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Membrane Filtration Characteristics in Membrane-Coupled Activated Sludge System: The Effect of Floc Structure on Membrane Fouling

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ABSTRACT

The membrane-coupled activated sludge (MCAS) process has many advantages over the conventional activated sludge system, but the inherent membrane fouling problem still remains to be solved. However, it is not yet advanced enough to understand the reliable fouling mechanism. The strength of the MCAS process lies in the almost complete removal of suspended solids from the activated sludge broth. But it has made us overlook the effect of sludge morphology and physiology on membrane flux which is one of the key factors in deciding the economical feasibility of the MCAS system. The aim of this study was to investigate membrane filtration characteristics in the MCAS process, especially to correlate floc structures of the activated sludge with membrane fouling. A series of ultrafiltrations with both hydrophilic and hydrophobic membranes using the stirred batch cell system was performed to assess flux behavior according to the floc structures of the activated sludges (normal, pinpoint, and bulking activated sludge). The order of fouling tendency was found to be normal sludge $<$ pinpoint sludge $<$ bulking sludge. Also, all the membranes behaved in the same way. The cake layer resistance (R_c) made up most of the total resistance (R_t), but the fouling resistance (R_f) was negligible in any floc structure. The key factors controlling the R_c were the shape and size of the activated sludge flocs and the porosity of the cake layer accumulated on the membrane surface. The hydrophobic

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membrane showed a greater fouling tendency than the hydrophilic membrane regardless of the microbial floc structures. The difference in fouling tendency between the two membranes was attributed to the hydrophobic interactions between the membrane and floc surfaces.

Key Words. Activated sludge; Membrane; Fouling; Bulking; Pinpoint floc; Hydrophobic interaction; Resistance in series model; Ultrafiltration

INTRODUCTION

The development of membrane technology has expanded its field of application to wastewater treatment streams. One of the possible modifications of conventional biological wastewater treatment processes is the replacement of the secondary sedimentation tank by membrane units. An activated sludge process coupled with membrane filtration as the solid–liquid separation step has many advantages over the conventional activated sludge process. These include minimum sludge wastage and reduced plant size by maintaining a high biomass concentration in the reactor, and eliminating a secondary sedimentation tank (1–3). This system is also capable of handling wide fluctuations in influent quality. The effluent can also be directly reused for nonpotable purposes since treatment efficiency is relatively high (4). Furthermore, an increased rate of nitrification can be achieved since a large amount of slow-growing nitrifying microorganisms can be retained in an aeration tank (5–7). Above all, the strong point of this process is that it is not directly affected by a sludge bulking problem.

Despite these advantages, the high energy consumption required to operate the filtration unit at a high suspended solids concentration has restricted the practical application of this process (8). One of the solutions for this problem may be to use this direct membrane separation within the reactor without recirculation (9, 10) because this will reduce energy requirements.

In recent reviews covering membrane applications to bioreactors, it was shown that membrane fouling was the most critical problem in this system. This fouling problem led to permeate flux decline, resulting in the necessity of frequent membrane replacement and cleaning procedures. Most studies concerning membrane fouling have been experimental investigations showing how the parameters, such as COD, MLSS, dissolved organic carbon (DOC), viscosity, and temperature, influence membrane fouling (11–14). But little attention has been paid to how the microorganisms themselves affect the performance of membrane filtration, probably because it has been acknowledged that all the microbial flocs can be completely rejected by the membrane in MCAB systems, and thus the anxiety about the carryover of unsettled flocs into the effluent would no longer be necessary. Nevertheless, although mem-

brane fouling can be largely affected by the characteristics of the microorganisms as well as the operating conditions of the membrane system, there is little information available on the effects on membrane fouling due to the structure of the activated sludge.

The purpose of this study was to evaluate how the floc structures (bulking, pinpoint, and normal floc) affect membrane filtration characteristics and to determine how sludge morphology would cause the least membrane fouling in MCAS processes.

MATERIALS AND METHODS

Cultivation of Activated Sludge

A synthetic wastewater was used throughout this study to ensure a consistent quality of influent to the membrane bioreactor. The synthetic wastewater was prepared from a sterile concentrated solution with the composition shown in Table 1. The concentrated feed solution was stocked in a refrigerator and diluted with tap water to the desired concentration before it was fed to the activated sludge reactor. Using a fill-and-draw technique, sludge was allowed to settle for 30 minutes and the supernatant was withdrawn and discarded. Then the reactor was refilled with fresh feed solution and aeration was restarted. These fill-and-draw processes were repeated every 12 or 24 hours. Aeration and mixing were provided through a porous stone diffuser delivering compressed air. An activated sludge had been acclimated to this synthetic wastewater for 3 months to achieve a steady-state prior to the membrane filtration experiments.

