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The effect of microbubbles on membrane fouling caused by different foulants

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ABSTRACT

Previously, we presented a water treatment technology using microbubbles (MBs) in membrane filtration in order to reduce membrane fouling and to improve the filtration flux. In this study, we investigated the effect of foulants in this system. Water samples from five rivers and three model foulant solutions were used, and water quality indicators such as dissolved organic carbon (DOC), ultraviolet absorbance (E260), and specific UV absorbance (SUVA) of these solutions were analyzed. Correlations between the flux enhancement by MB and DOC, E260, and SUVA in the five river water samples indicated that MBs were effective for river water samples with fewer foulants, and that an increase in the amount of highly aromatic hydrophobic substances in the river reduced the MB effect. In the experiments using humic acid, guar gum, and bovine serum albumin (BSA) as model foulants, it was found that MBs enhanced the reduction of fouling from the initial stage in the cases of humic acid and guar gum, while in the case of BSA, the MB effect was not clear in the initial stage and then became more pronounced. The relation of the flux enhancement by MB and DOC for the humic acid solution was roughly in agreement with the relations obtained from river water samples.

Keywords: Microbubble; Membrane fouling; Ultrafiltration; Filtration flux; Water quality

1. Introduction

Microfiltration and ultrafiltration (UF) membranes have been widely applied in drinking water production. In drinking water production using these membranes, the important issues are (1) the reduction of water production costs, (2) the enhancement of water quality, (3) safety and reliability, and (4) ease of maintenance. To reduce water production costs, suitable membrane module design and system optimization are necessary, as well as the development of new types of membranes. Membrane fouling control is essential for reducing water production costs as it reduces permeation flux and increases the running costs of the operation and the replacement of the module.

Many fundamental and applied studies have been conducted for the purpose of membrane fouling reduction. In surface water treatment, Cho et al. [1] elucidated that, where flux recovery by physical backwashing was difficult, the main substances causing irreversible fouling came from natural organic matter (NOM) such as humic acid, protein derived from bacteria, and colloidal polysaccharides in natural water.

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Yamamura et al. reported that polysaccharide-like materials in NOM caused irreversible membrane fouling [2–4]. Furthermore, Akamatsu et al. [5] studied the effect of free water and bound water adhered to these foulants and the membrane surface on membrane fouling.

On the other hand, membrane surface modifications have been widely investigated to reduce membrane fouling in surface water treatment. Razi et al. [6] grafted a zwitterionic monomer onto the membrane surface and demonstrated the reduction of fouling. The antifouling properties of polyvinylidene fluoride (PVDF) hollow fiber membranes dip-coated with a phosphorylcholine copolymer were reported by Nishigochi et al. [7]. They confirmed, by using a quartz crystal microbalance, that almost no bovine serum albumin (BSA) was adsorbed onto the phosphorylcholine-coated PVDF membrane. Modified PVDF membranes showed higher water flux than did the bare membrane after fouling.

Pretreatment of the feed water is also effective for membrane fouling reduction. Stoquarta et al. [8] demonstrated the effectiveness of activated carbon treatment in the membrane process. Mori et al. [9] reported a microfiltration system using ozone pretreatment. This system showed 3–4 times higher flux than that of a system without ozone for various types of feed water. Song et al. [10] reported a UV absorbance reduction of 71% and dissolved organic carbon (DOC) removal of 10% through surface water treatment at an ozone dosage of 3.0 mg/L. Ozone oxidation enabled the degradation and hydrophilization of macromolecular organic matter in surface water, which is responsible for membrane fouling. Muthukumaran et al. [11] studied the use of ultrasonic cleaning for membrane fouling reduction in the whey treatment. The formation, growth, and collapse of microbubbles (MBs), which increase permeability, via sonification might occur on the surface of the fouled membrane.

In our previous study, we introduced a water treatment technology using MBs in the membrane filtration system in order to reduce membrane fouling [12]. Pilot-scale experiments showed fouling reduction by MBs persisted for more than one month, and MBs were also effective for chemical-free cleaning of the fouled membrane. From the viewpoint of longer term

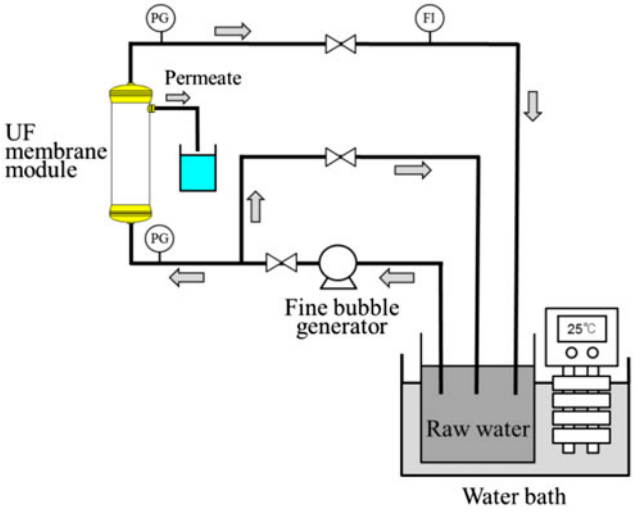

Fig. 1. Schematic diagram of membrane filtration equipment with MB generator.

