Effects of coagulation on submerged ultrafiltration membrane fouling caused by particles and natural organic matter (NOM)

LI YongHong[1,2], WANG Jun[1], ZHANG Wei[1], ZHANG XiaoJian[1] & CHEN Chao[1*]

[1] *Department of Environmental Science and Engineering, Tsinghua University, Beijing 100084, China;*
[2] *Department of Engineering, The Second Artillery Command College, Wuhan 430012, China*

Natural organic matter (NOM) and particles in source water are responsible for the majority of ultrafiltration (UF) membrane fouling that occurs during drinking water treatment. This study was conducted to (1) understand the UF membrane fouling phenomena caused by NOM and turbidity-causing particles and (2) investigate the effect of coagulation pretreatment on the alleviation of membrane fouling. In this study, kaolinite and humic acid (HA) were used to simulate the particles and NOM present in source water. The results revealed that the particles contributed to reversible fouling due to cake layer formation on the membrane surface, but that could be effectively reduced by increasing the frequency of physical backwashing. The results of the molecular weight (MW) distribution measurements, resin fractionation, SEM and FTIR analyses showed that HA primarily contributed to irreversible fouling. Taken together, the results of this study imply that the particles and NOM compounds present in source water may have different fouling behavior, and that particles may mitigate the irreversible fouling caused by HA. The addition of coagulant can lead to a higher rate of removal of large-sized hydrophobic compounds. Coagulation pretreatment can improve membrane performance and postpone membrane fouling development effectively, as well as retard the implementation of membrane chemical cleaning.

ultrafiltration, membrane fouling, NOM, particles, coagulation

Recently, ultrafiltration (UF) has been developed as an effective, economical and green technology, making it one of the most promising drinking water treatment processes currently available [1]. Despite these advantages, UF is still highly susceptible to membrane fouling, which reduces the performance of the UF membrane and restricts its more widespread application [2–4]. Thus, membrane fouling is one of the primary obstacles to wider application of UF process and the fouling mechanisms reported differ greatly [5]. Membrane fouling is generally dependent on the properties of the source water and the process parameters [6]. The interaction between the membrane surface and the substances dissolved in source water plays an important role in membrane fouling [7,8]. Turbidity-causing particles and natural organic matter (NOM) in surface water are considered to be two major contributors to membrane fouling. To develop more cost-effective strategies for the minimization of membrane fouling during actual application of UF process, a more comprehensive understanding of fouling phenomena by particles and NOM is necessary.

NOM is of concern during water treatment because it serves as a precursor for the formation of disinfection by-products (DBPs). Huang et al. [9] pointed out that the effects of hydrodynamic conditions on fouling were important, but to a lesser extent than the source of NOM. It has also been suggested that NOM plays a primary role in determining the membrane fouling rate and extent of fouling [10,11]. The reported results of the fouling behavior of hydrophobic

*Corresponding author (email: chen_water@tsinghua.edu.cn)

(HPO) and hydrophilic (HPI) fractions are also inconsistent. For example, it has been suggested [12,13] that the relatively hydrophilic NOM source water exhibited a greater flux decline than hydrophobic organics. However, others have concluded that the relatively higher molecular weight of hydrophobic compounds was responsible for the flux decline in UF [14,15]. Particulate fouling due to the deposition of colloids is a persistent problem in membrane filtration causing flux decline [16]. Recent studies have shown that turbidity-causing particles play an important role in membrane fouling [8,11,17]. However, these studies have primarily focused on pressured membrane modules with low filtration areas and sole UF process. Liu and Kim [18] showed that the submerged module performed better than the pressure module under all conditions. However, combined NOM-particle fouling in submerged UF membrane systems is still not fully understood and requires more advanced research. It is also necessary to investigate the mutual influences between particles and NOM in source water treated by submerged UF membrane. Furthermore, although coagulation pretreatment is a useful strategy to mitigate membrane fouling effects [19,20], Howe et al. and Maartens et al. [21,22] contend that coagulation pretreatment aggravated membrane performance in some cases, the effects of the addition of coagulant on membrane fouling caused by particles, NOM and combined NOM-particles have seldom been reported.

