Effects of hydrophilicity/hydrophobicity of membrane on membrane fouling in a submerged membrane bioreactor

Meijia Zhang a, Bao-qiang Liao b, Xiaoling Zhou a, Yiming He c, Huachang Hong a, Hongjun Lin a,⇑, Jianrong Chen a,1

a College of Geography and Environmental Sciences, Zhejiang Normal University, Jinhua 321004, PR China
b Department of Chemical Engineering, Lakehead University, 955 Oliver Road, Thunder Bay, Ontario P7B 5E1, Canada
c Department of Materials Physics, Zhejiang Normal University, Jinhua 321004, PR China

HIGHLIGHTS

• Membrane hydrophilicity is not directly relevant to interactions with flocs.
• Increasing surface charge highly raises electrostatic double layer interaction.
• Total interaction is continually repulsive when roughness is 300 nm.
• Strategy of improving membrane hydrophilicity cannot mitigate sludge adhesion.

GRAPHICAL ABSTRACT

Abstract

The interfacial interactions between sludge foulants and four different types of membranes were assessed based on a new combined calculation method. Effects of membrane surface hydrophilicity/hydrophobicity on the interfacial interactions were investigated. It was found that, membrane surface hydrophilicity/hydrophobicity was not directly relevant to the interfacial interactions with sludge particles. Increasing membrane surface zeta potential could significantly increase the strength of the electrostatic double layer (EL) interaction and the energy barrier. For membrane with a surface roughness of 300 nm, the total interaction was continuously repulsive in the separation distance coverage of 0–4 nm in this study. The results suggest that, under conditions in this study, designing membranes with a high zeta potential and certain roughness can significantly mitigate membrane fouling, whereas, the strategy of improving membrane surface hydrophilicity cannot alleviate sludge adhesion in the membrane bioreactor.

1. Introduction

Investigation of membrane fouling has been longstanding interest for the research community concerned with membrane bioreactor (MBR) technology since MBR was invented over four decades ago (Lesjean et al., 2011; Chen et al., 2012b; Wei et al., 2013). Numerous studies have indicated that sludge adhesion to form a cake layer is the main cause of membrane fouling in MBRs (Wang et al., 2007; Wang and Wu, 2009; Zhang et al., 2013). Therefore, considerable efforts have been made to understand and control sludge adhesion process in MBRs.

A number of factors, including hydrodynamic conditions, sludge properties and membrane properties, could affect the sludge adhesion process (Lin et al., 2014b). Hydrophilicity/hydrophobicity is a
primary membrane property. It is generally believed that hydrophilic membrane corresponds to lower membrane fouling potential than hydrophobic one (Kim et al., 2004; Weis et al., 2005; Santos and Judd, 2010). Based on this belief, improving membrane surface hydrophilicity was considered as an important strategy to mitigate membrane fouling in MBRs (Yu et al., 2005; Liu et al., 2013; Zhang et al., 2014a), although no solid theoretical base to support this belief. However, inconsistent results were also reported. For example, Choo and Lee (1996) found that the most hydrophobic polyvinylidene fluoride (PVDF) showed the smallest fouling tendency than the polysulfone (PSF) and cellulose acetate (CA) membranes. Chen et al. (2012a) reported that the flux decrease rate of the membranes followed the order CA > PVDF > polyether sulfones (PES) membranes, although CA membrane was most hydrophilic among them. The inconsistent results lead to the difficulty in understanding the exact roles of membrane hydrophilicity/hydrophobicity in sludge adhesion and membrane fouling. Literature analysis also showed that most of previous studies focused on the overall fouling behavior rather than the anatomized fouling steps such as sludge adhesion (Xiao et al., 2011).

