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Enhanced As(III) removal at low concentrations by the combined pre-oxidation and nanofiltration membrane process

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ABSTRACT

A specially designed combined process of pre-oxidation and nanofiltration membrane was utilized to remove As(III) in the contaminated water without reagent addition and sediment creation in this work. Different parameters that affected the rejections of As(III) and As(V) were discussed, including the initial concentration, pH of the feed, and the cross-flow filtration pressure. The rejection of As(III) was over 70% which was restricted to the high initial feed concentration, while it was around 40% under the other conditions. Whereas, the rejection of As(V) maintained above 95% in all tests. As a result, As(III) was first pre-oxidized before membrane filtration. The ozone and aeration oxidation methods were used to oxidize As(III). The effects of aeration time and oxidation time were studied. The experimental results indicated that the rejection of As(III) was increased to over 90% after pre-oxidation, and the permeate could meet the drinking water standard for As in China. Particularly, the aeration took a much longer oxidation time than ozone, but it was still a more suitable method as the pre-oxidation process because of its safety and easy operability.

Keywords: As(III) removal; Nanofiltration; Pre-oxidation; Ozone; Aeration

1. Introduction

Arsenic (As) is widespread in the natural environment and found in about 200 different minerals [1]. As, especially As(III) is highly toxic, fatal, and difficult to be degraded by normal biological processes. Wastewater containing As(III) would lead to serious environmental pollution and threat to human life and health. The previous national drinking water standard for As in China was 50 $\mu g\ L^{-1}$ (GB 5749-1985) [2]. However, since the year of 2007, the Chinese standard has been lowered to 10 $\mu g\ L^{-1}$ (GB 5749-2006) [3], the same as the standards of many authorities like European Commission and United States Environmental Protection Agency, facing the increasingly serious safety problems of drinking water.

There are a large amount of studies focusing on the As removal. The coagulation/precipitation [4] method is a conventional way to remove As, but the byproduct would generate new forms of pollution without proper treatment and disposal [5]. The adsorption method [6], like activated carbon [7],

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polyaluminum granulate (PAG) [8], magnetic iron oxide [9], and sawdust [10], could also be used to remove As from water. Nevertheless, adsorbent regeneration is a limitation for its extensive application. Among these methods, most of them emphasize particularly on the removal of As(V). As(III) is relatively difficult to be eliminated from water because of its electric neutrality [11].

Membrane processes are considered to be a promising technology for As(III) and As(V) removal [12], including membrane distillation [13], reverse osmosis (RO) [14–16], nanofiltration (NF) [5,17–21], ultrafiltration (UF) with pre-treatment like coagulation [22]. It is aware that the RO technology takes high operating pressure (over 10 MPa sometimes) and energy consumption, which means that the expenditure of money is considerable. NF is a kind of pressure-driven membrane technology between RO and UF, which has got fast development in recent years around the world [23]. In comparison with RO, NF just needs less than 2 MPa normally. The study of Vrijenhoek and Waypa [17] exhibited that As(V) was removed 60–90% by the Film Tech NF-45 membrane, while As(III) rejection was under 20% regardless of pH or As(III) concentration of feed waters. Figoli et al. [5] used two kinds of commercial NF membranes (N30F by Microdyn-Nadir and NF90 by Dow Chemical) to remove As(V) from synthetic water. They found out that among the parameters affecting the As (V) rejection, feed concentration was the key parameter for the As(V) removal. Uddin et al. [18] also studied two commercial polyamide NF membranes (NF-90 and NF-200) for As(III) and As(V) removal. In all tests, As(V) was better rejected than As(III), and the highest rejections obtained were over 98% for As(V) and around 65% for As(III).

According to the previous research results that mentioned before, compared with As(V), the As(III) rejection of NF membranes are commonly low. The idea that As(III) is pre-oxidized to As(V) before membrane filtration is theoretically feasible, if the As(V) rejection of the membrane is pretty high. Plus, the toxicity of the As solutions is reduced after the pre-oxidation owing to the less toxic and fatal As(V), which cannot be fulfilled in the RO process. Plenty of methods can be used to oxidize As(III), such as active chlorine [24], potassium permanganate [25,26], sodium hypochlorite [27], and chlorine dioxide [28]. However, these methods have the main disadvantage of being a health hazard as well as, if not monitored and managed properly [29]. Therefore, we wondered if the As (III) rejection would get higher after it was first oxidized to As(V) in the contaminated water before nanofiltration, which was rarely reported in the previous studies.

This work aims to find out whether the combined pre-oxidation and NF membrane process could effectively intercept As(III) in the contaminated water. The membranes used in the system were homemade polypiperazine–amide composite NF membranes, which were developed in our previous paper [30]. Different parameters that would affect the rejections of As(III) and As(V) were discussed, such as the initial concentration, pH of the feed, and the cross-flow filtration pressure. The ozone oxidation method was first used to oxidize As(III) because of its strong oxidizing property, no reagent addition, and sediment creation. Comparatively, the conventional aeration method was also used as the pre-oxidation process. Moreover, the effects of ozone and aeration oxidation were compared.