To prepare the activated sludges with different floc structures (bulking, pinpoint, and normal floc), bioreactors were operated in parallel at different hydraulic retention times (HRT) and food to microorganism (F/M) ratios as

TABLE 1
Composition of Synthetic Wastewater

Composition	Concentration (mg/L)
Glucose	16,000
Peptone	12,000
Yeast extract	1,600
$(NH_4)_2SO_4$	12,800
KH_2PO_4	2,560
$MgSO_4 \cdot 7H_2O$	3,200
$MnSO_4 \cdot 4–5H_2O$	288
$FeCl_3 \cdot 6H_2O$	16
$CaCl_2 \cdot 2H_2O$	320
$NaHCO_3$	2,000

TABLE 2
Operational and Sludge Characteristics of Activated Sludge Processes

	HRT (h)	F/M (g COD/g MLSS·d)	pH	SVI (mL/g)	SS_{30m}[a] (mg/L)	Turbidity[b] (NTU)	Floc size[c] (μm)	COD rejection[d] (%)
Normal floc	24	0.19	7.5	70	44	14	131	97
Pinpoint floc	24	0.26	7.0	30	130	47	81	93
Bulking floc	12	1.08	7.7	249	21	11	138	93

[a] Supernatant SS after 30 minutes settling.
[b] Supernatant turbidity after 30 minutes settling.
[c] Mean floc size based on volume frequency.
[d] COD rejection with YM30 membrane is defined as follows:

$$\text{COD rejection (\%)} = [(\text{influent COD} - \text{permeate COD})/\text{influent COD}] \times 100.$$

shown in Table 2. In addition, the aeration intensity was increased from 4000 to 6500 mL/min only when the pinpoint floc was required.

Analytical Methods

Once a day, just before the sludge wastage was removed, samples were withdrawn from the reactors and were analyzed for MLSS, pH, soluble COD, and SVI. Analysis of the above items for the influent, effluent, and mixed liquor samples was conducted using the procedures described in *Standard Methods* (15). Floc size analysis of the activated sludge was conducted using a particle size analyzer (MasterSizer/E, Malvern, UK).

Membranes and Filtration Apparatus

Asymmetric membranes made from various polymers, YM30, XM50, and PM30 membranes (Amicon Inc., USA), were used in this work. Some characteristics of each membrane are provided in Table 3. The relative hydropho-

TABLE 3
Characteristics of Membranes Used in Stirred Cell Experiments

	Membranes		
Characteristics	YM30	XM50	PM30
Molecular weight cutoff (Dalton)	30,000	50,000	30,000
Material of skin layer	Regenerated cellulose	Acrylic copolymer	Polysulfone
Contact angle with water (θ)	Totally wettable	59	66

bicity of each membrane was characterized by measuring the contact angle between water droplets and membrane surface with a goniometer (NRL, Rame Hart, USA). Based on the contact angle measurements, the order of the relative hydrophobicity was found to be PM30 > XM50 > YM30 membrane in decreasing order (Table 3).

The fresh membrane was first rinsed by letting it float skin-side down in ultrapure water for 90 minutes. The rinsing water was changed three times during this cleaning process.

Then the cleaned membrane was placed in a stirred batch cell (8200, Amicon Inc., USA). The experimental setup of the stirred cell is illustrated in Fig. 1. The permeate flux was determined by weighing permeates on an electronic top-loading balance connected to a personal computer equipped with an autoreading program. Transmembrane pressure (ΔP_T) was regulated at 1.4 bar using nitrogen gas, and the stirring speed was set at about 180 rpm for all tests in this work. The MLSS concentration of each activated sludge suspension was adjusted to 3500 ± 100 mg/L prior to membrane filtration in order to exclude the concentration effect on the permeate flux.

Measurements of Various Fluxes

In this article, initial water flux (J_{iw}), permeate flux (J), and final water flux (J_{fw}) are used to characterize the performance of membrane filtration.