Recent studies have reported that sludge adhesion to form a cake layer was mainly resulted from the interfacial interactions between sludge foulants and membrane surface (Feng et al., 2009; Hong et al., 2013; Su et al., 2013). While hydrodynamic forces forward the foulants close to membrane surface, it is the short-ranged interfacial interaction forces that are responsible for eventually binding the foulants to membrane surface (van Oss, 1995; Hong et al., 2013). The interfacial interactions comprised of Vander Waals (LW), electrostatic double layer (EL), and acid–base (AB) interaction could be generally described by the extended Derjaguin-Landau-Verwey-Overbeek (XDLVO) theory (van Oss, 1995). By using XDLVO approach, Wang et al. (2013) and Zhang et al. (2014b) presented a plausible explanation for the membrane fouling behavior caused by soluble microbial products (SMPs) and pH, respectively. Therefore, assessment of these interfacial interactions through XDLVO theory may provide a route map to track the exact roles of membrane hydrophilicity/hydrophobicity in sludge adhesion. However, most previous studies simply assumed an infinite smooth surface for the studied membranes (Feng et al., 2009; Wang et al., 2013). In fact, most commercial membranes used in MBRs were significantly varied in surface morphology or roughness (Mahendran et al., 2011; Chen et al., 2012a). It is expected that the interfacial interactions between sludge foulants and rough membrane surface are more complicated than those regarding smooth membrane surface. Fortunately, a novel method developed by the authors of this study allows to quantitative calculation of the interfacial interactions between sludge foulants and rough membrane surface (Lin et al., 2014a). A quantitative assessment of these interfacial interactions would shed significant lights on sludge adhesion and membrane fouling. Nevertheless, neither study assessed the effects of membrane hydrophilicity/hydrophobicity on the interfacial interactions between sludge foulants and rough membrane surface in MBRs.

This study aims to theoretically and experimentally assess the effects of membrane hydrophilicity/hydrophobicity on the interfacial interactions and sludge adhesion process in an MBR. A submerged MBR (SMBR) setup operated at stable period was continuously run to supply the sludge samples. A series of membranes with known surface properties were adopted for the calculation of the interfacial interactions.

2. Methods

2.1. Experimental setup

A lab-scale submerged MBR (SMBR) treating synthetic municipal wastewater was continuously run for over 200 days. The SMBR contained a reactor with 60 L effective volume, where a flat sheet PVDF membrane model with 0.1 m² effective filtration area was vertically located. Air flow rate and membrane flux were about 180 m³/h/m² and 30 L/m² h, respectively. The sludge retention time (SRT) was approximately 45.5 d. The synthetic municipal wastewater used in this study possessed a composition as follows: 300 mg COD/L glucose plus the following mineral medium: (NH₄)₂SO₄ (27 mg N/L); KH₂PO₄ (7 mg P/L); Na₂CO₃ (23 mg Na/L); Na₂HPO₄ (46 mg Na/L); MgSO₄ (7 mg Mg/L); CaCl₂ (6 mg Ca/L); FeCl₃ (4 mg Fe/L); ZnCl₂ (0.11 mg Zn/L); MnSO₄ (0.04 mg Mn/L); CuSO₄ (0.03 mg Cu/L); CoCl₂ (0.1 mg Co/L) and Na₂MoO₄ (0.02 mg Na/L). In this study, activated sludge obtained from a sequencing batch reactor (SBR) for real municipal wastewater treatment was used as inoculum. The sludge samples obtained during stable operation period of SMBR were used for interfacial interaction assessment.
2.2. Analytical methods

To ensure the representative of the sludge samples for measurements, the sludge suspension in the MBR during the stable operation period was used as sludge samples. Four types of membranes (CA, PES, PVDF and PP membrane) were used in calculation of the interfacial interactions. The surface properties of these membranes including surface tensions and zeta potential have been reported in previous studies (Feng et al., 2009; Chen et al., 2012a).

For measuring the contact angle of sludge foulants, sludge lawn was first prepared by filtering sludge suspension. The sludge lawn was fixed within two glass slides to form a relatively flat surface, and then dried in a desiccator for 24 h to remove surplus water. Static contact angles of the probe liquids on the sludge samples were measured by using a contact angle meter (Kino industry Co., Ltd, USA) based on the sessile drop method. Determination of the surface tension components of a solid substance needs three probe liquids. Among them, two should be polar and one should be apolar. The polar liquid is used to calculated the non-polar component of surface tension (van Oss, 1995). Moreover, high energy (polar and apolar) liquids are recommended to produce larger, more easily measured contact angles (van Oss, 1995). For these reasons, three liquids including ultrapure water, glycerol and diiodomethane were chosen as probe liquids in this study.