2. Experimental

2.1. Materials

Polysulfone (PSF) was bought from Dalian Polysulfone Plastic Manufacturing Co., Ltd, Piperazine (PIP, purity 99%) and trimesoyl chloride (TMC, purity 98%) were purchased from Shanghai Aladdin Reagent Co., Ltd. Na_2SO_4, NaCl, $MgSO_4$, $MgCl_2$, Na_3AsO_4, $NaAsO_2$ were all analytical reagent and used without any further purification.

The homemade polypiperazine–amide composite NF membrane prepared under the optimum condition [30] exhibited Na_2SO_4 rejection of 97.38% under 0.4 MPa at room temperature. In our previous work, the NF membranes were, respectively, soaked in absolute ethanol, diluted HCl, NaOH solution, and 80˚C water for a certain time to test their resistances of solvent, acid, alkali, and high temperature. According to the variation of Na_2SO_4 rejection and water flux, the NF membrane showed good solvent resistance, acid resistance, and high temperature resistant performance, while its alkali resistance still needed improvement.

2.2. Facilities

The fully recycling cross-flow membrane filtration device was designed to test the properties of the composite NF membranes to intercept As(III) and As(V) in the contaminated water, as exhibited in Fig. 1.

The portable conductivity meter (Rex DDBJ-350, Shanghai INESA & Scientific Instrument Co., Ltd) was used to measure the salt concentrations in permeate and feed solutions.

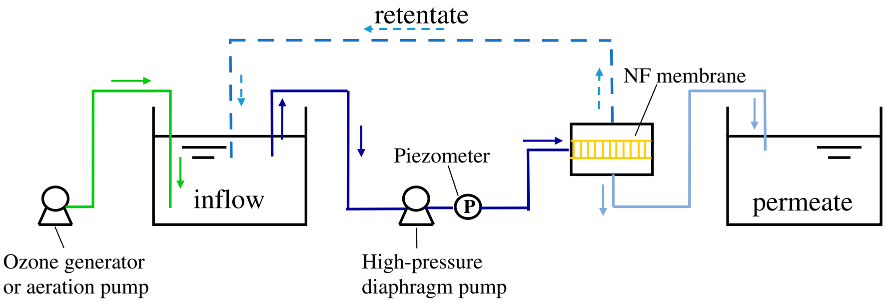

Fig. 1. Schematic diagram of experimental device.

The concentrations of As(III) and As(V) in water were determined by inductively coupled plasma atomic emission spectroscopy (Optima 8300, Perkin Elmer).

The portable multi-parameter controller (Rex DZB-718, Shanghai INESA & Scientific Instrument Co., Ltd) was used to measure the pH of the As solutions.

The ozone generator (CF-YG5, Beijing Makes Co., Ltd) and aeration pump (JZ-AP288, Minjiang Co., Ltd) were, respectively, utilized for ozone and aeration oxidation.

2.3. Methods

The sol–gel process [31] was utilized to prepare the ultrafiltration membrane as the support layer of the composite NF membrane. Then the polypiperazine–amide composite NF membranes were prepared by interfacial polymerization [32] action between PIP and TMC [30].

The salt rejection property of the homemade polypiperazine–amide composite NF membrane was measured according to its respective separation efficiency of Na_2SO_4, NaCl, $MgSO_4$, and $MgCl_2$.

The water flux was determined by direct measurement of permeate flow with cross-flow filtration way. The membranes were pressured at 0.4 MPa for 30 min with the As solution before the water flux and rejection were measured at room temperature. The water flux (J_w) of membranes was calculated as follows:

$$J_w\ (\mathrm{L\ m^{-2}\ h^{-1}}) = V_w\ A^{-1}\ t^{-1} \quad (1)$$

where V_w is the volume of the obtained permeate, A is the effective membrane area (17.71 cm^2 in this experiment), and t is the operating time. The As rejection (R) was calculated as follows:

$$R = (1 - C_p/C_0) \times 100\% \quad (2)$$

where C_p and C_0, respectively, represent As concentrations of the permeate and the inflow.

In the pre-oxidation process, the ozone or air, which was either generated by the ozone generator or aeration pump, was pumped into the As(III) solutions for a certain time before nanofiltration.

