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Fouling and Cleaning in an Ultrafiltration Membrane Bioreactor for Municipal Wastewater Treatment

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ABSTRACT

This paper investigates the fouling and cleaning of a tubular ultrafiltration membrane for the treatment of municipal wastewater. A bistage fouling hypothesis, i.e., channel clogging and gel layer forming, was introduced to elucidate the evolutional mechanism of ultrafiltration fouling. An effective method for channel-clogging prevention was developed, resulting in an extension of operation period to more than

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eight weeks. The multistep chemical cleaning protocol was tested to remove the gel layer from fouled membrane surface and further optimized in terms of cleaning temperature and NaClO concentration. It was found that the optimized chemical cleaning could restore the membrane's standard permeability to higher than 94% if taking the standard permeability of a new membrane as 100%.

Key Words: Fouling mechanism; Membrane bioreactor; Multistep cleaning; Municipal wastewater; Ultrafiltration.

INTRODUCTION

The combination of ultrafiltration (UF) membrane with a bioreactor is known as the membrane bioreactor[1] that offers distinct advantages over traditional biological processes, such as higher biodegradation efficiency, smaller footprint, better quality of treated water, and easy control of operating conditions.[2–4] However, membrane bioreactor has an inherent flaw, namely membrane fouling.[5] Despite its contribution to solute rejection, membrane fouling has been generally recognized as the causes of permeate flux decline and frequent physical/chemical cleaning.[6] Membrane fouling has been significantly limiting the widespread use of membrane bioreactor application.[2]

It has been suggested that the propensity of UF membrane to fouling is largely dependent on the feed quality, membrane type, and operating conditions[7]; most fouling studies are therefore focused on backwashing, back flushing, crossflow velocity, turbulence promoter, as well as two-phase slug flow etc.[8–12] The role of specific constituents (e.g., suspended solids, colloids, and dissolved molecules) in activated sludge has also been examined.[4,5] However, there is no publication available on the evolutional mechanism of UF membrane fouling in municipal wastewater treatment though such a mechanism can be prerequisite to minimizing the negative impact of membrane fouling on the performance of UF membrane bioreactor.

The major objective of this paper was to investigate the evolutional mechanism of UF fouling and the effectiveness of multistep cleaning in membrane bioreactor for municipal wastewater treatment. A bistage fouling hypothesis was introduced and verified by experimental evidences. To make it more effective, the multistep chemical cleaning protocol was further optimized in terms of cleaning temperature and NaClO concentration.

MATERIALS AND METHODS

Experimental Description

All the experiments were conducted in constant flux mode on the pilot shown in Fig. 1, where the 30-liter bioreactor, centrifugal pump 2, and UF module constituted a loop. Within the loop, activated sludge was circulated through membrane channels at a velocity of 3–4 $m\,s^{-1}$. At a constant hydraulic retention time of 5.0 hours, sludge retention times were in turn extended from 5, 15, to 30 days. During the 165 days of experiment, membrane fluxes of 75 and 150 $L\,m^{-2}\,h^{-1}$ were sequentially applied at elevated transmembrane pressures (TMP). When the TMP reached to 0.1 MPa, the membrane was thought as seriously fouled and a thorough chemical cleaning was performed to restore UF membranes' permeability. In the mode of cleaning, the cleaning tank would replace the bioreactor to form a cleaning loop together with pump 2, pump 3 and UF modules.

The tubular UF membrane used in this study was the seven-channel and 40 cm-long zirconia Kerasep™ X3 (Tech-Sep, F01703, Mirabel, France), having a channel diameter of 4.5 mm, surface area of 0.04 m^2, and molecular-weight-cut-off of 300 k Dalton. Its initial permeability was about 3.5 $L\,m^{-2}$-$h^{-1}\,(kPa)^{-1}$ on 20°C tap water test. The membrane permeability under such measuring conditions was hereinafter called standard permeability.

