# Quantitative analysis of biological effect on membrane fouling in submerged membrane bioreactor

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**Abstract** The objective of this study is to investigate solids concentration and extracellular polymeric substance (EPS) effects on the membrane fouling in the submerged membrane bioreactor. The relationship between the solids retention time (SRT) and the amount of EPS is observed in three lab-scale MBRs. Additionally, the EPS effect on membrane fouling is quantified by calculating the specific cake resistance ($\alpha$) using an unstirred batch cell test. By observing the sludge over a long period under various SRT scenarios, a wide range of EPS and membrane fouling data is obtained. These observations provide sufficient evidence of the functional relationship between SRT, EPS and $\alpha$. As SRT decreases, the amount of EPS bound in sludge floc becomes higher in the high MLSS condition ($>$5,000 mg/L). The amount of EPS in the sludge floc has positive influence on $\alpha$. A sigmoid trend between EPS and $\alpha$ is observed and the functional relationship obtained by dimensional analysis is consistent with the experimental results.

**Keywords** Bioreactor, EPS, fouling, membrane, SRT

## Introduction

The membrane bioreactor (MBR) is an activated sludge process with which membrane filtration is coupled instead of the settling process for liquid-solid separation. Because of the membrane filtration, the suspended solids are completely removed from the treated water to the extent that the effluent contains no bacteria in microfiltration or no virus in nanofiltration (Yamamoto *et al.*, 1989; Cote *et al.*, 1997; Ahn *et al.*, 1998). Nevertheless, one of the major obstacles that prevents widespread application of membrane filtration in wastewater treatment is the flux decline with time (Song, 1998; Mukai *et al.*, 2000). This phenomenon is the generally so-called membrane fouling. With specific regard to filtration of the activated sludge in MBR system, it is recognized that one of the important factors for fouling is the extracellular polymeric substance, EPS (Stec *et al.*, 1995; Chang and Lee, 1998; Nagaoka *et al.*, 1998). Leslie *et al.* (1993) and Hodgson *et al.* (1993) attributed low permeate flux and solute rejection of bio-fouled MF membranes to the EPS rather than to the colloidal nature of bacterial cells constituting the biofilm. The biosynthesis and extrusion from the attached cells of EPS also contributes to the initial rapid flux loss. However, these researches cannot show the relationship among the EPS production, the operation factors such as SRT and membrane fouling.

Moreover, the relationship between the sludge concentration and membrane fouling is unclear and controversial in the MBR system. Magara and Itoh (1991) reported that membrane fouling took place more rapidly at higher sludge concentrations. Manem and Sanderson (1996) also made the identical conclusion. However, other studies suggested that higher sludge concentration resulted in less fouling under identical conditions (Defrance and Jaffrin, 1999; Lee *et al.*, 2001). These contradictory results contributed to the view that the membrane fouling in MBR is related to not only sludge quantity but also quality as well as operating parameters in the MBR system (Lee *et al.*, 2003). The interaction between flux decline and solid concentration should be investigated simultaneously considering EPS. It is necessary that a continuous observation be performed, which tracks the variation of sludge characteristics and membrane fouling for different SRTs in real MBR system.

In order to investigate the relationship among SRT, EPS and membrane fouling, Chang and Lee (1998) cultivated activated sludges under nitrogen-deficient conditions different SRTs. The reduction of EPS content in activated sludges resulted in an equivalent reduction in the hydraulic resistance of the cake layer. Lee *et al.* (2003) also suggested that the different sludge characteristics induced by the different SRT had some contribution to microfiltration in submerged membrane bioreactors. The production and reduction of EPS was related to SRT of the activated sludges. However, the dynamic variation of EPS along with the membrane fouling is still not observed in real MBR systems, because existing studies conducted batch tests for evaluating the EPS contribution to membrane fouling. In this study, a continuous lab-scale experiment is performed to investigate the relationship among SRT, EPS and membrane fouling.

In summary, no researches can predict that how much EPS affects membrane fouling and to what degree the membrane is fouled, quantitatively. The interaction between flux decline and solids concentration considering EPS simultaneously has not been investigated so far. In this study, the specific cake resistance ($\alpha$) is measured to quantify the membrane fouling.