The zeta potential of the sludge foulants was measured according to the electrophoretic mobility method using a zetasizer nano analyzer (Malvern, UK). The measurements were conducted in the 0.01 mol/L NaCl solution. The membrane surface zeta potential was determined by a microelectrophoretic instrument (zeta 90 plus particle size analyzer, Brookhaven, UK). A Malvern mastersizer 2000 instrument was used to measure the particle size distribution (PSD) of the sludge suspension. Measurements were conducted at least in triplicate for each sample. The organics in supernatant were considered to be mainly SMPs because of the easy degradability of influent organics. SMPs were normalized as the sum of proteins and polysaccharides which were colorimetrically measured using phenol/sulfuric acid method and Folin method, respectively.

2.3. XDLVO approach

The LW, AB and EL interaction energy per unit area between two infinite planar surfaces at separation distance \( h \) \( (\Delta G^{\text{LW}}(h), \Delta G^{\text{AB}}(h) \text{ and } \Delta G^{\text{EL}}(h)) \) are given by the following equations in XDLVO theory (van Oss, 1995).

\[
\Delta G^{\text{LW}}(h) = \Delta G^{\text{LW}}_0 \frac{h^2}{h^2}
\]

\[
\Delta G^{\text{AB}}(h) = \Delta G^{\text{AB}}_0 \exp \left( \frac{h_0 - h}{\lambda} \right)
\]

\[
\Delta G^{\text{EL}}(h) = \kappa_c \gamma_{W} - \kappa_s \gamma_{W} + \frac{2 m_f}{2 m_f} \ln(1 - \coth(\kappa h)) + \frac{1}{\sinh(\kappa h)}
\]

where, \( h_0 = 0.158 \text{ nm} \) is the minimum separation distance where two planar surfaces are assumed to contact each other; \( \Delta G^{\text{LW}}_0, \Delta G^{\text{AB}}_0 \text{ and } \Delta G^{\text{EL}}_0 \) are the LW, AB and EL interaction energy per unit area between two infinite planar surfaces in contact, respectively. \( \Delta G^{\text{LW}}_0, \Delta G^{\text{AB}}_0 \text{ and } \Delta G^{\text{EL}}_0 \) can be obtained according to Eqs. (4)–(6), respectively (van Oss, 1995).

\[
\Delta G^{\text{LW}}_0 = -2 \left( \sqrt{\gamma^{\text{LW}}_m} - \sqrt{\gamma^{\text{LW}}_w} \right) \left( \sqrt{\gamma^{\text{LW}}_i} - \sqrt{\gamma^{\text{LW}}_w} \right)
\]

\[
\Delta G^{\text{AB}}_0 = 2 \left( \sqrt{\gamma^{\text{AB}}_m} + \sqrt{\gamma^{\text{AB}}_w} \right) \left( \sqrt{\gamma^{\text{AB}}_i} + \sqrt{\gamma^{\text{AB}}_w} \right)
\]
of the differential circular arc in the circular ring; \( f(r, \theta) \) is local amplitude directly below the circular arc as a function of the position of the differential circular arc defined by \( r \) and \( \theta \). In practice, it is basically impossible to get the antiderivative of the double integrals shown in Eqs. (12)–(14), because \( f(r, \theta) \) is generally complicated. Therefore, the interactions with rough surface cannot be precisely calculated. This is probably the reason why the interactions between a particle and rough membrane surface have not been quantitatively evaluated so far in previous studies, although SEI method has been established since 1997 (Bhattacharjee and Elimelech, 1997). By adopting the composite Simpson’s rule, the method developed in this study allowed to numerically estimate these double integrals, and thus could quantitatively evaluate the interactions with rough surface. In the composite Simpson’s rule, certain point \( x_i = a, x_i = x_1 + ih \) \( (i = 1, 2, ..., 2m + 1) \) and \( y_j = b, y_j = y_1 + jk \) \( (j = 1, 2, ..., 2n + 1) \) were used to subdivide the interval \([a, b]\) of variable \( x \) and the interval \([c, d]\) of variable \( y \) in a double integral, respectively. Where, \( h = (b-a)/2m \), and \( k = (d-c)/2n \). Defining \( f_{ij} \) as the function \( f(x_i, y_j) \), the double integral can be estimated by Eq. (15):

\[
\int_a^b \int_c^d f(x,y) \, dx \, dy = \sum_{i=1}^{m} \sum_{j=1}^{n} \left( \frac{f_{i-1,j} + 4f_{i,j-1} + f_{i,j} + f_{i-1,j+1}}{6} \right) h \cdot k
\]

where \( m \) and \( n \) are the number of segments for the variable interval of \( x \) and \( y \), respectively. The calculations were performed in MATLAB software. It was verified that the calculation error was negligible by setting \( m = n = 2000 \) or above in this study.