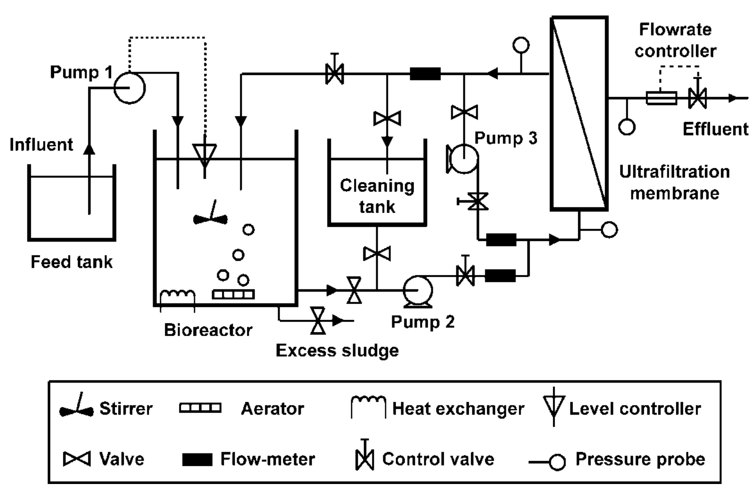


Figure 1. Schematic of UF membrane bioreactor.

Table 1. Characteristics of municipal wastewater.

Items	Typical	Range
COD, mg L^{-1}	200–800	50–2234
SS, mg L^{-1}	100–600	80–1327
NH_3-N, mg L^{-1}	10–30	10–40
Total coliform, CFU L^{-1}	10^5–10^6	10^5–10^7
Turbidity, NTU	50–70	50–80
pH value	7.5–8.5	7.5–8.5
Temperature, °C	15–25	15–25

The municipal wastewater was obtained from a local sewage station. According to Table 1, this wastewater could be classified as medium strength.[3]

Analytical Methods

To examine surfaces of new and fouled UF membranes, a scanning electronic microscope (SEM) (Model, Hitachi S-570, Hitachi Co., Tokyo, 101–8010, Japan) was employed. All the SEM samples were properly dried and preplated with gold prior to examination. An optical microscope (Model, Nikon Optiphot-2, Nikon Co., Tokyo, 100–8331, Japan) was used to view the fiber-zoogloea structure that caused channel clogging. Concentrations of metal ions such as Fe, Cu, Ca, and Mg were measured by atomic absorption spectrophotometer (Model, Shimadzu AA-6200). After each step of cleaning, membrane permeability was immediately determined *in situ* with the 20°C tap water.

RESULTS AND DISCUSSION

The Bistage Fouling Hypothesis

The pilot operation consisted of alternate filtration cycle and cleaning cycle. At the end of each filtration cycle, rapid decrease in membrane permeability and corresponding increase in TMP would be observed due to the membrane fouling.[13] The 165 days of experimental observation indicated that the evolutional mechanism of UF membrane fouling involved two correlative stages, i.e., channel clogging and gel layer forming.

Channel Clogging

The channel clogging represented the first stage of UF fouling, featured as serious formation of fiber-zoogloea structure at the inlet of membrane module. Our previous findings[14] showed that channel clogging was closely related to both the module geometry and fibrous matters contained in the activated sludge. Upon start of a filtration cycle, the fibrous matters began to accumulate at the inlet of UF module in the presence of sticky zoogloea. With the recirculation of activated sludge, a dense stack of fiber-zoogloea structure (see Fig. 2a) would eventually form within two days time, and the shape of such fiber-zoogloea structure fit the geometry of UF module very well, indicating that the UF module might be functioned as the "casting mold" during the early development of channel clogging. Depending on the severity of early channel clogging, relevant decrease of channel flowrate would be observed, primarily leading to deterioration of fluid dynamic conditions within membrane channels. It should be pointed out that there was still no discernible reduction in effective membrane surface area at the early development of channel clogging.

As a consequence of the aforementioned deterioration of fluid dynamic conditions, some fibrous foulants and sticky zoogloea would be gradually "squeezed" into the depths of membrane channels during the next few days, and inside the membrane channels were sometimes seen dense cylinders of fiber-zoogloea structure (see Fig. 2b) that could cause significant decrease in