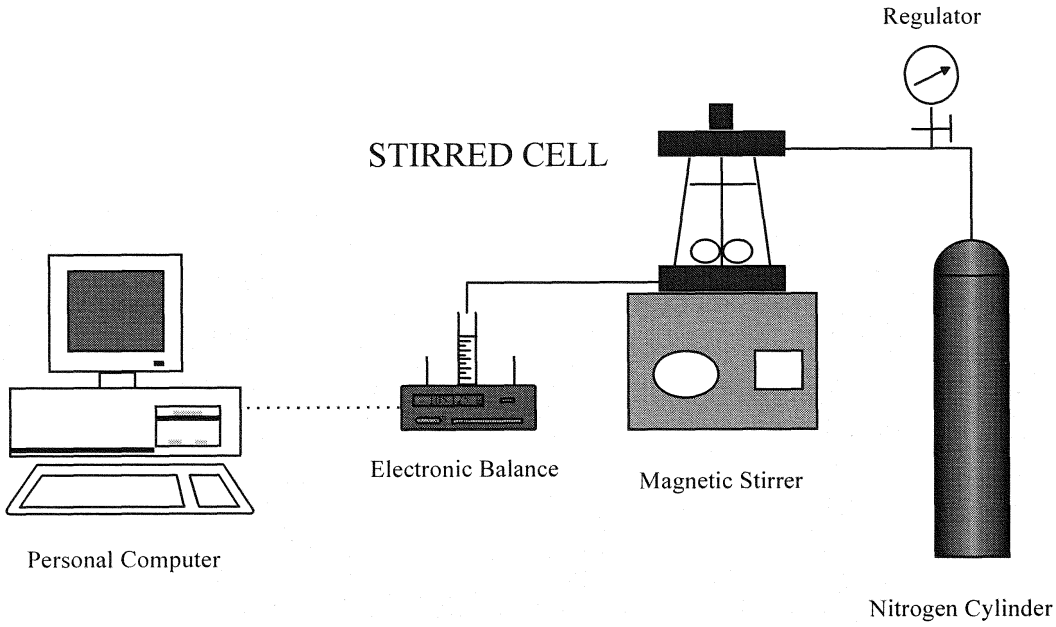

FIG. 1 Schematic diagram of stirred cell system.

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J_{iw} is simply a water flux through the cleaned membrane. Before filtrations of the activated sludge suspension, J_{iw} of each membrane was determined by filtering ultrapure water until a steady flux was reached. The stirred cell was then emptied and filled with the activated sludge suspension. Ultrafiltration was performed until the volume concentration factor equaled 5. The permeate flux at this moment is denoted J_5. Then the stirred cell was emptied again and refilled with ultrapure water. Surface rinsing of the tested membrane with ultrapure water was continued for 10 minutes without applying any pressure, and then the rinsing water was discarded. J_{fw} was determined by the ultrapure water right after the surface rinsing.

RESULTS AND DISCUSSION

Effect of Floc Structure on Membrane Fouling

In general, the floc structure of activated sludge is classified into three types according to the balance of floc-forming and filamentous bacteria, i.e., bulking, pinpoint, and ideal normal floc (16). Activated sludges with the above three kinds of floc structures were prepared by controlling HRT, F/M ratio, etc. The operational and sludge characteristic of each sludge at steady-state are summarized in Table 2. The sludge volume index (SVI) was used as one of the measures to monitor the floc structure in an aeration bioreactor. The SVI values of normal sludge, pinpoint floc sludge, and bulking sludge were 70, 30, and 249 mL/g, respectively. The suspended solid (SS) and turbidity of the supernatant after settling the mixed liquor for 30 minutes could be indirectly related to the floc structures, because it is known that the pinpoint floc sludge has a very turbid supernatant while bulking sludge has a relatively clean supernatant (17). In this experiment the supernatant of pinpoint floc sludge was very turbid (SS = 130 mg/L, turbidity = 47 NTU), whereas those of normal and bulking sludge were much less turbid (normal sludge: SS = 44 mg/L, turbidity = 14 NTU; bulking sludge: SS = 21 mg/L, turbidity = 11 NTU).

Microscopic observations of the mixed liquors revealed the floc structure of each activated sludge (Fig. 2). A proliferation of filamentous microorganisms was observed in the bulking sludge (Fig. 2c), while the filamentous bacteria were not observed in the pinpoint floc sludge (Fig. 2b). On the other hand, the normal activated sludge showed a good balance of the filamentous and the floc-forming bacteria (Fig. 2a). The pinpoint floc had the smallest floc size of about 80 μm, while the normal and bulking sludges had bigger but similar floc sizes of 131 and 138 μm, respectively.

