



## Role of adsorption in combined membrane fouling by biopolymers coexisting with inorganic particles



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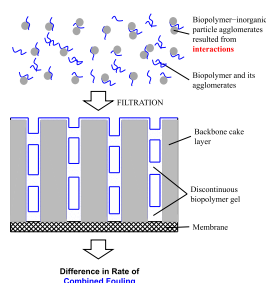
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### HIGHLIGHTS

- Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, kaolin and SiO<sub>2</sub> impacted differently on fouling by alginate.
- The fouling index order was in accord with that of adsorption capacity.
- Combined fouling layer comprised of main cake structure and local gels.
- Interactions of both physical and chemical natures played important roles.

### GRAPHICAL ABSTRACT



### ARTICLE INFO

#### Article history:

Received 2 July 2017

Received in revised form

19 September 2017

Accepted 24 September 2017

Available online 29 September 2017

Handling Editor: Xiangru Zhang

#### Keywords:

Ultrafiltration

Membrane fouling

Biopolymers

Inorganic particles

Water treatment

### ABSTRACT

This study was conducted in order to obtain a better understanding of the combined fouling by biopolymers coexisting with inorganic particles from the aspects of fouling index, fouling layer structure and biopolymer–particle interactions. Calcium alginate was used as the model biopolymer and Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, kaolin, and SiO<sub>2</sub> were used as model inorganic particles. Results showed that the combined fouling differed greatly among the four types of inorganic particles. The differences were attributed particularly to the different adsorption capacities for calcium alginate by the particles with this capacity decreasing in the order of Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, kaolin and SiO<sub>2</sub>. Particle size measurement and electron microscopic observation indicated the formation of agglomerates between calcium alginate and those inorganic particles exhibiting strong adsorption capacity. A structure was proposed for the combined fouling layer comprised of a backbone cake layer of alginate–inorganic particle agglomerates with the pores partially filled with discontinuous calcium alginate gels. The filterability of the fouling layer was primarily determined by the abundance of the gels. The strength of physical interaction between calcium alginate and each type of inorganic particle was calculated from the respective surface energies and zeta potentials. Calculation results showed that the extent of physical interaction increased in the order of Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, kaolin and SiO<sub>2</sub>, with this order differing from that of adsorption capacity. Chemical interactions may also play an important role in the adsorption of alginate and the consequent combined fouling. High-resolution XPS scans revealed a slight shift of electron binding energies when alginate was adsorbed.

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## 1. Introduction

Since the late 1980s, membrane filtration and ultrafiltration in particular has been increasingly applied in lieu of sand filtration as a central unit operation for drinking water production. The merits of membrane filtration include the consistently high quality of the filtered water in terms of turbidity and particle count, small footprint requirement, and adaptability to almost all raw water qualities (Crittenden et al., 2012; Liu, 2014; Ang et al., 2015). Membrane filtration relies on the sieving effect for the removal of colloidal and suspended particles which, problematically, leads to the accumulation of particles on the membrane surfaces. This is referred to as membrane fouling. Membrane fouling can substantially increase the operational cost and complexity of treatment as it can drastically increase the filtration resistance and may necessitate frequent membrane cleaning (Tijing et al., 2015; Meshram et al., 2016).

Significant effort has been devoted to the characterization and understanding of membrane fouling. The foulants on membranes typically include inorganic particles, organic substances and microorganisms (such as algae) (Peiris et al., 2013; Yamamura et al., 2014) with the actual composition depending on the raw water quality and the strategies employed for membrane fouling control (Lee et al., 2004; Guo et al., 2012; Dong et al., 2015; Hu et al., 2016). In most cases, inorganic particles are the major foulants in terms of mass while organic substances are the key foulants in terms of impact on throughput (Lee et al., 2006; Kennedy et al., 2008; Tian et al., 2013a). The fouling layer formed by inorganic particles usually exhibits good filterability and can be easily removed by air scouring and backwashing (Jermann et al., 2008a,b; Tian et al., 2013b; Schulz et al., 2016; Chang et al., 2017). In contrast, organic substances, biopolymers (e.g. polysaccharides and proteins) in particular, tend to form a gel-like fouling layer, which exhibits low hydraulic conductivity and is extremely difficult to remove (Kimura et al., 2004; Zhang et al., 2013; Wang et al., 2017). A correlation of membrane fouling rate with biopolymer concentration has been revealed in a number of previous studies (Lee et al., 2006; Tian et al., 2013a; Kimura et al., 2014; Quang et al., 2016).

