

## Mechanism of membrane fouling control in the magnetic activated sludge (MAS) process

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### ABSTRACT

The components of activated sludge liquor are a primary cause of membrane fouling. In this study, magnetic powder ( $\text{Fe}_3\text{O}_4$ ) was added to a membrane bioreactor (MBR) to form a magnetic activated sludge (MP-MBR). The addition of 650 mg/L magnetic powder to MBR extended the membrane operation period, and the cleaning period was 3 d longer in the MP-MBR than the control MBR. The addition of magnetic powder to the MBR process reduced membrane fouling by decreasing the content of soluble microbial metabolites (SMPs) and extracellular polymeric substances (EPSs) content, decreasing the zeta potential of the activated sludge, and increasing the average sludge particle size.

*Keywords:* MBR; Membrane fouling; Magnetic powder; EPS; Sludge particle size

### 1. Introduction

Membrane bioreactors (MBRs) are widely used for the treatment of municipal and industrial wastewater [1,2]. However, the performance of MBR filtration inevitably decreases with filtration time, and membrane fouling is a major obstacle to the expanded application of MBRs [3,4]. The characteristics of the activated sludge mixed liquor have a significant impact on membrane fouling, including biological flocs formed by a large range of living or dead microorganisms and soluble and colloidal compounds [5,6]. Adding coagulants, adsorbents or flocculants can improve the characteristics of activated sludge and alleviate membrane fouling [7,8].

As an adsorbent or flocculant, magnetic particles have attracted increasing attention in studies of bio-degradation processes, particularly activated sludge processes [9–11]. Magnetic activated sludge processes are a modification of the conventional activated sludge process in which ferromagnetic powder ( $\text{Fe}_3\text{O}_4$ ) is added to create a magnetic

activated sludge [12]. The magnetic particles can be applied as an adsorbent or flocculant to improve the removal of organics. Sakai [13] observed that the addition of magnetic powder in activated sludge helped prevent activated sludge expansion and enhance removal performance. Ren [14] concluded that nano-magnetic particles strengthen the structure of microbial flocculation and increase pollutant removal ability by enhancing microbial activity. Lin [15] discussed the performance of magnetic powder activated sludge in industry wastewater treatment and noted that the addition of magnetic powder to the sludge enhances performance with respect to sedimentation, microbial activity and the concentration of pollutants. Similarly, Chen [16] observed a good sedimentation effect and high degradation efficiency of organic pollutants in magnetic granular sludge. Ni [17] determined that magnetic powder has a positive effect on the properties of activated sludge and a negative effect on extracellular polymeric substances (EPSs) under an impact load.

The performance of magnetic powder has been evaluated in previous studies [13,16], but the effects of magnetic powder on the mixed liquor properties in MBR, which greatly impact

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fouling mitigation, remain unclear. In this study, magnetic powder was added during the MBR process to form magnetic activated sludge, and the effects of magnetic powder on membrane fouling were examined. Possible mechanisms involved in membrane fouling reduction were also observed and examined by determining the transmembrane pressure (TMP) and the characteristics of the active sludge.

## 2. Materials and methods

### 2.1. Operation of MBRs

The MBR process for oily wastewater treatment involves a continuous operation mode. Magnetic powder was added to the MBR to initiate the MP-MBR process. We studied the MBR with and without magnetic powder using the experimental apparatus shown in Fig. 1.

The MBR process was used for oil wastewater treatment with the following operation: raw oily water was pumped from the raw water tank into the MBR process. The COD<sub>Cr</sub> of the oily wastewater was approximately 500 mg/L, and the oil concentration was controlled at 50 mg/L. An electronic timer was used to control the water pump, which was run for 8 min and then stopped for 2 min. A vacuum gauge in the water pipe was used to monitor the membrane pressure; the diaphragm was cleaned when the TMP reached the critical value of 50 kPa. The MBR process operated at a constant flux of 16.2 L/(m<sup>2</sup>·h). Continuous cycle aeration was used, and the total aeration rate was 33 L/min. The hydraulic retention time (HRT) was 6 h. The temperature in the reactor was approximately 25°C, and the pH was maintained within a range of 6.5–7.5.

Activated sludge was collected from the secondary clarifier of the Daqing oil sewage treatment plant, Heilongjiang, China. The mixed liquor suspended solids (MLSS) concentration was maintained constant at approximately 16000 mg/L.