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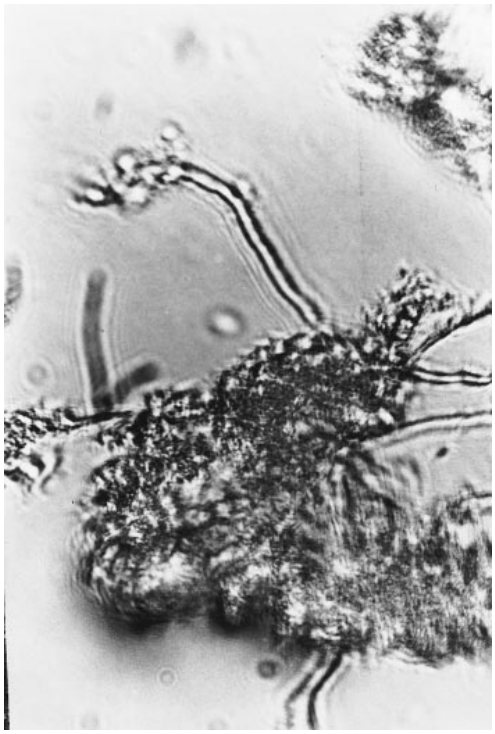
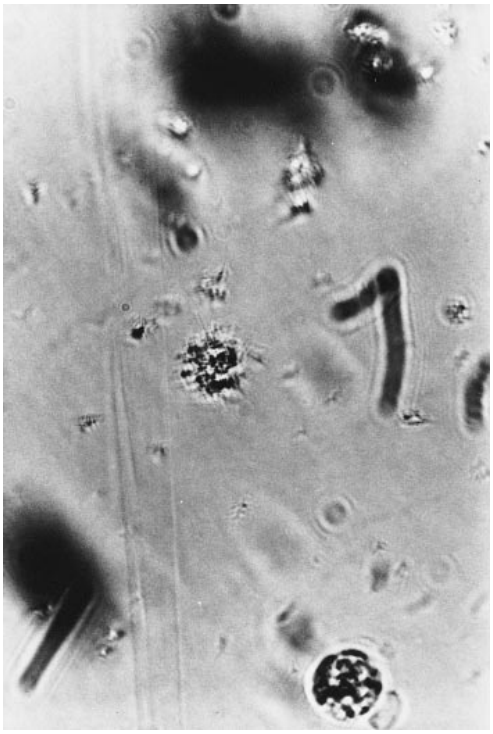


(a)



(b)

(c)

FIG. 2 Microscopic observations of the various activated sludge floc structures: (a) normal floc, (b) pinpoint floc, (c) bulking floc.

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A series of ultrafiltrations with membranes of different hydrophobicity was conducted to assess the flux behaviors according to the floc structures of activated sludges. A ratio of permeate flux to initial water flux, J/J_{iw}, was plotted as a function of concentration factor (Figs. 3a, 3b, 3c) because the initial water flux value (J_{iw}) of each membrane was different from each other. During the ultrafiltration of the bulking sludge with the YM30 membrane, the permeate flux decreased to about 12% of J_{iw} at a concentration factor (CF) of 3 where the flux reached steady-state, while that of the normal sludge reduced only to about 55% of J_{iw} at the same CF. As for the pinpoint floc sludge, it declined to about 40% at the same CF. The other two membranes (XM50 and PM30) also showed the same trends of flux decline, but the extent of flux decline for XM50 and PM30 was significantly different from that of YM30 membrane. This effect will be further discussed in a later section.

Permeate water quality was very good regardless of the membrane fouling tendency. For instance, the COD rejection efficiency for the YM30 membrane ranged from 93 to 97%, as shown in Table 2.

In summary, the order of flux decline was found to be normal sludge < pinpoint floc sludge < bulking sludge, regardless of membrane materials.

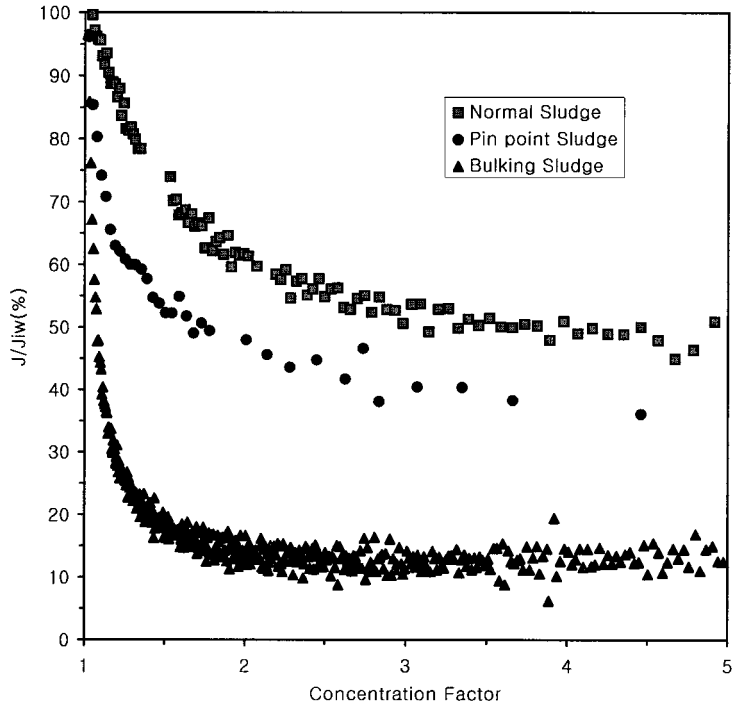

FIG. 3a Flux declines according to floc structures during ultrafiltration of activated sludge broth with YM30 membrane.