This study focused on the effects of turbidity-causing particles, NOM and NOM-particles present in source water on submerged UF membrane fouling during UF and coagulation-UF process respectively. Additionally, the effect of the addition of particles on NOM membrane fouling was investigated. Based on the experimental filtration study, a more comprehensive understanding of membrane fouling caused by particles and NOM was obtained by analysis of the molecular weight (MW) distributions of NOM, XAD resin fractionation, and X-ray energy dispersive spectra (EDS), as well as by scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FTIR).

1 Materials and methods

1.1 Experimental source water

Kaolinite and humic acid (HA) were used to simulate the turbidity-causing particles and NOM in surface water. The concentrations of kaolinite and HA stock solution were 5 g/L and 1.7 g/L. Premixed solutions of kaolinite and HA were used to simulate the combined fouling by source water containing NOM and particles. Each stock solution was prepared in advance and stirred overnight to give stable constituents. The synthetic source water was a diluent of the stock solution using tap water.

1.2 UF membrane and coagulant

Four immersed modules of hollow fiber UF membrane

(polyvinylidene fluoride (PVDF), 0.03 μm, Tianjin Motimo Membrane Technology LTD) with a total filtration area of 4 m^2 were applied. The membrane's internal and external diameters are 0.6 mm and 1.1 mm. During the filtration phase, the source water is filtered from outside the fiber to the internal channel using a diaphragm pump. During the backwashing phase, pressurized backwashing water is sent from the inside of the fiber to the outside using a backwashing pump. Aluminum sulfate was used as a coagulant and dosed in the pipeline before the coagulation tank and the contact time was 5 min.

1.3 Experimental equipment and approach

A schematic diagram of the UF experimental set-up is shown in Figure 1. The system consisted of UF membrane modules, a source water preparation system, a coagulant dosage system and an air-water backwashing system. The UF membrane modules were directly submerged in the membrane tank and driven by a diaphragm pump to provide negative pressure suction. Four perforated pipes were placed at the bottom of the membrane tank to dislodge particles through aeration. Air was intermittently supplied by an air compressor.

The stock solution was diluted in the raw water tank with tap water. The concentration of the source water was controlled by adjusting the flow rate of the tap water and stock solution, after which the source water was allowed to flow into the coagulation tank and the membrane tank sequentially by gravity. The water was next drawn into the UF membrane and then the permeate tank through the collection pipe by suction pump. During the experiment, the membrane was run through a cycle of 105 s sole UF and 15 s UF combined with aeration simultaneously. The membrane reactor was operated at a flux of 57–60 L/ (m^2 h) and the air compressor was operated at a flow rate of 1.5 m^3/h. The water flushing flux and air flushing strength for the physical cleaning of the membrane were maintained at 2.0 times the operating flux and 6.25 L/(m^2 s). Prior to the beginning of each experiment, the UF membrane modules were cleaned by strengthening hydraulic backwashing and the membrane tank was emptied. The UF membrane performance was evaluated by examining the decrease in the normalized membrane specific flux (J_{SF}/J_{SF0}).

1.4 Analytical methods

The HA-contaminated source water was analyzed for UV absorbance at 254 nm, total organic carbon (TOC), dissolved organic carbon (DOC, pre-filtration through a 0.45 μm membrane), the MW distribution and XAD fractionation. UV absorbance was measured in an ultraviolet gradient spectrophotometer (Lengguang 752N, China) and a UV-VIS detector was used for sample detection at 254 nm. TOC and DOC were analyzed in a total organic carbon
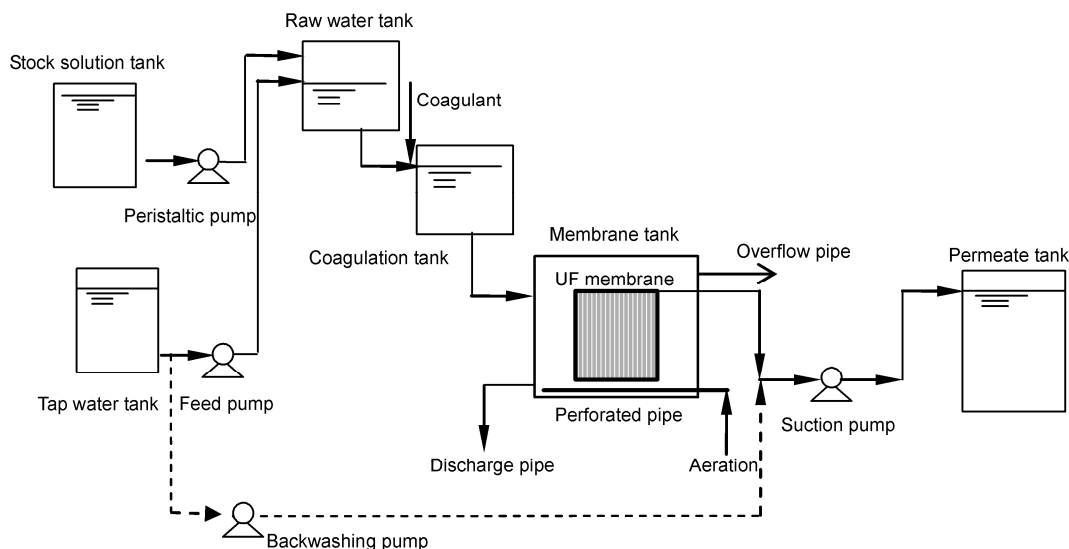
Figure 1 Schematic diagram of the UF experimental equipment.