## Materials and methods

### Lab-scale submerged membrane bioreactor

The lab-scale consists of the flat sheet microfiltration membrane module (Yuasa Co-operations, Japan) with the effective filtration area of 0.1 $m^2$/module. The membrane is made of polyethylene with hydrophilic coating, and its nominal pore size is 0.25 μm. The membrane module is fully immersed and symmetrically placed in the reactor. The airlift is installed underneath the membrane module to provide dissolved oxygen in the reactor as well as to control membrane fouling by hydraulic shear force and agitation. For generating permeate, outside-in type filtration is induced by the suction pump. Thus, the transmembrane pressure (TMP) of membrane is monitored as negative value. The experiment conditions are described in Table 1. Three MBRs are operated with different SRT, while the other factors are maintained as identical. Since there is no return activated sludge, SRT is calculated simply by dividing the sludge wasting volume into the volume of MBR reactor regardless of solids concentration in the system. The sludge wasting volume of each reactor corresponds to 700, 280 and 70 mL/day for SRT 8, 20, and 80 days, respectively. The activated sludge from the local municipal wastewater treatment plant is seeded into the three reactors after screening.

The synthetic wastewater is introduced to observe the production and degradation of EPS and investigate its effect on membrane fouling. In order to prevent the

**Table 1** Operating conditions of lab-scale SMBR system

| Operating factors | Descriptions |
|---|---|
| Flux | 15 LMH (L/$m^2$/hour), constant flux mode |
| HRT | 4.5 hours |
| SRT | 8, 20 and 80 days for MBR 1, 2 and 3, respectively |
| Reactor Volume | 5.6 L |
| Starting MLSS | 6,000 mg/L |
| Aeration | 15 L/min/module, continuous aeration |
| Operation cycle | 12 minutes (10 min suction with 2 min rest) |
| F/M ratio | 0.1 kgCOD/kgMLVSS |

decomposition of organic matters, the feed that is maintained at 4°C. Glucose is introduced as carbon source. Four minerals ($MnSO_4 \cdot 7H_2O$, $FeCl_3 \cdot 6H_2O$, $CaCl_2$ and $MgSO_4 \cdot 7H_2O$) and phosphate buffer are supplied. The concentration of COD, total nitrogen and total phosphorus in this synthetic wastewater are 330 $mgCOD_{cr}$/L, 56 mgN/L and 10 mgP/L, respectively. Since there are no particles in the synthetic wastewater, the change of solids content such as MLSS in the MBR would be affected only by SRT and biological growth or decay.



#### EPS extraction and measurement

EPS concentration of the activated sludge is measured every three days. The wasting sludges are collected from the lab-scale MBR system for EPS extraction and measurement. The bound EPS in activated sludge floc is extracted by a cation exchange resin procedure. This method is referred mainly from Frølund *et al.* (1996) and modified to be suitable for this research. The cation exchange resin (CER) is DOWEX 508, 20–50 meshes in the sodium form (Aldrich 42878-7). The amount of EPS is analyzed by determining total organic carbon (TOC) content in the extracted EPS solution. TOC-Analyzer ($V_{CPN}$-6000, Simadzu, Japan) is used to measure TOC.

#### Batch cell test for calculating specific cake resistance

In order to determine $\alpha$ of the activated sludge floc, the dead end filtration experiment is performed using the system called Amicon Cell (Amicon™, USA). In this study, the unstirred batch test is performed to eliminate the cross-flow effect on the cake layer formation caused by stirring. The nitrogen gas is injected into the vessel to keep the constant TMP across the membrane. The constant pressure across the membrane is established. Therefore, the permeate flux decreases as the membrane is fouled. The membrane is directly cut from the large flat sheet membrane module to have the size and shape suitable for the vessel. This large module (0.84 $m^2$) is manufactured for the field plant and has the identical material characteristic as the small one (0.1 $m^2$) installed in the lab-scale MBR system operated in this study.