3. Results and discussion

3.1. Salt rejection property

The rejection property of Na_2SO_4, NaCl, $MgSO_4$, and $MgCl_2$ is exhibited in Fig. 2. It could be seen that the SO_4^{2-} rejection of the polyamide NF membranes is obviously much higher than the Cl^- rejection. In detail, the rejections of Na_2SO_4, $MgSO_4$, $MgCl_2$, and NaCl, respectively, were 97.38, 94.53, 45.66, and 37.47% in average. As it is known that the nanofiltration membranes depend on not only sieving, but also electrostatic repulsion (Donnan effect), while the polyamide membrane is negatively charged [33]. In this case, the Donnan effect functions dominantly, so the rejection of the monovalent ion Cl^- is lower than the one of the divalent ion SO_4^{2-}. The transit of Cl^-

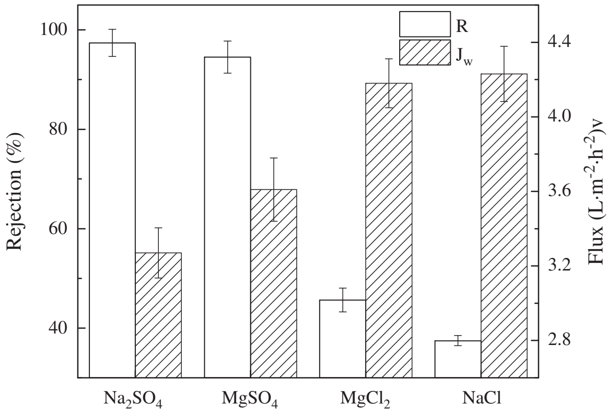

Fig. 2. Salt rejection property of the polyamide composite NF membrane.

accelerates the mass transfer, therefore, more water molecules pass through the membranes. Ultimately, the water flux of Na_2SO_4, $MgSO_4$, $MgCl_2$, and NaCl shows the opposite variation trend of the rejection.

3.2. Effects of experimental conditions on As(III) removal

3.2.1. Initial concentration

Taking it into consideration that As(III) is highly toxic and fatal, the initial concentration of As(III) was 50–250 μg L^{-1}, while the pH of the As(III) solutions were not adjusted (pH 7) under 0.4 MPa at room temperature. As shown in Fig. 3, the rejection and water flux are both gradually increased along with the concentration increasing, which displays different trends from the other studies. It is not sure whether different trends from other references are owing to the entirely homemade composite NF membranes. In the studies of our co-workers [34,35], TiO_2 nanoparticles were added in polysulfone (PSF) membrane casting solution to prepare PSF/TiO_2 UF membranes with super-high flux and BSA rejection. On the basis of the PSF/TiO_2 hybrid membranes, we developed the polyamide/TiO_2 composite NF membranes by interfacial polymerization [30]. The different trends might be owing to that the different porosity and hydrophilicity of the support layer (PSF/TiO_2 hybrid membrane) of the composite NF membrane affects the solute and water passing through the NF membrane.

According to the dissolution–diffusion model of membrane, the water flux (F) and salt flux (F_S) could be, respectively, calculated as follows [15,36]:

$$F = A(\Delta P - \beta\Delta\pi) \tag{3}$$

$$F_S = B(\beta C_1 - C_2) \tag{4}$$

where A and B are the permeation coefficients of water and salt, respectively, ΔP is the operating pressure, β is the concentration polarization factor, $\Delta\pi$ is the osmotic pressure, C_1 and C_2 are the salt concentrations on both sides of membrane.

When ΔP remains the same, the increased As(III) concentration makes the higher $\Delta\pi$ owing to the concentration polarization effect. But the concentration polarization factor is decreased. The interaction of the osmotic pressure and concentration polarization factor results in the improvement of water flux. The low As concentrations have effect on the water flux, which have been found in some references [5,11,19]. When the variation extent of F_S is much less than that of F, the As(III) concentration of the permeate gets lower and lower, leading to the gradual growth of As(III) rejection.

3.2.2. pH

Fig. 4 exhibits that when pH was 6–10, As(III) was 50 μg L^{-1} under 0.4 MPa at room temperature, the rejection of As(III) changes relatively slightly and maintains around 40%, while the water flux is substantially increased. It is correlated with the states of As(III) and As(V) ions in the aqueous solutions [11,37,38]:

$$H_3AsO_3 + H_2O \Leftrightarrow H_2AsO_3^- + H_3O^+;\ pK_a = 9.10 \tag{5}$$

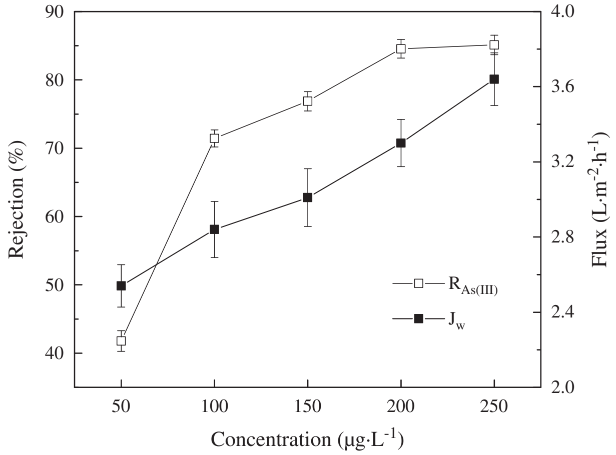

Fig. 3. Effect of initial concentration on the As(III) rejection and water flux.

