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Pore fouling of microfiltration membranes by activated sludge

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Abstract

Pore fouling of four microfiltration membranes was investigated for the filtration of activated sludge using sonication as the means to remove the cake layer from membrane surface. Results show that pore fouling was affected by membrane's microstructure, pore openings and hydrophilicity. For the track-etched polycarbonate (PC) membrane, there was no measurable pore resistance (R_p) to filtration. For the three membranes of sponge-like microstructure, the respective R_p values were 0.3×10^{11}, 1.3×10^{11}, and 16.4×10^{11} m^{-1} for polyvinylidene fluoride (PVDF), mixed cellulose esters (MCE), and polyethersulfone (PES). The PES membrane had the highest R_p due to its large pore openings (18–20 μm), whereas MCE had higher R_p than PVDF because of its higher hydrophilicity. The R_p of PES membrane accounted for 86% of the total resistance for its filtration of activated sludge; the corresponding values were merely 2% for PVDF and 11% for MCE. It increased with the suspended solids level of the activated sludge to the power of 0.56. Results further demonstrated that R_p-dominant type of membranes, such as PES, was unsuitable for the membrane bioreactors.

Keywords: Activated sludge; Fouling; Membrane; Microfiltration; Pore

1. Introduction

The membrane bioreactor (MBR) process has become widely accepted for wastewater treatment [1]. In this process, pollutants are degraded by the activated sludge and the clean effluent is obtained by filtering through a membrane under pressure. Compared to the traditional activated sludge process, the MBR process not only allows wastewater to be treated at higher sludge concentrations and loading rates [2], but also reduces the sludge yield [3] and improves the effluent quality [1]. However, the application of MBR process has been hampered by the problem of membrane fouling [4], which results in the increase of operational cost and the shortening of membrane life [5].

Fouling is mostly caused by the deposition of colloidal matters inside the membrane porous structure and the formation of a cake layer on the membrane surface, resulting in the increase of filtration resistance. Filtration resistance R (m^{-1}) may be expressed, according to Darcy's law, by the following equation:

$$R = \frac{\Delta P}{\mu J} \tag{1}$$

where ΔP is the trans-membrane pressure gradient (N m^{-2}), μ the viscosity of the permeate (N s m^{-2}), and J the permeation flux (m s^{-1}). For the filtration of activated sludge, the total permeation resistance, R_t, may be expressed as the sum of three components:

$$R_t = R_m + R_p + R_c \tag{2}$$

where R_m is the intrinsic membrane resistance, R_p the pore fouling resistance, and R_c the cake layer resistance [6,7].

Of the four resistances in Eq. (2), R_m and R_t can be readily measurable individually; but R_p and R_c cannot. R_m may be determined from the J and ΔP data for the permeation of pure water alone through the membrane using Eq. (1). Similarly, R_t of activated sludge may be determined from the corresponding data of sludge filtration. The fouling resistance, which is the sum of R_p and R_c, may then be determined from the differ-

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ence between R_t and R_m. The fouling resistance is dependent on conditions of the sludge such as concentration and size distribution of colloids, characteristics of membrane such as pore size and chemical nature, as well as process conditions such as ΔP and cross-flow velocity, etc. [8].

In order to further differentiate R_p and R_c in fouling resistance, one has to conduct a separate filtration experiment after removing the cake layer on the membrane. In the absence of the cake layer, the fouling resistance represents the R_p alone. As a result, the R_c value may then be determined from Eq. (2). The cake layer may be removed from membrane surface by rinsing [9,10] or scrubbing [11]. However, microscopic examinations have shown that these tactics could not remove the cake layer thoroughly; consequently, the estimated R_p and R_c values were not reproducible. Without a reliable means to remove the cake layer thoroughly and reproducibly is likely the reason for the conflicting conclusions in literature: R_c was claimed by some as the predominant resistance in membrane fouling of activated sludge [9,10,12], while R_p by others [13].

On the other hand, sonication has recently been shown as an effective means to remove the cake layer from the membrane surface [14,15]. This study was thus conducted to evaluate the R_p values of four membranes for activated sludge filtration using sonication as the means for cake removal. Results are then used to correlate pore fouling to various characteristics of the membranes.

