Membrane and Water Treatment, *Vol. 10, No. 2 (2019) 179-189*
DOI: https://doi.org/10.12989/mwt.2019.10.2.179

Investigation of influence of temperature and solid retention time on membrane fouling in MBR

Atoosa Mirzavandi[1], Hossein Hazrati*[1,2] and Sirous Ebrahimi[1]

[1]*Faculty of Chemical Engineering, Sahand University of Technology, Tabriz, Iran*
[2]*Environmental Engineering Research Center, Sahand University of Technology, Tabriz, Iran*

(Received August 23, 2018, Revised February 12, 2019, Accepted February 20, 2019)

Abstract. This study aimed to investigate the effect of temperature and solid retention time (SRT) on membrane fouling in a membrane bioreactors (MBRs). For this purpose, a lab-scale submerged MBR system was used. This system operated at two SRTs of 15 and 5 days, three various temperatures (20, 25 and 30˚C) and hydraulic retention time (HRT) of 8 h. The results indicated that decreased the cake layer resistance and increased particles size of foulant due to increasing temperature and SRT. Fourier transform infrared (FTIR) analysis show that the cake layer formed on the membrane surface, contained high levels of proteins and especially polysaccharides in extracellular polymeric substances (EPS) but absorbance intensity of EPS functional groups decreased with temperature and SRT. EEM analysis showed that the peak on the range of Ex/Em=220-240/350-400 in SRT of 15 and temperature of 30 ˚C indicates the presence of fulvic acid in the cake. In addition, as the temperature rise from 20 to 30˚c, concentration of soluble microbial products (SMP) increased and COD removal reached 89%. Furthermore, the rate of membrane fouling was found to increase with decreasing temperature and SRT.

Keywords: submerged membrane bioreactor, solid retention time, temperature, membrane fouling

1. Introduction

MBR technology has been utilized for wastewater treatment since 1969. This technology combines a bioreactor as well as separation membrane. Bioreactor has the same function as aerated tank and instead of settling tank, the separation membrane is used. MBRs can be generally classified in two categories: Submerge MBRs and side stream MBRs. Submerge MBRs due to the saved energy have been preferred recently. The advantages of MBR are: Independent of HRT and SRT, fine control of SRT, High concentration of MLSS, Low production of excess sludge, decrease F/M ratio, and decrease of operational space. Membrane bioreactor like another membrane systems have limitations (Kertesz 2014). Fouling of membrane is the most important limitation factor in the membrane process. Recent researches (Faust *et al*. 2014, Ma *et al*. 2013, Silva *et al*. 2016, Wang *et al*. 2010) reported that Extra Cellular Polymeric Substances (EPS) and Soluble Microbial Product (SMP) including polysaccharide, protein, humic acid as well as nucleic acid are the primitive reasons of fouling in membrane bioreactor process.

EPS is a microbial product having a matrix structure which plays a pivotal role in providing microbial flocs from individual cells. EPS includes two layer, tightly and loosely bound in inner and outer layer, respectively. The compounds and unique structure of EPS makes EPS to be stick at the surface (Hazrati *et al*. 2018, Hazrati and Shayegan 2016). Therefore, the subsequent result could be membrane fouling as the result of the interactions between membrane and EPS (Chen *et al*. 2016, Lin *et al*. 2014, Nam *et al*. 2015, Teng *et al*. 2018b).

If EPS is the main membrane foulant, floc adhesion and cake formation can be considered as a second stage of membrane fouling in MBRs. 4 forces exerted on Single floc nearby the membrane in the disturbed sludge suspension include: 1-the permeate drag force, 2-the inertial lift force, 3-the net gravity force (gravity force minus buoyant force), 4-the shear force and Brownian diffusion force (extremely small to be ignored). The motion of floc depends on the predominant forces on it in MBRs (Qu *et al*. 2018, Teng *et al*. 2018a, Teng *et al*. 2019). The hydrodynamic conditions are closely associated with aeration intensity, bubble size, sludge concentration, suspension viscosity, and membrane module configuration. Increased shear force by improved hydrodynamic conditions could control membrane fouling. However, their effects on SMP release and floc size should also be considered. It has been recently revealed that chemical potential mechanism related with foulant/cake layer filtration is mainly responsible for the filtration resistance (Chen *et al*. 2016, Teng *et al*. 2018b, Zhang *et al*. 2018).

Membrane fouling can be visible by the increment of transmembrane pressure (TMP) or a decrement in flux according to the operation mode (constant flux or constant pressure) (Abdoli *et al*. 2018). Solid retention time (SRT) and hydraulic retention time (HRT) are of two important operation parameters which effect on microbial characteristics and EPS product, SMP, and flocs.

One of the parameters that describe membrane fouling

*Corresponding author, Ph.D.
E-mail: h.hazrati@sut.ac.ir

ISSN: 2005-8624 (Print), 2092-7037 (Online)