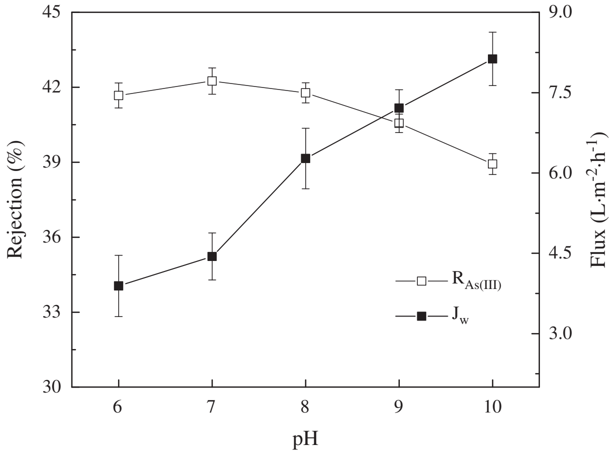

Fig. 4. Effect of pH on the As(III) rejection and water flux.

$$H_3AsO_4 + H_2O \Leftrightarrow H_2AsO_4^- + H_3O^+;\ pK_{a1} = 2.19 \quad (6)$$

$$H_2AsO_4^- + H_2O \Leftrightarrow HAsO_4^{2-} + H_3O^+;\ pK_{a2} = 6.94 \quad (7)$$

$$HAsO_4^{2-} + H_2O \Leftrightarrow AsO_4^{3-} + H_3O^+;\ pK_{a3} = 11.50 \quad (8)$$

According to Eq. (5), with the pH range of 6–10 in this study, the neutral H_3AsO_3 molecules mainly exist at pH below 9.10, which is disadvantageous for the negatively charged NF membranes to remove As(III). The removal of H_3AsO_3 mainly depends on the sieving of NF membranes, when the molecular weight of H_3AsO_3 is lower than the molecular weight cut-off (MWCO) of the NF membranes (600 Da in this study). The rejections of As(III) should be theoretically improved owing to the production of more $H_2AsO_3^-$ when pH increases to above 9. Nevertheless, as mentioned in Section 2.1, the alkali resistance of the homemade polyamide NF membranes was not good. The compact net structure of the polymer layer was damaged in alkaline environment. When pH is 7–10, the rejection of As(III) is decreased as a result. The increase of water flux might be owing to the electroviscous effect [39], which is a physical phenomenon that happens when the electrolyte solution passes through the charged capillary pores. When pH gets higher, the influence of electroviscous effect is less, so the water flux is enhanced.

3.2.3. Operating pressure

The effect of operating pressure on As(III) rejection and water flux is shown in Fig. 5, under the conditions of As(III) 50 μg L^{-1}, pH 7 at room temperature, the water flux is increased, while the rejection of As (III) is decreased. This is because the Donnan effect is not dominant since the neutral H_3AsO_3 molecules mainly exist. Under this circumstance, the interception of As(III) primarily relies on the sieving of the NF membranes. With the pressure increasing, more H_3AsO_3 and water molecules pass through the membranes. When the growth extent of H_3AsO_3 is more than that of water, the As(III) concentration of the permeate gets higher and higher, leading to the gradual decrease of As(III) rejection.

3.3. Effects of experimental conditions on As(V) removal

3.3.1. Initial concentration

On account that As(V) is not as toxic and fatal as As(III), the range of the initial As(V) concentration was broadened to 0.1–5.0 mg L^{-1}, when the pH of the As (V) solutions was about 7 under 0.4 MPa at room temperature. Fig. 6 shows the similar variation trend with Fig. 3. Based on Eq. (7), when pH was around 7, there are more $HAsO_4^{2-}$ generated due to the increase of As (V) concentration. Compared with the monovalent ion $H_2AsO_4^-$, the divalent ion $HAsO_4^{2-}$ is more easily intercepted by the negatively charged NF membrane. Consequently, the rejection of As(V) is increased.