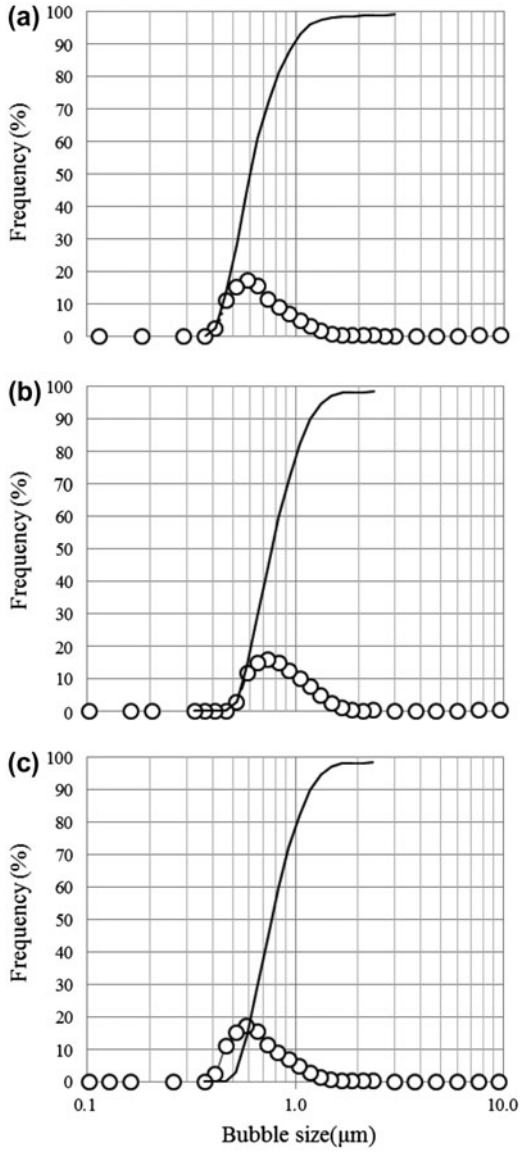

Fig. 2. Distribution of MB size in: (a) pure water; (b) Toga River water; and (c) humic acid solution (5 ppm). Full line: cumulative distribution curve.

effects of MBs on the membrane, there was no change in the tensile strength and molecular weight (MW) cut-off after one month of exposure.

The flux enhancement from membrane fouling reduction could be achieved through a higher cross-flow velocity of 0.16 m/s. However, we obtained higher flux even at a lower cross-flow velocity of 0.01 m/s when MBs were present [12]. Regarding energy efficiency, the power consumption of the MB generator was 1.09 times higher than that of a general centrifugal pump. Even though the power consumption for this MB generator is higher, the filtration flux was enhanced by a factor of almost two. Thus, this MB generating system can reduce the total water treatment costs. Therefore, using MBs is better than generating higher cross-flow velocity.

The possible mechanisms for fouling reduction by MBs are (1) the detachment of the cake layer by MBs, (2) the adsorption of foulant on the MB surface, (3) the reduction of cake layer resistance by MB presence in the cake layer, and (4) decomposition of organic matter by radicals generated from the collapse of MBs. To elucidate the mechanism for the reduction of membrane fouling by MBs, more fundamental studies are necessary.

In this work, we investigated the effect of foulants on membrane fouling reduction by MBs. The effectiveness of MB incorporation was investigated in raw water samples from five rivers. In addition, feed solutions containing model foulants such as humic acid, guar gum, and BSA were used in these experiments. DOC, ultraviolet absorbance (E260), specific UV absorbance (SUVA), and turbidity were measured for each water sample, and the effectiveness of MBs was investigated based on these data.

2. Experimental

2.1. MB generation

A pressurized dissolution-type apparatus (OM4-MDG-045, AuraTec Co., Ltd., Fukuoka, Japan) was used as a MB generator [13]. The mixture of water and air supplied at the inlet of the pump by suction was pressurized in the tank, where dissolved air was supersaturated. MBs were generated by discharging the pressure using a reducing valve. The water containing MBs was supplied from a nozzle to a hollow fiber membrane. The size and population of the MBs depended on the pressure in the tank and the discharging degree.

2.2. Hollow fiber membrane

Cellulose acetate UF hollow fiber membranes (Daicen Membrane-Systems Ltd., Tokyo, Japan) were used for filtration. The membrane (inner/outer fiber diameters: 0.80/1.30 mm) was hydrophilic, and displayed a low foulant adsorption. The nominal MW cut-off (as determined by protein rejection) was 150,000 Daltons. One fiber was cut to 110 mm (effective membrane area: 0.00028 m^2) and used for pure water filtration and raw water filtration.

2.3. Membrane filtration measurement

Surface water samples from the Akashi, Ibo, Muko, Sumiyoshi, and Toga Rivers (Hyogo, Japan) were used for membrane filtration measurement. Model filtration solutions were prepared by dissolving humic acid from Aldrich Chemical Co., guar gum from Ina Food