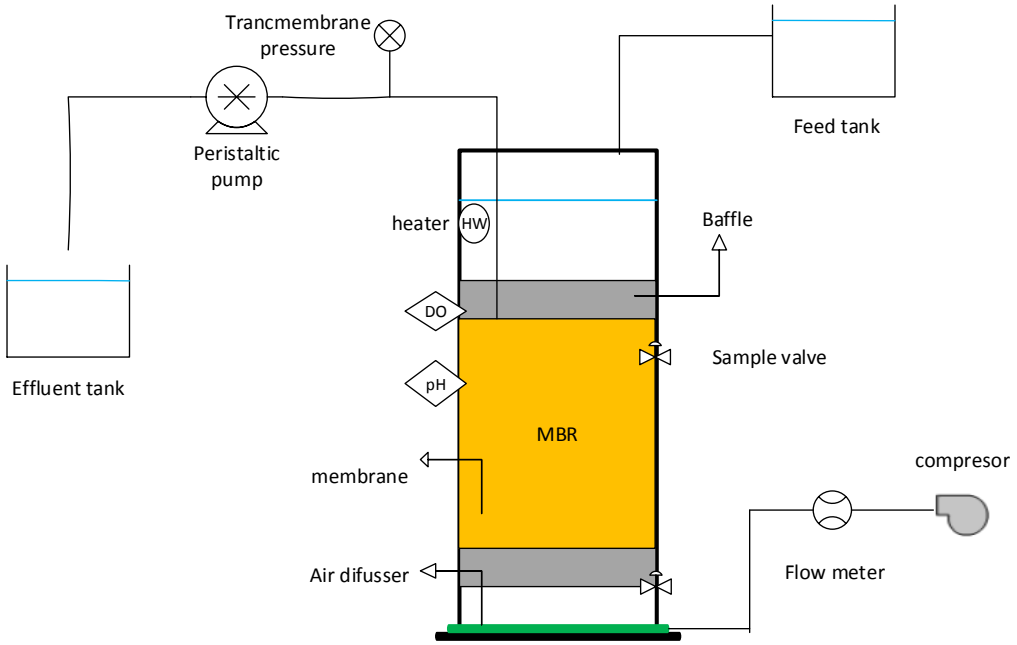


Fig. 1 A lab scale MBR

in bioreactor membrane is microbial characteristics which is affected by operation parameters. Two vital parameters directly influence on microbial characteristics are SRT and temperature**.** SRT is an operating parameter to control the sludge production rate and constant biomass concentration in the MBR, therefore, choosing the best SRT for better performance of MBR is a goal of processes**.** Hung *et al*. reported that in a longer SRT and higher SMP production induced more pore blocking in membrane (Huang *et al*. 2011). Whereas, Faust *et al*. has shown that shorter SRT result in more membrane fouling by poor bio flocculation (Faust *et al*. 2014). Ana *et al*. investigations on the affection of SRT on sludge composition and EPS in MBR, illustrate that in high SRT, while the overall EPS content of suspended biomass was increased, a decrease in cake layer was detected that resulted in the reduction of membrane fouling (Silva *et al*. 2016). Temperature is another environmental parameter that effects on biological wastewater treatment and also on membrane fouling in MBR. The researchers were showed that permeability was reduced by the increase in membrane flux resistance at temperatures <15°C (Arévalo *et al*. 2014) . The other work was studied the influence of temperature variation on EPS and found that when the temperature increased the EPS bound's concentration was decreased (Gil *et al*. 2010). In addition, it was reported that extracellular polymer substances (EPS) and soluble microbial products (SMPs) increased due to decreasing temperature, which triggered membrane fouling as evidenced by the trans-membrane pressure (TMP) increase rate (Ma *et al*. 2013). Also, results of van den Brink *et al*. showed that increased membrane fouling at low temperature because of released polysaccharide and submicron particle from sludge flocs (van den Brink *et al*. 2011). Furthermore, the extent of membrane fouling increased with an increase in the operating temperature, whereas temperature shocks temporarily decreased fouling resistance (Gao *et al*. 2012).

With due attention to result of recently researches the same viewpoint about effect of SRT and temperature on SMP and EPS production have not been introduced. Moreover, investigations on the effect of temperature and SRT at the same time on EPS and membrane fouling have been less. So in this paper we focus on effect of these two parameters on the production of EPS and membrane fouling in MBR**.**

2. Materials and methods

2.1 Experimental set-up and operation condition

A lab scale MBR with a total working volume of 7 L and dimensions of 22×7×60 cm^3 was used in this research (Fig. 1). The bioreactor was equipped with a polyethylene flat sheet membrane with a pore size of 0.4µm and a surface area of 0.1 m^2 (Kubota, Japan). The sides of MBR were two layer which the warm / cold water can flow for controlled temperature. Air diffuser placed blow the membrane in order to supply oxygen demanded by the microorganisms (biological processes) and reduced foulants on the membrane surface. To unify air movement on the membrane surface a plate as a baffle was settle behind the membrane. The channel width between the membrane and baffle was 7 mm.

The sludge used in the MBR was supplied from Tabriz Petrochemical Company with mixed liquor suspended solid (MLSS) concentration of 5000 mg/L (after concentration). The sludge was allowed to acclimate to the operating conditions was fed with synthetic wastewater. The synthetic wastewater had the following composition (mg/L): C_2H_5OH: 350; K_2HPO_4: 35; KH_2PO_4: 45; Urea: 560; $MgSO_4{\cdot}7H_2O$: 13; $CaCl_2{\cdot}2H_2O$: 7; $FeCl_3$: 5; $NaHCO_3$: 500.

The MBR was operated at an overall hydraulic retention

time (HRT) of 8 h. For investigated of influence of solid retention time (SRT) and temperature on membrane fouling, the SRT and temperature were considered of 15 and 5 days 20±1, 25±1 and 30 ±1°C respectively. The Chemical oxygen demand (COD) of synthetic wastewater was 1200 mg/L. Dissolved oxygen concentration in the MBR was controlled at 3-6 mg/L (HANNA Instruments, HI9142). The pH of MBR was controlled at 6.5±0.1 by addition of $NaHCO_3$, using a pH transmitter (2500, Mettler Toledo, Switzerland). In the MBR, continuous aeration at 8 L/min was applied.

2.2. System start up

The MBR was initially filled with 6 L synthetic wastewater and it was operated on batch mode. At the end of each batch, the membrane-filtered effluent was obtained by suction using a peristaltic pump (Master flex, Cole-Parmer, USA) connected to the module at a constant flux of 2×10^{-3} $m^{-3}/$ m^2.s. This work has been repeated every day until the end of each periods (each periods was 30 days). The effect of three temperatures (20, 25 and 30°C) was investigated at SRT 15 days and then the best temperature was test at SRT of 5 days. At the end of each periods the membrane module was taken out from the MBR and was chemically cleaned. Chemical cleaning of the membrane was performed according to the instructions outlined by Kubota Corporation. At first, using a dilute solution of sodium hypochlorite for cleaned organic fouling (2 h) and then, using oxalic acid for cleaned inorganic fouling (1h) (Zonoozi *et al*. 2015). Because of different SRTs were test in this research, specific volume of sludge discharged from the MBR at the end of each cycle.

The amount of waste sludge was determined according to the following relation (Van den Broeck *et al*. 2012):

$$SRT = \frac{V\ MBR}{V\ waste/day} \quad (1)$$

V_{MBR} is the volume of the MBR (m^3) and $V_{waste/\ day}$ is the volume of waste sludge per day (m^3 / day).

2.3 Analytical methods

2.3.1. Resistance analysis

Analyzing filtration resistances makes it easy to understand the fouling phenomena in MBRs. A series of filtration experiments and calculations for each resistance value with the filtration data provides us an insight into what kind of fouling existed and was predominant. Fouling resistance calculated by Darcy' s equation (Park *et al*. 2015):

$$J = \frac{\Delta P}{\mu . R_t} \quad (2)$$

$$R_t = R_M + R_P + R_C \quad (3)$$

Where J (m^3/m^2.s) is the membrane permeate flux, ΔP (Pa) is the transmembrane pressure, μ (kg / m. s) is the viscosity of permeate, R_t (m^{-1}) is the total filtration resistance, R_m (m^{-1}) is the intrinsic membrane resistance, Rp (m^{-1}) is the pore clogging resistance and Rc (m^{-1}) is the cake resistance. At the end of each periods, for measurement each resistance value these steps were followed (Hazrati and Shayegan 2016):

1) R_T was estimated by measuring TMP (AUTONICS, PSA-V01P) at the end of periods.
2) R_P was evaluated by measuring TMP after cleaning cake layer on the membrane surface.
3) R_M was determined by measuring TMP with clean membrane (after chemical cleaning).
4) Put R_T, R_M and R_P in equation (3) and then calculate R_C.