A frame plate membrane module from Shanghai Snapp Membrane Separation Technology Co., Ltd., was used. The membrane was composed of polyvinylidene fluoride (PVDF) and had a total area of 0.1 m<sup>2</sup> and a normal pore size of 0.1 μm.

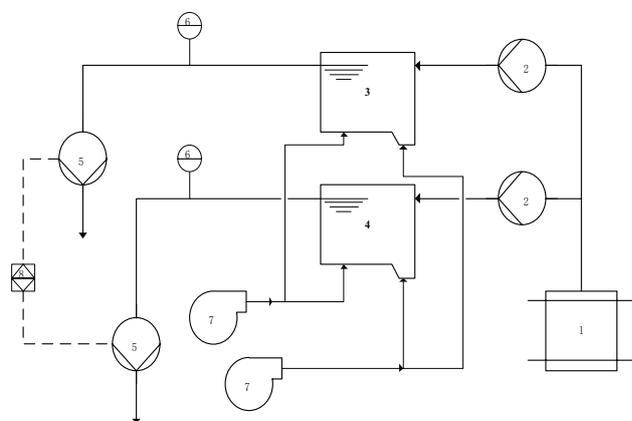


Fig. 1. Schematic of MBRs used for oily wastewater treatment. Raw water tank; 2. Feed pump; 3. MP-MBR; 4. MBR; 5. Effluent pump; 6. Vacuum gauge; 7. Blower; 8. Electronic timer.

Fe<sub>3</sub>O<sub>4</sub> powder, which is a common magnetic material with weak magnetic properties and strong adsorption ability for its surface area, was used in this study. The powder size range was 1 μm to 12 μm, with 50% within the size range of 2 to 4 μm. The magnetic powder was pretreated as follows: 20 g of magnetic powder (with an Fe<sub>3</sub>O<sub>4</sub> content greater than 98%) was added to a 1-L beaker and mixed with deionized water. The magnetic powder was settled for 30 min using a round magnet placed on the bottom of the beaker, and the supernatant was removed. The settling step was repeated twice, and the beaker was then placed in an oven at 105°C for 24 h. The magnetic powder was added to a 1-L beaker and placed on a permanent magnet for 30 min to magnetize it before addition to the MBR process [18].

The dosage of magnetic powder added to the MBR process was determined by a jar test similar to that described by Wang [19]: 100 mL of oil wastewater was treated with different concentrations of magnetic powder from 0 mg/L to 1250 mg/L. After mixing by agitation at 150 rpm for 24 h and standing for 30 min, the COD<sub>Cr</sub>, sludge settling ratio, and oil removal rate were analyzed. The optimum dosage of 650 mg/L was used in subsequent experiments.

### 2.2. Experimental methods

The zeta potential of the supernatant was measured using a Zeta-meter (ZetaSizer3000, England). The sludge particle size was determined using a laser particle size analyzer (MasterSizer, UK, Malvern). The organic matter contents was analyzed using a fluorescence spectrophotometer (FP-6500, JASCO, Japan).

Proteins and polysaccharides are the largest components of EPSs and thus can be used to infer EPS content. Polysaccharides were measured using the anthrone sulfuric acid method; proteins were measured using ultraviolet visible spectrophotometry.

## 3. Results and discussion

After several months of operation, both MBR processes exhibited stable oil removal performance. Magnetic powder (650 mg/L) was added to one of the MBR reactors to form magnetic activated sludge, hereafter referred to as the MP-MBR process. Both reactors were operated under the same conditions (detailed in Section 2.1). The changes in TMP and the characteristics of the activated sludge in both processes were investigated.

### 3.1. Effect of magnetic activated sludge on TMP changes

TMP is an important factor that can be used to evaluate system performance. At a constant flow rate, the TMP is directly related to the rate of membrane fouling. The discrepancy in TMP between the MP-MBR and MBR is illustrated in Fig. 2.