### Results and discussion

#### EPS and MLSS variation for different SRT

The mixed liquor suspended solids (MLSS) changes with different SRT. MLSS of each reactor is about 2,500, 4,000 and 8,000 mg/L for SRT 8, 20 and 80, respectively at steady state. The variation of bound EPS concentration is illustrated in Figure 1. Until 40 days

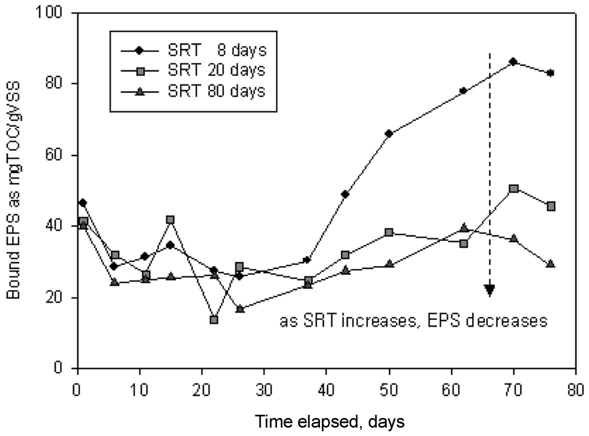


**Figure 1** Variation of bound EPS for the different SRT with time elapsed

elapse, there is no difference in the EPS concentration for different SRT. However, after 40 days, the EPS concentration of MBR #1 is much higher than those of other two reactors. After 80 days, the bound EPS concentrations per biomass (bound EPS) of three reactors are measured as 26.33, 40.90 and 82.59 mg TOC/gVSS for SRT 80, 20 and 8 days, respectively. As shown in Figure 1, as SRT increases, the bound EPS tends to decrease.

Figure 2 represents the relationship between the bound EPS and MLSS with different SRT. The amount of EPS tends to decrease with MLSS. It is noted that the amount of EPS tends not to change much above a certain value of MLSS concentration (5,000 mgMLSS/L). However, EPS variation is wide below 5,000 mgMLSS/L. The EPS amount ranges from 20 to 80 mgTOC/gVSS in this region. As presented in Table 2, the statistics show that in the broad variation region (below 5,000 mgMLSS/L), the Pearson correlation coefficient between MLSS and EPS concentration becomes $-0.51$ (negative correlation). This analysis implies that as MLSS increases, EPS tends to decrease non-linearity. Also, the paired sample *t*-test confirms that the estimated Pearson coefficient is reliable $(P(t_c \leq t|H_0) < 0.05)$.

However, above 5,000 mgMLSS/L, the bound EPS concentration ranges from 20 to 45 mgTOC/gVSS. This fluctuation is relatively small compared with the broad variation region. As shown in Figure 2, although MLSS increases from 5,000 to 8,000 mg/L, the bound EPS scatters within $30 \pm 10$ mgTOC/gVSS. In addition, there seems to be no clear correlation between EPS and MLSS. The Pearson correlation coefficient between MLSS and EPS concentration is estimated as almost zero (0.03). This means that there is no correlation between the MLSS and the bound EPS amount. This estimation is also confirmed to be reliable, because $P(t_c \leq t|H_0)$ value is less than 0.05.

Consequently, at high level of MLSS (>5,000 mg/L) SRT seems to be less sensitive to the bound EPS amount rather than at low level of MLSS (<5,000 mg/L). This phenomenon is attributed to the fact that the EPS itself would be substrate for microbial growth (Lu *et al.*, 2001; Laspidou and Rittmann, 2002). At high level of biomass population (high MLSS condition), substrate in the influent is exhausted more quickly, and then the microorganisms may utilize the EPS bound in the floc as well as released from cell lysis for their metabolism. In this study, the microbial population seems to not only exhaust the influent substrate but also consume the bound EPS, above 5,000 mgMLSS/L. Thus, the difference in the bound EPS amount with different SRT is less significant