2. Material and methods

2.1. Activated sludge and membranes

Activated sludge sampled from a local municipal wastewater treatment plant (Stanley, Hong Kong) was used in this study. The sludge contained 4800 mg l^{-1} of suspended solids (SS) and 4100 mg l^{-1} of volatile suspended solids. Four flat-sheet microfiltration membranes with a nominal pore size of 0.2–0.22 μm were used in this study. These membranes from Millipore (Bedford, MA, USA) were made of different polymeric substances, including polycarbonate (PC; GTTP 14250), polyvinylidene fluoride (PVDF; GVWP 09050), mixed cellulose esters (MCE; GSWP 09000), and polyethersulfone (PES; GPWP 09050).

2.2. Filtration

All experiments were conducted at room temperature in a complete-mix filtration cell with a membrane area of 31.7 cm^2 and a working volume of 200 ml (Model 8200, Amicon) and a constant ΔP of 14 kN m^{-2} commonly used in MBR process [16,17]. Fig. 1 illustrates the schematic set up of the filtration system. Throughout the filtration, the mixed liquor volume was kept unchanged by automatically replacing the lost filtrate volume with de-ionized water from a pressurized vessel. The mixed liquor was stirred with a rod, which was suspended 3 mm above the membrane surface. The permeate flux was determined from the weight of the collected filtrate which was continuously measured by an electronic balance connecting to a data logger.

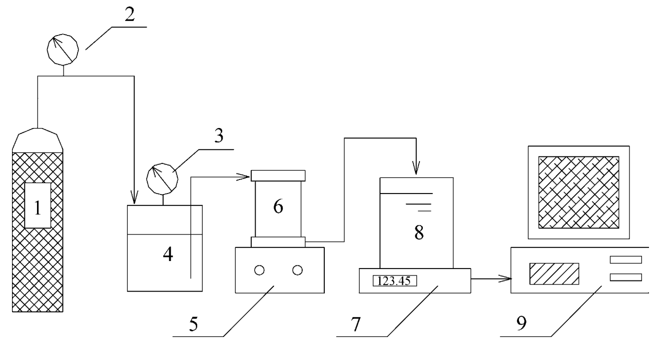

Fig. 1. Schematic diagram of a stirred cell unit for membrane filtration. (1) Nitrogen cylinder, (2) regulator valve, (3) pressure gauge, (4) feed vessel, (5) magnetic stirrer, (6) stirred cell, (7) balance, (8) permeate vessel, (9) personal computer.

There were two sets of filtration experiments in this study. The first set was conducted to determine various resistance values of four membranes, following the pattern as illustrated in Fig. 2 (not on scale). Each experiment was conducted for 8–19 cycles until steady state was reached. Each cycle was composed of three steps: filtration of de-ionized water through membrane (15 min), filtration of activated sludge (50 min), and removing the cake layer by sonication (Branson 8200, USA) at 355 W for 5 min. The sonication intensity had been proven unaffecting the permeability virgin membranes from a series of preliminary tests. In the first cycle, the resistance of the virgin membrane to water permeation increased initially, and became constant in about 10 min. The resistance at the end of the 15-min step represented the R_m of the membrane. The filtration resistance was then increased in the second step as water was replaced by activated sludge, and then leveled off after about 30 min. The resistance at the end of 50-min sludge filtration step represented the R_t. After removing the cake layer deposited on the membrane surface by 5 min of sonication, the cycle was repeated.

Under ideal situation, the membrane's resistance after sonication should be a constant representing R_p, if sonication removes only the cake layer. However, results in this study showed that such resistance initially increased with the num-

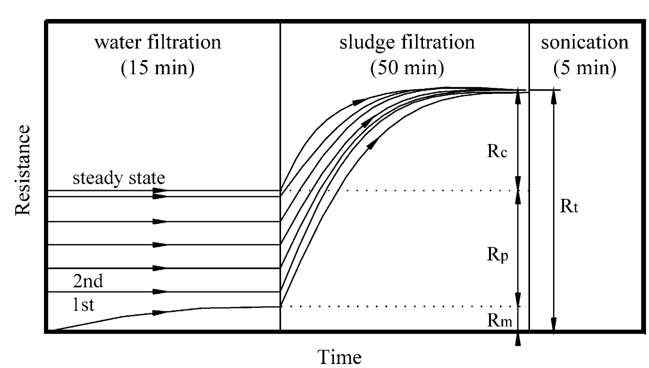

Fig. 2. Filtration cycles from which R_m, R_p, and R_c were estimated.

ber of cycles. The resistance finally leveled off after 6–14 cycles to a constant value, based on which the R_p value for each membrane was calculated. R_c was then lastly calculated from Eq. (2) using the determined values of R_m, R_t, and R_p.