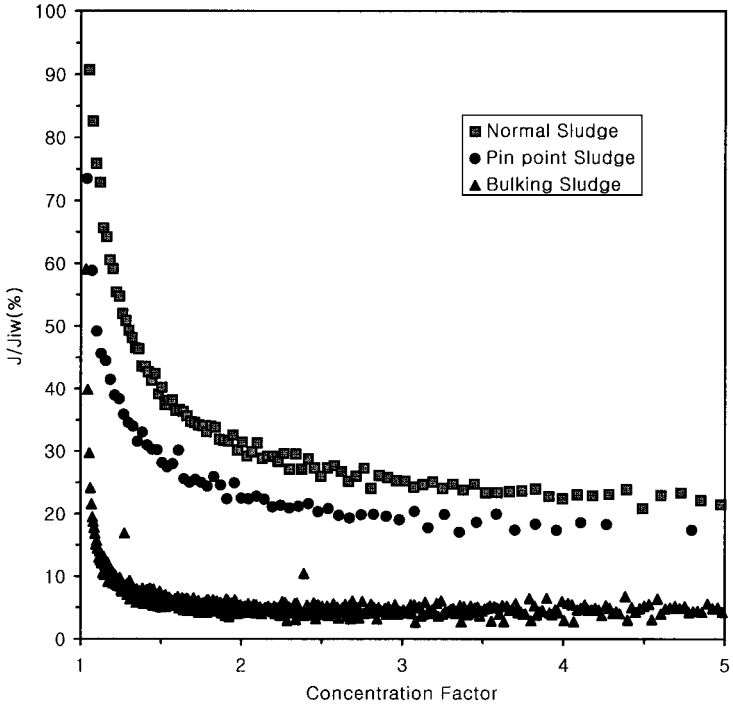

FIG. 3b Flux declines according to floc structures during ultrafiltration of activated sludge broth with XM50 membrane.

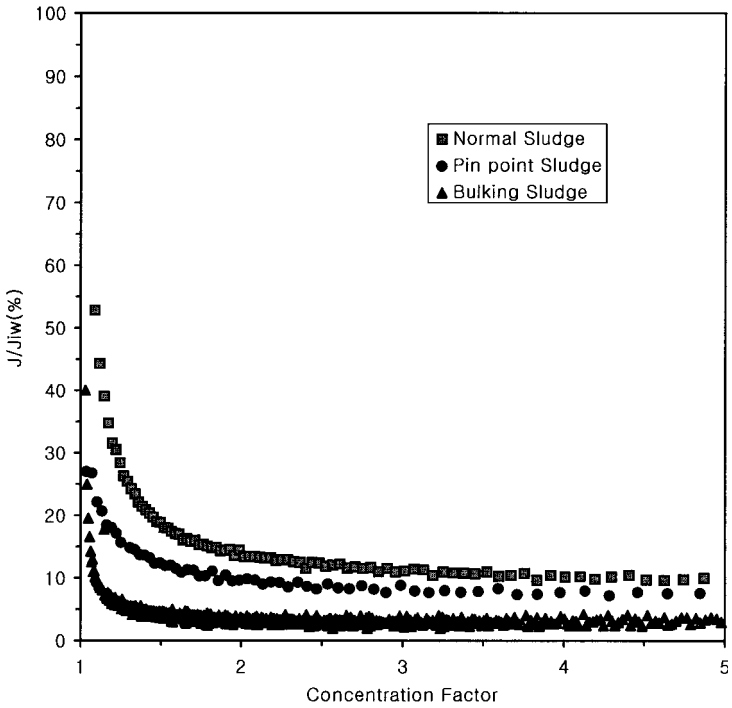

FIG. 3c Flux declines according to floc structures during ultrafiltration of activated sludge broth with PM30 membrane.

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Estimation of Membrane Fouling

A resistance-in-series model was applied to evaluate the characteristics of membrane fouling. According to this model, the permeate flux (J) took the following form:

$$J = \Delta P_T/(\eta R_t) \quad (1)$$

$$R_t = R_m + R_c + R_f \quad (2)$$

where ΔP_T is the transmembrane pressure, η is the viscosity of the permeate, R_t is the total resistance, R_m is the intrinsic membrane resistance, R_c is the cake resistance formed by the cake layer deposited over the membrane surface, and the fouling resistance, R_f, is the resistance caused by pore plugging and/or solute adsorption onto the membrane surface and pores. Each resistance value (R_m, R_c, and R_f) can be obtained from Eqs. (3)–(5) and the experimentally determined J_{iw}, J_{fw}, and J_5 values.