### 3. Results and discussion

#### 3.1. Interfacial properties of the sludge foulants and membranes

Various sludge samples were taken from the MBR reactor during the stable operation period (from day 68 to day 198). In such a period, the sludge properties were quite stable, which corresponded to a low variability. 6 sludge samples were subjected to contact angle and zeta potential measurements, and the average values were used to calculate the surface tensions based on the Young’s equation. The results are present in Table 1. The sludge samples had relatively high \( \gamma^\circ \) component and absolute value of zeta potential. The data of sludge samples are comparable with those reported by other researchers (Feng et al., 2009; Su et al., 2013), indicating the representativeness of the sludge samples in this study for MBRS. The sludge suspension in the MBR had a LW of about 10\( ^m \), which corre-

<table>
<thead>
<tr>
<th>Materials</th>
<th>( \gamma^{SW} ) (mJ m(^{-2}))</th>
<th>( \gamma^\circ ) (mJ m(^{-2}))</th>
<th>( \gamma^- ) (mJ m(^{-2}))</th>
<th>( \Delta \gamma_{sw} ) (mJ m(^{-2}))</th>
<th>Zeta potential (mV)</th>
<th>References</th>
</tr>
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<tbody>
<tr>
<td>Sludge foulants</td>
<td>37.90</td>
<td>0.08</td>
<td>18.02</td>
<td>-19.36</td>
<td>-23.0</td>
<td>This study</td>
</tr>
<tr>
<td>PES membrane</td>
<td>41.18</td>
<td>0.19</td>
<td>3.01</td>
<td>-55.76</td>
<td>-17.4</td>
<td>Chen et al. (2012a)</td>
</tr>
<tr>
<td>PP membrane</td>
<td>24.02</td>
<td>0.11</td>
<td>1.74</td>
<td>-70.39</td>
<td>-15.8</td>
<td>Feng et al. (2009)</td>
</tr>
<tr>
<td>PVDF membrane</td>
<td>45.4</td>
<td>0.12</td>
<td>2.23</td>
<td>-71.19</td>
<td>-15.5</td>
<td>Chen et al. (2012a)</td>
</tr>
</tbody>
</table>

For simplification, smooth surface was first assumed for the four membranes. In order to exclude the effects of zeta potential on the interfacial interactions, same zeta potential value of 17.4 mV was used in the calculation. Fig. 1 shows the profiles of the interfacial interactions between a sludge foulant (floc) and the smooth surface of the four membranes. For all the four membranes, the LW and AB interactions are continuously attractive, and the EL interaction is continuously repulsive. With the decrease in the separation distance, the total interaction shows a three-stage profile characterized by a slow increase followed by a repulsive energy barrier and a rapid decrease. The profile of the total interaction suggests that the successful adhesion of a sludge floc on the membrane surface should surmount an energy barrier. As shown in Fig. 1, membrane surface hydrophobicity could affect the strength and distribution of LW and AB interactions. For example, when separation distance is at 4 nm, the AB interaction is \(-640, -619, -700\) and \(-730\) kT for CA, PES, PVDF and PP membrane, respectively. However, no obvious effect of hydrophobicity on the total interaction could be drawn. The energy barrier for the four membranes follows the order: PP > PES > PVDF > CA. While PP membrane is much more hydrophobic than PES and CA membranes, it is most difficult to be fouled by the foulant adhesion based on the interfacial interaction calculation. This result indicates that, membrane surface hydrophobicity/hydrophobicity may not be a fundamental factor influencing the foulant adhesion on smooth membrane surface. It is worth noting that, in order to exclude the effects of other factors, a general treatment for investigating effect of a factor on membrane fouling is to set other factors at a constant value. Therefore, in this study, the calculations were all performed by treating the foulant as a single species although the activated sludge particles (flocs) in MBR is of complexity.

It is generally believed that improving the membrane surface hydrophilicity is an important strategy to mitigate membrane fouling in MBRS (Yu et al., 2005; Liu et al., 2013; Zhang et al., 2014a). Meanwhile, it has long been suspected that, membrane fouling behavior is irrelevant to the membrane surface hydrophilicity/hydrophobicity in MBRS (Choo and Lee, 1996; Chen et al., 2012a). However, both sides of the argument were based on observations of limited experiments or even intuition rather than theoretical derivations. This study quantitatively analyzed the interfacial interactions between foulants and membrane surface based on XDLVO theory, thus providing a more solid conclusion regarding the effects of membrane surface hydrophilicity on membrane fouling in MBRS.