The second set of experiments was conducted using only PES and PVDF membranes to simulate the MBR operation for 18 cycles of filtration. Each cycle consisted of four steps: 10 min of filtration of activated sludge, 5 min of sonication, decanting the mixed liquor, and replenishing with a new batch of activated sludge. Unlike the first set, the step of water permeation was skipped.

2.3. Scanning electron microscopic (SEM) images

Surfaces of fouled membrane in this study were analyzed using a SEM (Cambridge Stereoscan 360). The fouled membranes were fixed in a 2.5% glutaraldehyde solution overnight, followed by stepwise dehydration in a graded series of water/ethanol solutions and critical-point drying with carbon dioxide [18]. Lastly, the dried samples were sputter coated with gold/palladium prior to SEM observation.

3. Results and discussion

3.1. Membrane surface and morphology

Fig. 3a–d are the scanning electron micrographs of membrane surface at the end of the last cycle at the same degrees of magnification. These figures illustrate that all the membrane surfaces were in general free of foulants, confirming that sonication is an effective means for the removal of cake layer. In addition, Fig. 3a–d further illustrate the morphological differences of the four membranes. The PC membrane (Fig. 3a), which was made by the track-etch process, had a dense structure with small but uniform cylindrical pores of 0.20 μm [19,20]. In sharp contrast, the other three membranes had the interwoven sponge-like microstructure. Among these three membranes, PES (Fig. 3d) had the largest pore openings of up to 18–20 μm, whereas MCE (Fig. 3b) and PVDF (Fig. 3c) membranes have the comparable pore openings of 0.5–3 μm. These pore openings estimated based on the micrographs are substantially higher than the 0.22 μm reported by Millipore using undisclosed methods.

Table 1
Characteristics of membranes and resistances on filtration of activated sludge

Material	PC	PVDF	MCE	PES
Porosity (%)	10 [21]	70[a]	75[a]	Not available
Wettability (cos θ)	0.34 [22]	0.41 [23]	0.84 [24]	0.71 [23]
R_m ($\times 10^{11}$ m^{-1})	1.2	1.0	0.9	0.6
R_p ($\times 10^{11}$ m^{-1})	0.0	0.3	1.3	16.4
R_c ($\times 10^{11}$ m^{-1})	14.4	11.6	10.3	2.1
R_t ($\times 10^{11}$ m^{-1})	15.6	12.9	12.5	19.1

[a] Data from membrane manufacturer Millipore.

3.2. Determination of resistance values

Table 1 summarizes the relevant characteristics of the four membranes and the respective resistance values. The R_m values were relatively low, varying from 0.6×10^{11} m^{-1} for the PES membrane (the pore size of which being 18–20 μm)