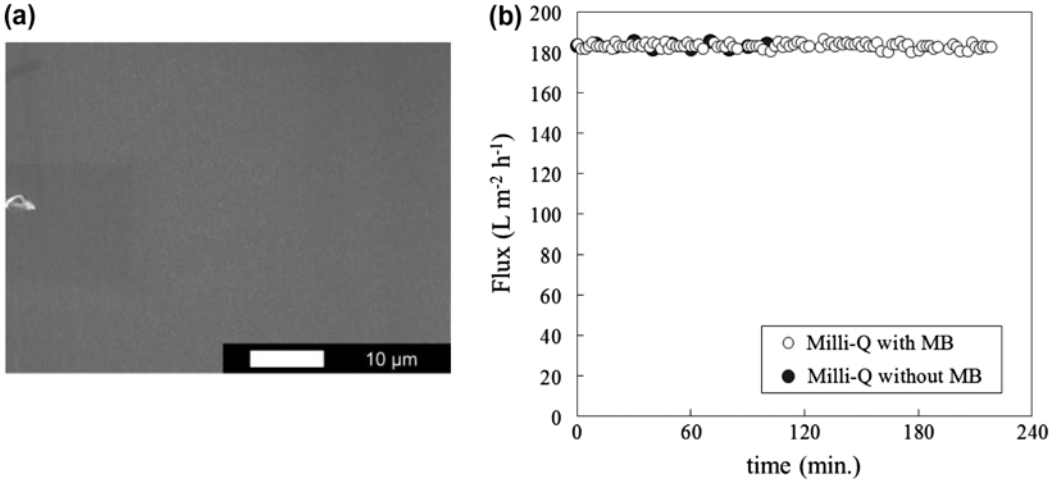

Fig. 3. SEM images of hollow fiber inner surface (a) and filtration flux in the Milli-Q water with and without MBs (b).

Industry Co., and BSA from Wako Pure Chemical Industries in deionized (DI) water. The concentrations of the humic acid, guar gum, and BSA solutions were 5, 1, and 1 ppm, respectively. Humic acid, guar gum, and BSA were selected as model foulants because they have been adopted in many studies as examples of

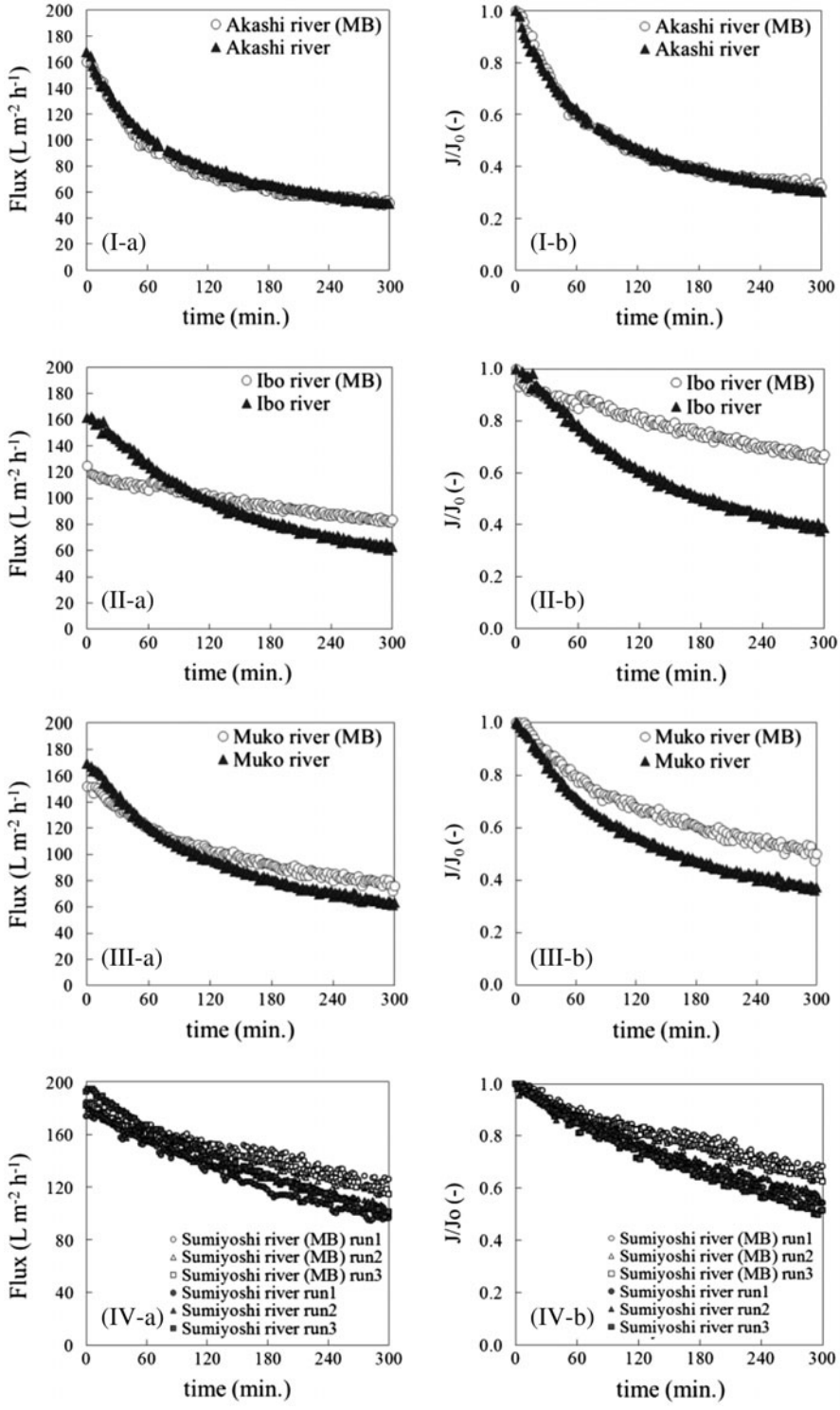

Fig. 4. Effect of MB addition on filtration flux (a) and relative flux J/J_0 (b) in the Akashi (I), Ibo (II), Muko (III), Sumiyoshi (IV) and Toga (V) River samples.