analyzer (Shimadzu TOC-VCPH, Japan), and the MW distributions were determined by high performance size exclusion chromatography (HPSEC) using an HPLC pump (Agilent 1100) equipped with an autosampler. XAD polymeric resin was used to designate organic matter as hydrophilic (HPI) and hydrophobic (HPO) [23].

An Ankersmid Eyetech Series particle analyzer (Ankersmid, Netherlands) was employed to determine the particle size and quantity distribution. The turbidity was measured using a turbidimeter (HACH-2100P, USA). Morphological analysis of the fouled membranes before and after physical backwashing was conducted by SEM (JSM-6460LV, Japan). The dried membrane samples were gold plated with ionic sputtering prior to analysis. Inorganic elements on the membranes were analyzed using EDS (Oxford). To investigate the chemical structures of the organic foulants present on the membrane surface, a FTIR spectrometer (Nicolet Magana IR-560) was utilized in the range of 650–4000 cm^{-1}. Each spectrum was collected by compiling 32 scans at a resolution of 4 cm^{-1}.

2   Results and discussion

2.1   Effect of coagulation on particle fouling in the UF

Kaolinite containing source water with turbidities ranging from 10–2000 NTU was adopted to further investigate the fouling characteristics of the immersed UF membrane in response to turbidity-causing particles. Experimental results demonstrated a positive correlation between the turbidity of the source water and the rate at which the J_{SF}/J_{SF0} ratio decreased. J_{SF} remained almost the same as the initial value after 5 h of ultrafiltration of 20 NTU raw water, while it decreased to about 80% of J_{SF0} after 1 h of ultrafiltration of 2000 NTU raw water (shown in Figure 2). These findings

imply that increased turbidity resulted in a higher filtration resistance and a greater decrease of J_{SF}. However, J_{SF} could be recovered completely through air-water backwashing, regardless of the turbidity of the source water. These findings implied that the submerged UF membrane has a high potential to handle high turbidity source water. The particles could only form loose physical deposits rather than firm chemical bonds or sticky materials on the membrane surface. Therefore, high turbidity would not be obstacles for UF operation with backwashing.

The outer surfaces of the new and fouled membranes before and after physical backwashing were observed using SEM. The surface of the new membrane was clean and smooth. Conversely, the membrane surface fouled by kaolinite (Figure 3(a)) was covered with a loose cake layer composed of kaolinite particles, but this layer was removed almost completely after backwashing. Inorganic elements on the membranes were analyzed by EDS, which revealed that the new membranes contained 20.3% and 79.7% carbon and fluorine (based on weight). The total O, Al, Si, and Ca composition on the fouled membrane prior to backwashing was found to be 75.77%, while the percentages of C and F decreased to 0 and 24.23%. However, the percentages of C
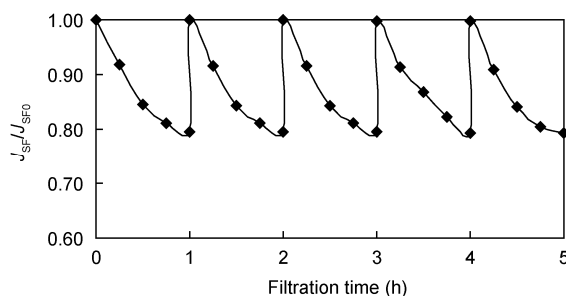

Figure 2 Decrease in normalized membrane specific flux during ultrafiltration of source water with high turbidity (i.e. 2000 NTU).

and F were restored to 15.27% and 81.95% after backwashing, with only a small amount of residual Si (2.78% by weight) being detected.

