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Integrity of PVC membranes after sequential cleaning with hypochlorite and citric acid

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ABSTRACT

The integrity of polyvinylchloride membranes after sequential cleaning with sodium hypochlorite (NaOCl) and citric acid (CA) was investigated by measuring transmembrane pressure (TMP), permeability to bovine serum albumin (BSA), the particle size distribution of the effluent, and attenuated total reflectance-Fourier transform infrared spectra. Cleaning with both NaOCl and CA was found to be equally effective in recovering flux regardless of the order in which they were applied. The TMP was reduced from 120% of the value for a clean, new membrane to 92–105% after cleaning. The BSA entrapment rate was in the range 70–78% for fouled membranes after cleaning. Hypochlorite was much more effective at removing foulants than CA but caused damage to the membranes. CA to some extent mitigated the damage caused by NaOCl and partially repaired membrane integrity. Sequential cleaning processes left smaller pore sizes compared with cleaning using NaOCl alone. Applying the CA first was found to be more appropriate for prolonging membrane life. NaOCl might be used at a mass fraction greater than 1% without compromising the integrity of the membrane if it is used with CA.

Keywords: Polyvinylchloride membranes; Integrity; Cleaning; Hypochlorite; Citric acid; Water purification

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1. Introduction

Ultrafiltration (UF) has been widely used in water treatment because of its relative simplicity, low energy consumption, and lack of chemical additives. In addition, UF can achieve high separation efficiency without phase transitions or intermediate products [1–3]. Membrane fouling is, however, a key problem in UF, reducing permeation performance, leading to a flux decline and increased working pressure and energy consumption. Operational methods such as optimizing the hydrodynamic conditions [4], improving the membrane structure [5], pretreatment, and cleaning are required to minimize the well understood mechanisms of membrane fouling [6,7].

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Natural organic matter (NOM) and organic micromolecules are usually the main components of membrane fouling during drinking water treatment. Their adsorption leads to the formation of a gel layer [6,8]. In particular, it has been suggested that humic compounds are the most detrimental [9,10]. Most NOMs cannot be effectively removed by physical cleaning. So fouled membranes require intermittent chemical cleaning.

Common cleaning chemicals include sodium hydroxide, sodium carbonate and hydrochloric acid, critic acid (CA), sodium hypochlorite, and hydrogen peroxide. Hypochlorite is widely used because it is cost-effective and efficient, but frequent cleaning affects the properties and functions of UF membranes and damages the membrane integrity. It has been shown that hypochlorite cleaning results in good recovery of transmembrane pressure (TMP), but leads to more severe fouling in subsequent operation [4].

Wolff and Zydney have demonstrated that the pore size of a polysulfone membrane increased by more than four times after prolonged cleaning with bleach [11]. In a fouling experiment, polyethersulfone (PES) membranes have been shown to suffer chain scission and reduced membrane integrity after long-term exposure to NaOCl [12]. Other researchers have proposed that chain scission in PES membranes is caused by the attachment of radicals at the sulfone and isopropylidene bridge [13]. In addition, it has been reported that hypochlorite exposure causes scission of PES polymer and membrane pitting, which removes the active filtration layer and exposes the larger pores in the asymmetric structure [14,15].

Both acids and alkalis have been used to remove organic foulants. Lim and Bai's (2003) research shows that backwashing, sonication, and chemical cleaning with alkali and acid can achieve almost complete flux recovery [16]. Foulants derived from hydrophobic NOM sources were cleaned more effectively than foulants from relatively hydrophilic NOM sources in terms of flux recovered by acid and caustic cleaning. Liu's group has shown that when a membrane is fouled by iron oxides, cleaning with CA is very effective because it can dissolve the oxide precipitates and form a complex with the iron [17]. It can also prevent "salt bridge" formation by removing divalent cations and improve the cleaning of membranes fouled by organic foulants [18]. But PES membranes age in acid and other cleaning solutions, and PES membranes appear to be incompatible with solutions containing chlorine [19]. Attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy shows that the C–S bonds in PES are broken and Cl–S bonds form after long-term treatment with alkaline bleach.