2.3.2. SMP and EPS extraction

EPS were extracted from the mixed liquor in the MBR according to the thermal treatment method (Tseng *et al*. 2015). According to this method, separated EPS into loosely bound EPS (LB EPS) and tightly bound EPS (TB EPS). 50 ml of each well- mixed sludge sample were transferred to a 50-ml polypropylene centrifuge tube. After centrifugation (101,SIGMA) at 4000×g for 5 min, the supernatant was discarded (the supernatant contained SMP). The sludge pellet was resuspended in 0.05% sodium chloride (NaCl) solution at 50°C and reconstituted back to 50 ml. The suspension was sheared with a vortex mixer for 1 min. It was then centrifuged at 4000×g for 10 min. The EPS contained in this supernatant was considered to be LB EPS. A solution containing 0.05% NaCl was transferred to the decanted sludge pellet to form a 50-ml suspension at 50°C, which was sheared again with a vortex mixer for 1 min and then heated to 60°C in a water bath where it was kept for 30 min. The EPS in this supernatant (considered to be TB EPS) was separated by centrifugation at 4000×g for 15 min (Tseng *et al*. 2015).

2.3.3. Analysis of proteins

The Lowry method was used for protein quantitation. For following Lowry method to be in need of this solutions and reagent (Waterborg 2002): 1. Complex-forming reagent: Prepare immediately before use by mixing the following stock solutions in the proportion 100:1:1 respectively: Solution A: 2% (w/v) Na_2CO_3 in distilled water, Solution B: 1% (w/v) $CuSO_45H_2O$ in distilled water, Solution C: 2% (w/v) sodium potassium tartrate in distilled water. 2. NaOH with normality of 2. 3. Folin reagent (commercially available): Use at 1 N concentration. To 0.1 mL of sample or standard (Bovine serum albumin (BSA) from was used as standard) add 0.1 mL of 2 N NaOH. Hydrolyze at 100°C for 10 min in a heating block or boiling water bath. Cool the hydrolysate to room temperature and add 1 mL of freshly mixed complex-forming reagent. Let the solution stand at room temperature for 10 min. Add 0.1 mL of Folin reagent, using a vortex mixer, and let the mixture stand at room temperature for 30–60 min (do not exceed 60 min).

Read the absorbance (UNIC, UV2100 SPECTROPHOTOMETER) at 750 nm if the protein concentration was below 500 μ g/mL or at 550 nm if the protein concentration was between 100 and 2000 μ g/mL (Waterborg 2002).

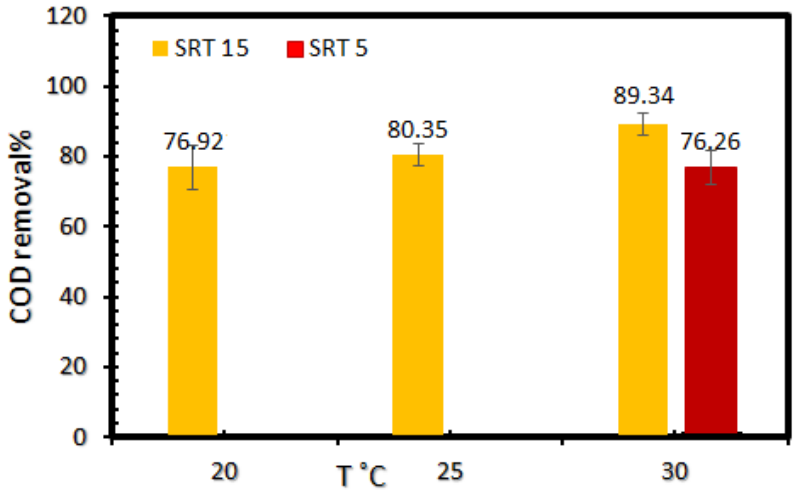


Fig. 2 COD removal at different temperature and SRT

2.3.4. Analysis of carbohydrates

The Anthrone method is a colorimetric method of determining the concentration of the total sugars in a sample. Glucose was used as standard (Zuriaga-Agustí *et al*. 2013). 1 mL of sample is mixed with 2 mL of anthrone reagent diluted in sulfuric acid and then placed in a bath at 100°C during14 min until the reaction is completed. When the solution is cooled, the absorbance is measured at 625 nm (the anthrone reagent is ready right before analysis. 0/1 gr (0/1%) anthrone reagent is diluted in 100 mL concentrate sulfuric acid (98%) and protected from the light) (Zuriaga-Agustí *et al*. 2013).

2.3.5. Analysis of Fourier transform infrared spectrometer (FTIR)

FTIR analysis was used to characterize the major functional groups of organic matters in cake layer that formed on membrane surface (Wang *et al*. 2009). The cake layer that removed from the membrane surface was dissolved in 500 mL pure water. After that, about 50 mL of the solution were centrifuged for 10 min at 9000 rpm. The foulants pellet were placed in incubator for 48 h at 55°C. The dry foulants used for FTIR analysis (Hazrati and Shayegan 2016).

2.3.6. Analysis of Excitation- Emission Matrix (EEM)

EEM analysis was used to study the chemical and physical characteristics of organic matters in foulants that formed on membrane surface (Hazrati *et al*. 2017, Van den Broeck *et al*. 2012). Extracted EPSs from foulants and used for EEM analysis. The EEM was determined by LS 55; PerkinElmer. A three dimensional EEM spectra was obtained by collecting wavelength of both excitation over range of 200-400 nm and emission of 200-600 nm in stepwise 10 nm (Hazrati and Shayegan 2016).

2.3.7. Other analysis

Particle Size Distribution (PSD) analysis was determined by Laser Particle Sizer-ANALYSETTE22-NanoTec –FRITSCH With a detection range of 0.1-100 µm. COD, MLSS and MLVSS were estimated according to the standard methods (Association and Association 1989).

Table 1 Resistance was measured at the end of each period

SRT	T (°C)	R_T* 10^{11} (m^{-1})	RP* 10^{11} (m^{-1})	RM*10^{11} (m^{-1})	RC*10^{11} (m^{-1})
15	20	34.1	7.12	6.6	20.4
15	25	25.1	7.0	6.5	11.5
15	30	17.5	6.9	6.5	4.1
5	30	32.6	14.6	6.7	11.3

3. Results and discussion

3.1. COD removal

Fig. 2 shows COD removal at different temperatures and SRTs. Based on the results, COD removal was variable in different SRTs. For SRT of 15 days, it reached to maximum of 89% at 30°C. For SRT of 5 days, COD removal was 76.26%. This lower removal (76.26%) can be due to lower concentration of biomass as more sludge was discarded.

Fig. 3 depicts MLSS concentrations in different periods. As it can be seen, for SRT of 15 days, MLSS concentration varied from 3000 to 5000 mg/l which reached to 2000 mg/l for SRT of 5 days. Although removal efficiency was 76.26% for SRT of 5 days, but as the organic load of this condition was higher than the conventional situation, efficiency of 76.26 is also acceptable and shows microbialactiveness of the sludge. In the other words, MBR has a high potential for wastewater treatment due to having high concentration of biomass.