A coagulant dose lower than 4 mg/L slowed the TMP increase and mitigated membrane fouling effectively when source water containing particles at a turbidity of 100 NTU was ultra-filtrated [24]. The rates at which the TMP increased were 0.013, 0.002, 0.008, and 0.047 kPa/min when 0, 1, 4, and 8 mg/L alum were added. The SEM image (Figure 3(b)) of the membrane fouled by particles in the coagulation-UF process with 1 mg/L alum addition revealed that the cake layer was looser and thinner when compared to no coagulant addition. However, a thicker and more compact cake layer formed in response to the addition of 8 mg/L alum Figure 3(c), resulting in the highest filtration resistance and TMP increase rate for all alum concentrations tested. These results revealed that the structure of the cake layer formed by coagulated flocs combined with particles greatly influences the filtration resistance in UF, and that the addition of coagulants should be optimized during the UF of source water.

2.2 Effect of coagulation on NOM fouling during UF

The HA and combined effects of HA and kaolinite fouling, as well as the effects of the addition of coagulant on UF membrane fouling were examined. HA (about 8 mg/L of

COD_{Mn}) and HA-kaolinite solution (about 8 mg/L of COD_{Mn}, 20NTU of turbidity) were treated by UF process with alum dosages of 0 and 1.5 mg/L. As shown in Figure 4, backwashing could not recover the membrane specific flux completely. These results indicate that irreversible fouling occurs during ultrafiltration of source water containing NOM. In other words, NOM made a large contribution to irreversible fouling. Control and prevention of irreversible fouling is much more important during the actual application of UF. Therefore, a greater understanding of irreversible fouling caused by NOM constituents in source water is essential.

Membrane fouling is generally divided into reversible fouling and irreversible fouling. The total membrane fouling of a cycle n is the sum of the irreversible and reversible fouling, which is given here by the difference in J_{SF}/J_{SF0} between the beginning and end of a cycle. Reversible fouling is represented by the part of the specific flux restored by physical backwashing, which is equivalent to the difference in J_{SF}/J_{SF0} before and after backwashing. The irreversible fouling is accumulated fouling that cannot be restored by backwashing, which equals the difference in J_{SF}/J_{SF0} between the beginning of the treatment cycle and the beginning of the next cycle [8]. The average total membrane fouling, irreversible fouling and irreversible fouling represented by J_{SF}/J_{SF0} is presented in Table 1.
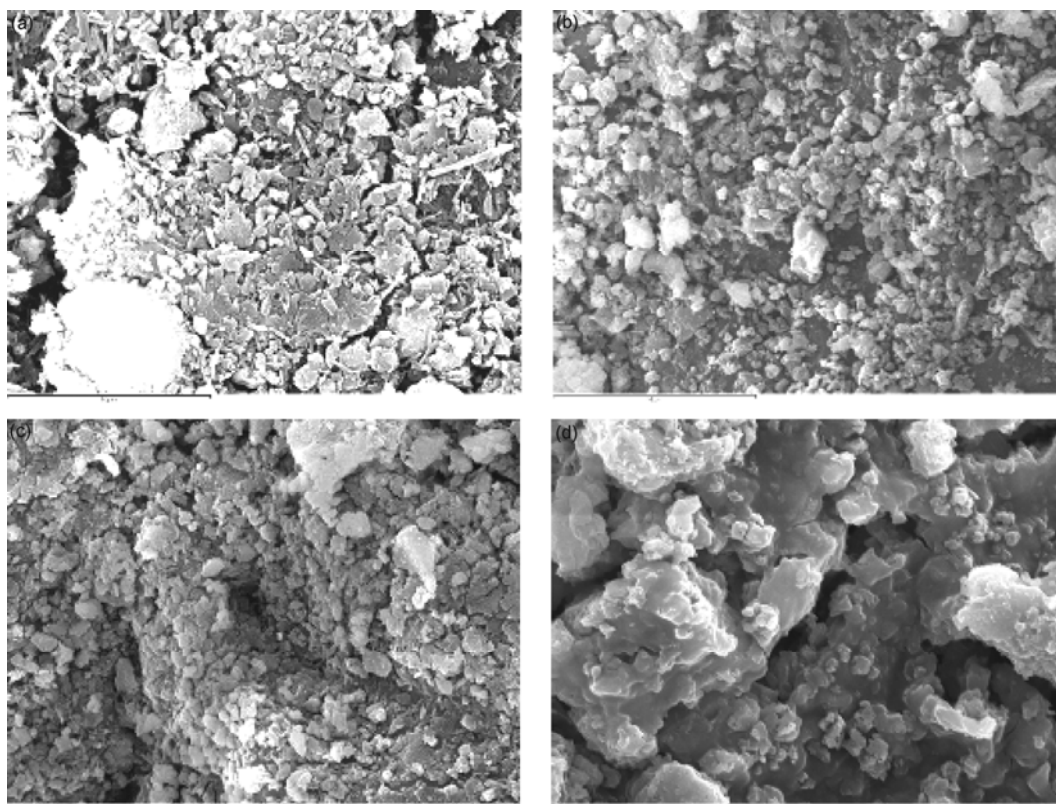