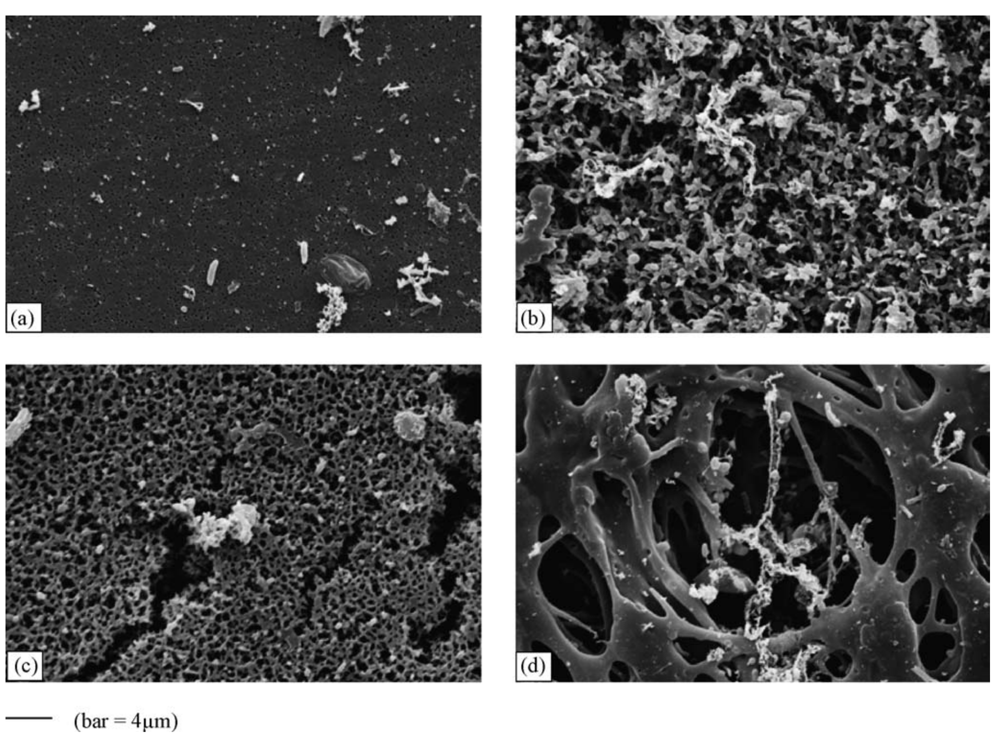

Fig. 3. SEM images of membrane surfaces after sonication: (a) PC, (b) PVDF, (c) MCE, and (d) PES membranes.

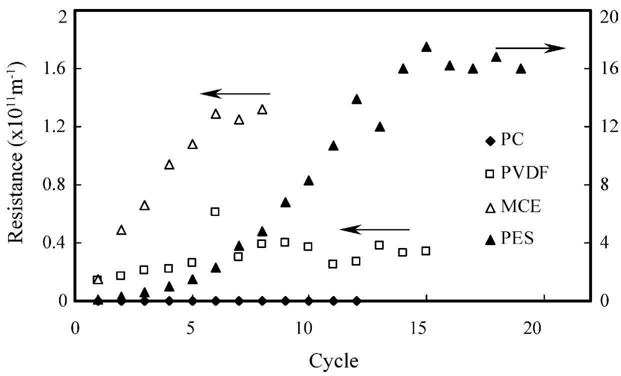

Fig. 4. Post-sonification pore resistance at various filtration cycles.

to $1.2 \times 10^{11}\,\mathrm{m}^{-1}$ for the PC membrane (0.2 μm). It shows that R_m decreased with the increase of pore size, as would be expected hydrodynamically. The R_t values varied from $12.5 \times 10^{11}\,\mathrm{m}^{-1}$ for MCE to $19.1 \times 10^{11}\,\mathrm{m}^{-1}$ for PES. Overall, R_m of all membranes accounted for only 3–8% of the R_t.

Fig. 4 illustrates the pattern of pore resistance after sonication at various filtration cycles. It shows a general trend that the pore resistance after sonication of a membrane increased initially, and leveled off to a steady-state value after a certain number of cycles. The R_p listed in Table 1 were the steady-state resistances shown in Fig. 4. Fig. 4 also illustrates that there was no measurable pore resistance, and thus little pore fouling, for PC. The R_p values varied substantially from nil for PC, to $0.3 \times 10^{11}\,\mathrm{m}^{-1}$ for PVDF, $1.3 \times 10^{11}\,\mathrm{m}^{-1}$ for MCE, and $16.4 \times 10^{11}\,\mathrm{m}^{-1}$ for PES. The R_c values in Table 1 were lastly determined from Eq. (2) using the determined values of R_m, R_t and R_p.

Results in Table 1 show that the fouling resistance of PES membrane was R_p-dominant, whereas that of the other three membranes was R_c-dominant.