Despite the general ease of filterability of inorganic particulate assemblages, the coexistence of inorganic particles with biopolymers could have a substantial influence on fouling development (Sioutopoulos and Karabelas, 2012; Lin et al., 2014; Chen et al., 2015). A number of studies have confirmed that some inorganic particles (such as silica) may aggravate membrane fouling resulting from retention of biopolymers (Jermann et al., 2007, 2008; Huang et al., 2009; Schulz et al., 2016). The adverse effect of inorganic particles was confirmed in a recent study in which kaolin was dosed into biologically treated water collected from a full-scale water plant (Ao et al., 2016). In contrast, some other inorganic particles, such as ferric (hydr)oxides, have been shown to greatly alleviate membrane fouling by biopolymers (Jung et al., 2006; Qiao et al., 2008; Amjad et al., 2015; Chen et al., 2015). These inorganic particles appear to behave in a manner similar to ferric iron-based coagulants, which are widely used not only for natural organic matter removal but also for membrane fouling control.

The underlying mechanisms governing the effect of inorganic particles on fouling require further clarification. It is hypothesized that if the particles are able to strongly interact with biopolymers to form particle–biopolymer agglomerates, a more filterable cake-like fouling layer rather than gel-like fouling layer may be formed with inorganic materials such as ferric (hydr)oxides likely to belong to this category of particles. In contrast, if the particle–biopolymer interactions are weak or non-existent, the dispersion of particles in the biopolymer matrix might be expected to lead to the formation of a highly impermeable fouling layer. Silica particles may belong to

this category. This study was conducted to test the above hypothesis, with the principal aim being to better understand the combined fouling by biopolymers and inorganic particles, particularly from the viewpoint of particle–biopolymer interactions and the impact on the fouling layer structure. The inorganic particles commonly encountered in the feed water to UF membranes include those in raw waters such as clays, silica, and ferric and aluminum oxides, and those resulting from the hydrolysis of the ferric and aluminum salt coagulants. Possible interactions between particles and biopolymers may include those of a physical nature such as the nonpolar van der Waals and electrostatic double layer forces and the polar Lewis acid–base force, and those of a chemical nature such as covalent bonding. Different particles may interact with biopolymers by different mechanisms and to different strengths. Results of this study should assist in the development of more cost-effective membrane fouling control strategies for ultrafiltration processes.

## 2. Materials and methods

### 2.1. Model biopolymer and particles

Alginate, a polysaccharide, was used as the model biopolymer and is known to form a gel in the presence of calcium ions. The alginate stock dispersion ( $1.00 \text{ g L}^{-1}$ ) was prepared by first adding 1.00 g sodium alginate (Sigma-Aldrich), 2.92 g NaCl, 0.17 g  $\text{NaHCO}_3$  and 0.2 g  $\text{NaN}_3$  into 0.9 L high-purity water (Milli-Q, Millipore), followed by magnetic stirring for 12 h, then adding 0.28 g  $\text{CaCl}_2$ , followed by further magnetic stirring for 12 h with the volume of the mixture, finally, increased to 1.00 L. The alginate working dispersion ( $100 \text{ mg L}^{-1}$ ) was obtained by ten-fold dilution of the stock dispersion using ultrapure water. (Selection of the working dispersion concentration was arbitrary as it would not affect the structure and filterability of the formed fouling layer). The working dispersion had a pH of  $\sim 7.5$  and an ionic strength of 5 mM.

$\text{Fe}_2\text{O}_3$  (AR, Xilong Chem.), kaolin (Ziyi Co.),  $\text{SiO}_2$  (AR, Xilong Chem.) and  $\text{Al}_2\text{O}_3$  (AR, Xilong Chem.) were used as the model inorganic particles. The working dispersion of alginate coexisting with each type of inorganic particle was prepared by adding 10 mL alginate stock dispersion and 2.00 g inorganic particles into 90 mL ultrapure water, followed by magnetic stirring for 5 min and ultrasonication for 5 min, then adding 1 M  $\text{NaHCO}_3$  or 10 mM HCl for pH adjustment to  $\sim 7$ , and finally bringing the volume to 100 mL. The alginate and inorganic particle concentrations were  $100 \text{ mg L}^{-1}$  and  $20 \text{ g L}^{-1}$ , respectively. (The inorganic particle concentration was deliberately chosen to be much higher than the alginate concentration because the feed water to UF membranes typically has a concentration of inorganic particles several orders of magnitude higher than that of biopolymers (Ao et al., 2016)). For comparison, a working dispersion of each type of particle ( $20 \text{ g L}^{-1}$ ) was also prepared in an identical manner except that no alginate stock dispersion was added. Each working dispersion was prepared immediately prior to filtration study.

### 2.2. Characterization of particles in working dispersions

The particle size distribution (PSD) and zeta potential of the particles in the working dispersions of alginate and inorganic particles were determined using a dynamic laser scattering instrument (Delsa Nano C, Beckman, USA). Because of the possible interactions between alginate and inorganic particles, alginate–particle agglomerates may be formed with a much larger particle size than the original particles. The PSD of the agglomerates was determined using a laser diffraction particle size analyzer (LS13320, Beckman, USA).

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