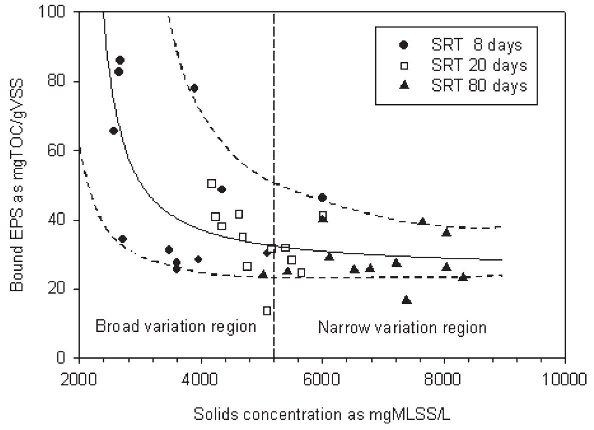


**Figure 2** Relationship between the bound EPS and the MLSS varied with different SRT

**Table 2** Paired sample t-test for the correlation between the MLSS and the bound EPS concentration (5% significance level, two-tail test)

| Factor | Broad variation region | Narrow variation region |
|---|---|---|
| Average | 46.31 | 29.39 |
| Pearson coefficient | −0.51 | 0.029 |
| *t*-value | 19.19 | 25.53 |
| $(P(t_c \leq t \mid H_0)$ | $2.87 \times 10^{-12}$ | $1.81 \times 10^{-16}$ |
| *t*-critical value, $t_c$ | 1.75 | 1.72 |



above that MLSS level. This means that the substrate-loading rate to the microorganisms is also considered important factor.

#### EPS effect on membrane fouling

In order to observe membrane fouling, TMP change of three reactors is monitored. Because these lab-scale MBRs are operated in constant flux mode (15LMH), TMP decreases along with membrane fouling. Considering flux fluctuation during a constant flux mode filtration, TMP is normalized to the condition that flux is 15LMH and temperature 20 °C. Figure 3 represents the variation of normalized TMP of three MBRs with the accumulated permeates volume. In an MBR system, the suspended particles including activated sludge floc consists of main foulant in a microfiltration. Therefore, it is conceivable that the higher MLSS causes the greater membrane fouling and faster flux decline. However, Figure 3 shows that normalized TMP ($TMP_N$) of SRT 8 days (MBR #1) decreases more rapidly than the others do. It is noted that although MLSS of MBR #1 is lower than the other two MBRs, the greater membrane fouling is observed. Before the permeate volume becomes 1,500 L (40 days), there is no significant difference in $TMP_N$ along with the different SRT conditions. However, after 1,500 L, $TMP_N$ of the MBR #1 (SRT 8 days) decreases precipitously to −120 kPa, while $TMP_N$ of MBR #2 and #3 remains almost identical with each other ranging from −5 to −8 kPa. At 1,800 L (60 days), three membranes are taken out from the reactor and the surface of each membrane is washed using tap water. Then, all the membranes are soaked fully in the 5,000 ppm sodium hyperchloride (NaOCl) solution for 3 hours. After this step, the membrane module is cleaned by deionized water several times. However, despite physical and chemical cleaning, the membrane installed in MBR #1 starts being fouled rapidly at the beginning as shown in Figure 3.

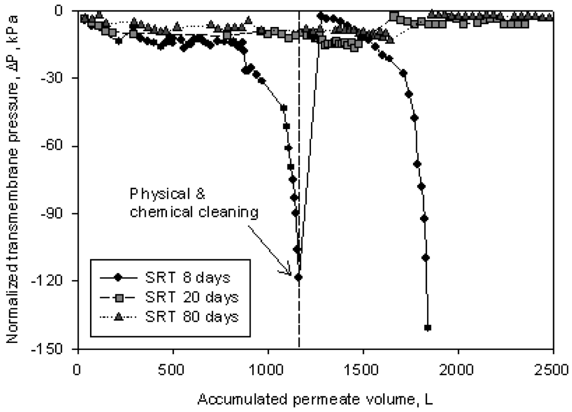


**Figure 3** Normalized TMP of three MBRs with permeate volume

The EPS effect on membrane fouling is illustrated clearly in Figure 4. This figure is depicted only using the results from MBR #1 (SRT 8 days). Until permeate is accumulated to 850 L, the bound EPS amount seems not to change significantly and TMP is fluctuated between −10 and −15 kPa. In this initial fluctuation stage, the cake layer begins to form, and then the membrane starts being fouled. However, the shear strength induced by aeration prevents the cake layer growth from proceeding to foul the membrane severely. The cake growth by convection and the cake detachment by aeration are balanced with each other showing little change of TMP. However, at the beginning of the phase I, EPS concentration increases almost linearly along with accumulated permeate volume. TMP decreases rapidly with the increasing of EPS. Thus, it seems that as the bound EPS amount enlarges, the hydraulic resistance of membrane increases.