Various studies have reported that chemical cleaning in sequence or combination is effective in removing membrane fouling [20–22]. Consistent membrane cleaning sequence tests have shown that an alkali cleaning followed by an acid is more effective than the reverse for membranes treating surface water [23,24]. Sodium hypochlorite and CA are membrane cleaning chemicals commonly used in drinking water treatment, and it is reasonable to suggest cleaning fouled membranes with those two chemicals in sequence. Caustic/chlorine cleaning followed by CA cleaning has been shown to restore more than 94% of the original flux of a membrane [17]. However, the effects of various sequences on the restoration and integrity of membranes remain unknown. To ensure the quality of the effluent, the effect of sequential chemical cleaning on membrane integrity must be investigated.

To date, PES, polysulfone, and polyvinylidene fluoride membranes have been the most intensively studied. Few published studies have investigated treating polyvinylchloride (PVC) membranes, despite the fact that PVC membranes are durable, efficient, and economical, and are widely applied in drinking water treatment. In this study, TMP and the rate of entrapment of bovine serum albumin (BSA) were used to characterize the performance of PVC membranes and their integrity after sequential cleaning with sodium hypochlorite and CA.

2. Materials and methods

2.1. Materials

2.1.1. Membrane

Hollow-fiber (tube) PVC membranes (Lisheng Co. Ltd, Hainan Province, P.R. China) were used in all experiments. The characteristics of the membrane

Table 1
Characteristics of the membrane and suggesting working conditions

Average pore size	Effective film diameter	Membrane surface area	Molecular weight cut-off	Suggested working pH	Maximum working pressure	Suggested working flux
0.01 μm	1.45 mm	48 m^2	50 kDa	1–13	80 kPa	10–70 L/(m^2 h)

material and the operating conditions suggested by the manufacturer are presented in Table 1. The material was assembled into modules consisting of five hollow tubes 30 cm long, with an effective working area of 0.01 m^2 for each module. The membranes were washed before use with pure water to remove the protective agents (sodium dodecylsulfate and glycerine) from the surface. They were then used to filter pure water at $30\,L/(m^2\,h)$ for 20 min and soaked in pure water for 20 h to eliminate residual agents. The TMP for each membrane was then determined after 10 min of filtration at $30\,L/m^2\,h$ with pure water, and this was defined as that module's TMP_0.

2.1.2. *Water*

The feed water was taken from a river in Guangzhou, China. The water's characteristics are presented in Table 2. The feed water was supplemented with agents including peptone (80–120 mg/L), beef extract, and BSA (MW 67,000 Da) for testing. The mass ratio of water to the chemical agents in simulation water was 1:5. These solutions were termed "supplemented river water."

2.2. *Analytical methods*

2.2.1. *Transmembrane pressure*

The TMP was monitored during this study to show changes in the membrane. The TMP is related to the flux according to the Hagen–Poiseuille equation: under certain conditions, the flux and TMP have a linear relationship. TMP was measured after 10 min of filtration at $30\,L/m^2\,h$.

2.2.2. *BSA entrapment rate*

The membrane's rejection of BSA was monitored. Solutions of BSA (0.2 g/L) were used because of the low susceptibility of BSA to conformational variations in response to changes in temperature and pH.

The concentration of BSA and the solution's absorbance at 280 nm are linearly related, so the feedstock and effluent from the membrane were analyzed using UV-vis spectroscopy to determine their BSA concentrations. The UV-vis spectra were measured after 10 min of BSA solution filtration at $30\,L/m^2\,h$. The BSA entrapment rate was calculated from the two BSA concentrations.

2.2.3. *Particle size distribution*

The particle size distributions of the effluents (PSDEs) were measured using a size distribution and zeta potential meter (Zetasizer Nano ZS90, Malvern, UK), again after 10 min of filtration at $30\,L/m^2\,h$. The volume of each sample was at least 10 mL and the samples were analyzed immediately.

2.3. *Experimental procedures*

2.3.1. *Preparation of fouled membranes*

Fouled membranes were prepared by filtrating supplemented river water at $30\,L/m^2\,h$ and 25℃. Each 90 min of filtration included a single 60 s backwash with ultrafiltered water. Filtering continued until the module's TMPi reached 1.5 times its TMP_0. The fouled membranes were then washed carefully with tap water to remove the gel layer, and then backwashed five times with pure water for 1 min at 10 min intervals. Finally, the membrane's TMPi was again measured with pure water.