$$R_m = \Delta P_T/(\eta J_{iw}) \quad (3)$$

$$R_f = \Delta P_T/(\eta J_{fw}) - R_m \quad (4)$$

$$R_c = \Delta P_T/(\eta J_5) - (R_m + R_f) \quad (5)$$

In order to evaluate the reproducibility of each R value obtained using the above method, ultrafiltrations of the same activated sludge suspension were repeated three times (Runs 1–3) with YM30 membranes to obtain the statistical data shown in Table 4. The relative standard deviations for R_c and R_f were 12 and 6%, respectively. Although each membrane had a different R_m value, they showed very similar results of R_c and R_f values governing the overall membrane fouling. From this statistical test, it was confirmed that the resistance values of R_c and R_f obtained by the above method are reliable.

Various resistances at different floc structures and membrane materials are

TABLE 4
Statistical Evaluation of the Reproducibility of Each Resistance Value

	R_m (10^{11} m^{-1})	R_c (10^{11} m^{-1})	R_f (10^{11} m^{-1})	R_t (10^{11} m^{-1})
Run 1	26.6	19.4	0.31	46.3
Run 2	26.9	21.0	0.32	48.2
Run 3	17.6	24.5	0.34	42.5
Mean	—	21.6	0.32	45.7
Standard deviation (s)	—	±2.6	±0.02	±2.9
Relative standard deviation (%)	—	12	6	6

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TABLE 5
A Series of Resistances with Changes of Floc Structure and Membrane Material

	R_m (10^{11} m^{-1})	R_c (10^{11} m^{-1})	R_f (10^{11} m^{-1})	R_t (10^{11} m^{-1})	R_c/R_t (%)
YM30:					
Normal sludge	23	24	0.3	47.3	50.7
Pinpoint sludge	20	35	0.3	55.3	63.3
Bulking sludge	23	250	0.3	273.3	91.5
XM50:					
Normal sludge	10	67	6	83	80.7
Pinpoint sludge	9	79	7	95	83.2
Bulking sludge	9	499	6	514	97.1
PM30					
Normal sludge	5	100	6	111	90.1
Pinpoint sludge	4	198	9	211	93.8
Bulking sludge	6	562	10	578	97.2

given in Table 5. The cake resistance (R_c) appeared to be a controlling factor of the total resistance (R_t) as implied by R_c/R_t. R_c/R_t was greater than 51%, but the fouling resistances (R_f) were negligible regardless of the floc structures and membrane materials. The order of fouling was found to be bulking sludge $>$ pinpoint floc sludge $>$ normal sludge, and all the membranes behaved in the same way.

The large values of R_c/R_t clearly indicate that cake layer formation should be the major factor for the overall flux decline. In other words, the membrane fouling tendency seems to be closely related to the characteristics of the activated sludge flocs, e.g., the size, shape, specific surface of the flocs, etc.

To provide insight into the differences in cake layer formation, the Carman–Kozeny equation was applied to the resistance-in-series model.

$$R_c = \frac{LkS^2(1-\varepsilon)^2}{\varepsilon^3} \tag{6}$$

$$= Lk\left(\frac{6}{\psi d}\right)^2 \frac{(1-\varepsilon)^2}{\varepsilon^3} \tag{7}$$

where L is the cake layer thickness, S is the specific surface of the cake media ($S = 6/\psi d$), k is the Kozeny constant, ε is the porosity of the cake layer, ψ is the sphericity, and d is the cake media diameter.

From Eqs. (6) and (7), the cake layer resistance (R_c) is seen to be a reciprocal square function of the media diameter (d). Considering the mean floc size of normal activated sludge (131 μm) and pinpoint floc sludge (81 μm), as

shown in Table 2, the floc size difference would be one of the factors for the higher R_c of the pinpoint floc sludge than of the normal sludge.

On the other hand, the R_c value of the bulking sludge was about 5 to 10 times greater than that of the normal activated sludge in respect to the membrane materials although the floc sizes for both activated sludges were very similar (normal sludge = 131 μm, bulking sludge = 138 μm). It was reported that the bulking sludge had a higher surface area per unit volume of microorganism than normal sludge (18). Therefore, it is expected that the bulking sludge has a larger specific surface of the cake, S ($= 6/\psi d$), than the normal sludge because it must have a smaller sphericity, ψ, which is defined as the ratio of the surface area of the equivalent-volume sphere to the actual surface area.

Nakanishi et al. (19) studied the effect of shape of microorganisms on membrane filtration characteristics. They found that the cake layer porosity of elliptical-shaped cells such as *M. glumitacus* and baker's yeast was around 0.3, whereas those of rod-shaped cells such as *E. coli*, *R. spheroides*, and *B. circulans* ranged from 0.1 to 0.23.

