lu et al., 2011; Yang et al., 2012). SFA is able to measure molecular forces and time-dependent interfacial effects in complex fluids, which provides useful information on intermolecular interaction mechanisms as well as molecular or surface deformations in situ and in real time. For SFA measurements, thin PA films were prepared on mica surfaces via interfacial condensation polymerization (Jeong et al., 2007; Kim et al., 2009). HA, BSA, and LPS were used as model organic and/or bacterial foulants (Fig. 1) considering their following characteristics: (1) HA, a molecular mixture that includes carboxylic acids, quinones, phenols, catechols, and sugars, is the major organic foulant in natural waters; (2) BSA is one kind of well-studied protein abundant in water systems and notorious for adsorption onto many kinds of surfaces, which is selected as a model system to study protein fouling on PA membrane; and (3) LPS are major surface components of Gram-negative bacteria and occupy 75% of the surface of *E. coli*, which contain a truncated lipid A chain and inner and outer core polysaccharides. In this work, the interaction forces between PA films and different foulant (i.e., HA, BSA, LPS) were directly measured using the SFA under different aqueous solution conditions (e.g., salt concentration) in an asymmetric configuration (i.e., foulant-deposited PA films vs. PA films) (Fig. 2 a–c). The adsorption behaviors of these organic foulants on PA surfaces were also directly monitored using the SFA in a symmetric configuration (i.e., PA films vs. PA films) (Fig. 2 d). These interactions were examined as a function of both contact time and solution chemistry (mainly ionic strength), and the interaction mechanisms between PA films and foulants were discussed in terms of the interaction forces and interaction dynamics measurements.

2. Materials and methods

2.1. Materials

m-Phenylenediamine (MPD, >99%, Fluka (Sigma-Aldrich), St. Louis, MO, USA) and 1,3,5-benzenetricarbonyl trichloride (TMC, >98%, Aldrich, St. Louis, MO, USA) were used for interfacial condensation polymerization to prepare PA thin-film layer on back silvered thin mica sheets (ruby mica blocks, grade 1, S&J Trading Inc., NY, USA). Polystyrene (PS) (MW $\sim 10^6$, $M_w/M_n \sim 1.10$) was obtained from Polysciences (PA, USA), 3-aminopropyltriethoxysilane (APTES) (98%) was purchased from Alfa Aesar (USA), sodium chloride (NaCl, $\geq 99.5\%$) was from Sigma-Aldrich (USA), toluene (HPLC grade) and hexane (HPLC grade) were from Fisher Scientific (Canada). Aqueous solutions were prepared in 18.2 M Ω deionized water (Millipore, Mississauga, ON, Canada) and filtered through 0.22 μm hydrophilic PVDF filters (Fisher Scientific, Canada). Organic solutions were filtered through 0.2 μm hydrophobic PTFE filters (Fisher Scientific, Canada) prior to use.

Both HA and BSA were purchased from Sigma-Aldrich, USA and LPS was provided by Invivogen, Canada. HA has an average molecular weight of ~ 20 kDa and contains carboxylic acids with pK_a values of 3.39, 4.78 and 6.06 (Aleixo et al., 1992; Avena et al., 1999). Based on the manufacturer's specifications, BSA has a molecular weight of around 66 kDa and a pI of 4.7. The LPS used was ultrapure LPS prepared from *E. coli* K12, with an average molecular weight of 7 kDa, containing phosphate groups ($\text{pK}_{a\text{s}}$ 2.2 and 7.2) and carboxyl groups.

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