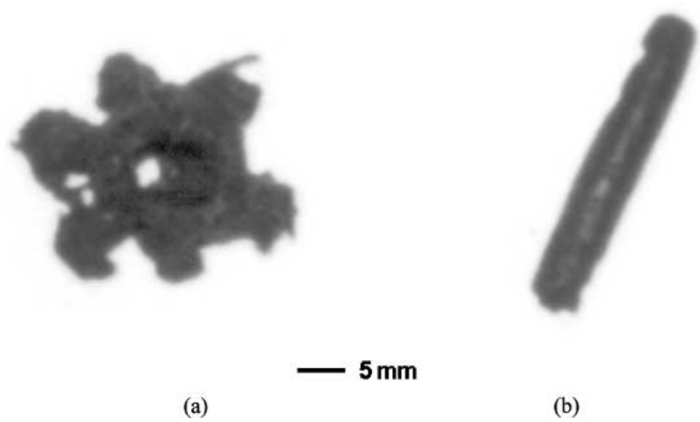


Figure 2. (a) Fiber-zoogloea structure at the UF membrane inlet, and (b) a cylinder formed within membrane channels.

effective membrane surface area. Supposing that only three of the seven channels were completely clogged, the effective membrane surface area would decrease by approximately 43%. At constant membrane flux, corresponding decrease in UF permeate flow would be observed and the design capacity of membrane bioreactor therefore became unachievable. According to our observation, the earlier development of channel clogging (see Fig. 2a) took about 2–3 days while the formation of dense cylinders inside membrane channels (see Fig. 2b) required 5–7 days. Upon the natural occurrence of channel clogging, the filtration cycle of such a UF pilot was generally as short as 7–10 days. In other words, membrane cleaning had to be performed every 7–10 days in order that the UF pilot could achieve its design capacity. Thus, the cleaning cost and operational complexity would be inevitably elevated.

To investigate the component of fiber-zoogloea structure, the UF module was dismantled after the filtration cycle for immediate sampling. Under the SEM, it could be clearly seen that such fiber-zoogloea structure was composed of various fibrous matter and zoogloea (Fig. 3a and 3b) that were either cross-linked or closely stuck to each other. In

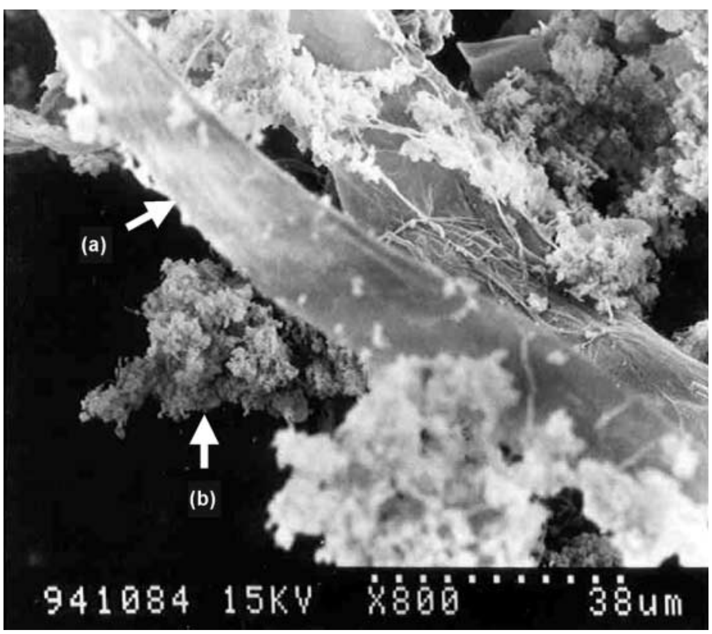


Figure 3. (a) Fibrous matters and (b) Zoogloea.

the formation of fiber-zoogloea structure, these fibrous matters might serve us the internal skeleton, just as a steel bar does in reinforced concrete, while the role of sticky zoogloea was to conglutinate the fibrous matters together. Both the zoogloea and fibrous matters played indispensable roles in channel clogging. As zoogloea was also the major contributor to biological treatment of municipal wastewater and fibrous matters were too small to be removed by simple pretreatment, it might not be realistic for us to protect the UF membrane module from channel clogging through elimination of zoogloea or fibrous matters or both from activated sludge. However, an innovative method for channel-clogging prevention was introduced in the following discussion.

Gel Layer Forming

The forming of gel layer on the UF membrane surface represented the second stage of fouling evolution with respect to the time elapsed. The major foulants were presumably separate/conjunctive microorganisms, extracellular polymeric substances (EPS) matrix, and metal ions, while the molecular basis of gel layer forming was extremely intricate and still poorly understood so far.[7] In the present study, however, gel layer forming was investigated from the angle of experimental evidence.