2.3.2. *Membrane cleaning*

Sixteen fouled membrane modules were divided into five groups and soaked in the cleaning solutions in a set order. Cleaning with NaOCl followed by CA is referred to as "pre-Cl" cleaning. Cleaning with CA followed by NaOCl is referred to as "pre-CA." About 1 and 2% solutions of NaOCl, and 5 and 10% CA solutions were tested.

In routine use, membranes are typically chemically cleaned every 1–3 months, for 3–5 h each time. In these experiments the membranes were soaked for 1–4 days. One day of exposure was considered equivalent to one year of routine use and cleaning. Earlier studies have shown that membrane degradation is closely related to the dose of cleaning agent, which is equal to the concentration multiplied by the

Table 2
River water characteristics

Parameter	Turbidity (NTU)	Temperature (℃)	pH	CODcr (mg L^{-1})	UV_{254} (cm^{-1})
Range	15.5–108.9	25.8–32.7	7.02–7.48	14.09–60.71	0.170–0.206

exposure time. In these experiments, a constant NaOCl concentration was maintained by renewing the solutions every two days [4].

After exposure, the membranes were carefully removed from the solutions and rinsed with distilled water for 10 min, and then soaked in distilled water overnight to remove any residual chlorine trapped in the membrane. The membranes were then characterized in terms of their TMP, BSA entrapment rate, PSDE, and their membrane integrity as indicated by the ATR-FTIR spectrum.

3. Results and discussion

3.1. TMP

The ratio of TMP to TMP_0 was used to express the changes in TMP after cleaning. The pure water TMPs for the new and fouled membranes after different cleaning sequences using NaOCl and CA are presented in Fig. 1.

Fig. 1 shows that regardless of whether the membrane was new or fouled, sequential cleaning with NaOCl and CA in any sequence caused substantial decreases in TMP. This means that the flux was effectively restored and even became larger than that measured before cleaning. Clearly, all of the sequential cleaning processes were effective for removing membrane fouling.

The initial TMP/TMP_0 value of a new membrane before it had been exposed to cleaning agents was by definition 100%. Sequential cleaning reduced the TMP/TMP_0 value markedly into the 66–77% range. The average reduction in TMP for new membranes after their first cleaning was about 28%. The TMP/TMP_0 value of 140% represents increases in the TMP of fouled membranes after filtering river water. The TMP/TMP_0 value for fouled membranes after backwashing is 120%. Physical cleaning could reduce the TMP by 20%. After cleaning with NaOCl and CA, the value dropped from 120 to 92–105%. The average reduction in TMP for fouled membranes after cleaning was about 22%. The TMP/TMP_0 value for cleaned membranes was then approximately equal to that of new membranes. So sequential cleaning could almost fully restore the flux of fouled membranes. The TMP of cleaned new membranes was markedly lower than that of cleaned fouled membranes, which may indicate that new membranes suffer more severe damage in cleaning.

Although no significant differences in TMP reduction were observed between the various cleaning sequences and mass fractions, but one contrary result should be noted. In the case of new membranes, pre-CA cleaning reduced TMP/TMP_0 slightly more than pre-Cl cleaning by about 5%, whereas in the case of fouled membranes, pre-Cl cleaning reduced TMP much more than pre-CA cleaning did.

3.2. BSA entrapment rate

Fig. 2 illustrates the BSA entrapment rates of new and fouled membranes after sequential cleaning, indicating the condition of the membrane surfaces and their effective pore sizes. For new membranes, the BSA entrapment rate after cleaning was in the range 63–70%. The BSA entrapment rate was in the range 70–78% for fouled membranes. That the BSA entrapment rates of fouled membranes were higher

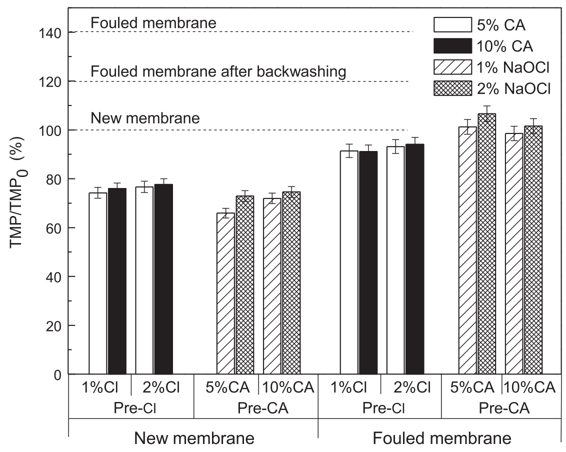

Fig. 1. TMP decline of new and fouled membranes after sequential cleaning with NaOCl and CA.