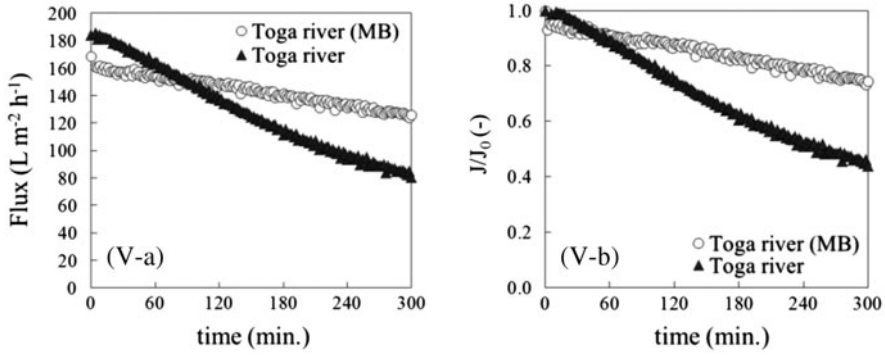

Fig. 4. (*Continued*).

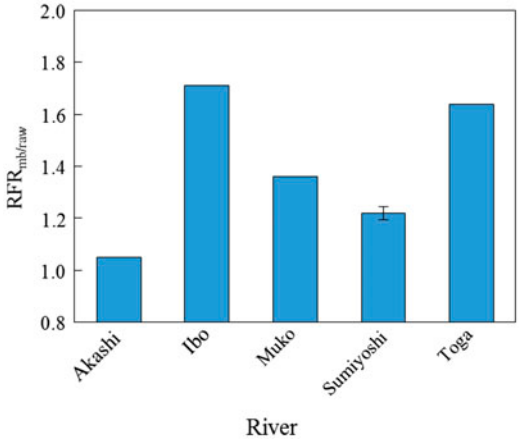

Fig. 5. Relative flux ratio ($RFR_{mb/raw}$) for the five river water samples.

humic substances, polysaccharides, and proteins, respectively. The MW of the humic acid used was several hundred to hundreds of thousands. The MWs of guar gum and BSA were 200,000–300,000 and 66,000, respectively.

Fig. 1 shows a schematic diagram of the filtration equipment with the MB generator. The filtration system consisted of two loops: a recirculation loop for cross-flow filtration, and a loop for returning excess water to the tank. Firstly, pure water was introduced into a tank kept at 25°C and then supplied to the hollow fiber membrane; the water flowed through the lumen side (0.8 mm in diameter) of the hollow fibers. Water was filtered through the membrane and recirculated to mix with the feed water. Cross-flow UF was conducted at a flow velocity of 0.06 m/s, which corresponded to a Reynolds number of 50 and a transmembrane pressure of 50 kPa. The volumetric flow rate was determined by measuring the volume of permeate collected at various time intervals. The pure water flux J_0 was calculated using the equation $J = Q/A$, where Q is the volumetric flow rate (L/h) and A is the effective membrane area (m^2). After the pure water filtration, a river water or model foulant solution was filtered with the same hollow fiber membrane, and the water flux J was obtained. Filtration tests for Sumiyoshi river water were performed twice. Relative flux (RF) was calculated as J/J_0. Filtration was carried out in two modes: with MBs (by opening the air suction valve) and without MBs (by closing the air suction valve). To investigate the flux enhancement effect of MBs (the MB effect) quantitatively, the normalized relative flux ratio ($RFR_{mb/raw}$) was defined as:

Table 1
Water quality and relative flux ratio ($RFR_{mb/raw}$) of the five river water samples

River	DOC[a] (mg/L)	E260[a] (1/cm)	SUVA ($m^{-1}(mg/L)^{-1}$)	Turbidity (NTU)	$RFR_{mb/raw}$[a] (–)
Akashi	1.92 ± 0.01	0.058 ± 0.001	3.00	1.18	1.05
Ibo	0.48 ± 0.02	0.009 ± 0.001	1.84	0.41	1.71
Muko	1.38 ± 0.01	0.032 ± 0.001	2.33	0.39	1.36
Sumiyoshi	0.85 ± 0.01	0.008 ± 0.010	0.94	0.10	1.22 ± 0.046
Toga	0.61 ± 0.01	0.015 ± 0.001	2.37	0.41	1.64

[a]Mean ± standard deviation.

$$RFR_{mb/raw} = RF_{mb}/RF_{raw} \quad (1)$$

where RF_{mb} and RF_{raw} indicate relative fluxes after 300 min filtration with and without MBs, respectively. Since the membrane is generally backwashed every hour for 1 min in surface water treatment, the filtration time of 300 min was sufficient. Larger $RFR_{mb/raw}$ values mean the MBs yield greater flux enhancement and prevent membrane fouling.

2.4. Water quality analytical methods

Three river water samples were filtered through a 0.45-micron PTFE filter before water quality analyses. The data were averaged and reported as a mean ± standard deviation except for turbidity. DOC concentrations were determined by a total organic carbon (TOC) analyzer (TOC-VCSH, Shimadzu Co., Kyoto, Japan). Before measuring the DOC, raw water was filtered under vacuum by a glass fiber filter with a pore size of 1 μm. E260 of the solutions was measured at 260 nm using a spectrophotometer (V-650KE, JASCO Co., Tokyo, Japan). E260 was used as an index of humic substances concentration. SUVA values were determined by dividing the E260 by the DOC concentration as follows:

$$\text{SUVA } (m^{-1}(mg/L)^{-1}) = \text{E260 } (cm^{-1}) \times 100/\text{DOC } (mg/L) \quad (2)$$

SUVA correlates well with the aromaticity and hydrophobicity of the organic carbon [14]. Turbidity was measured using a turbidity meter (TN-100, Eutech Instruments, Singapore).