Once the filtration cycle started, some foulants (i.e., separate/united microorganisms, EPS matrix, and metal ions) in the turbulent region would pass through the boundary layer and reach the UF membrane via convective diffusion transport, and concurrently, other foulants on the UF membrane surface, driven by tangential-flow shearing force, would return the turbulent region across boundary layer (see Fig. 4). These two reverse processes struck a dynamic balance. When the balance was lost, the gel layer would thicken to some extent due to the fact that the membrane surface velocity associated with the tangential-shearing force was set constant in this study. With the buildup of gel layer on the membrane surface, TMP approach 0.1 MPa gradually and a typical gel layer would be seen under the SEM thereafter (see Fig. 5). Compared with the new membrane (see Fig. 6), it was obvious that a fouled UF membrane exhibited significant reduction in porosity because of the formation of gel layer. Figure 7 gives another evidence of gel layer forming, where zone A indicated the residual gel layer by a striking contrast with zone B, the NaClO-cleaned membrane surface. Further magnification of zone B (see Fig. 8) revealed that there was no discernible difference between

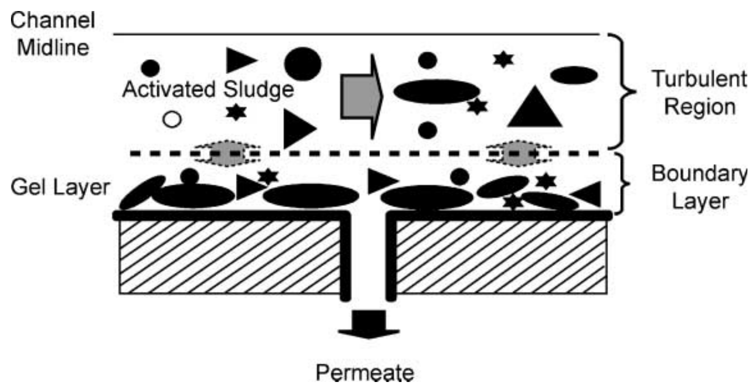


Figure 4. Description of gel layer forming.

the cleaned and new UF membrane (see Fig. 5), demonstrating the effectiveness of NaClO cleaning. However, the remaining portion of gel layer as shown in zone A needed to be removed by subsequent HNO_3 cleaning. From the above analysis, it could be concluded that the macroscopic effect of gel layer forming was actually to place a barrier

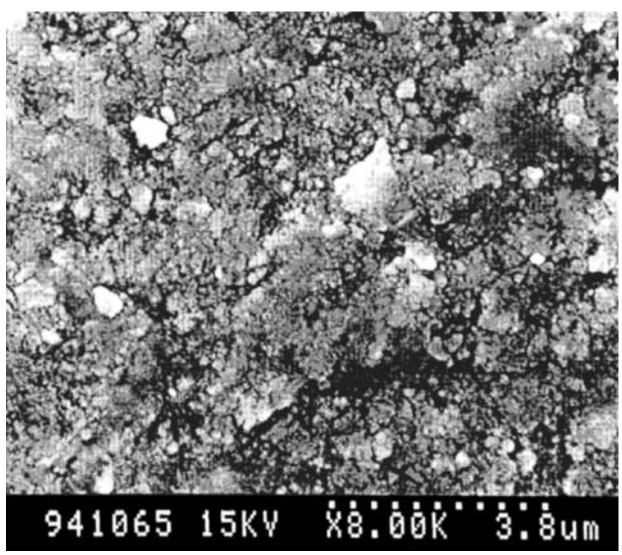


Figure 5. The fouled UF membrane surface.