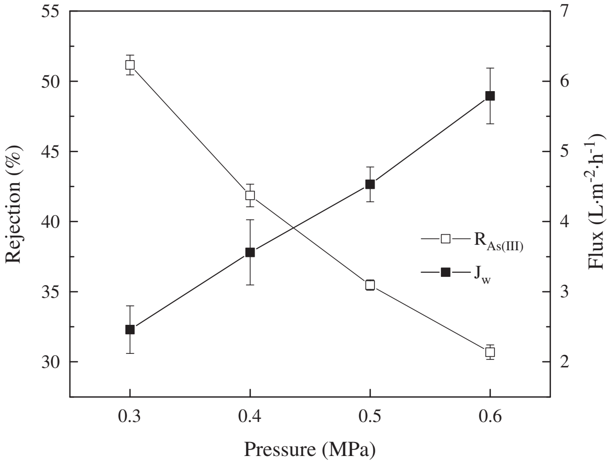

Fig. 5. Effect of pressure on the As(III) rejection and water flux.

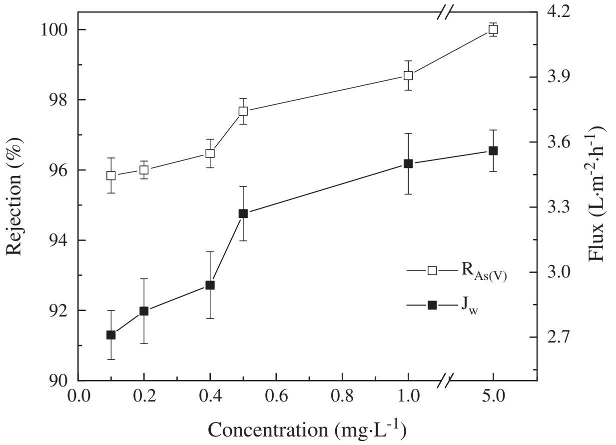

Fig. 6. Effect of initial concentration on the As(V) rejection and water flux.

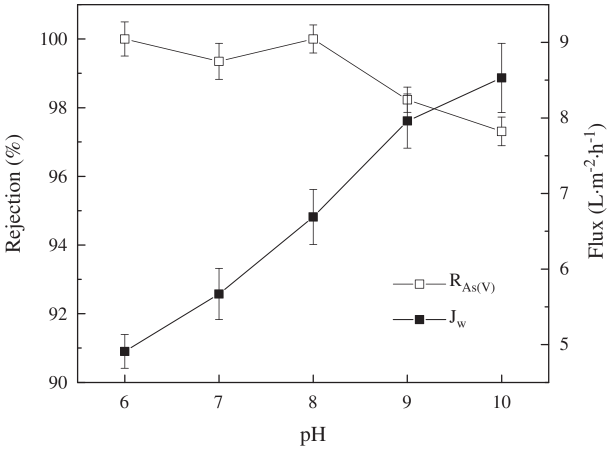

Fig. 7. Effect of pH on the As(V) rejection and water flux.

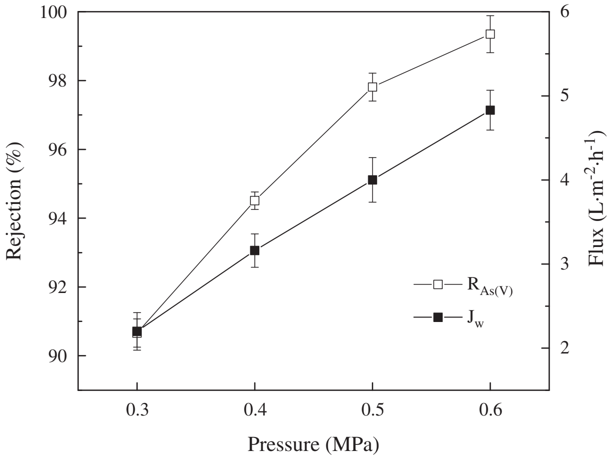

Fig. 8. Effect of pressure on the As(V) rejection and water flux.

3.3.2. pH

Fig. 7 shows that when pH was 6–10, As(V) was 200 μg L^{-1} under 0.4 MPa at room temperature, the rejection of As(V) also changes relatively slightly, while the water fluxes are substantially increased. Unlike the rejection of As(III) maintains around 40%, for As(V), it changes between 97 and 99%. According to Eqs. (6)–(8), with the pH range of 6–10 in this study, the normal existential states of As(V) are negative ions as $H_2AsO_4^-$ and $HAsO_4^{2-}$, which is advantageous for the negatively charged NF membranes to remove As(V) owing to the electrostatic repulsion primarily as mentioned in Section 3.1. The increase of water flux also might be owing to the electroviscous effect [39], which is a physical phenomenon that happens when the electrolyte solution passes through the charged capillary pores. When pH gets higher, the influence of electroviscous effect is less, so the water flux is enhanced.

3.3.3. Operating pressure

The effect of operating pressure on As(V) rejection and water flux is shown in Fig. 8, under the conditions of As(V) 200 μg L^{-1}, pH 7 at room temperature. The rejection of As(V) and the water flux are both gradually improved along with the increase of pressure. Based on the dissolution–diffusion model of membrane, according to Eq. (3), when ΔP is increased and $\Delta \pi$ remains constant, F is approximately linearly increased. Thus, water flux is raised. When the variation extent of F_S is less than that of F, the As(V) concentration of the permeate gets lower and lower, leading to the gradual growth of As(V) rejection.