3.2. Resistance distribution

In this study, resistance was measured at the end of each period as shown in Table 1. As the data suggest, increase of temperature at SRT of 15 days resulted in decrease of cake layer resistance (20.4, 11.5 and 4.1, respectively). But Roorda *et al*. reported opponents of these results (Gil *et al*. 2010), they found that as the temperature increased (10°C to 40°C) the microbial activities, SMP in media and cake resistance were increased too. For sludge retention time of 5 days and at temperature of 30°C, cake resistance (11.3 m^{-1}) was almost equal with the resistance at 25°C for SRT of 15 days (11.5 m^{-1}); but its pore resistance was higher. Decrease

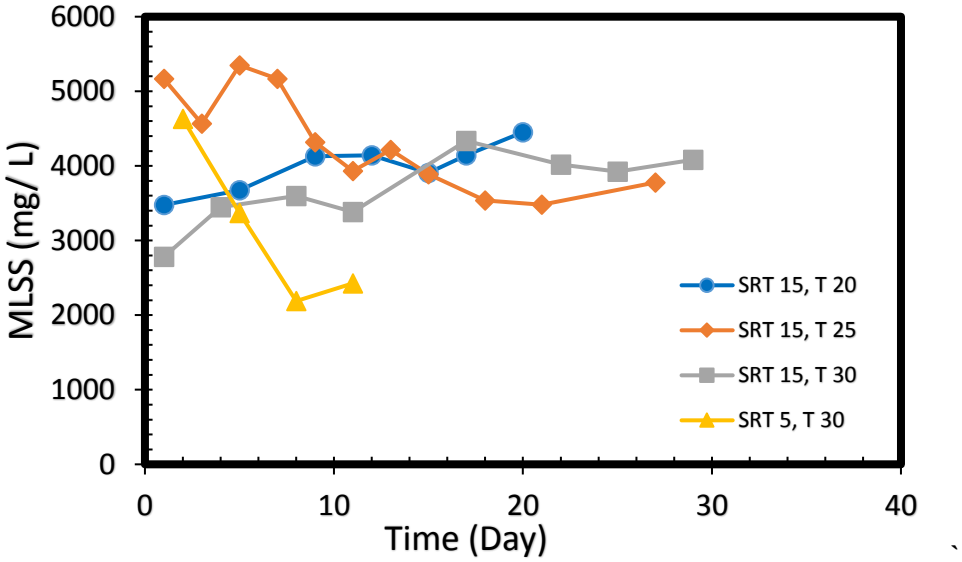


Fig. 3 MLSS variation at different temperature and SRTs

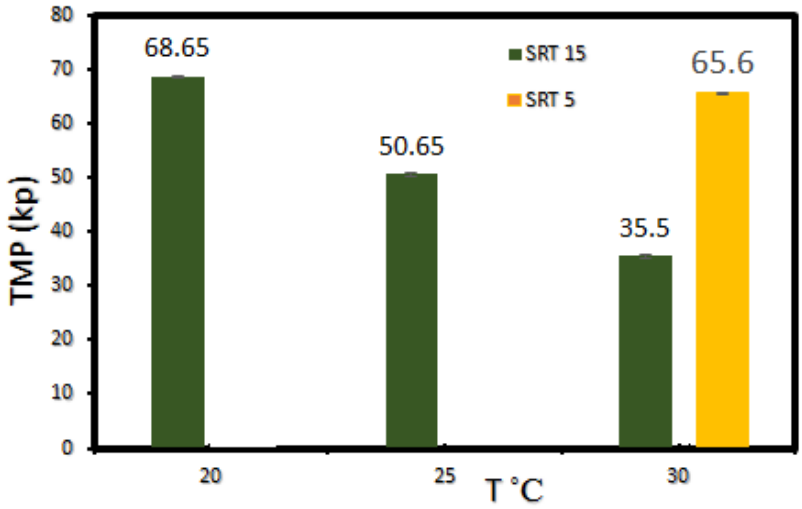


Fig. 4 Increase of TMP with decline of temperature

in SRT from 15 to 5 at 30°C resulted in enhancement of total resistance. The reason could be increase of pore resistance in SRT of 5 days. In fact, in such situation, pore fouling played an important role in membrane fouling. The reason could be presence of smaller particles in the cake layer which could enter pores.

3.3. TMP variations

Fig. 4 shows increase of TMP with decline of temperature. Temperature reduction will increase sludge viscosity and decrease flocs size which will lead to TMP enhancement and can be attributed to increase fouling. For SRT of 5 days and temperature of 30°C, SMP was higher and the particles were smaller; so TMP increased in comparison with STR of 15 days at 30°C. This is agree with the results of Ana *et al*. expressing that reduction of SRT will increase the cake resistance (Silva *et al*. 2016). For SRT of 5 days, biomass concentration and cloth were declined so, resistance and TMP increased.

Fig. 5 indicates that TMP has different trend with changing SRT and temperature. For SRT of 15 days, temperature decline resulted in TMP increase with higher slope. For SRT of 5 days, this slope increase was faster. The reason could be explained according to Table 1. As the Table 2 suggests, increase of temperature in SRT of 15 days resulted in decrease of total resistance; so lower TMP would be imposed during filtration. For SRT of 5 days, total resistance increased again which was mainly due to pores resistance. More pore blockage in this SRT will cause less amount of water passing through the system. To maintain the flux, system will increase the pressure which resulted in TMP enhancement.

3.4. Particle size distribution

Figs. 6 (a-d) present the size distribution of the particles precipitated on the membrane in the range of 0.1-100 µm. 8% of particles were in the range of 20-30µm in SRT of 15