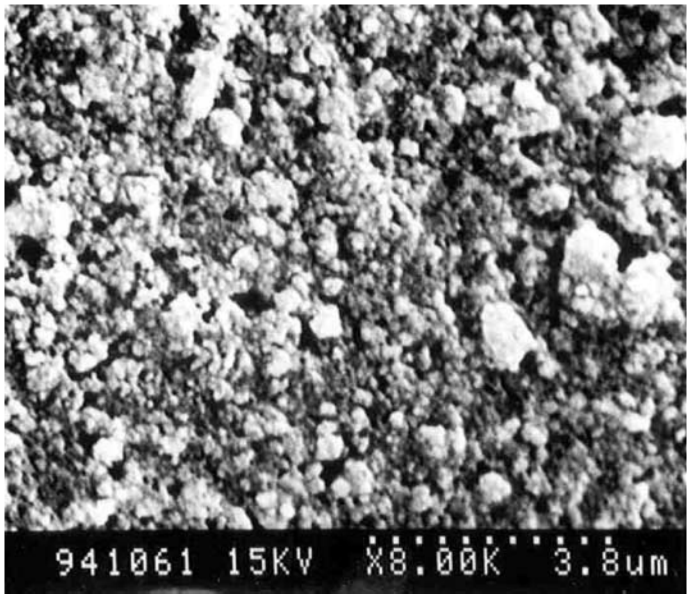


Figure 6. The new UF membrane surface.

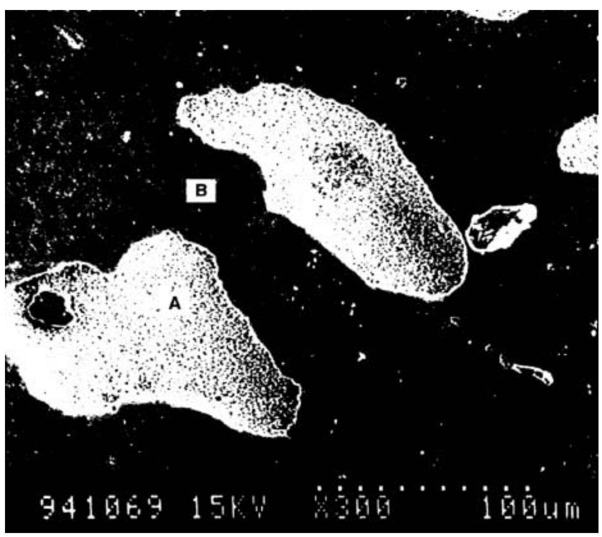


Figure 7. The fouled UF membrane after alkaline cleaning.

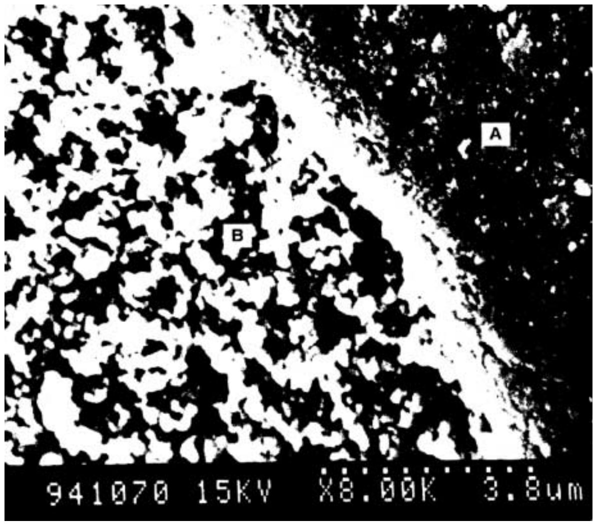


Figure 8. The magnification of zones A and B.

between the activated sludge to be filtrated and the porous surface of UF membrane, thereby elevating the filtration resistance and fluid friction. As a consequence, the convective fluid motion proximal to the membrane surface would slow down and an observable increase in TMP would be encountered. Once TMP attained 0.1 MPa, a thorough membrane cleaning had to be undertaken so that the membrane bioreactor could be operated to its design capacity.

Provided the early channel clogging was properly prevented, an interesting phenomenon that occurred at the very beginning of the filtration cycle was the initial modification to UF membranes, probably by the EPS matrix or metal ions or both. Such a modification was monitored through the variation of permeate COD vs. filtration time. As shown in Fig. 9, the UF permeate COD showed a rapid decrease from 47.5 mg/L to 3.9 mg/L during the first 20 minutes. Followed was a platform and no further decrease of permeate COD was observed. In other words, the initial modification was completed in 20 minutes for a UF membrane bioreactor. Similar results were also reported on microfiltration membrane bioreactor[15]; however, the time required for initial modification was nearly double (i.e., about 40 minutes).