3.4. Effects of pre-oxidation methods on As(III) removal

3.4.1. Ozone oxidation

Based on the previous results, the rejection of As(III) was around 40% under most conditions, while for As(V), it is maintained above 95% in all tests. Therefore, the idea that As(III) was pre-oxidized to As(V) before nanofiltration was theoretically feasible. The ozone oxidation method [40] was first used to oxidize As(III) because of its strong oxidizing property, no reagent addition and sediment creation. The gas flow rate of the ozone generator was 4 L min^{-1}, and the ozone output was 4 g h^{-1}. When the As(III) concentration of the inflow was 50 μg L^{-1}, the one of the permeate was 9.67 μg L^{-1} after pumping ozone for about 30 min, which could meet the drinking water standard for As in China, as shown in Fig. 9(b). When the As(III) concentration was 100 μg L^{-1}, it took 20 min to meet the standard. As explained in Section 3.2.1, when the As(III) concentration was increased, the rejection was improved. The shortened time might be due to the different intermolecular interactions between As(III) and water molecules under different concentrations. So the ozone oxidation time was shortened when the As(III) concentration was raised from 50 to 100 μg L^{-1}.

Along with the extension of ozone oxidation time, more and more As(III) compounds are oxidized to As(V); thus, the rejection is increased and the As concentration of the permeate is lowered to under 10 μg L^{-1}. The solubility of ozone is much higher than the ones of oxygen and air [40]. Plus, ozone decomposes quickly and generates oxygen, which forms microbubbles in the water. The bubbles promote the mass transfer, resulting in the increase of the water flux.

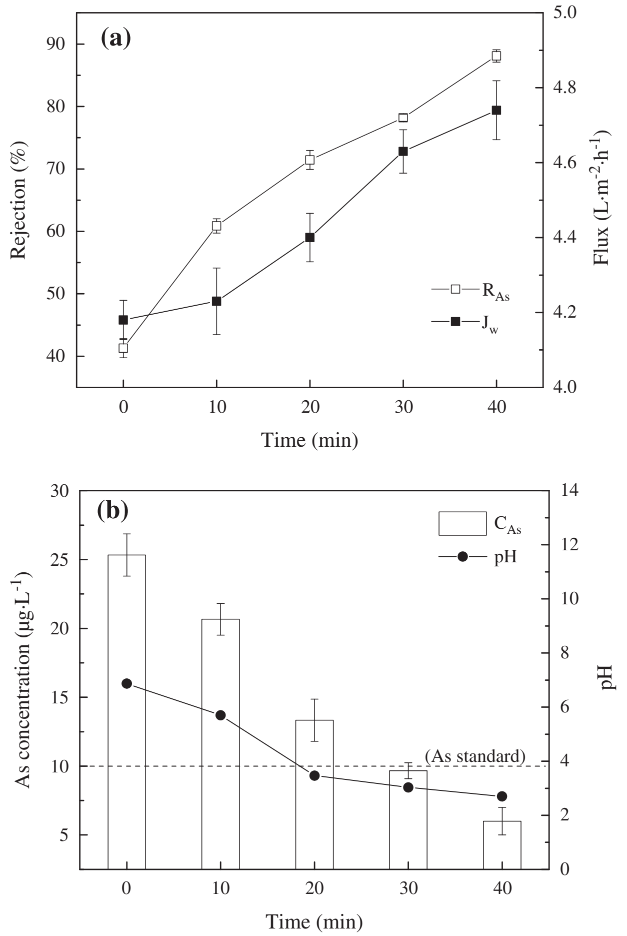

Fig. 9. Effect of ozone oxidation time on (a) AsIII) rejection ($C_{\mathrm{As}} = 50\ \mu\mathrm{g\ L}^{-1}$) and water flux and (b) As concentration of permeate and pH.