In this study, whenever the TMP exceeded 50 kPa, the fouled membranes were cleaned using a chemical method. As shown in Fig. 2, the control MBR required a cleaning period after 13 d of operation, 3 d sooner than MP-MBR. The rate of membrane fouling was expressed by the change in transmembrane pressure per unit time, defined in units

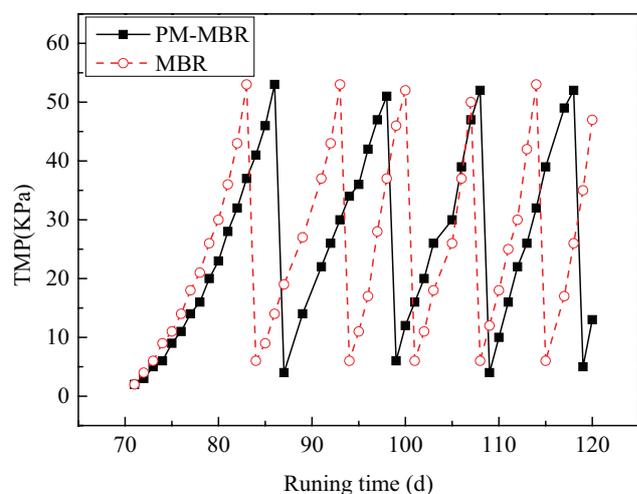


Fig. 2. Membrane fouling rates in the MP-MBR and MBR processes.

of kPa/d. Compared with the control MBR, the average fouling rate of TMP was decreased in the MP-MBR. Taking the first operation as an example, the average fouling rate of TMP was 3.31 kPa/d, which is 23% lower than in the MBR process.

The significantly lower fouling rate in the MP-MBR indicates that integrating magnetic powder into the MBR system is a promising means of alleviating the negative effects of membrane fouling, thereby prolonging operation periods for MBRs. Liu [19] observed that the membrane-fouling rate was lower in MBRs filled with aerobic granular sludge including magnetic seed than in MBRs filled with conventional activated sludge. Similarly, Chen [16] observed that microbial species were more numerous in an MBR reactor and that the addition of magnetic particles decreased the membrane-fouling rate. To better understand the mechanism by which the addition of magnetic powder slows the rate of membrane fouling, the characteristics of the activated sludge were analyzed

### 3.2 Changes in organic matter in the MP-MBR and MBR processes

The changes in organic matter in both bio-treatment processes were analyzed. Samples of raw oily water, sludge mixture and effluent water from both reactors were filtered using qualitative filter paper. Next, the samples were analyzed using a three-dimensional fluorescence spectrophotometer, a useful spectral fingerprint technology used in fields ranging from environmental inspection to biochemical analysis. Five indicator organic compounds were measured in the samples: tyrosine, tryptophan, fulvic acid, SMPs and humic acid. The amounts of these indicators in the samples were calculated using the volume integral method [20]. Fig. 3 shows the results of three-dimensional fluorescence analysis for the various organic compounds.

In raw water, the fluorescence intensities of tyrosine, tryptophan, fulvic acid, SMPs and humic acid were  $1.3 \times 10^6 \text{ AU}\cdot\text{nm}^2$ ,  $2.2 \times 10^6 \text{ AU}\cdot\text{nm}^2$ ,  $2.3 \times 10^6 \text{ AU}\cdot\text{nm}^2$ ,  $9.6 \times 10^6 \text{ AU}\cdot\text{nm}^2$ ,  $12.4 \times 10^6 \text{ AU}\cdot\text{nm}^2$ , respectively, and both MBR

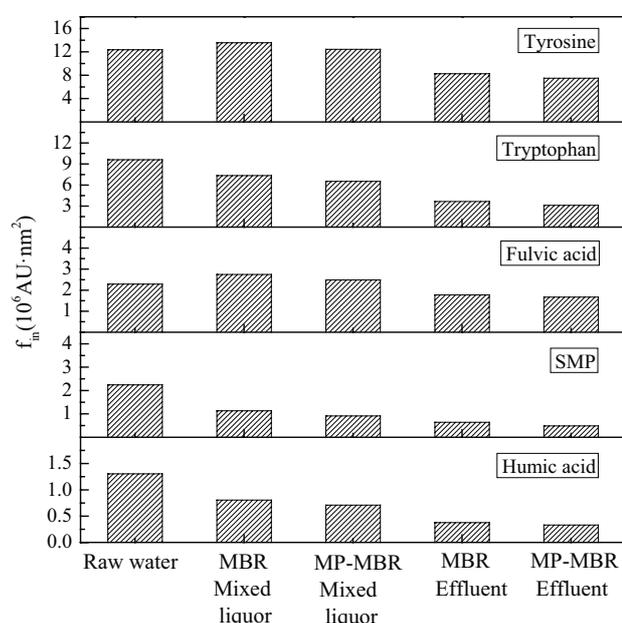


Fig. 3. Three-dimensional fluorescence spectrum analysis for the MP-MBR vs. the MBR process.