The membrane surface zeta potential could be significantly varied with the membranes used in MBRS. For example, the membrane...
used in the MBR in this study possessed a surface zeta potential of \(-31.0\) mV. Fig. 2 shows the profiles of the interfacial interactions under conditions of membrane surface zeta potential = \(-31.0\) mV.

The profiles in Fig. 2 are similar to those in Fig. 1. However, the strength of EL and total interaction is significantly different from Fig. 1. For all the four membranes, when membrane surface zeta potential...
potential increases from −17.4 to −31.0 mV, the strength of energy barrier increases for over 2 times, and the strength of the attractive stages is weaken. As EL interaction stems from surface charge, and provides the repulsive force impeding foulant adhesion, it can be concluded that, increasing membrane surface charge is much more important than improving membrane surface hydrophilicity for membrane fouling control in MBRs.

3.3. Effects on interfacial interactions with rough membrane surface

The interfacial interactions between foulants and rough membrane surface are much more complicated than those with a smooth membrane surface. The rough membrane surface shows the morphology in the form of a series of asperities and valleys (Fig. 3). Basically, there are three possible interaction scenarios associated with foulants (Fig. 3). For the soluble foulants such as SMPs in MBRs (Fig. 3(a)), rough membrane surface means the increased interacting surface area because SMPs are considered as chain-like substances, and are flexible enough to fit membrane surface morphology when they interact with each other. This suggests that rough membrane surfaces would increase the strength of interactions with soluble foulants. Chen et al. (2012a) reported that rough surfaces favor the deposition of soluble compounds. For the particle foulants, the interaction profiles would depend on the particle size (Fig. 3(a) and (c)). In this study, the SMPs in supernatant during the stable operation period was averaged at 8.8 ± 3.7 mg/L (6.7 ± 3.5 mg/L proteins and 2.1 ± 0.6 mg/L polysaccharides) (10 measurements in 130 days), and only accounted for about 0.1% of the total biomass of sludge suspension. This indicated that membrane fouling caused by SMPs was not significant. The PSD of the sludge suspension and the roughness of the membranes were in the range of 1.5–564 nm and 100–300 nm, respectively. The mismatch in size between foulants and roughness, together with the low SMPs content, suggested that the main form of interfacial interactions fell into the scenario shown in Fig. 3(c).

Fig. 4(a)–(c) shows the schematics of a sludge foulant particle on the top of a rough membrane surface. Although the randomness of surface roughness precludes a rigorous mathematical model describing surface morphology, it is possible to propose a hypothetical surface topology that represents some pertinent statistical properties of the rough membrane surface. Fig. 4(d) shows the schematic of an asperity on membrane surface. As suggested in Fig. 4(d), it may be reasonable to implement a sine function as the surface defining function for the roughness. Accordingly, \( f(r, \theta) \) could be described by Eq. (16). Some previous studies also suggested a similar equation for \( f(r, \theta) \) (Whitehead and Verran, 2006; Verran et al., 2010).

\[
  f(r, \theta) = s \sin(\pi r \cos \theta / 2a + \varphi)
\]

Fig. 5 shows the profiles of the interaction energies between a foulant particle and the rough surface of the four membranes. As compared with the data in Fig. 1, the strength of all the three interactions and total interaction is dramatically decreased for more than 10 times for the rough membrane surface. Furthermore, the interactions for the rough membrane surface are more short-ranged than those for smooth membrane surface. The energy barrier for the four membranes follows the order: PP > PES > CA > PVDF. Whereas, the absolute value of interaction energy in contact (separation distance = 0.158 nm) for the four membranes follows the order:
PP > PVDF > CA > PES. These results also suggest the irrelevance of the membrane surface hydrophilicity/hydrophobicity to the adhesion of foulant particle in the MBR.