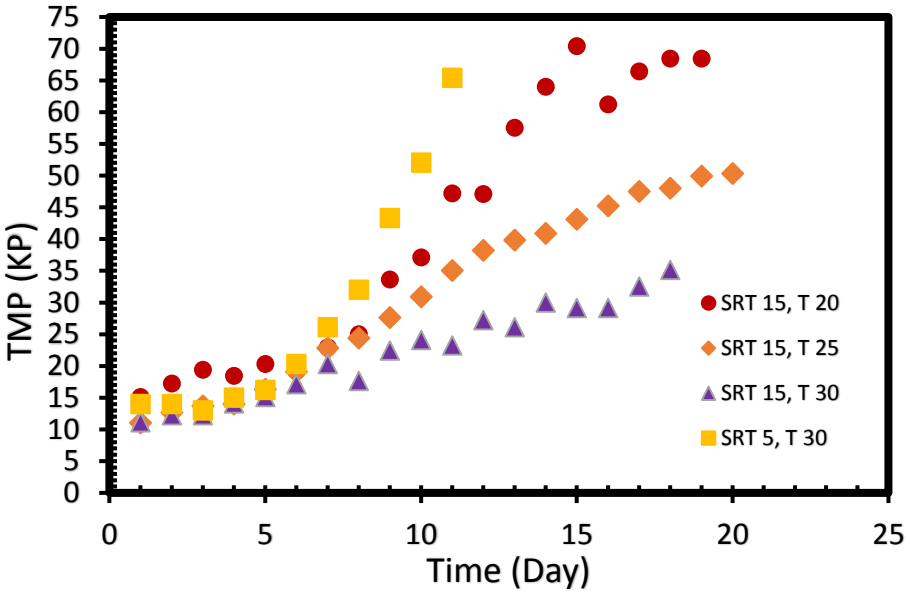
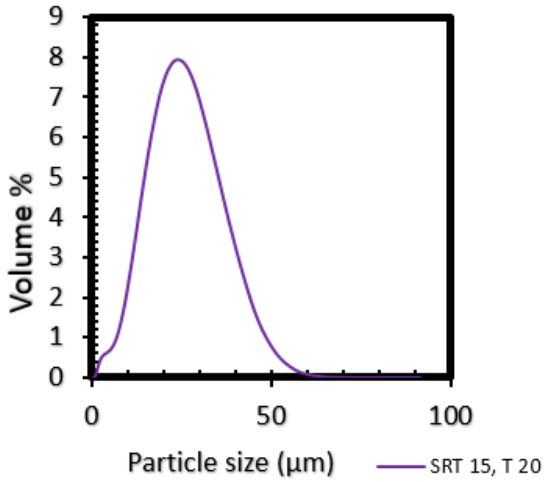
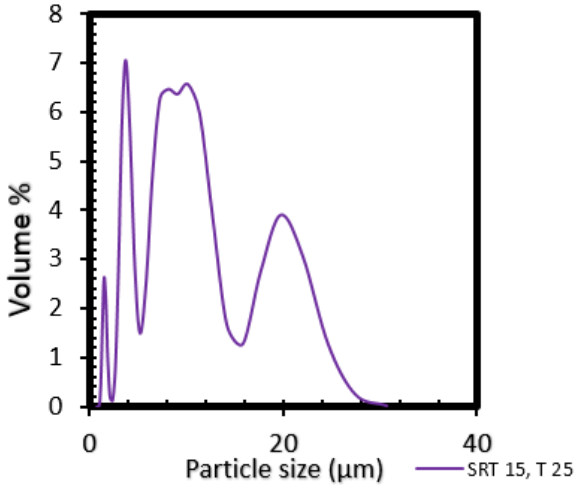


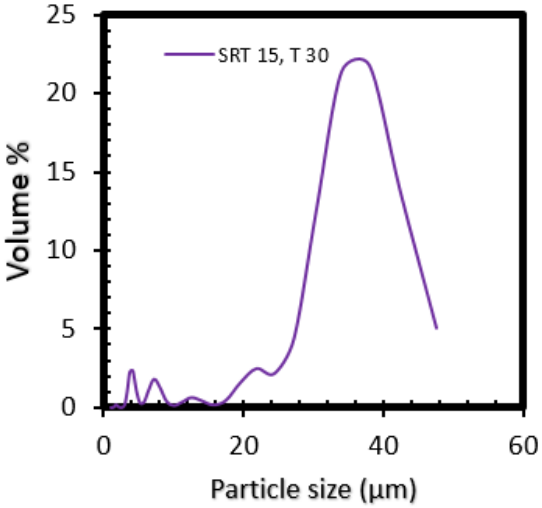
Fig. 5 Different of TMP with SRT and temperature