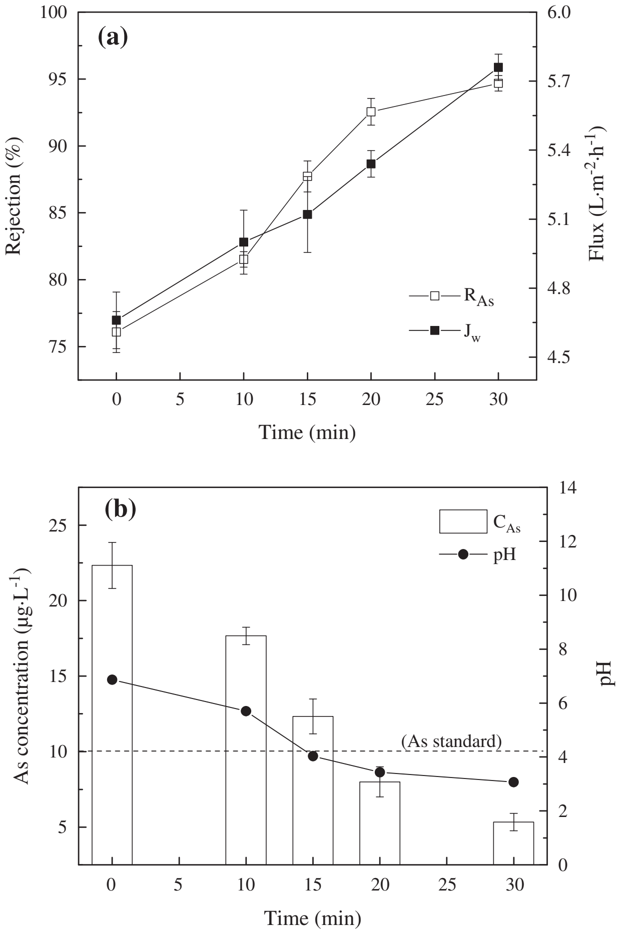

Fig. 10. Effect of ozone oxidation time on (a) As(III) rejection ($C_{\mathrm{As}} = 100\ \mu\mathrm{g\ L}^{-1}$) and water flux and (b) As concentration of permeate and pH.

Ozone oxidation can simultaneously improve the As (III) rejection and water flux fast and effectively. However, as shown in Figs. 9(b) and 10(b), when the As concentration of the permeate can meet the standard, the pH is decreased to 3–4, which cannot meet the water quality standard for pH [3]. Moreover, ozone oxidation is costly and would accelerate the aging process of the membranes [41].

3.4.2. Aeration oxidation

Although ozone oxidation has achieved good results as a pre-oxidation process for nanofiltration, it cannot be avoided that ozone is hazardous to human health [29]. Besides, the pH was lowered beyond the water quality standard after pumping ozone. We were inspired by the activated sludge process, in which the aeration could offer oxygen to the sludge to degrade the pollutants. So, it was suspected if the conventional aeration method also could be used as pre-oxidation process.

The gas flow rate of the aeration pump was 40 mL min^{-1} (power = 4 W), which was far less than the rate of the ozone. But a ceramic aerator was used to uniformly disperse the air in the experiments, which could improve the aeration efficiency to some extent. When the As(III) concentration of the inflow was 50 $\mu\mathrm{g\ L}^{-1}$, the one for the permeate was 6.67 $\mu\mathrm{g\ L}^{-1}$ after aeration for 70–80 min, which could meet the drinking water standard for As in China as Fig. 11(b) exhibited. When the As(III) concentration was 100 $\mu\mathrm{g\ L}^{-1}$, it took 50–60 min to meet the standard as shown in Fig. 12(b). As explained in Section 3.2.1, when the As(III) concentration was increased, the rejection was improved as well. So the aeration oxidation time was shortened when the As(III) concentration was raised from 50 to 100 $\mu\mathrm{g\ L}^{-1}$. Along with the extension of aeration time, dissolved oxygen is

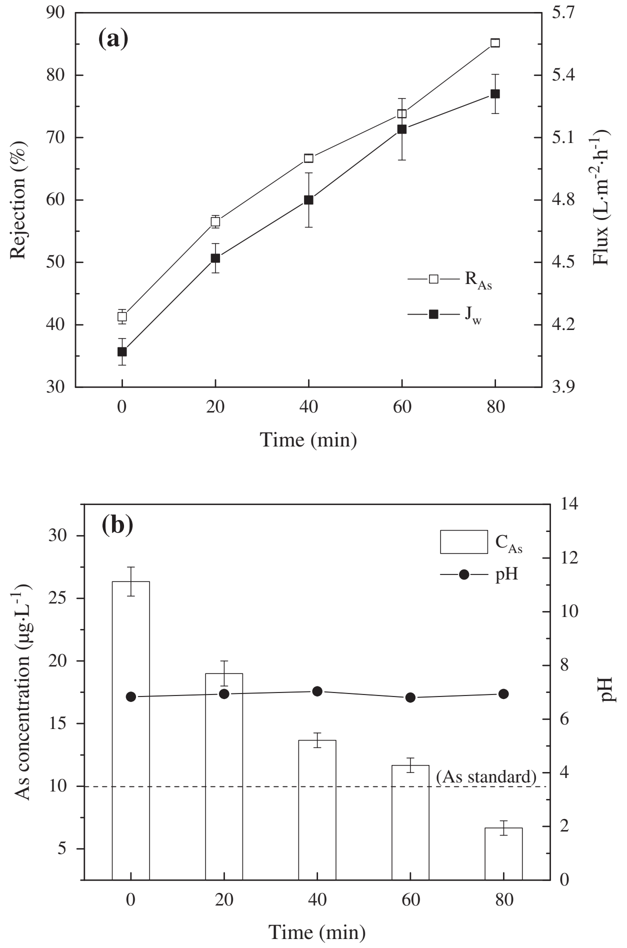

Fig. 11. Effect of aeration time on (a) As(III) rejection (C_{As} = 50 μg L^{-1}) and water flux and (b) As concentration of permeate and pH.