When the membrane surface roughness is changed from 100 nm to 300 nm, the profiles of the interaction energies are also significantly changed (Fig. 6). It is interesting to note that, for all the four membranes, the total interaction is continually repulsive when the separation distance is in the range of 0–4 nm. This means that the membrane surface with such a roughness has a strong ability of anti-adhesion for the sludge particle foulants in this study. Designing a membrane with strong anti-adhesion ability has been the long-lasting objective for the research community. The study suggested that this objective could be achieved by designing membrane surface roughness. Furthermore, the total energy in contact (separation distance = 0.158 nm) for the four membranes is repulsive, and follows the order: PP > PES > CA > PVDF. Again, no direct correlation between membrane surface hydrophilicity/hydrophobicity and interaction energies can be found in this case. These results indicated that designing membrane surface roughness is much more important than improving membrane surface hydrophilicity for membrane fouling mitigation in MBRs.

To date, extensive efforts have been devoted into improving membrane surface hydrophilicity in order to mitigate membrane fouling in MBRs (Yu et al., 2005; Liu et al., 2013; Zhang et al., 2014a). Improved hydrophilicity of membrane can be achieved by the modification measures including coating, blending and grafting (Kang and Cao, 2014). Some studies claimed that the improved hydrophilic membranes could improve anti-fouling ability in MBRs (Yu et al., 2005; Zhang et al., 2008; Liu et al., 2013). However, these studies did not measure the changes of other membrane surface properties (such as zeta potential and roughness) caused by membrane modification. According to this study, changes of membrane surface zeta potential and roughness rather than hydrophilicity may be the exact causes of the improved anti-fouling ability in MBRs. Based on the theoretical and experimental assessment of the interfacial interactions, the exact roles of membrane hydrophilicity in foulant adhesion in MBRs were explored in this study. It was also suggested that, the strategy of improving membrane surface hydrophilicity cannot mitigate sludge particle adhesion in MBRs.

### 3.4. Experimental evidence for the findings

Some experimental results in literature can verify the findings in this study. One direct evidence comes from the study of Chen et al. (2012a), where the flux decrease rate of the membranes followed the order CA > PVDF > PES membranes by using the membranes same to this study. In nature, the front surface of lotus leaf has attracted considerable attention due to its super anti-adhesion ability. Lots of studies reported that the super anti-adhesion ability was not related with the bulk hydrophobicity, but mainly resulted from the surface morphology (Wang et al., 2009; Zhang et al., 2012). Observation through electron microscope showed that the front surface morphology of lotus leaf was similar to the hypothesized membrane surface morphology in this study (Wang et al., 2012a). By simulating the lotus leaf surface structure, some materials with high anti-adhesion ability have been fabricated (Wang et al., 2014; Zhang et al., 2014a). Swain and Andelman (1999) also reported that the adhesion energy with rough surfaces decreased as compared with planar surfaces. Bani-Melhem and Elektorowicz (2011) reported that applying an intermittent direct current (DC) field between immersed circular perforated electrodes around a membrane filtration module could significantly reduce membrane fouling. All of these studies demonstrated the unimportance of surface hydro-
philicity, and the importance of surface roughness and surface charge for adhesion of foulant particles. However, the conventional adhesion mechanisms, where the surface hydrophilicity/hydrophobicity takes a decisive role, cannot explain these experimental phenomena. By quantitatively evaluating the interfacial interactions between foulant and membrane surface in this study, these phenomena could be satisfactorily explained, and, on the other hand, the phenomena serve as the solid experimental evidence for the conclusions obtained in this study.

It should be noted that current conceptions regarding surface hydrophobicity do not include the curvature (morphology) of the two contacting surface. If the two contacting surfaces are infinite planar, surface hydrophilicity should have an important effect on the interaction strength between two same planar surfaces in contact (van Oss, 1995). This is probably the reason why many studies believed or observed the importance of hydrophobicity in membrane fouling. However, for the interactions between a spherical particle and membrane surface, this conclusion will be changed as indicated in this study. In this regard, the opposite results in literature could also be satisfactorily explained.

4. Conclusions

This study theoretically and experimentally investigated effects of membrane hydrophilicity/hydrophobicity on the interfacial interactions between particle foulants and membranes using a new combined calculation method. The results showed the irrelevance of the membrane surface hydrophilicity/hydrophobicity to the total interfacial interaction with large foulant particles. As compared to membrane hydrophilicity, membrane surface zeta potential and roughness exerted more important effects on these interactions under conditions in this study. It was suggested that, the strategy of improving membrane hydrophilicity cannot mitigate particle adhesion, whereas, designing membranes with high zeta potential and certain roughness may significantly alleviate membrane fouling in MBRs.

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