Figure 3 SEM images of fouled UF membranes: (a) fouled by kaolinite with no added coagulant (5000×); (b) fouled by kaolinite and 1 mg/L coagulant (5000×); (c) fouled by kaolinite and 8 mg/L coagulant (5000×); (d) fouled by HA-kaolinite (5000×).

Table 1 Comparison of J_{SF}/J_{SF0} before and after backwashing in source water containing HA and HA-kaolinite that was ultra-filtrated by UF alone and coagulation-UF

Type of fouling	HA Al 0 mg/L	HA Al 1.5 mg/L	HA-kaolinite Al 0 mg/L	HA-kaolinite Al 1.5 mg/L
Average total fouling	0.199	0.147	0.209	0.147
Average reversible fouling	0.050	0.053	0.108	0.068
Average irreversible fouling	0.149	0.094	0.101	0.079
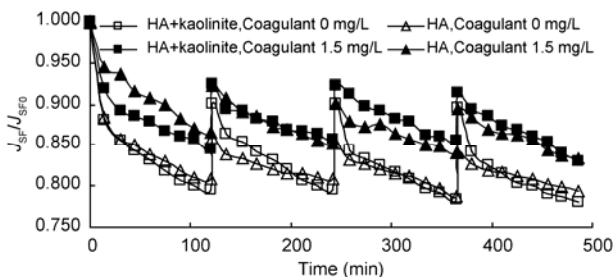
Figure 4 Comparison of the decrease in normalized membrane specific flux for HA and HA-kaolinite containing source water ultra-treated by UF with coagulant doses of 0 and 1.5 mg/L

As shown in Figure 4, the percentage of irreversible fouling was 74.9% in the case of HA containing source water. These findings indicate that fouling initially occurred in response to narrowing and blockage of the membrane pore [25], which eventually became completely covered by a NOM gel layer. The continuous operation of the membrane causes compression of the NOM gel layer as the filtration time increases [26]. The fouling caused by pore blockage and the NOM gel layer could not be completely recovered by backwashing. However, a greater decrease in specific flux at the end of every cycle and a higher specific flux recovery after backwashing during each cycle was observed in the ultra-filtrated HA-kaolinite containing source water. The average total fouling represented by J_{SF}/J_{SF0} in HA-kaolinite was 0.010 higher than that in HA, while the average irreversible fouling was 0.048 lower than that in HA. The ratios of the average reversible and irreversible fouling in ultra-filtrated HA and HA-kaolinite were 0.34 and 1.08. Overall, these results imply that the addition of kaolinite to source water containing HA increased the total fouling, increased reversible fouling caused by the cake layer, and reduced irreversible fouling caused by HA.

The findings presented here can be explained by the rapid formation of a cake layer consisting of a porous structure composed of inorganic particles on the membrane surface at the beginning of filtration. The cake layer could then prevent parts of HA from being adsorbed into the membrane pores and trap some organic matter. As filtration progressed, the cake layer became thicker and more compact, which led to a significant increase in total membrane fouling. However, no significant synergistic effect was observed during combined HA-kaolinite fouling in the present study, which differs from the findings of a study conducted by Jermann et al. [8]. The reversible fouling that occurred during combined particle-NOM fouling was

much higher than the reversible fouling observed in NOM or particle fouling, while the irreversible fouling was almost the same as that observed in the NOM fouling in the study conducted by Jermann et al. These contradictory results may be due to the flat sheet membrane adopted in the experiment conducted by Jermann et al., which is susceptible to the concentration of particles. Conversely, submerged hollow fiber membranes have a tolerance of high turbidity.