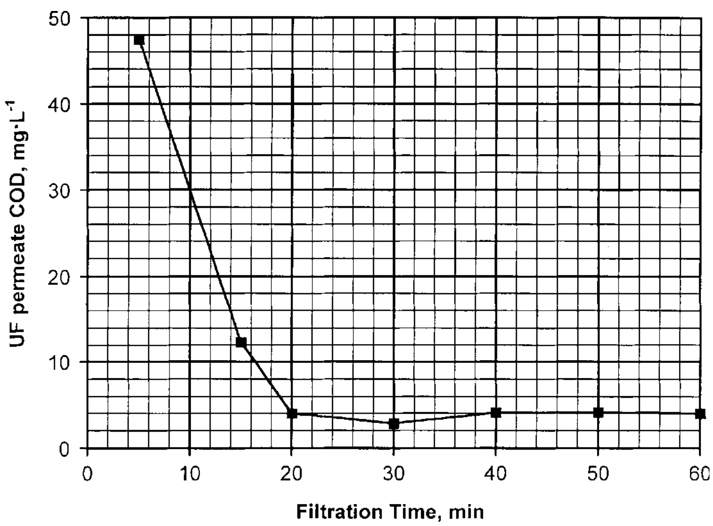
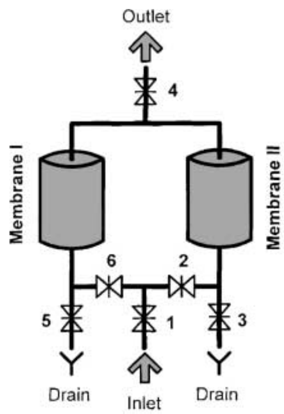


Figure 9. The initial modification: UF permeate COD vs. filtration time.

Membrane Cleaning

Prevention of Channel Clogging

As mentioned above, the development of early channel clogging took about 2–3 days. If the responsible foulants for channel clogging such as fibrous matters and sticky zoogloea were daily removed from the inlet of the UF membrane modules prior to the mature formation of fiber-zoogloea structure, channel clogging could be prevented therewith. Taking continuous filtration as a restriction, the preventive cleaning introduced in this study is illustrated in Fig. 10. In filtration mode, activated sludge from the bioreactor flowed through valve 1 → valve 2 and 6 → membrane I and II → valves 4 and then back to the bioreactor. When the membranes I and II were to be cleaned, the activated sludge would circulate along the direction of valve 1 → valve 2 → membrane II → membrane I → valve 5 and valve 1 → valve 6 → membrane I → membrane II → valve 3 for several seconds, respectively. Lots of cleaning practices had proven that if such a cleaning was performed once a day, channel clogging could be completely prevented and as a result, the filtration cycle would see an extension by five to eight fold, or more than 8 weeks on average. In addition to making overall operation easy and membrane performance stable, the use of cleaning agents could be minimized. In fact,



Valve	Normal	Clean Membrane I *for* 2–3 s	Clean Membrane II *for* 2–3 s
1	Open	Open	Open
2	Open	Open	Close
3	Close	Close	Open
4	Open	Close	Close
5	Close	Open	Close
6	Open	Close	Open

Figure 10. The prevention of channel clogging.

this preventive cleaning was also applicable to the channel clogging encountered in microfiltration membrane bioreactor.[14]

It should be pointed out that even with preventive cleaning, the UF pilot produced permeate as usual in terms of quality and quantity. Technically, such cleaning could be realized *in situ* by a programmable logic control unit and a patentable cleaning device may be hidden.

Chemical Cleaning

Unlike the aforementioned preventive cleaning, the target of chemical cleaning was the gel layer deposited on the UF membrane surface. According to Tech-sep® recommendations, some reagents such as NaOH, HCl, and formulated Ultrasil® may be applied for chemical cleaning. Considering the specific composition of gel layer, however, a multistep cleaning procedure was proposed in this study, i.e., permeate rinsing, NaClO cleaning, and HNO_3 cleaning in sequence. The purpose of permeate rinsing was to wash away loosen deposits on the internal surface of the cleaning loop while the NaClO cleaning was aimed to preliminarily breach the gel layer (mainly EPS matrix). Some large pieces of EPS matrix inside membrane channels would be scoured away by high-speed circulation. The role of HNO_3 cleaning was to further break the remaining EPS matrix into smaller pieces through dissolving the chemically combined metal ions. These smaller pieces of EPS matrix were then peeled off the membrane surface by the circulation-shearing forces so that UF membrane permeability could be restored satisfactorily. The entire chemical cleaning took about 40 minutes. If the standard permeability of a new UF membrane were regarded as 100%, residual standard permeability of a fouled UF membrane was statistically in the range of 25–30%. After multistep cleaning, however, the standard permeability could be restored to more than 94%, indicating the high efficiency of the chemical cleaning proposed here.

