



Performance of adsorption pretreatment in mitigating humic acid fouling of ultrafiltration membrane under environmentally relevant ionic conditions



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HIGHLIGHTS

- HA fouling and its control under a wide range of ionic conditions was examined.
- HA fouling decreased at first and increased afterwards as the ionic strength rising.
- HA removal efficiency of MAR was influenced more notably by ionic environments.
- MAR and PAC exhibited different adsorption preferences for HA fractions.
- MAR outperformed PAC in HA fouling control under most ionic environments.

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ABSTRACT

This study comprehensively investigated humic acid (HA) fouling of ultrafiltration (UF) membrane and the efficiency of adsorption pretreatment in its control under environmentally relevant ionic conditions. Two types of adsorbents, mesoporous adsorbent resin (MAR) and powdered activated carbon (PAC), were examined, and the ionic conditions investigated included ionic strength varying from 1 mM to 500 mM and different ionic composition at three ionic strength levels. HA fouling decreased at first and increased afterwards as the ionic strength rose, and the influence of ionic composition varied between different levels of ionic strength. Moreover, the efficiency of physical cleaning (imposing surface shear stress and backwashing) decreased with the elevation of ionic strength and calcium ions because of the intensification of the HA-membrane and HA-HA interactions. With the increase of ionic strength and calcium concentration, HA removal efficiency of MAR pretreatment increased more rapidly than that of PAC pretreatment. Meanwhile, due to the characteristics of the adsorbents' pore structure, MAR pretreatment efficiently removed HA that would deposit on/within the membrane while PAC pretreatment preferentially adsorbed HA that would pass through the membrane. Therefore, MAR pretreatment significantly outperformed PAC pretreatment in HA fouling control under most environmentally relevant ionic conditions.

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1. Introduction

With the improvement in membrane material and the decrease of membrane costs, the application of ultrafiltration (UF) in water industry has increased significantly in the past decades [1]. High performance in

the removal of colloids, particles and organisms makes UF an attractive technology for drinking water production and wastewater reclamation [2,3]. In recent years, UF has also been extensively employed as pretreatment for nanofiltration and reverse osmosis in the desalination of brackish water and seawater [4–7]. However, membrane fouling caused by natural organic matter (NOM) remains a major obstacle for more widespread application of UF [8,9].

Solution chemistry (e.g., solution pH, ionic environments) has a profound impact on NOM fouling because it could influence the interfacial properties of NOM and membrane and thus the physical and chemical interactions between them [10–12]. In the water industry, although solution pH remains neutral in most situations, ionic environments (including ionic strength and ionic composition) of surface water,

Abbreviations: HA, humic acid; MAR, mesoporous adsorbent resin; MW, molecular weight; MWCO, molecular weight cutoff; NOM, natural organic matter; PAC, powdered activated carbon; PES, polyethersulfone; TFI, total fouling index; TMP, trans-membrane pressure; UF, ultrafiltration.

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wastewater, brackish water and seawater varied substantially [6,13]. Plenty of studies have been carried out to examine the effects of ionic environments on NOM fouling, however, consistent conclusions have not been reached yet. For example, although many studies reported that both high ionic strength and the presence of divalent ions would aggravate membrane fouling [14–16], Tian et al. [17] found that 10 mM Na⁺ alleviated membrane fouling caused by humic acid (HA) and bovine serum albumin, and Liu et al. [18] observed that HA fouling decreased with the elevation of ionic strength and calcium concentration. Besides, some studies showed that HA fouling increased at first and decreased afterwards as calcium concentration rose [19,20]. Therefore, further studies are necessary to explore NOM fouling under environmentally relevant ionic strength and to examine the influence of divalent ion concentration at different levels of ionic strength.

As an efficient technology for NOM removal, adsorption pretreatment is widely employed upstream of UF to mitigate membrane fouling as well as to improve the permeate water quality [21]. Several types of adsorbents, including powdered activated carbon (PAC) which is a frequently-used type of adsorbent in water treatment and mesoporous adsorbent resin (MAR) that is an adsorbent specially developed for UF membrane fouling control, have been explored to integrate with UF process [21,22]. A lot of studies have been conducted to investigate the efficacy of different types of adsorbents in retarding membrane fouling caused by NOM with different sources [22–27]. These studies substantially contributed to the development and application of the hybrid adsorption/UF process, but most of them were carried out with fresh surface water or synthetic water with similar ionic condition. Similar to the interactions between NOM and membrane, those between NOM and adsorbents and the consequent NOM removal efficiency are also significantly affected by ionic environments [28,29]. Therefore, the efficiency of adsorption pretreatment in mitigating NOM fouling might vary for brackish water, seawater and wastewater with different salinity, but few studies could be found in the literature dealing with this topic.