A significant increase in the specific flux was observed in the coagulation-UF process. The total fouling in HA and HA-kaolinite treated by coagulation-UF was 0.052 and 0.062 lower than that observed in response to sole UF, while the irreversible fouling was 0.055 and 0.021 lower. These results indicate that the addition of coagulant can effectively decrease the irreversible fouling caused by NOM. This positive effect was attributed to the formation of a loose cake layer by NOM flocs intercepted by the UF membrane. As such, the irreversible fouling caused by NOM was effectively reduced.

During the coagulation-UF process, the total fouling was almost equivalent in ultra-filtrated HA and HA-kaolinite containing source water. However, the irreversible fouling in ultra-filtrated HA-kaolinite was 0.015 lower than that in HA. These results confirmed that the addition of kaolinite into HA-containing source water can mitigate some of the irreversible fouling caused by NOM. Although reversible fouling by NOM-particles is higher than that caused by particles, it can be minimized by increasing the backwashing frequency.

The outer surface of the membrane fouled by HA-Kaolinite (Figure 3(d)) was covered completely by a tight and compact layer. The HA gel bound to kaolinite particles tightly. In addition, HA decreased the porosity of the cake layer, and the outer surface did not reappear in response to backwashing. EDS analysis of the backwashed membrane indicated that 13.7% of the O and Si was retained on the membrane surface. The UF membrane was finally cleaned with sodium hydroxide, hydrochloric acid, and sodium hypochlorite sequentially. The chemical cleaning resulted in a 32% improvement in the J_{SF} of the fouled membrane, which was nearly equivalent to the J_{SF} of the new membrane. The SEM image revealed that the outer surface of the chemically cleaned membrane was completely exposed, indicating that chemical cleaning is an efficient method for flux recovery when necessary.

The FTIR spectra of the new membrane (Figure 5-4) revealed absorption peaks indicative of stretching vibration of C—H and C=C at 2933 and 1402 cm^{-1}. The absorption
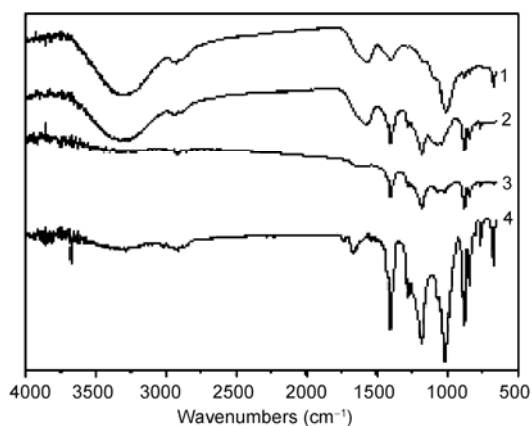
Figure 5 FTIR spectra of membranes: 1, Backwashed membrane fouled by HA; 2, backwashed membrane fouled by HA-kaolinite; 3, chemically cleaned membrane; 4, new membrane.

peaks of the $-CF_2$ stretching vibration of PVDF were shown at 1277 and 1182 cm^{-1}. The vibration absorption peaks at 796, 841 and 1277 cm^{-1} were typical of PVDF and belonged to the alpha phase, amorphous state and beta phase. The FTIR spectra of the membrane fouled by HA (Figure 5-1) and HA-kaolinite (Figure 5-2) showed almost no characteristic peaks of PVDF, while new absorption peaks were adsorbed at 1600 and 3320 cm^{-1} when compared with that of the new membrane. The abatement of a PVDF characteristic peak indicates that the outer surface of the fouled membrane was covered by organic foulants. The absorption peak at 1600 cm^{-1} indicates that the organic foulants may contain stretching vibration of the aromatic nucleus $-C=C-$ and (or) $C=C$ functional groups. The smooth absorption peak at 3320 cm^{-1} indicates the existence of functional groups of $-OH$ (alcohol or phenols) and (or) N–H. There was almost no difference between the FTIR spectra of the membrane fouled by HA and HA-kaolinite, which implies that particle addition could not affect the properties of organic pollutants. The FTIR spectra of the chemically-cleaned membrane (Figure 5-3) did not show absorption peaks of $-C=C-$, $C=C$, or $-OH$ which suggests that the majority of the organic foulants on the membrane surface were removed by chemical cleaning. Thus, it can be concluded that sodium hydroxide, hydrochloric acid and sodium hypochlorite effectively recover membrane performance.