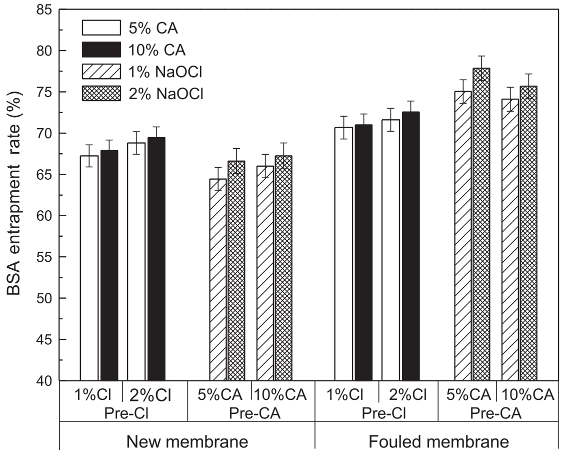

Fig. 2. BSA entrapment rate of new and fouled membranes after sequential cleaning with NaOCl and CA.

than those of new membranes is consistent with the TMP declines presented in Fig. 1. Slightly higher BSA entrapment rates were obtained by pre-Cl cleaning in the case of new membranes and by pre-CA cleaning in the case of fouled membranes. This pattern is also consistent with the TMP declines in reported Fig. 1. There were again no significant differences observed in BSA entrapment rates between the various mass fractions in the same sequence. The consistency between BSA entrapment rates and TMP decline demonstrates that the changes in TMP were directly correlated with pore size. The higher BSA entrapment rate for fouled membranes can presumably be attributed to retention of foulants in the pores.

3.3. TMP decline contribution

The reduction of membrane TMP (i.e. TMP_i) after cleaning with the first and second chemical agent was recorded. The contribution of each chemical in the cleaning sequence could then be calculated and the results are presented in Fig. 3. The contributions of NaOCl and CA are again expressed as the ratio between TMP_i and TMP_0. A negative value indicates that TMP decreased after cleaning, indicating the removal of foulants. Conversely, a positive value indicates that TMP increased after cleaning and implies a probable reduction in pore size. It can be seen that NaOCl always increased the magnitude of TMP reduction. This can be explained by its strong oxidizing capacity, which effectively removed foulants and enlarged the pore size of new membranes. It is notable that although all TMPs were reduced after the complete cleaning sequences, CA yielded less reduction in some cases. CA may play a role in membrane repair rather than cleaning in certain circumstances.

In the case of new membranes (Fig. 3(a)), hypochlorite led to a much better TMP improvement, which offset the negative contribution of CA in the pre-Cl sequence, leading eventually to an overall TMP decline. The big TMP declines induced by NaOCl cleaning were caused by severe damage to the hydrophilic groups and enlarged pore sizes, which resulted in gross surface defects. Subsequent exposure to CA may result in its being adsorbed on the affected surface, blocking pores, and increasing the TMP. With the pre-CA sequence, CA may simultaneously clean and protect the smooth surface of new membranes, while definitely hindering subsequent cleaning by NaOCl. This may explain the less effective contribution of NaOCl than that observed with the pre-Cl sequence.