This seems to be attributed to two reasons: (1) rate of cake growth becomes higher than that of cake detachment by aeration; and (2) although the cake layer growth is balanced with the shear rate, characteristic of the cake layer is changed to increase the hydraulic resistance. This means that the mass of cake layer is not varied, while $\alpha$ is changed. In this experiment, the only factor affecting floc characteristic and $\alpha$ is the amount of bound EPS.

In order to confirm that abrupt pressure drop is attributed to EPS increase, the fouled membrane is cleaned strongly (90% restoration is confirmed) and TMP of cleaned membrane is observed again (phase II). In the phase II, the membrane is also fouled rapidly and TMP decreases with the bound EPS amount still increasing. While, in the case of MBR #2 (SRT 20 days) and #3 (SRT 80 days), abrupt decrease of TMP is not observed (Figure 5). During the phase I and II, TMP of both MBR #2 and #3 are maintained at around −5 kPa, even though the bound EPS of both reactor increase from 20 to 45 mgTOC/gVSS. The low level of bound EPS causes no serious increasing of TMP of both MBR #2 and #3. Compared to MBR #1, MBR #2 and #3 shows relatively low and slightly increasing of the bound EPS in the phase I and II. Therefore, the abrupt or rapid decrease of TMP is not observed. Lee *et al.* (2003) also reported that the total amount of EPS was not varied significantly with the SRT of 20, 40 and 60 days.

Consequently, the bound EPS in the activated sludge flocs is closely related with the membrane fouling. As the amount of bound EPS enlarges, the absolute value of TMP increases negatively. This variation of TMP is attributed directly by the change of hydraulic resistance of the cake layer. As the amount of bound EPS in the sludge floc is higher, the total hydraulic resistance increases more rapidly. EPS affect characteristics of activated sludge floc such as floc size and structure density. EPS makes a floc more

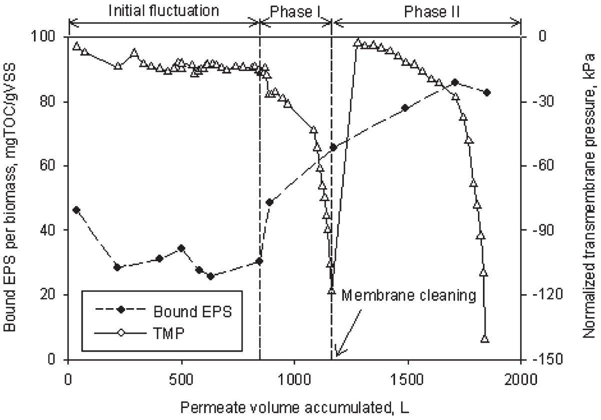


**Figure 4** The bound EPS effect on membrane fouling in case of MBR #1 (SRT 8 days)

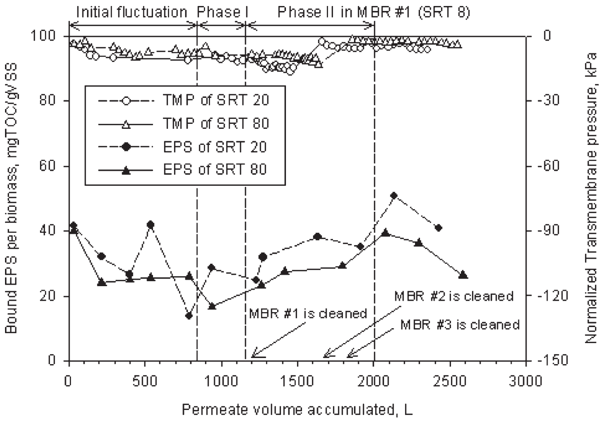


**Figure 5** The bound EPS effect on membrane fouling of MBR #2 and #3 (SRT 20 and 80 days)

dense and large. Accordingly, the characteristic of the cake layer is changed to increase the hydraulic resistance. This implies that α becomes higher.