In this study, a comprehensive investigation was carried out to examine NOM fouling and the performance of adsorption pretreatment in its control under environmentally relevant ionic conditions. A commercial HA widely used in previous studies was employed as model NOM. Ionic strength varying from 1 mM to 500 mM was investigated, and the influence of calcium concentration was studied at three levels of ionic strength (10, 100 and 500 mM), respectively. Fouling control efficiency of adsorption pretreatment was evaluated by calculating its efficiency in reducing total fouling index (TFI). The efficiencies of two types of adsorbents, MAR and PAC, were systematically compared. Meanwhile, the change in fouling resistance distribution and mass balance of HA during UF was analyzed to gain some insights into the mechanisms of HA fouling reduction and to explain the different performance of MAR and PAC.

2. Materials and methods

2.1. Humic acid solutions and adsorbents

HA purchased from Sigma-Aldrich Chemical Co. (USA) was employed in this study. To prepare HA stock solution, 2 g of HA was added to 800 mL of 0.01 M NaOH solution and it was stirred for 24 h to ensure dissolution. The solution was then diluted to 1000 mL and the pH was adjusted to 7.0 using HCl. Salt solutions with different ionic environments were prepared by adding 1 mM NaHCO₃, appropriate amount of NaCl and CaCl₂ to Milli-Q water. NaCl was used to adjust ionic strength and CaCl₂ was used to alter ionic composition. For each experiment, HA stock solution was diluted with prepared background salt solution to obtain a 10 mg/L HA solution with specific ionic environment, and then the pH was adjusted to 7.5 ± 0.1 with 0.1 N HCl and NaOH. All HA solutions were stored at room temperature (25 ± 1 °C) for 2 h before use.

Two types of adsorbents, a polymeric adsorbent and an activated carbon, were used in this study. MAR is a type of mesoporous polymeric adsorbent synthesized following the method proposed by Clark et al. [30]. Briefly, polyethersulfone (PES, Veradel 3000P, Solvay, USA) was dissolved in the mixture of N-methylpyrrolidone and propionic acid (Bench Chemicals, China), and then the polymer solution was injected into Milli-Q water under stirring to form a suspension of MAR particles [22]. MAR was washed thoroughly to remove the solvent before use. Wood-based PAC purchased from Bench Chemicals (Tianjin, China) was used as a representative of activated carbons commonly used in water treatment. Textural properties of MAR and PAC are given in Table S1. It can be seen that MAR and PAC were abundant in mesopores and micropores respectively, and the surface area of MAR was much smaller than that of PAC.

2.2. UF membranes and experimental setup

Flat sheet PES membranes (OM100076, Pall, USA) with a nominal molecular weight cutoff (MWCO) of 100 kDa were used in this study. The membrane was negatively charged with a zeta potential of −16.88 mV and the water contact angle of the membrane was 58.2° [31].

Membrane filtration experiments were conducted in a filtration cell (Amicon 8400, Millipore, USA) in dead-end mode (the stirring paddle inside the cell was not used during filtration) at room temperature (25 ± 1 °C). During filtration, UF membrane was placed on the bottom of the cell with glossy side towards the feed solution and the effective filtration surface area was 42 cm². The UF cell was operated in constant-flux mode with a peristaltic pump used as the suction pump and the permeate flux was kept at 150 L/(m² h). A pressure transducer (PTP708, Tuopo Electric, Foshan, China) was mounted between the cell and the suction pump to monitor the trans-membrane pressure (TMP). The transducer was connected to a computer and the data was automatically logged every 5 s.

2.3. Filtration experiments

For each ionic condition, adsorption pretreatment was conducted by adding the adsorbent to HA solution in conical flasks and shaking them in a rotary shaker at 120 rpm and 25 °C. Adsorbent dosage was 50 mg/L and the contact time was set at 30 min. The adsorbent particles were removed before UF by filtering the mixed solutions through 0.45 μm mixed cellulose filters (Taoyuan, China), and the filtrate, which was denoted as MAR/PAC-treated HA, was used as UF feedwater. Meanwhile, UF experiments were also carried out with raw and 0.45 μm prefiltered HA, respectively. The comparison between the results obtained during UF of raw and 0.45 μm prefiltered HA revealed the fouling behavior of HA aggregates larger than 0.45 μm, while the comparison between MAR/PAC-treated HA and 0.45 μm prefiltered HA reflected the influence of adsorption pretreatment.

To evaluate fouling reversibility and gain some insights into the fouling mechanisms, membrane fouling resistance was operationally classified into four parts based on the resistance-in-series model [32,33]: loosely-attached external fouling resistance ($R_{ef,t}$), strongly-attached external fouling resistance ($R_{ef,s}$), reversible internal fouling resistance ($R_{if,r}$) and physically irreversible fouling resistance (R_{irr}). The procedure of filtration and subsequent physical cleaning were performed as described in our previous work [33] except that the corresponding background salt solution rather than Milli-Q water was used for backwashing and determination of membrane resistance. Membrane fouling rate was assessed using TFI and “fouling control efficiency” was defined as the reduction percentage of TFI due to pretreatment. The procedure of UF experiments and the calculations of TFI and fouling control efficiency were described in detail in the Supplementary information.

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