3.3. Membrane characteristics affecting R_p

Results in this study show that pore fouling is affected by three characteristics of membrane: structure, pore openings and hydrophilicity. The PC membrane had little pore resistance, as shown in Fig. 4. This is likely due to the membrane's uniform, small cylindrical pore configuration, which makes it difficult for foulants to deposit inside the pores. In contrast, the other three membranes of sponge-like microstructure are more vulnerable to pore fouling due to their porous network. This concurs with a recent study, which reported similar observations [11].

Fig. 4 illustrates that pore resistance after sonication initially increased with the number of filtration cycles, suggesting that sonication in the initial cycles removed not just the cake layer but also some loosely bound foulants deposited inside the pores. After several cycles, all the loosely bound foulants were removed and the pores were packed with the tightly bound foulants that could no longer be removed by sonication. The post-sonication pore resistance became constant afterwards.

Of the three membranes of sponge-like microstructure, PES had a R_p value of $16.4 \times 10^{11}\,\mathrm{m}^{-1}$, substantially higher than the respective R_p values of 1.3×10^{11} and $0.3 \times 10^{11}\,\mathrm{m}^{-1}$ for MCE and PVDF. This is likely due to the effect of pore openings. Foulants had a much higher tendency to deposit inside the porous structure of the PES membrane with large pore openings (up to 18–20 μm) than inside the MCE and PVDF membranes (pore openings 0.5–3 μm). The more foulant deposits resulted in the higher the R_p.

For membranes of similar microstructure, porosity and pore opening, R_p is likely affected by the membrane hydrophilicity. Membranes of higher hydrophilicity tend to be more vulnerable to deposition of foulants of hydrophilic nature. Since activate sludge contains substantial amount of hydrophilic extracellular polysaccharides [25], the more hydrophilic MCE (with a wettability of 0.84) thus had a higher R_p than the less hydrophilic PVDF (wettability of 0.41), as observed.

3.4. Effect of activated sludge strength on R_p

Two additional sets of experiments were conducted to determine the effect of activated sludge strength on R_p. A low-strength ($2400\,\mathrm{mg\,l^{-1}}$ of SS) sludge was prepared by diluting the activated sludge with an equal amount of water, whereas a high strength sludge ($9600\,\mathrm{mg\,l^{-1}}$ of SS) was prepared by concentrating the sludge by settling. The R_p values of PES for the filtration of these two sludges were determined following the same aforementioned procedures. Results in Fig. 5 illustrates that the R_p value increased with the mixed liquor suspended solids (MLSS) level of the sludge which may be expressed as:

$$R_p = 6.35[\mathrm{MLSS}]^{0.56} \quad (3)$$

The positive correlation between R_p and MLSS found in this study concurs with the findings of several previous studies [2,4,26,27]. On the other hand, some other studies showed that R_p was independent of MLSS [28–31]. The discrepancy is likely due to the difference in the membrane characteristics, such as pore opening and hydrophilicity, which were not specified in most previous studies.

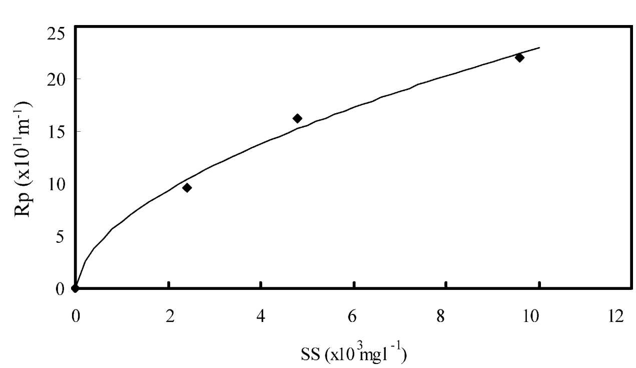

Fig. 5. Increase of R_p for PES membrane with the SS level of activated sludge.