processes exhibited good performance for the removal of these organics. As shown in Fig. 3, the removal rates of tyrosine, tryptophan, fulvic acid, SMPs and humic acid in the effluent were, respectively, 74.7%, 78.2%, 67.8%, 39.3%, and 71.4% for the MP-MBR and 71.1%, 22.6%, 27.1%, 62%, and 33.1% for the MBR. These results demonstrate that the MP-MBR removed organic matter more efficiently. The membrane has a significant effect on pollutant removal, and the removal rates of the above organic compounds by the membrane were, respectively, 29.1%, 18.8%, 35.7%, 35.6%, and 39.9% for the MP-MBR and 32.9%, 21.8%, 38.3%, 42.6%, and 42.9% for the MBR. The removal of organics by membrane filtration was lower in the MP-MBR than in the MBR, probably because the content of organics on the MP-MBR membrane was lower.

Analysis of the activated sludge liquor samples revealed accumulation of fulvic acid and humic acid in the reactors. For the MP-MBR, the amounts of fulvic acid and humic acid were 8.6% and 0.6%; these quantities were much higher in the MBR, at 20% and 9.8%, respectively. The contents of tyrosine, tryptophan, fulvic acid, SMPs and humic acid were lower in the MP-MBR activated sludge liquor sample (19.5%, 12%, 9.5%, 11.2% and 8.4%, respectively) than in the MBR sample.

EPSs are used as construction materials for microbial aggregates, including biofilms, flocs and activated sludge liquors. Many studies have indicated that EPSs play a major role in fouling [21]. Proteins and polysaccharides are the major components of EPSs [22] and were measured as an indication of the amount of EPSs. The changes in EPS concentration are shown in Fig. 4.

Fig. 4 shows that the EPSs are composed of polysaccharides and proteins, with a high percentage of polysaccharides. The EPS concentration in the activated sludge liquor of both reactors followed the same trend, with the EPS

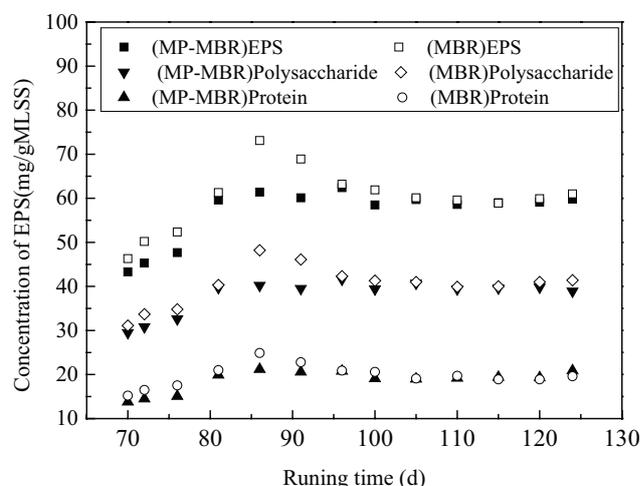


Fig. 4. Change in EPS concentration in the MBR vs. the MP-MBR process.

concentration increasing and then decreasing slowly over time. The maximum EPS concentrations in the MBR and MP-MBR were 73.1 mg/g MLSS and 62.4 mg/g MLSS at 96 d and 86 d, respectively. EPSs are mainly derived from an increase in substrate and consequent stimulation of microbial cells. Normal growth and metabolism can also produce EPSs under conditions of no external pressure. Initially, the sludge concentration in the reactor was low, and the sludge load was high; the major organic compounds in this experiment were easy to degrade, favoring full degradation of the substrate and increasing the microbial metabolism of EPSs. As time increased, the sludge growth rate in the reactor also increased, while the usable matrix and the load decreased. Having no other source of energy, the microorganisms began to consume EPSs, and thus the concentration of EPSs decreased.