2.5. Distribution of MB size and scanning electron microscopy (SEM) observation of fouled membrane

The MB sizes were measured using a nanoparticle size analyzer (SALD-7500 nano, SHIMADZU Co., Kyoto, Japan). The MBs were generated by an MB generator using (1) pure water, (2) Toga River water, and (3) a 5-ppm humic acid sodium solution. The sample water containing MBs was then supplied to the analyzer, and the MB sizes were measured.

To obtain dry membranes for surface observation by scanning electron microscopy (SEM), the hollow fiber membranes were freeze-dried in a freeze dryer (FD-1000, Tokyo Rikakikai Co., Japan). The dry hollow fiber membranes were fractured in liquid nitrogen and treated by osmium coating. The inner surfaces of the hollow fiber membranes were observed by field emission (FE)-SEM (JSM-7500F, JEOL, Tokyo, Japan) with an accelerating voltage of 5 kV.

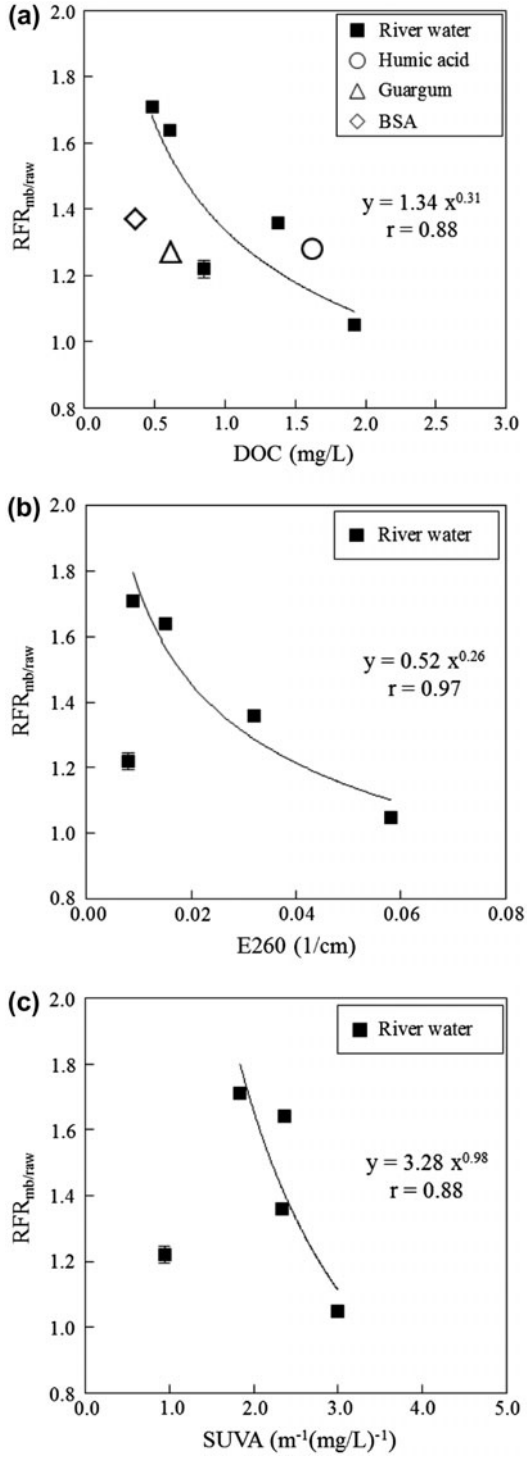

Fig. 6. Influence of water quality on relative flux ratio ($RFR_{mb/raw}$): (a) DOC; (b) E260; and (c) SUVA. Note: Error bars in the river water quality graphs are too small to appear on the graphical scale.