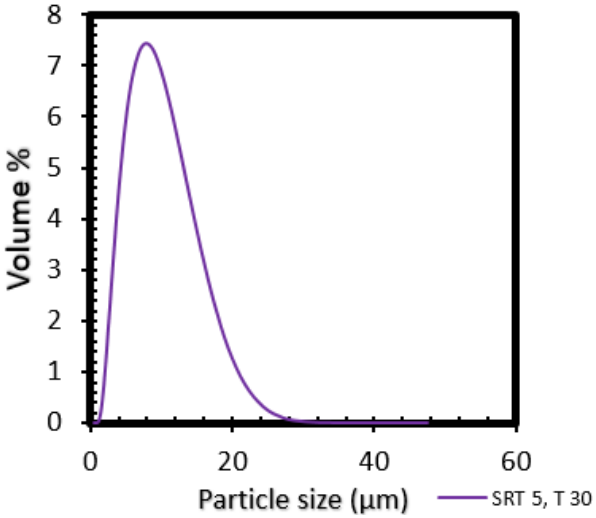
(a) SRT 15 and T 20°C



(b) SRT 15 and T 25°C



(c) SRT 15 and T 30°C



(d) SRT 5 and T 30°C

Fig. 6 Size distribution of the particles precipitated on the membrane

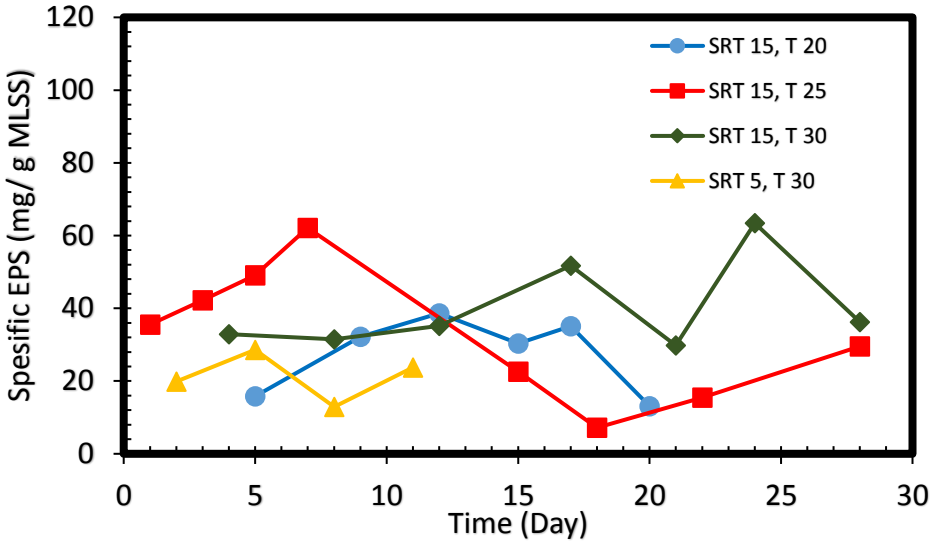


Fig. 7 The variation of EPS concentration in MBR

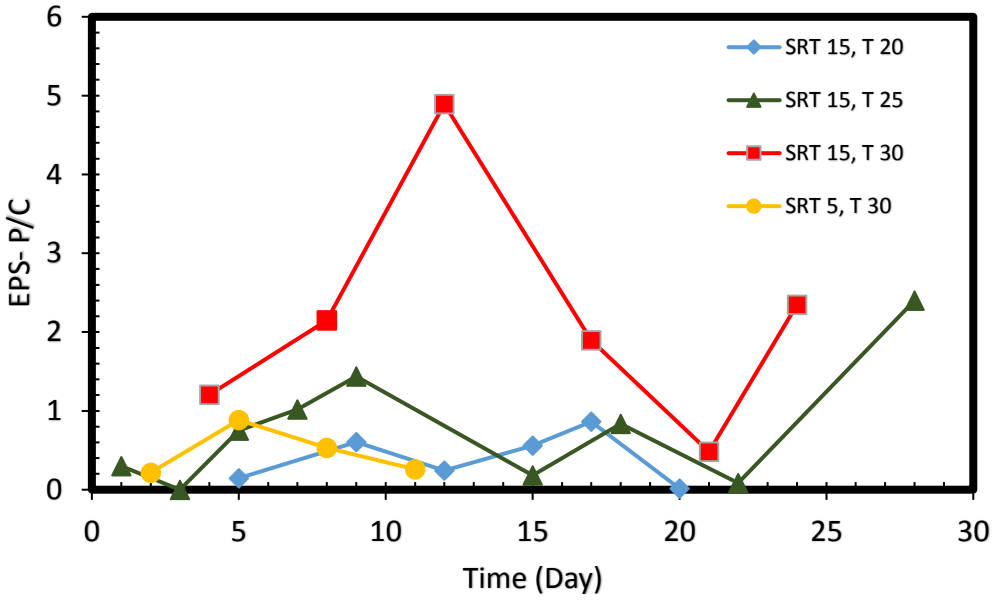


Fig. 8 Protein to polysaccharides (P/C) ratio in EPS

days and at temperature of 20°C. In SRT of 15 days and at temperature of 25°C, 2.5% of the particles have the size ranging from 1.5 to 1.7 μm. 2-7% of the particles have the size ranging from 3-3.78 µm, the size of 1-4% of them varied from 5.26 to 6.56 µm and about 7% of them were in the range of 7.5-10.2 µm. 1.5-4% of particles size varied from 14.2 to 30.6 µm. For SRT of 15 days and temperature of 30°C, about 21% of particles have the size ranging from 34 to 38 µm. Among these three graphs showing particle size in three different temperatures for SRT of 15days, the particles were larger for the case of 30°C; hence lower fouling was created. Results of resistance and TMP also confirmed this trend. For SRT of 5 days and at 30°C, the size of 7% of the particles varied from 6.4-10 µm which is smaller than the particle size in case of SRT=15 days and temperature of 30°C. This smaller size gave rise to more membrane fouling and hence increased TMP. The smallest particles in SRT of 15 days and temperatures of 20, 25 and 30°C were 0.728, 0.898 and 0.95, respectively. All of them were larger than the pore size (0.45µm). For the case of SRT=5 days and T=30°C, the smallest particles had the dimension of 0.488 µm which is near to the size of pores. S it was reported before, for this SRT value, the resistance was mainly due to pores blockage.

3.5. EPS in liquid mixture

Fig. 7 illustrates EPS concentration of the active sludge inside the bioreactor. It can be seen that EPS concentration has no specific trend. For SRT of 15 days and different

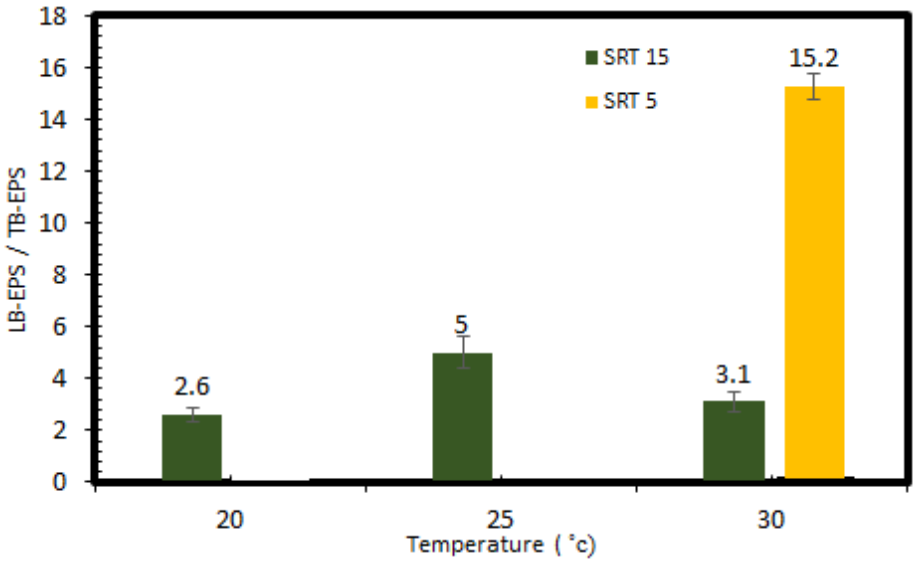


Fig. 9 Ratio of loose to tight bonds in EPS

temperatures, it first increased, then decreased followed by a final increase. Its value was higher at 30°C. Theoretically, it can be explained that increase of temperature resulted in enhancement of metabolic activity which elevated production of EPS. High temperature may also break the clothes giving rise to release of EPS in the form of SMP (Hazrati *et al*. 2016). EPS concentration observed in SRT of 5 days was lower than the one recorded for SRT=15 days. This indicates that in addition to temperature, SRT can also affect EPS concentration while Faust *et al*. (Faust *et al*. 2014) expressed oppositely. They believed that SRT has no impact on EPS.

Fig. 8 demonstrate protein to polysaccharide (P/C) ratio of activated sludge. For SRT=15 days, P/C increased by temperature rise reflecting high levels of protein relative to polysaccharide. According to the studies (Gao *et al*. 2012, Lin *et al*. 2009), EPS is mainly composed of protein and high ratio of P/C will cause more attachment of protein to activated sludge giving rise to formation of flocs. Moreover, due to hydrophobicity of proteins, they tend to form flocs. That's why larger flocs were formed in SRT=15 and T=30°C; so had been less membrane fouling.

3.6. Loose and tight bonds ratio in EPS

Fig. 9 demonstrates the ratio of loose to tight bonds in EPS. Regarding the results, in SRT=15 days and at two temperatures of 20 and 30°C, these ratios were almost equal but at 25°C it was slightly larger. However, for SRT of 5 days, loose bonds had concentration far more than the tights ones (about 15 times). Therefore, it can be said that these loose bonds play a crucial role in membrane fouling which tight ones did not have significant impact (Lin *et al*. 2014). In this regard, high resistance in SRT of 5 days could be attributed to this observation.

3.7. Loose and tight bonds in EPS

Table 2 lists the concentration of loose and tight bonds for different SRT and temperatures. For SRT=15 days, increase of temperature resulted in decline of loose and tight bonds concentration. Based on the previous studies expressing that loose bonds have the highest impact on fouling (Lin *et al*. 2014), lowest value of these bonds were observed at 30°C which led to resistance decrease at 30°C and therefore less fouling. For fixed temperature (30°C, reduction of SRT caused a significant increase in loose bonds concentration which confirmed increase of resistance and therefore fouling in SRT of 5 days compared with SRT=15 days.