SMPs and EPSs play important roles in membrane fouling [23,24]. In this study, the addition of magnetic powder caused the concentration of SMPs and the maximum EPS concentrations to decrease by 11.2% and 5.4%, respectively. This result indicates that the magnetic powder helped absorb the organic substances. The majority of the magnetic powder particles ranged in size from 2 to 4  $\mu\text{m}$ , providing a large surface area for adsorption in wastewater. In the MBR, these adsorbents had the potential to adsorb dissolved organic polymers, notably SMPs, hence reducing the membrane-fouling propensity. Xing [25] determined that the adsorption of AR73 was a fast physical adsorption on the surface of nano-magnetic particles and was consistent with a pseudo-second order kinetic equation. Yeana [26] evaluated the effect of magnetite particle size on adsorption and observed that the maximum adsorption capacities for arsenite and arsenate increased with decreasing magnetite particle size. Other adsorbents can also be applied in MBR processes to reduce organic fouling and biofouling; powdered activated carbon (PAC) can serve as a medium for bacterial attachment and subsequent growth, hence reducing attachment at the membrane surface and pores [27].

In addition, the microorganisms were more active under the weak magnetic field, and the organic materials

were degraded more completely [28]. As shown in Fig. 3 and Fig. 4, the variations of SMPs and EPSs in the MP-MBR were similar to those in the MBR. In general, the SMP and EPS concentrations were lower in the MP-MBR than in the MBR. Some studies have reported that microbial cell enzyme activity is enhanced under a weak magnetic field. Nan [29] added magnetic powder to a bio-reactor to treat 120–130 mg/L phenol wastewater, and the results showed that the biofilm colonization time of the magnetic powder-containing bio-reactor was decreased by 1–2 d and that the biomass increased compared to the bioreactor without magnetic powder. Tu [30] demonstrated that the addition of magnetic powder improved the biodegradation removal rate for copper, and the maximum copper removal rate increased by 23.4% when the  $\text{Fe}_3\text{O}_4$  concentration was 4 g/L. In this study, the addition of magnetic powder created a weak magnetic field that increased the microorganisms' ability to metabolize SMPs and EPSs, leading to lower SMP and EPS concentrations in the MP-MBR than in the MBR, which reduced the interception of the membrane and slowed the rate of membrane fouling [31].

### 3.3 Changes in sludge particle size in the MP-MBR and MBR processes

The sludge particle size is related to membrane filterability, which can be reduced by small floc particles [32]. During the stable operation stage, samples of the sludge mixture were withdrawn from the MP-MBR and MBR reactors, and the particle size was measured using a laser particle size analyzer. The results are shown in Fig. 5.

As shown in Fig. 5, the ranges and distributions of the particle sizes of the activated sludge differed significantly between the MBR and MP-MBR. In the MP-MBR, 59.5% of sludge particles were larger than 80  $\mu\text{m}$ , 24.7% higher than in the MBR. This result indicates that the sludge particles were larger in the MP-MBR than in the MBR.

Some studies have shown that the resistance of the cake layer is related to the size of the sludge particles in the reactor. The practical implication is that the larger the floc size, the better the fouling reduction. Fig. 5 indicates that 49% of

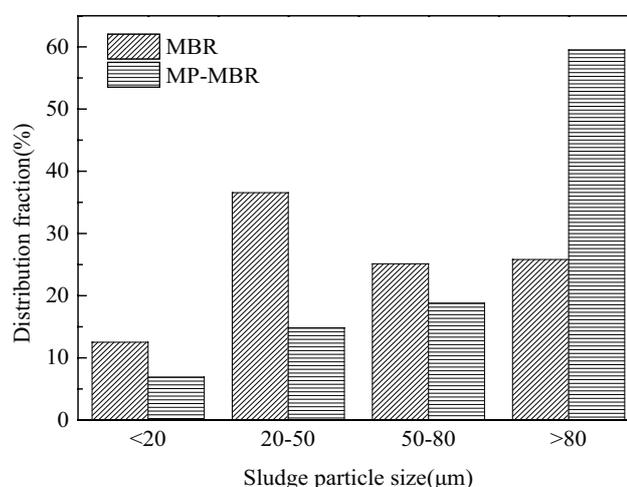


Fig. 5. Particle size distribution of the activated sludge in the MBR versus the MP-MBR process.

the particles in the MBR were smaller than 50  $\mu\text{m}$ , which is 27.4% higher than in the MP-MBR. Lu [33] also observed improvement of the floc structure and settling characteristics of the sludge, with a reduced likelihood of sludge bulking, when ferromagnetic powder ( $\text{Fe}_3\text{O}_4$ ) was added to the aerobic tank of the conventional activated sludge process. This improvement occurred because the surface charge and surface area properties of the magnetic particles enabled the creation of a scaffold and unification with Zoogloea. Microorganisms tend to aggregate and form flocs in biological wastewater treatment systems. The size of the flocs formed aids the liquid-solid separation of the treated water from the MLSS [34]. In addition, the magnetic field enhanced microbial activity, improving biological adsorption. The sludge flocculation created close links that could not easily be aerated by shear force. The addition of magnetic powder increased the sludge particle diameter. As the sludge particle size increased, the chance of collisions between the sludge particles decreased. Thus, the number of microparticles in the reactor that could lead to membrane fouling was reduced, slowing membrane fouling [35]. In short, the increase in floc size with the addition of magnetic powder improved filtration by reducing fouling.