Three important parameters governing standard permeability recovery were the cleaning temperature, NaClO concentration, and HNO_3 concentration. To investigate effects of cleaning temperature and NaClO concentration, the HNO_3 concentration remained constant at 1% (w/w) in this study. The effect of cleaning temperature on the recovery of membrane standard permeability is presented in Fig. 11. It could be seen that the residual permeability for group 50–60°C was 10% higher than that for group 40–50°C. On average, permeate rinsing could restore the standard permeability to 25% and 40% for groups 40–50°C and 50–60°C, respectively, where the standard permeability for group 50–60°C was still 15% higher than that for group 40–50°C. After NaClO cleaning, the standard permeability for group 40–50°C increased to 80% while for the group 50–60°C, the standard permeability reached 66% only, implying that for a fouled UF membrane

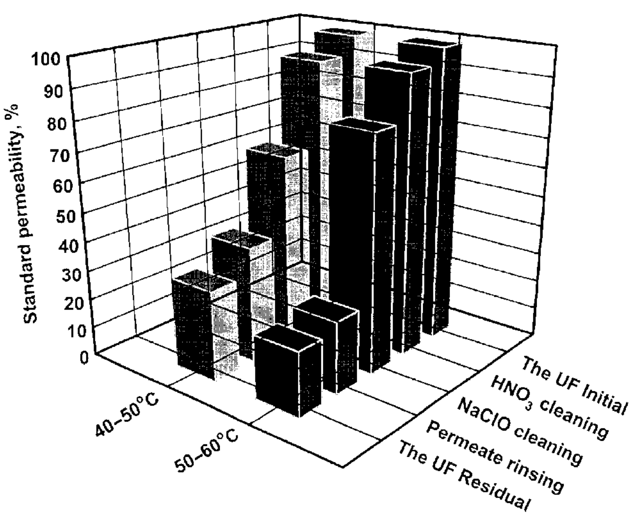


Figure 11. The effect of temperature.

the NaClO cleaning was much more effective at a relatively low temperature. After HNO_3 cleaning, however, the membrane standard permeability for both groups exceeded 94%. A conclusion could be drawn that the cleaning temperature within the range of 40–60°C had no discernible effect on the final recovery of membrane standard permeability. From the viewpoint of saving energy, the cleaning temperature of 40–50°C was recommended.

At HNO_3 concentration of 1% (w/w) and cleaning temperature of 40–50°C, the effect of NaClO concentration on standard permeability recovery is shown in Fig. 12. The residual standard permeability for groups 0.3% NaClO and 0.5% NaClO were 31% and 50%, respectively, with a difference of 19%. The permeate rinsing resulted in an observable increase of 9% and 5% for groups 0.3% NaClO and 0.5% NaClO, but the difference in standard permeability stayed at 15%. After the NaClO cleaning, however, the membrane standard permeability was restored to 66% and 61% for groups 0.3% NaClO and 0.5% NaClO, indicating that NaClO cleaning was rather conclusive regardless of the residual standard permeability. However, subsequent HNO_3 cleaning restored the standard permeability for groups 0.3% NaClO and 0.5% NaClO to 96% and 94%, respectively. It was obvious that NaClO concentrations within the range of 0.3–0.5% had no substantial effect on the recovery of UF membrane standard permeability. Targeting less