Table 2 lists the concentration of loose and tight bonds for different SRT and temperatures

SRT (day)	T (˚c)	LB-EPS (mg / L)	TB-EPS (mg / L)
15	20	16.7±1.95	6.7±1.95
15	25	4.2±0.6	27.3±2.1
15	30	1.7±0.08	3.4±0.4
5	30	11.8±1.5	8.6±0.8

Table 3 Concentrations of protein and polysaccharide of cake layer

SRT (day)	T (˚c)	EPSp (mg / L)	EPSc (mg / L)
15	20	1.1±0.2	13.3±0.7
15	25	22.4±2.8	9.5±1.1
15	30	1.5±0.5	4.9±1.0
5	30	4.2±0.9	15.9±0.8

3.8 Protein and polysaccharide of cake layer

Concentrations of protein and polysaccharide of cake layer are provided in Table 3. As the results suggest, increase of temperature reduced the polysaccharide concentration for case with SRT=15 days. But its concentration was still higher than protein expect at 25°C. For fixed temperature (30°C), decrease of SRT (from 15 to 5 days) resulted in increase of polysaccharide content.

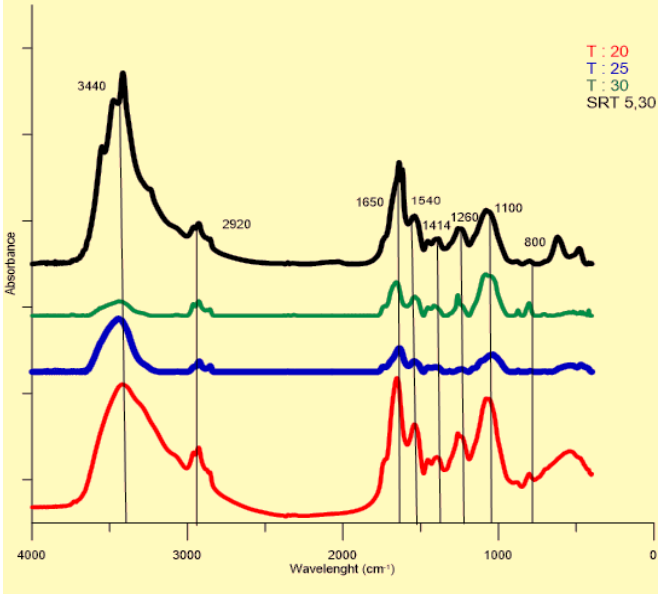
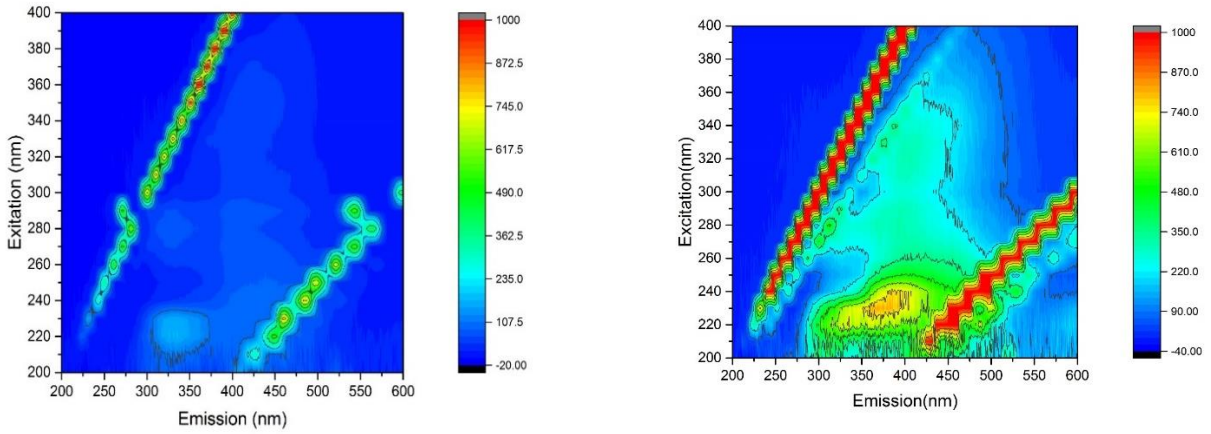


Fig. 10 FTIR spectra of membrane foulants at termination of each operating period



(a) SRT 15 and 25°C (b) SRT 15 and 30°C

Fig. 11 Fluorescence EEM of membrane foulants

Therefore, it can be concluded that reduction of temperature and SRT can increase polysaccharide concentration; this compound is also more effective on fouling. FTIR results in next section also confirms that.

3.9. FTIR analysis

The peaks intensity in FTIR spectrum (Fig. 10) indicates absorbance intensity. For SRT of 15 days, increase of temperature declined the absorbance intensity which can be a sign of reduced polysaccharide and protein composition of the cake. This means that most of compounds were biodegraded by increase of temperature. With comparing the peaks of SRT= 15 and 5 days, it can be concluded that decrease in SRT resulted in enhancement of polysaccharide and protein compounds in cake layer. This could be generalized to the results of previous analyses; implying that reduction in temperature and SRT will increase polysaccharide and protein compounds which are among the major effective parameters on fouling.

Regarding the peaks and identified functional groups, a high portion of EPS composition is polysaccharide. According to Zhongbo *et al*. (Zhou *et al*. 2012) this can be explained that protein compounds can be degraded easier and faster (in comparison with polysaccharides) so polysaccharides are chief composition of the medium.

3.10. EEM analysis

Figs 11 (a-b) present EEM spectroscopy results of the organic compounds in cake layer. At 25°C, the peak at Ex/Em=220-230/330-350 indicates aromatic protein compounds and the one in Ex/Em=280/330-350 shows tryptophan protein (Zhang *et al*. 2011). This is agree with EPS_p test of cake layer at 25°C. The peak on the range of Ex/Em=220-240/350-400 in Fig 11 (b) indicates the presence of fulvic acid in the cake (Zhang *et al*. 2011). Regarding protein concentration of cake layer in Table 3, protein concentration was very low at 30°C but it reached to its maximum concentration at 25°C; which was also confirmed by EEM test.

4. Conclusion

This study investigates the effect of SRT and temperature on reducing the membrane fouling in MBR systems. The results of this study are as follows:

• Increase of temperature and SRT resulted in decline of cake layer resistance; while particle and floc size was increased which decreased the membrane fouling.

• Among proteins and polysaccharides, higher presence of polysaccharides can introduce them as the main factor in membrane fouling.

• Increase of temperature and SRT decreased the concentration of loose bonds in EPS which is one of the factors in fouling

• In comparison with EPS, SMP plays a more prominent role in fouling.

References

Abdoli, S.M., Shafiei, S., Raoof, A., Ebadi, A., Jafarzadeh, Y. and Aslannejad, H. (2018), "Water flux reduction in microfiltration membranes: A pore network study", *Chem. Eng. Technol.,* **41** (8), 1566-1576.

American Public Health Association (APHA) and American Water Works Association (AWWA) (1989), *Standard Methods for the Examination of Water and Wastewater*, APHA, AWWA, U.S.A.