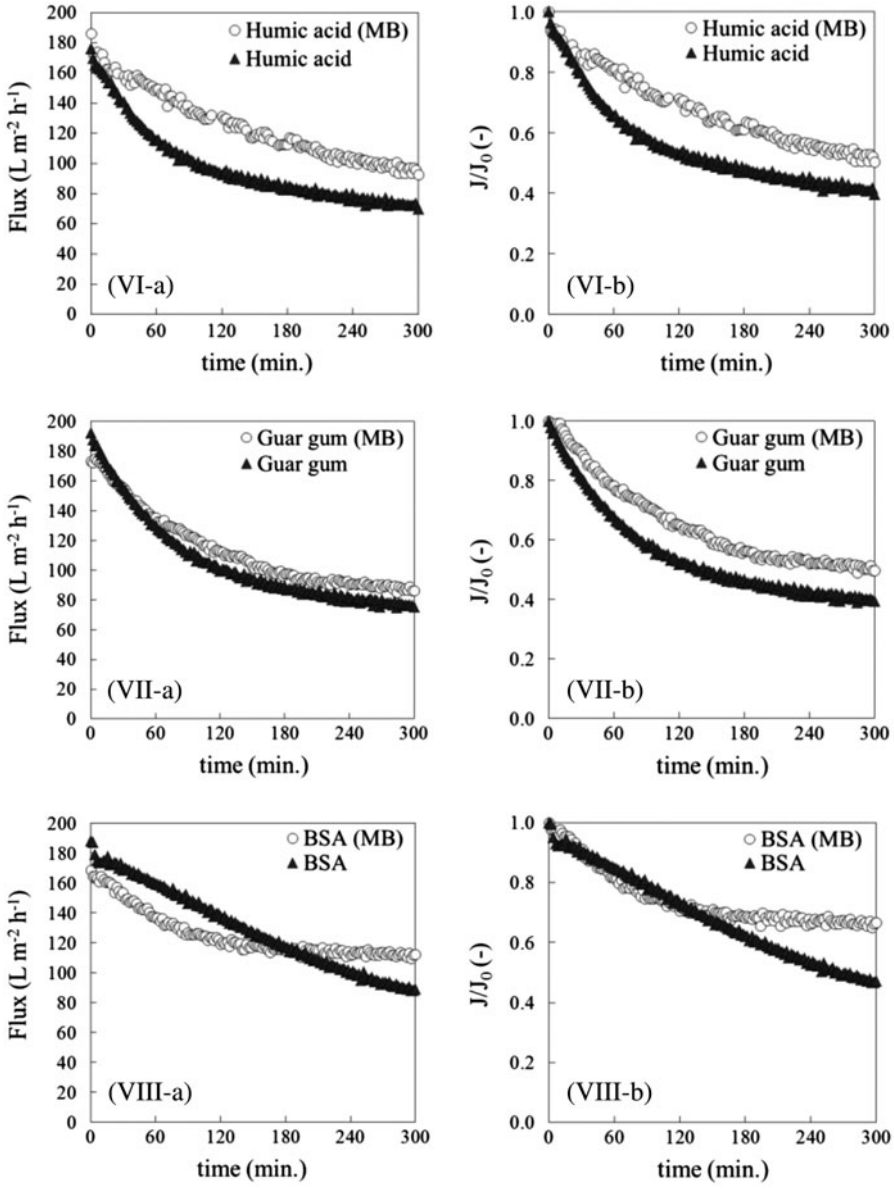

Fig. 7. Effect of MB addition on filtration flux (a) and relative flux J/J_0 (b) in humic acid (VI), guar gum (VII), and BSA (VIII) solutions.

3. Results and discussion

3.1. Distribution of MB size

Fig. 2 shows the distribution of MB size in pure water, Toga River water, and 5-ppm humic acid solution. The full line shows the cumulative distribution curve. The MBs had a narrow size distribution, and their diameters were 0.4–1.1 μm in water sample from any source. The largest sizes were approximately 0.6 μm in pure water and humic acid solution and 0.7 μm in water from the Toga River.

3.2. MB effect on various river water filtrations

Fig. 3(a) shows the inner surface structure of the hollow fiber membrane. The bare membrane surface before fouling was smooth, and no pores can be seen at this magnification because a UF membrane was used. Fig. 3(b) shows filtration fluxes for Milli-Q water with and without MBs. This result indicates that MBs have no effect on the pure water flux.

Fig. 4 shows the changes in filtration flux (a) and relative flux J/J_0 (b) using raw water from the Akashi

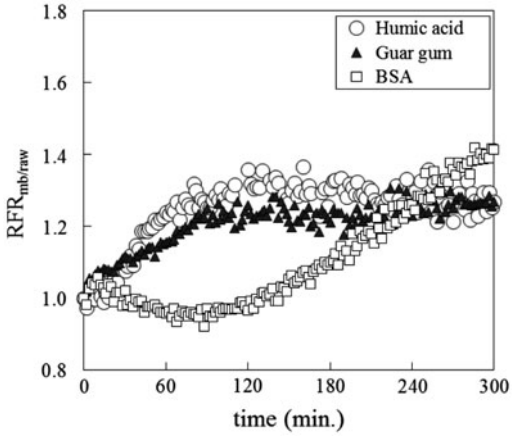

Fig. 8. Relative flux ratio ($RFR_{mb/raw}$) changes in filtrations using model foulants.

(I), Ibo (II), Muko (III), Sumiyoshi (IV), and Toga (V) Rivers. Open and closed symbols show the data in the cases with and without MBs, respectively. From the results of J/J_0 in Sumiyoshi River water (run 1, run 2, and run 3), the reproducibility of the filtration flux can be confirmed. In most cases, the introduction of MBs was effective in reducing membrane fouling. These results are in agreement with our previous work using a pilot-scale apparatus [12].

However, MBs showed no clear effect on fouling reduction in water from the Akashi River. These results indicate that the MB effect depends on the location of the river, and therefore, on the variable quality of the water. This dependence on water quality is discussed later.

Fig. 5 shows the normalized relative flux ratio ($RFR_{mb/raw}$) in the five river water samples. The highest $RFR_{mb/raw}$ of 1.71 was obtained in the water from the Ibo River. In the data from the Ibo River showing high RFR values, start fluxes were different in the cases with and without MBs (Fig. 4(II)). The Toga River data were similar to the Ibo River data. It was assumed that reversible membrane fouling in the initial stage was accelerated by the MBs that absorbed the foulants (Fig. 4(II-b), (V-b)). Foulants absorbed by the MBs could not permeate the membrane and formed cake layers. However, the subsequent flux reduction was slow, and because cake layers containing MBs were loose, the cake layer detachment occurred. The $RFR_{mb/raw}$ of Ibo and Toga River samples became higher than those of Akashi, Muko, and Sumiyoshi River samples after 300 min. In the case of water from the Akashi River, $RFR_{mb/raw}$ was almost unity, showing that the introduction of MBs had no pronounced effect.