In this context, the porosity of the bulking sludge should be smaller than of the normal sludge since the normal and the bulking sludge flocs, as shown in Fig. 2, seem to resemble elliptical- and rod-shaped cells, respectively. According to Eq. (6), a small change in porosity leads to a large variation of cake resistance. Hence, the substantial flux decline for the bulking sludge compared with normal sludge could be attributed to both the smaller porosity originating from the proliferation of filamentous bacteria and the larger specific surface with an assumption of equal floc size.

The MCAS system does not suffer from a solid–liquid separation problem as indicated by many other researchers. This is one of the most important advantages of this process over conventional activated sludge. However, if filamentous bulking occurs at the aeration tank or pinpoint flocs proliferate, a severe flux decline can be anticipated during the operation of this system. Therefore, maintaining the normal activated sludge floc is very important for minimizing flux decline in the MCAS system, as well as for good settling in the conventional activated sludge system.

Effect of Membrane Hydrophobicity on Fouling

Figure 4 shows the flux declines during ultrafiltrations of normal activated sludge with the three different membranes. The extent of flux decline is significantly different depending on the membrane materials. The PM30 membrane, which has strong hydrophobicity, showed the highest flux declines among the three membranes used. The hydrophilic YM30 membrane showed comparatively little flux decline whereas the less hydrophobic XM50 mem-

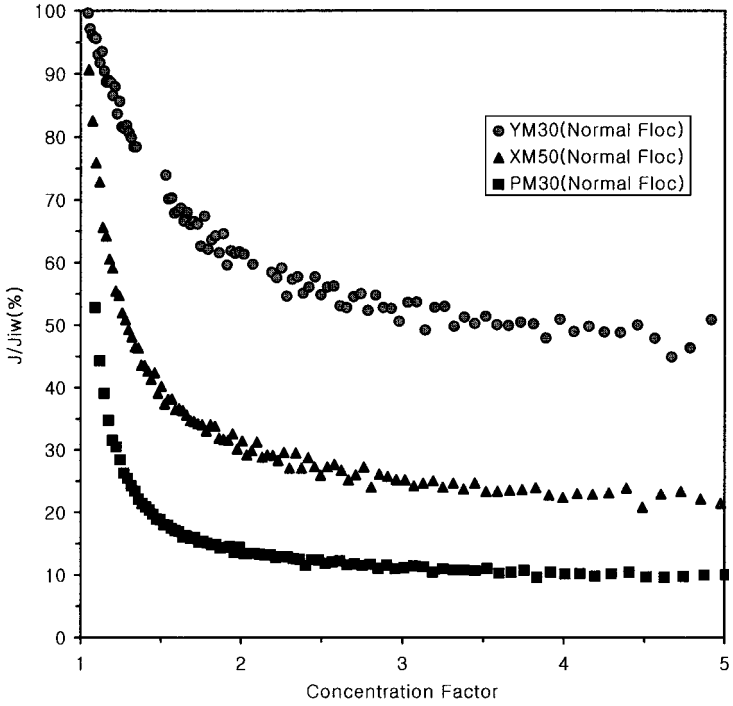

FIG. 4 Flux vs concentration factor according to membrane material.

brane showed an intermediate flux decline. Accordingly, the degree of flux decline can be said to be intimately related to membrane hydrophobicity. The same flux pattern was also observed during ultrafiltration of a foaming activated sludge (20).

The above results could have been caused by hydrophobic interaction between the membrane surface and the activated sludge floc surface. The importance of hydrophobic interaction has been recognized by many other researchers. For example, Fletcher and Loeb (21) reported that many bacteria are known to adhere preferentially to the hydrophobic substrata rather than hydrophilic ones. Ridgway et al. (22) indicated that the hydrophobic interaction between bacterial cell surface components and the reverse osmosis membrane surface plays an important role in bacterial adhesion and biofilm formation.

However, hydrophobic interaction might not be the only reason for this phenomenon because the fouling tendency for the different membranes depends not only on the membrane hydrophobicity but also on the membrane surface properties. The membranes used in this study do not have the same surface properties. For example, there is a little difference in molecular weight cutoff (MWCO) between the XM50 membrane (50,000 daltons) and the YM30 and PM30 membranes (30,000 daltons). Furthermore, the surface

porosity of the YM30 membrane (~50%) is quite different from that (>10%) of the PM30 membrane (23, 24).

In order to confirm whether the above result in Fig. 4 really comes from hydrophobic interaction, membrane filtration of the normal sludge was conducted with two membranes having the same surface pore size and porosity but different hydrophobicities. The GVWP and GVHP (MILLIORE) membranes were selected for this purpose. Both membranes had the same pore size (0.22 μm), the same surface porosity (75%), and were made from the same material (polyvinylidene difluoride, PVDF). On the other hand, the surface hydrophobicity of the membranes was different. Contact angle measurements with water droplets showed that the contact angle of GVWP was 61° (hydrophilic) and that of GVHP was 118° (hydrophobic).