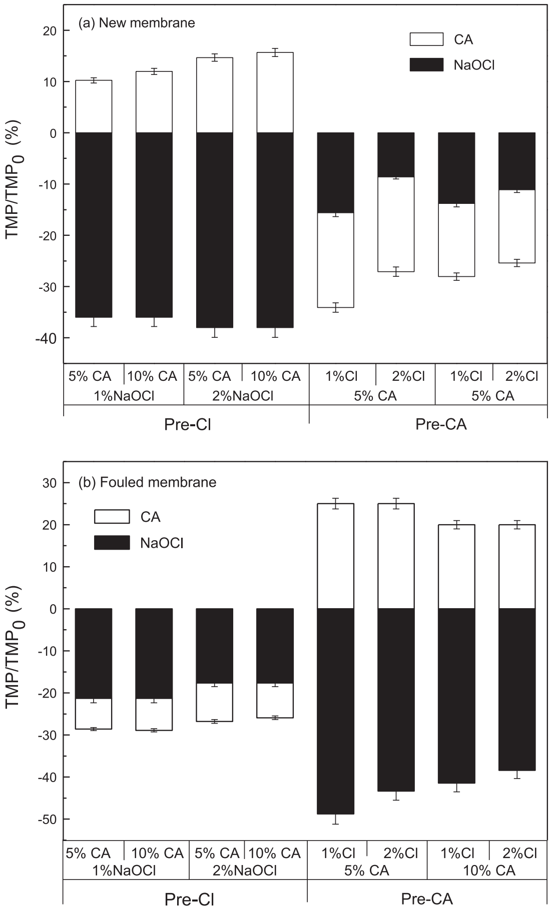

Fig. 3. TMP decline contribution analysis.

In the case of fouled membranes (Fig. 3(b)), both NaOCl and CA contributed to the TMP reduction in the pre-Cl sequence. NaOCl contributed more at all concentrations than CA, but was not as much as in the case of new membranes. This indicates that less damage is caused by NaOCl in the presence of foulants. Subsequent CA treatment mainly resulted in cleaning rather than repair. The negative contribution of CA in the pre-CA sequence may have been caused by a ligand-exchange reaction of CA with the foulants [18]. CA may bind to the “gross surface” formed by foulants. Such binding definitely thickened the caked layer and led to more severe fouling. The data suggest that this kind of caked layer may be easily removed by subsequent NaOCl cleaning, as evidenced by the greater contribution that NaOCl made to TMP decline.

Note that relatively less TMP improvement was observed where CA made negative contributions, i.e. in the pre-Cl sequence for new membranes and the pre-CA sequence for fouled membranes, as shown by comparing Figs. 1 and 2. It can be inferred that CA either blocked the enlarged pores in new membranes or protected foulants in the pores.

3.4. ATR-FTIR spectra

The ATR-FTIR spectra after sequential cleaning with 2% NaOCl and 10% CA are shown in Fig. 4. Some intense bands typical for PVC membrane are clearly present, such as 3,385 cm^{-1} for O–H bonds, 2,964 cm^{-1} for CH_3, 2,915 cm^{-1} for C–H, 1,640 cm^{-1} for C=C, 1,241 cm^{-1} for C–O–C, and 1,025 cm^{-1} and 967 cm^{-1} for the C–C skeletal vibrations.

For new membranes (Fig. 5(a)), there were no new or absent peaks in the membranes' spectra after cleaning compared with fresh, new membranes, but the peak strength was reduced for most peaks. After the pre-CA sequence, the peaks corresponding to the O–H (3,385 cm^{-1}) and C=C (1,653 cm^{-1}) bonds were dramatically reduced. This is direct evidence for damage to the membrane surface structure caused by chemical cleaning. In contrast, for the pre-Cl sequence, the reduction in peak strength was not as great. This might be explained by the above-mentioned repair effects of CA, which to some extent repaired the damage to hydrophilic groups caused by exposure to the NaOCl. In contrast, CA played a cleaning rather than a repairing role in the pre-CA sequence (Fig. 3(a)).