3.3. Effect of water quality on MB effect

Table 1 shows the water quality and the normalized relative flux ratio ($RFR_{mb/raw}$) of the five river water samples. Ibo River water showed the lowest DOC, while Sumiyoshi River water showed the lowest E260, SUVA, and turbidity. Thus, the water quality in these two rivers is high. On the other hand, the highest DOC, E260, SUVA, and turbidity were obtained from Akashi River water, showing that the water quality in this river is poor.

Fig. 6 shows the influence of water quality indicators such as DOC, E260, and SUVA on the $RFR_{mb/raw}$. Error bars in the river water quality graphs are too small to appear on the graphical scale. Correlations were obtained between $RFR_{mb/raw}$ and DOC (correlation coefficient: $r = 0.88$), E260 ($r = 0.97$), and SUVA ($r = 0.88$). Correlation lines in the cases of E260 and SUVA were drawn through four points. The data of Sumiyoshi River in Fig. 6(b) and (c) deviated from the correlation lines. Although E260 and SUVA of Sumiyoshi River were low, $RFR_{mb/raw}$ was not so high. The foulants except humic substances (i.e. polysaccharides) may be included in the Sumiyoshi River. More detailed data about water quality is necessary to clarify this exception in the correlation. $RFR_{mb/raw}$ decreased with increasing DOC concentration, as shown in Fig. 6(a). An increase in DOC concentration indicates degradation of the water quality. Therefore, it was confirmed that the use of MBs is a more effective method for fouling reduction in higher quality water samples. In addition, the increase in E260 and SUVA also brought about a decrease in $RFR_{mb/raw}$. SUVA values lower than 2 generally indicate a high fraction of hydrophilic non-humic matter with low E260. SUVA values between 2 and 4 indicate a mixture of hydrophobic humic substances and hydrophilic non-humic matter, and values higher than 4 are indicative of the presence of highly aromatic and hydrophobic humic substances [14]. This indicates that the presence of humic substances and highly aromatic hydrophobic substances reduces the MB effect. Therefore, the introduction of MBs was not effective for the low-quality water from the Akashi River, which showed an E260 of 0.058 and a SUVA of 3.00.

Yamamura et al. showed that hydrophilic substances such as polysaccharides or proteins are the main contributors to membrane fouling [15]. Therefore, filtration experiments using polysaccharides and proteins as model foulants were conducted to confirm the MB effect. Fig. 7 shows the effect of MBs on filtration flux (a) and relative flux J/J_0 (b) in the filtration experiments using model foulants. The results for humic acid and guar gum show similar