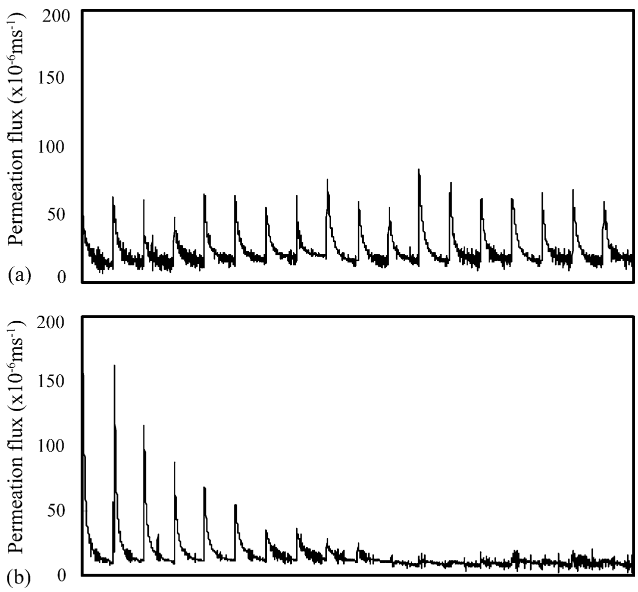

Fig. 6. Patterns of permeation fluxes for (a) PVDF and (b) PES membranes.

3.5. Filtration of R_p- and R_c-dominant membranes

Results in Table 1 show that membrane fouling may be classified into two types: R_p-dominant and R_c-dominant. The PES membrane, which has the sponge-like microstructure with large pore openings, belongs to the former, whereas the remaining three, the latter.

A MBR system is normally operated alternately between sludge filtration and membrane cleaning [32–34]. Filtration experiments were conducted in parallel to compare the filtration performance of PVDF, representing the R_c-dominant membranes, and PES, representing the R_p-dominant membranes. Each filtration test was conducted for 18 cycles, each comprised the following: activated sludge filtration (10 min), sonication (5 min), decanting mixed liquor, and replenishing with fresh activated sludge. Permeation flux was continuously monitored throughout each experiment.

Fig. 6a and b illustrate the respective permeation flux patterns for the filtration of activated sludge by the PDVF and PES membranes. In each cycle, water permeation flux gradually decreased during sludge filtration because of the gradually buildup of cake resistance. However, after cake layer removal by sonication, permeation flux of the R_c-dominant PDVF membrane was almost fully recovered in each cycle, whereas that of the R_p-dominant PES diminished rapidly. This is because sonication was effective to remove the cake layer on the PDVF membrane surface, but ineffective to remove the foulants deposited inside the PES porous sponge-like microstructure. The average permeation flux for the PDVF membrane maintained at $24 \pm 2 \times 10^{-6}\,\mathrm{m\,s^{-1}}$ throughout the 18 cycles, whereas the flux of PES decreased steadily from the initial 31×10^{-6} to $10 \times 10^{-6}\,\mathrm{m\,s^{-1}}$ by cycle 12, and leveling off at the level afterwards.

Results in Fig. 6a and b have two implications. Firstly, MBR systems should not use R_p-dominant membranes, such as PES, because conventional membrane cleaning techniques, such as sonication [15], backwashing [35] cannot effectively remove foulants deposited inside the pores. Secondly, permeation flux of R_p-dominant membranes steadily decreased in the initial cycles due to the gradually buildup of pore fouling; thus estimation of permeation flux of these membranes cannot be relying on the data of initial cycles.

4. Conclusions

Based on results of this study, the following conclusions may be drawn:

1. Sonication is an effective means for the removal of cake layer from the membrane surface.
2. The track-etched PC membrane did not have measurable pore resistance. For the three membranes with sponge-like microstructure, the R_p were $0.3 \times 10^{11}\,\mathrm{m^{-1}}$ for PVDF, $1.3 \times 10^{11}\,\mathrm{m}$ for MCE, and $16.4 \times 10^{11}\,\mathrm{m^{-1}}$ for PES membranes. R_p accounted for 86% of the total permeation resistance for the filtration of activated sludge by PES, but only 2% for PVDF and 11% for MCE.
3. Membrane's microstructure, pore openings and hydrophilicity affect the pore resistance. For the three sponge-like membranes, PES had the highest R_p due to its large pore openings (18–20 μm). MCE had higher R_p than PVDF because of its higher hydrophilicity.
4. The R_p of PES membrane increased with the MLSS level of the activated sludge to the power of 0.56.
5. MBR system should use the R_c-dominant type of membranes, such as PC, PVDF and MCE, but avoid the R_p-dominant type of membranes, such as PES.

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