#### 3.4. Change in zeta potential of the activated sludge mixed liquor in the MP-MBR and MBR processes

The zeta potential of activated sludge can be used to predict the fouling tendency, and its variability in the MP-MBR and MBR batch experiments was therefore investigated. The detection of the zeta potential of the sludge in the two reactors is shown in Fig. 6.

As shown in Fig. 6, during the operation period, the value of the zeta potential of the two reactors decreased during the early stage and remained at a relatively low level in the later stage. The zeta potential of the control MBR changed from  $-24$  to  $-35$  mV, and its mean value was  $-31$  mV, whereas the zeta potential of MP-MBR changed from  $-18$  to  $-28$  mV, with a mean value of  $-24.33$  mV. Compared with the MBR process, the zeta potential in the MP-MBR was 27.4% lower, indicating that the magnetic field had

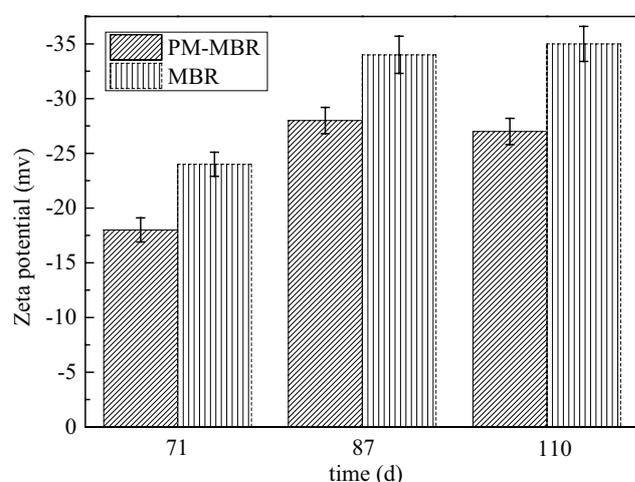


Fig. 6. Zeta potential of the activated sludge mixed liquor in the MBR versus the MP-MBR process.

a very significant role in reducing the zeta potential in the mixed liquor colloid. As shown in Fig. 6, a lower zeta potential was obtained due to the addition of magnetic powder by neutralizing the negative charge on the surface of the flocs, which resulted in the formation of larger flocs, improved aggregation, and enhanced porosity of the cake layer deposited on the membrane [36]. The change in zeta potential value may also be attributable to enhancement of the microbial activity for EPS removal by the weak magnetic field [37]. The decrease in negatively charged EPS content shown in Fig. 4 would lead to a higher zeta potential, which could affect the filterability performance of the MBRs. Zhou et al. [38] also observed that a magnetic field could indeed reduce the zeta potential of activated sludge. The maximum sludge zeta potential reduction (46.5% to 51.4%) was achieved when the center field magnetic field was approximately 0.4 T.

#### 4. Conclusions

By studying the effect of magnetic powder on the TMP and the characteristics of active sludge (i.e., SMP, EPS, sludge particle sizes and zeta potential of activated sludge), this work provides a more in-depth understanding of the impact of magnetic powder on sludge characteristics and its corresponding effect on mitigating membrane fouling. A few highlights are worth noting:

The coexistence of charge neutralization, adsorption and bio-oxidation plays an important role in mitigating membrane fouling in the MP-MBR. The addition of magnetic powder improved dehydrogenase activity and stimulated microbes to produce less EPSs and SMPs. The addition of a magnetic field also decreased the zeta potential and increased the particle size, which markedly improved the filtration performance of the MBR.

This study demonstrates that the addition of magnetic powder to the MBR process is a promising technique for membrane fouling control and has a characteristics of less infrastructure investment, flexible operation and so on. Magnetic activated sludge process has the potential to become the next generation of MBR systems because of its potential ability to significantly alleviate membrane fouling.

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