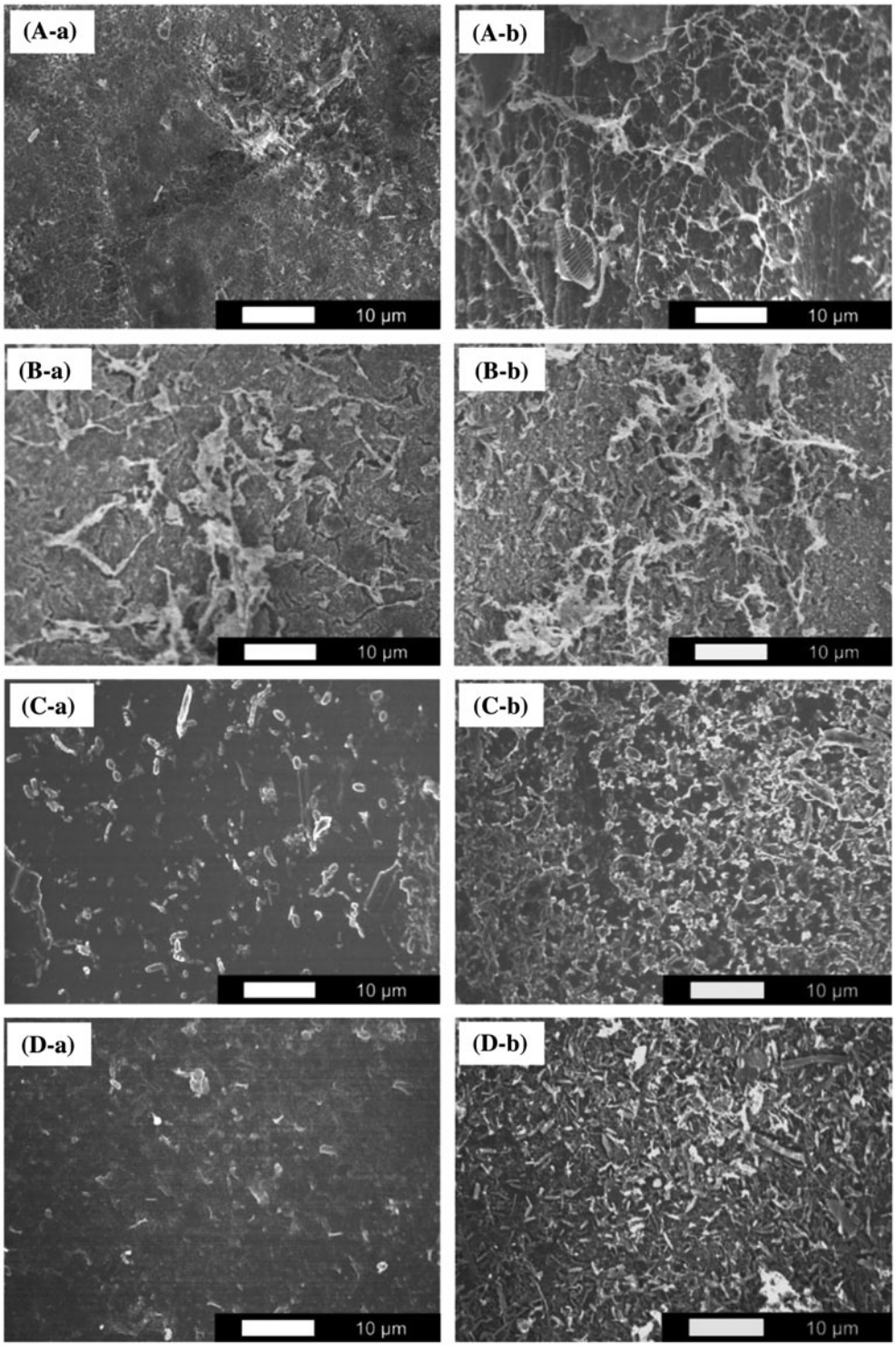

Fig. 9. SEM images of hollow fiber inner surfaces after filtration for 300 min. (A) Toga River, (B) humic acid, (C) guar gum, and (D) BSA; (a) without MB and (b) with MB.

behavior to those of Muko River water (III-a, and b) shown in Fig. 4. Thus, it was confirmed by these model foulant experiments that the introduction of MBs reduces the membrane fouling caused by humic substances and polysaccharides. In the BSA filtration experiments, membrane fouling was not affected by

the introduction of MBs before 150 min; however, the fouling was clearly reduced after 150 min.

The normalized relative flux ratio ($RFR_{mb/raw}$) changes in the experiments using model foulants are shown in Fig. 8. In the cases of humic acid and guar gum, $RFR_{mb/raw}$ initially increases and reaches a constant value. However, in the case of BSA, $RFR_{mb/raw}$ is almost unity at the initial stage and then increases. Although the MW_S of humic acid and guar gum are high, the MW of BSA (66,000) is lower than the MW cut-off of the membrane (150,000). Thus, membrane fouling initially occurs inside the pore in the case of the BSA filtration, while humic acid and guar gum are deposited on the membrane surface, even in the initial stage of the fouling. Since MBs cannot penetrate into the membrane pores owing to their large size, the effect of MBs was not observed in the early stage of the filtration. As the fouling occurs, BSA blocks the pores and deposits on the membrane surface, as confirmed in Fig. 9(d). In this situation, MBs can attack the deposited layer and reduce membrane fouling.

Fig. 9 shows SEM images of the inner surfaces of the hollow fibers of fouled membranes with and without MBs. Compared to the original smooth structure shown in Fig. 3(a), deposited layer formation was confirmed in all membranes. In all four water cases, the introduction of MBs brought about rougher and more porous deposited layers, while relatively flat deposited layers were formed under the conditions without MBs. The rough structures may be formed because of the presence of MBs and have higher porosities than the flat deposited layers. The formation of porous structures because of the presence of MBs causes less permeability reduction.

Table 2 shows the DOC concentration and the relative flux ratio ($RFR_{mb/raw}$) obtained in the experiments is shown in Fig. 7. E260 and SUVA calculated from E260 are not shown in this table because the E260 values of these three solutions were too low to be measured accurately. Plots of DOC vs. $RFR_{mb/raw}$ for the three solutions are also shown in Fig. 6(a).

The relation of $RFR_{mb/raw}$ and DOC for the humic acid solution was roughly in agreement with the relations obtained from river water samples. This is reasonable because the organic matter in river water mainly comprises humic substances. Although the DOC of the guar gum and BSA solution was the same as those for the Ibo and Toga Rivers, the $RFR_{mb/raw}$ values of these model foulant solutions were lower. This suggests that guar gum and BSA are materials that reduce the MB effect. Further study is necessary to clarify the reason for the difference of MB effects between model foulant solutions and river samples.

Table 2
DOC concentration and relative flux ratio ($RFR_{mb/raw}$) of model foulant solutions

Model foulant	DOC (mg/L)	$RFR_{mb/raw}$ (−)
Humic acid (5 ppm)	1.62	1.28
Guargum (1 ppm)	0.62	1.27
BSA (1 ppm)	0.36	1.37

4. Conclusions

We aimed to investigate the effect of foulant types on the reduction of membrane fouling by MBs. Water samples from five rivers and model foulant solutions were used. Correlations between the flux enhancement by MB and DOC, E260, and SUVA were observed in the five river water samples. Furthermore, from experiments using humic acid, guar gum, and BSA as model foulants, it was revealed that an MB effect was observed in the initial stage of fouling in the cases of humic acid and guar gum, while in the case of BSA, MBs had little effect in the initial stage, but a marked fouling reduction was observed in the later stage. This suggests that the small molecules of BSA initially penetrated the membrane pores. MBs are not effective here as they are too large to penetrate the pores. In the later stage of filtration, a BSA layer formed on the membrane surface and the MB effect became pronounced because MBs can attack the deposited layer. In the relationship between the MB effect and DOC, the data for the humic acid solution was roughly in agreement with the relations obtained from river water samples.

The possible mechanisms for fouling reduction by MBs are (1) the detachment of the cake layer by MBs, (2) the adsorption of foulant on the MB surface, (3) the reduction of cake layer resistance by MB presence in the cake layer, and (4) decomposition of organic matter by radicals generated from the collapse of MBs.

Our next aim is to elucidate the mechanism of the MB effect on fouling reduction.

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