improved. More and more As(III) compounds are oxidized to As(V); thus, the rejection is gradually increased and the As concentration of the permeate is lowered to under 10 μg L^{-1}. In the meanwhile, there are a large number of micro-bubbles in the water because of aeration, which can promote the mass transfer, so the water flux is raised as well.

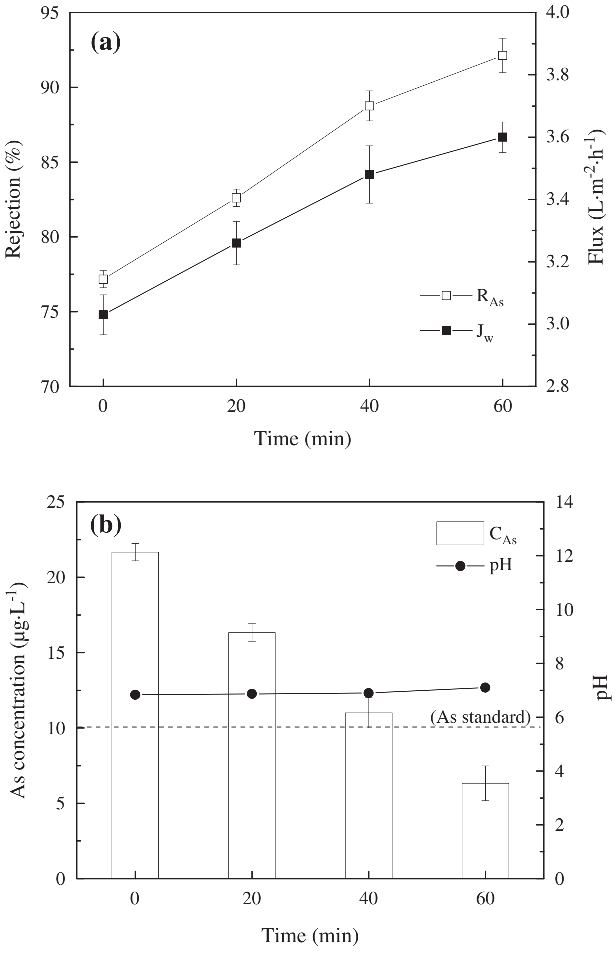

Fig. 12. Effect of aeration time on (a) As(III) rejection (C_{As} = 100 μg L^{-1}) and water flux and (b) As concentration of permeate and pH.

The effects of ozone and aeration oxidation are compared, as shown in Table 1. The gas flow rate of the aeration pump is far less than the rate of the ozone, but the ceramic aerator could uniformly disperse the air in the solutions, which could improve the aeration efficiency to some extent. Nonetheless, aeration still takes a much longer oxidation time than

Table 1
Comparison between ozone and aeration oxidation on the As(III) removal results

				C_{As} = 50 μg L^{-1}		C_{As} = 100 μg L^{-1}	
Method	Toxicity	Flow rate	Ceramic aerator	Time (min)[a]	pH[b]	Time (min)[a]	pH[b]
Ozone	High	4 g h^{-1}	Not used	30	3.0	20	3.4
Aeration	Low	40 mL min^{-1}	Used	80	6.9	60	7.1

[a] Time for As(III) removal to meet the As standard.

[b] pH of the As solutions.

ozone. On the other hand, the aeration does not generate the byproducts and seriously change the pH of the solutions. It is also safer and easier to operate and control. As a result, aeration is still a more suitable and suggested method as the pre-oxidation process.

4. Conclusions

The homemade polypiperazine–amide composite nanofiltration membranes were used in the combined pre-oxidation and NF membrane process to remove As(III) in the contaminated water. The results indicated that the initial concentration affected the As rejection the most significantly. The rejection of As(III) was over 70% which was restricted to the high initial feed concentration, while it was around 40% under the other conditions. Whereas, the rejection of As(V) maintained above 95% in all tests. As a result, As(III) was first pre-oxidized before filtration. The ozone and aeration oxidation methods were used to oxidize As (III). The rejection of As(III) was increased to over 90% after pre-oxidation, and the permeate could meet the drinking water standard for As in China. Particularly, the aeration took a much longer oxidation time than ozone, but it was still a more suitable method as the pre-oxidation process because of its safety and easy operability.

Acknowledgments

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