2.3 Organic removal by UF and coagulation-UF processes

The MW distribution for the removal rate of HA by UF and the coagulation-UF process (1.5 mg/L alum dose) is shown in Figure 6. When compared with the sole UF process, coagulation-UF significantly improved the NOM removal rate of organics with an MW 1–5 kDa of 63% to 71% and those greater than 10 kDa from 85% to 99%. However, the removal rate of organics with a MW lower than 1 kDa was
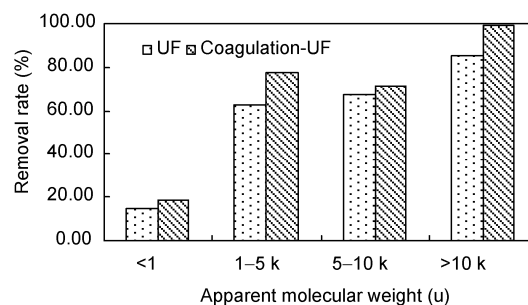

Figure 6 MW distribution of HA removal by UF and coagulation-UF processes.

increased by only 3.7%. These results indicate that the addition of coagulant can significantly improve the rate of the removal of organics with MW higher than 10 kDa, as well as the removal of organics with a MW between 1–5 kDa. Moreover, low-MW organic matter (<1 kDa) may be trapped within the loose cake layer, partially reducing the irreversible fouling.

The UV$_{254}$ distribution of the HPI and HPO fractions in HA-containing source water and permeate water treated by UF and coagulation-UF are shown in Figure 7. The ratio of HPI/HPO was 0.113 in source water and 0.391 and 0.556 in the permeate water treated by UF and coagulation-UF. The Coagulation-UF process improved the total UV$_{254}$ removal rate by 10.6% when compared to UF alone, and the removal rate of the HPO fraction increased by 9.2%. These results indicate that coagulation pretreatment primarily contributes to a greater removal of the HPO fraction, which led to mitigation of the irreversible fouling caused by the HPO fraction.

3 Conclusions and suggestions

UF membrane fouling caused by particles is predominately reversible because of the removable property of the cake layer on the membrane surface by air-water backwashing. The cake resistance increased in proportion to the turbidity of the source water and can be effectively reduced by shortening the filtration period. Additionally, the submerged hollow-fiber UF membrane tolerated high turbidity source water.
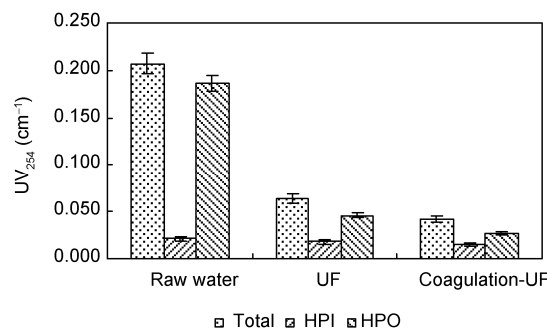

Figure 7 Composition of source water containing HA and permeate water treated with UF and coagulation-UF.

HA primarily contributed to irreversible fouling, however, the combined effects of particles and HA could not be regarded as additive simply. Conversely, adding particles to the HA solution was found to mitigate the irreversible fouling by NOM, but it did not mitigate the reversible fouling by HA. This was because the particles dotted on the NOM gel layer attached to the membrane surface reduced the adhesiveness of foulants and blocked the entry of organic molecules into the membrane pore. In this case, the deposition of kaolinite into the NOM-fouled layer resulted in a compact fouling cake, but the adsorption and gel resistance caused by the organics were alleviated.

Coagulation pretreatment was found to be capable of effectively mitigating membrane fouling due to the shape and size of flocs influencing the cake structure. Taken together, these findings will serve as a useful guide to coagulation pretreatment to alleviate membrane fouling. The addition of coagulant should target elimination of the irreversible fouling caused by NOM, especially the HPO fraction, which contributed more to membrane fouling according to the XAD fractionation results. These results suggest that coagulation pretreatment could be optimized by selecting the appropriate dosage to prevent formation of a sticky and compact NOM gel layer on the membrane surface by coagulant flocs.

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