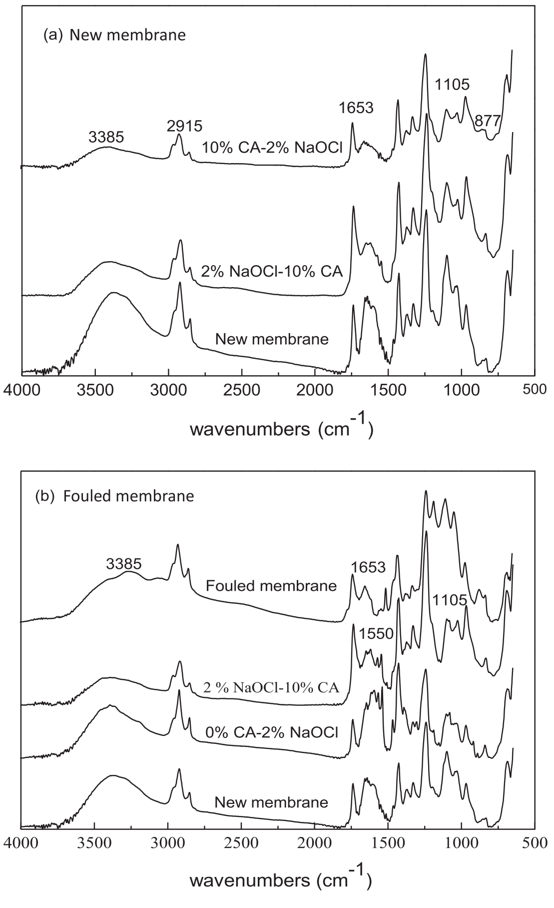

Fig. 4. ATR-FTIR spectra after exposure to NaOCl and CA.

In the case of fouled membranes, foulants, especially aliphatics, were retained on the membrane surface after backwashing and strengthened the bonds around 3,385 cm^{-1} (for the O–H vibration), and from 1,200 to 1,000 cm^{-1} (for the C–O–C and C–C vibrations). Pre-CA cleaning reduced almost all bond strengths, except the intensities around 1,550 cm^{-1} (for the N–H vibration). The reduction of intensity at 1,738 cm^{-1} (for the C=O vibration) implies damage to the surface structure. The shape of the spectrum after pre-CA cleaning was similar to the spectrum of the original new membrane. This illustrates that pre-CA cleaning can effectively remove aliphatics without marked damage to the membrane surface. This could be considered as an advantage of the negative contribution of CA. The proposed bonding of CA and foulants, as discussed above, may protect the membrane surface from attack by radicals released by NaOCl [25].

Regarding the pre-Cl sequence, the intensity around 3,385 cm^{-1} (for the O–H vibration) was significantly decreased compared with the spectra of the original membranes and the membranes after pre-CA cleaning, while the peaks at 1,738 cm^{-1} (for the C=O vibration) and 1,250 cm^{-1} (for the C–O–C vibration) were strengthened. This indicates that O–H bonds were broken in the fouled membrane and the surface structure was significantly altered by NaOCl treatment [13]. Even the subsequent CA treatment made a positive contribution (Fig. 3(b)), and may have further aggravated the damage.

Because of the similar TMP improvements after both sequences, pre-CA cleaning could be an appropriate choice for practical operations and promoting long-term maintenance of membrane integrity.

3.5. Effluent particle size distribution

The PSDEs from fouled membranes are shown in Fig. 5. The PSDE from a fouled new membrane is shown in Fig. 5(a) and the results from a fouled membrane after cleaning are presented in Fig. 5(b) for comparison.

The PSDE of the fresh new membrane was 13–32 nm. The average particle size passed by new membranes after sequential cleaning at any mass fraction and with either sequence was much larger than that of a new membrane. Chemical cleaning apparently enlarged the pore size of new membranes, but the pre-Cl sequence resulted in smaller pore sizes than pre-CA. The peaks narrowed when the concentration of CA was five rather than 10%. This may be a further evidence for the repair role of CA. The results