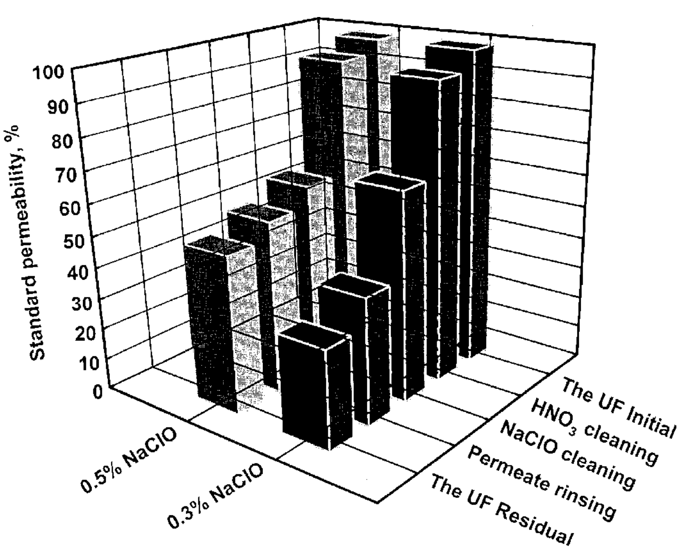


Figure 12. The effect of NaClO concentration.

chemical use, however, NaClO concentration of 0.3% was preferable in the multistep cleaning of UF membrane when applied to municipal wastewater treatment.

In brief, the recommended multistep cleaning included permeate rinsing, NaClO cleaning at 0.3% (w/w), and HNO_3 cleaning at 1% (w/w). The temperature applied was 40–50°C.

The Deposition Within the Cleaning Loop

The cleaning loop comprised the cleaning tank, membrane module, pumps, and PVC pipeline. Although the membrane module represented approximately 10% of the overall internal area of the cleaning loop, the deposition within the cleaning loop could be, to some extent, a reflection of those foulants that finally deposited on the UF membrane surface. In view of the chemical complexity of these foulants, COD were monitored as a surrogate parameter for organic matter while some metal ions such as Ca, Cu, Fe, and Mg, were measured as an indication of inorganic substances. As given in Table 2, a COD increase before/after NaClO cleaning was found to be from 3 mg/L to 46.8 mg/L but there was no discernible concentration change in metal ions, reaffirming that the major role of NaClO cleaning was to preliminarily breach the EPS matrix so that some large pieces of EPS matrix could be washed away from the UF membrane surface. However, COD concentrations before/after HNO3 cleaning showed no observable difference, indicating that HNO3 cleaning was not effective for organic matter removal. With regard to metal ions before/after HNO3 cleaning, Ca, Cu, and Fe exhibited increases of 115 times, 2.4 times, and 2 times, respectively, while the concentration of Mg remained nearly the same. It could be concluded that the HNO_3 cleaning was effective for the removal of metal ions such as Ca, Cu, and Fe, indirectly confirming the contribution of metal ions to the gel layer

Table 2. Chemical analysis on depositions within the cleaning loop.

Sample description		Concentration as $mg\,L^{-1}$				
		COD	Ca	Cu	Fe	Mg
NaClO cleaning	Before	34.3	40	0.20	0.0	7.6
	After	46.8	38	0.18	0.0	7.8
HNO_3 cleaning	Before	2.9	0.52	0.72	0.2	51
	After	2.6	60	1.73	0.4	52

forming on UF membrane surface. No obvious change in Mg concentration before/after HNO_3 cleaning might imply that the Mg played a minor role in the gel layer forming on UF membrane surface.

CONCLUSIONS

Fouling and cleaning of a ceramic tubular UF membrane for municipal wastewater treatment was investigated. A bistage fouling hypothesis, namely channel clogging and gel layer forming, was introduced and further verified during 165 days of experimental evidences. To get rid of the channel clogging, a preventive method was proposed and proven to be very effective. Without the occurrence of channel clogging, the filtration cycle achieved a five to eight fold increase, attaining to more than 8 weeks. The optimized multistep cleaning was recommended as permeate rinsing, 0.3% NaClO (w/w) cleaning, and 1% HNO_3 (w/w) cleaning at a temperature of 40–50°C. Such a cleaning could restore the UF membrane's standard permeability from 25–30% to more than 94% if taking the standard permeability of a new UF membrane as 100%. Further chemical analysis indicated that NaClO cleaning and HNO_3 cleaning were very effective in the removal of organic foulants and metal ions such as Ca, Cu, and Fe, respectively. The metal ion Mg might play a minor role in the formation of gel layer on the UF membrane surface.

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