#### Effect of dissolved EPS on membrane fouling

The dissolved EPS in the lab-scale MBR comes from two origins. One is induced by cell lysis and the other by the soluble EPS released from activated sludge floc. These matters are non-biodegradable or very slowly biodegradable matters compared with the glucose in synthetic wastewater. Therefore, it can be assumed that there are almost no substrate residuals from glucose in the supernatant. The dissolved EPSs are treated to be lumpsome and analyzed by measuring the dissolved organic carbon (DOC) concentration of supernatant in the bioreactor. After collecting a mixed-liquor directly from the bioreactor, supernatant is obtained by removing the activated sludge using the centrifuge and 0.45 μm filtration. The EPS released naturally from the cell or floc can be included in this measurement. It should be noted that this dissolved EPS differs from the bound EPS, which is entrapped on the microorganisms or microbial floc. Figure 6 shows the variation of the dissolved organic matters for the different SRT with the operation time elapses. It seems that as SRT decreases, DOC concentration also becomes lower. Since the amount of biomass (MLSS) in SRT 8 days is small compared with the other cases, the organic matter induced by cell lysis becomes less than in SRT 20 and 80 days. However, as previously discussed, the amount of bound EPS is much higher in SRT 8 days.

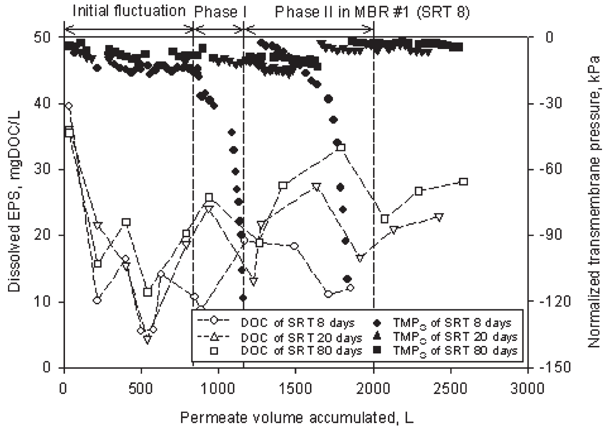


**Figure 6** Scatter plot for investigating the relationship between DOC and membrane fouling

**Table 3** Relative contribution of various fractions in sludge to membrane fouling at different SRT

| SRT | 20 days (Lee et al., 2003) | 40 days (Lee et al., 2003) | 60 days (Lee et al., 2003) | 60 days (Defrance et al., 2000) |
|---|---|---|---|---|
| Fractions | | | | |
| Supernatant | 37% | 28% | 29% | 35% |
| Suspended solids | 63% | 72% | 71% | 65% |

This means that the releasing of bound EPS from a sludge floc does not occur seriously in this condition.

Compared with the relationship between bound EPS and $TMP_N$ (Figure 4), DOC seems not to affect membrane fouling significantly (Figure 6). Lee *et al.* (2003) reported that the relative contribution of supernatant to overall membrane fouling was slightly higher at SRT 20 days than at 40 and 60 days. However, no remarkable contribution was found in fouling caused by the supernatant. Defrance *et al.* (2000) also reported that dissolved matters including colloidal particles had little effect on membrane fouling in membrane bioreactor with microfiltration. The suspended solids such as activated sludge floc contributed to hydraulic resistance predominantly. Table 3 summarizes results of these studies. It is noted that the suspended solids in MBR consist of the activated sludge, and the bound EPS is major component affecting the characteristic of activated sludge floc. Therefore, it is concluded that the membrane fouling is affected more by the bound EPS of activated sludge floc rather than the dissolved organic matter. This conclusion means that this study neglects the internal fouling of the membrane such as adsorption in membrane pores.