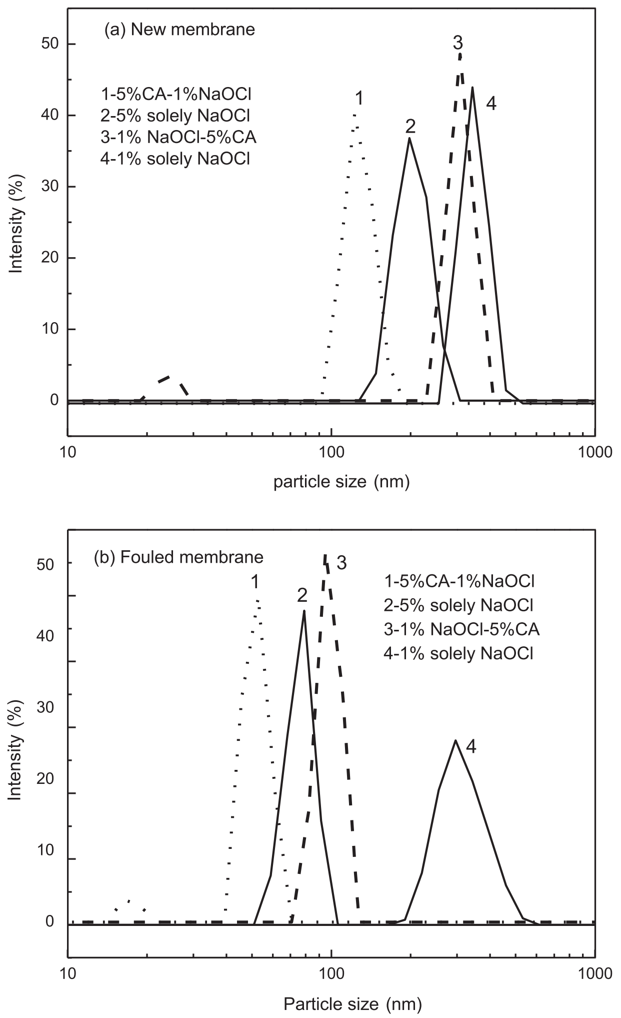

Fig. 5. Particle size distributions of effluent from new membranes and from fouled membranes after cleaning.

are consistent with the negative contribution of CA after the pre-Cl sequence when using a clean new membrane. A higher mass fraction of CA may enhance the repairing effect.

The average particle sizes passed by fouled membranes were smaller than those of new membranes for the same mass fraction and sequence. This indicates that some foulants were not completely removed by chemical cleaning. It is also worth mentioning that the pre-CA sequence cleaning left smaller pore sizes than pre-Cl. This is the converse of the case for the new membranes and is again consistent with the above assertion that citric acid made a negative contribution in the pre-CA sequence when cleaning a fouled membrane (Fig. 3(b)). The PSDE results support the suggestion that adsorption of CA onto foulants may protect the surface structure from further damage during subsequent NaOCl treatment.

NaOCl cleaning at a mass fraction of 1% left a wide PSDE with a range 100–500 nm, which is far larger than that resulting from sequential cleaning. A previous investigation revealed that a higher mass fraction of NaOCl (>1%) results in twisting and wrinkling of the membrane surface, although it also leads to a relatively narrow PSDE [26]. Considering the long-term integrity of membranes, mass fractions higher than 1% are not recommended for cleaning PVC membranes when NaOCl is employed by itself [26]. According to the results of this study, when applied with CA, a NaOCl mass fraction of 1% could be used in practice without compromising the integrity of the membrane. Pilot- or full-scale trials are needed to evaluate the long-term effects of sequential cleaning on membrane integrity.

4. Conclusions

The pre-NaOCl and pre-CA cleaning sequences were found to be equally effective in restoring flux, as evidenced by the similar TMP declines observed. Sequential cleaning reduced the TMP/TMP$_0$ values from 100 to 66–77% for new membranes and from 120 to 92–105% for fouled membranes. The BSA entrapment rate after cleaning was in the range 63–70% for new membranes and 70–78% for fouled membranes. The consistency between the observed TMP improvements and the BSA entrapment rates indicates a positive correlation between TMP decline and pore size after sequential cleaning. Hypochlorite was observed to be much more effective than CA for cleaning foulants, but hypochlorite also caused damage to the surface structure.

CA might help repair fouled membranes when used in the pre-CA sequence. It may mitigate the damage to a membrane's surface structure caused by NaOCl and improve the membrane's integrity.

The particle size distribution data clearly show the advantages of sequential cleaning compared with using NaOCl alone with respect to pore size enlargement. The ATR-FTIR spectra show that C–H, O–H and C=O bonds are broken in chemical cleaning. The pre-CA sequence could be a more appropriate choice than the pre-Cl sequence for prolonging membrane life. A mass fraction of NaOCl greater than 1% could be used in practice if it is used in combination with CA. Pilot- or full-scale trials are needed to evaluate the long-term effects of sequential cleaning on membrane integrity.

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