Figure 5 compares flux declines during the filtration of normal activated sludge with GVWP and GVHP membranes. It clearly shows that the flux decline with the hydrophobic membrane is much greater than that with the hydrophilic membrane. As a result, it is concluded that hydrophobic interaction is one of the key factors controlling flux decline in membrane filtration of activated sludge.

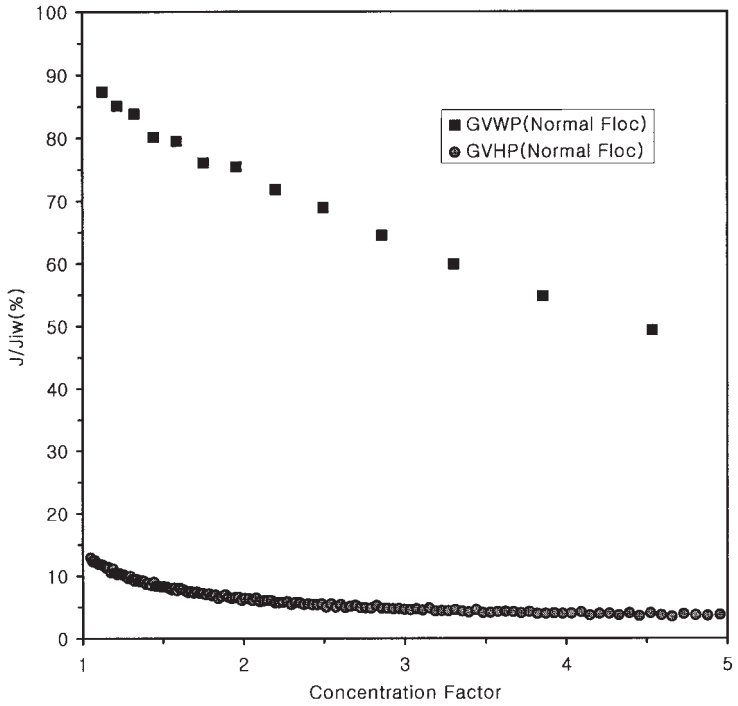

FIG. 5 Comparison of flux pattern between hydrophobic and hydrophilic membrane in microfiltration of activated sludge.

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Microbial cell surfaces are known to have hydrophobic molecules such as proteins or lipids (25). Therefore, hydrophobic interaction may result from the attractive forces existing between the complex surface components of activated sludge floc and the membrane surface. As a consequence, the hydrophobic membrane exhibited more fouling tendency than the hydrophilic membrane in the MCAS system.

CONCLUSIONS

In this study the membrane filtration characteristics in the MCAS system were investigated under various floc structures of activated sludge, and the following conclusions were drawn:

1. The tendency for membrane fouling was analyzed using resistance in series (R_f, R_c, R_t) model, and the experimental method used to obtain each resistance value proved to be reliable from the statistical point of view.
2. The cake layer resistance (R_c) appeared to be the controlling factor of the total resistance (R_t) whereas the fouling resistance (R_f) was negligible in any floc structure and membrane material.
3. The order of fouling tendency was found to be bulking sludge, pinpoint floc sludge, and normal sludge, regardless of the membrane materials. The key factors controlling the overall membrane fouling tendency were the shape, the size of the activated sludge flocs, and the porosity of the cake layer deposited on the membrane surface.
4. Although the MCAS system does not suffer from the solid–liquid separation problem, maintaining a normal activated sludge floc is very important for stable operation of this system.
5. The hydrophobic membrane always exhibited a greater fouling tendency during the membrane filtration of activated sludge than the hydrophilic membrane due to hydrophobic interaction.

NOMENCLATURE

d	cake media diameter (m)
J	permeate flux ($L \cdot m^{-2} \cdot h^{-1}$)
J_{iw}	initial water flux ($L \cdot m^{-2} \cdot h^{-1}$)
J_{fw}	final water flux ($l \cdot m^{-2} \cdot h^{-1}$)
J_5	flux at the volume concentration factor of 5 ($L \cdot m^{-2} \cdot h^{-1}$)
k	Kozeny constant
L	cake layer thickness (m)
R_c	cake layer resistance (m^{-1})
R_f	fouling resistance (m^{-1})
R_m	membrane resistance (m^{-1})
R_t	total resistance (m^{-1})

S	specific surface (m^{-1})
η	dynamic viscosity of permeate (Pa·s)
ψ	sphericity
ε	porosity of cake layer
ΔP_T	transmembrane pressure (Pa)

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