#### The bound EPS effect on specific cake resistance

Figure 7 shows the bound EPS effect on $\alpha$. As the bound EPS amount in the sludge floc increases, $\alpha$ value also increases. Previously, it was shown that as the SRT is higher, the bound EPS amount increases (Figures 1 and 2). Based on this result, the functional relationship between the bound EPS and $\alpha$ value is established empirically. For this work, the dimensional analysis is performed to organize the variables related with this function reasonably (Buckingham, 1914; Frank, 1994). In this study, it is assumed that the specific resistance ($\alpha$, $m \cdot kg^{-1}$) is affected by four variables such as the bound EPS (EPS, $kg \cdot m^{-3}$), the biomass concentration (MLVSS, $kg \cdot m^{-3}$), the transmembrane

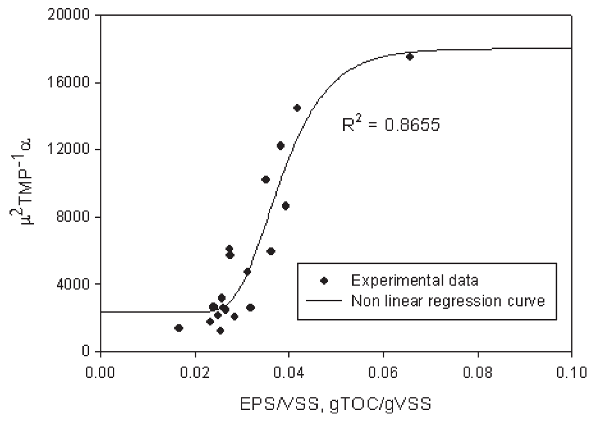


**Figure 7** Bound EPS effect on the specific cake resistance ($\alpha$) determined by unstirred dead end filtration test ($\Delta = 0.5$ psi, activated sludges from three lab-scale SMBRs is used)

pressure (TMP, $kg \cdot m^{-1} \cdot sec^{-2}$) and the permeate viscosity ($\mu$, $kg \cdot m^{-1} \cdot sec^{-1}$). In order to express the functional relationship among these variables, all the variables are introduced in the dimensional analysis. The relationship between MLVSS, EPS, TMP, and $\mu$ is assumed as; $\alpha$ = Function (EPS, MLVSS, TMP, $\mu$). Here, the number of variables is five, and that of dimension is three (mass, length, and time). Therefore, by the Buckingham PI theory, at least two-independent non-dimensional factor can be determined. Finally, the power series of two non-dimensional factors can be determined and the relationship among all the variables is expressed as; $\mu^2 \cdot TMP^{-1} \cdot \alpha = fn(EPS \cdot MLVSS^{-1})$. The non-dimensional analysis result should correspond to the previous observation for TMP and MLVSS. This shows that $\alpha$ can be expressed as function of the bound EPS per unit mass of particle. Additionally, $\alpha$ is proportional to TMP.

Figure 7 presents the sigmoid trend between the EPS and $\alpha$. This means that the contribution of the EPS on $\alpha$ can be divided into three different regions. First, the amount of EPS has no effects on $\alpha$ (below 20 mgEPS/gMLVSS). In second, the increasing of EPS results in the high value of $\alpha$. The specific cake resistance changes sensitively with the variation of EPS. At last, no more increasing of $\alpha$ is observed (above 80 mgEPS/gVSS). The EPS contribution on cake layer seems to be already reflected fully enough to increase more specific cake resistance value.



## Conclusions

As SRT decreases, the amount of EPS bound in sludge floc becomes higher. However, in the high MLSS condition (>5,000 mg/L), the bound EPS concentration is not sensitive to SRT. This phenomenon is attributed to the fact that the EPS itself can be the substrate for microbial growth. The bound EPS in the activated sludge floc is closely related with the membrane fouling. As the amount of bound EPS increases, the absolute value of TMP increases. It is concluded that as the amount of bound EPS increases, the absolute value of TMP increases. It is concluded that as the amount of bound EPS in the sludge floc is higher, the cake layer resistance increases more rapidly. The bound EPS affects the membrane fouling more than the dissolved organic matters does as long as the adsorption mechanism does not take place. As the bound EPS amount in the sludge floc increases, the specific cake resistance is higher. The sigmoid trend between the EPS and the specific cake resistance is observed and the functional relationship obtained by dimensional analysis is consistent with the experimental results.

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