Arévalo, J., Ruiz, L., Pérez, J. and Gómez, M. (2014), "Effect of temperature on membrane bioreactor performance working with high hydraulic and sludge retention time", *Biochem. Eng. J.,* **88**, 42-49.

Chen, J., Zhang, M., Li, F., Qian, L., Lin, H., Yang, L., Wu, X., Zhou, X., He, Y. and Liao, B.-Q. (2016), "Membrane fouling in a membrane bioreactor: High filtration resistance of gel layer and its underlying mechanism", *Water Res.,* **102**, 82-89.

Faust, L., Temmink, H., Zwijnenburg, A., Kemperman, A. and Rijnaarts, H. (2014), "High loaded MBRs for organic matter recovery from sewage: Effect of solids retention time on bioflocculation and on the role of extracellular polymers", *Water Res.,* **56**, 258-266.

Gao, W., Qu, X., Leung, K. and Liao, B. (2012), "Influence of temperature and temperature shock on sludge properties, cake layer structure, and membrane fouling in a submerged anaerobic membrane bioreactor", *J. Membrane Sci.,* **421**, 131-144.

Gil, J.A., Túa, L., Rueda, A.C., Rodríguez, M. and Prats, D. (2010), "Influence of temperature variations on the cake resistance and EPS of MBR mixed liquor fractions", *Desalination Water Treat.,* **18** (1-3), 1-11.

Hazrati, H., Jahanbakhshi, N. and Rostamizadeh, M. (2018), "Fouling reduction in the membrane bioreactor using synthesized zeolite nano-adsorbents", *J. Membr. Sci.,* **555**, 455-462.

Hazrati, H., Moghaddam, A.H. and Rostamizadeh, M. (2017), "The influence of hydraulic retention time on cake layer specifications in the membrane bioreactor: Experimental and artificial neural network modeling", *J. Environ. Chem. Eng.,* **5** (3), 3005-3013.

Hazrati, H. and Shayegan, J. (2016), "Influence of suspended carrier on membrane fouling and biological removal of styrene and ethylbenzene in MBR", *J. Taiwan Institute Chem. Eng.,* **64**, 59-68.

Hazrati, H., Shayegan, J. and Mojtaba Seyedi, S. (2016), "The effect of HRT and carriers on the sludge specifications in MBR to remove VOCs from petrochemical wastewater", *Desalination Water Treat.,* **57**(46), 21730-21742.

Huang, Z., Ong, S.L. and Ng, H.Y. (2011), "Submerged anaerobic membrane bioreactor for low-strength wastewater treatment: Effect of HRT and SRT on treatment performance and membrane fouling", *Water Res.,* **45**(2), 705-713.

Kertesz, S. (2014), "Industrial dairy wastewater purification by shear-enhanced membrane filtration: The effects of vibration", *Membr. Water Treat.,* **5**(2), 73-86.

Lin, H., Xie, K., Mahendran, B., Bagley, D., Leung, K., Liss, S. and Liao, B. (2009), "Sludge properties and their effects on membrane fouling in submerged anaerobic membrane bioreactors (SAnMBRs)", *Water Res.,* **43**(15), 3827-3837.

Lin, H., Zhang, M., Wang, F., Meng, F., Liao, B.-Q., Hong, H., Chen, J. and Gao, W. (2014), "A critical review of extracellular polymeric substances (EPSs) in membrane bioreactors: Characteristics, roles in membrane fouling and control strategies", *J. Membr. Sci.,* **460**, 110-125.

Ma, Z., Wen, X., Zhao, F., Xia, Y., Huang, X., Waite, D. and Guan, J. (2013), "Effect of temperature variation on membrane fouling and microbial community structure in membrane bioreactor", *Bioresour. Technol.,* **133**, 462-468.

Nam, A., Kweon, J., Ryu, J., Lade, H. and Lee, C. (2015), "Reduction of biofouling using vanillin as a quorum sensing inhibitory agent in membrane bioreactors for wastewater treatment", *Membr. Water Treat.,* **6**(3), 189-203.

Park, H.D., Chang, I.S. and Lee, K.J. (2015), *Principles of Membrane Bioreactors for Wastewater Treatment*, CRC Press, Florida, U.S.A.

Qu, X., Cai, X., Zhang, M., Lin, H., Leihong, Z. and Liao, B.-Q. (2018), "A facile method for simulating randomly rough membrane surface associated with interface behaviors", *Appl. Surface Sci.,* **427**, 915-921.

Silva, A.F., Antunes, S., Saunders, A., Freitas, F., Vieira, A., Galinha, C.F., Nielsen, P.H., Crespo, M.T.B. and Carvalho, G. (2016), "Impact of sludge retention time on the fine composition of the microbial community and extracellular polymeric substances in a membrane bioreactor", *Appl. Microbiol. Biotechnol.,* **100**(19), 8507-8521.

Teng, J., Shen, L., He, Y., Liao, B.-Q., Wu, G. and Lin, H. (2018a), "Novel insights into membrane fouling in a membrane bioreactor: Elucidating interfacial interactions with real membrane surface", *Chemosphere,* **210**, 769-778.

Teng, J., Shen, L., Yu, G., Wang, F., Li, F., Zhou, X., He, Y. and Lin, H. (2018b), "Mechanism analyses of high specific filtration resistance of gel and roles of gel elasticity related with membrane fouling in a membrane bioreactor", *Bioresour. Technol.,* **257**, 39-46.

Teng, J., Zhang, M., Leung, K.-T., Chen, J., Hong, H., Lin, H. and Liao, B.-Q. (2019), "A unified thermodynamic mechanism underlying fouling behaviors of soluble microbial products (SMPs) in a membrane bioreactor", *Water Res.,* **149**, 477-487.

Tseng, L., Gori, R. and Rosso, D. (2015), "Effects of activated sludge process conditions on the production of extracellular polymeric substances: results of yearlong monitoring in a warm climate", *Environ. Eng. Sci.*, **32**(7), 582-592.

van den Brink, P., Satpradit, O.-A., van Bentem, A., Zwijnenburg, A., Temmink, H. and van Loosdrecht, M. (2011), "Effect of temperature shocks on membrane fouling in membrane bioreactors", *Water Res.,* **45**(15), 4491-4500.

van den Broeck, R., Van Dierdonck, J., Nijskens, P., Dotremont, C., Krzeminski, P., van der Graaf, J.H.J.M., van Lier, J.B., Van Impe, J.F.M. and Smets, I.Y. (2012), "The influence of solids retention time on activated sludge bioflocculation and membrane fouling in a membrane bioreactor (MBR)", *J. Membr. Sci.,* **401-402**, 48-55.

Wang, Z., Wu, Z. and Tang, S. (2009), "Extracellular polymeric substances (EPS) properties and their effects on membrane fouling in a submerged membrane bioreactor", *Water Res.,* **43** (9), 2504-2512.

Wang, Z., Wu, Z. and Tang, S. (2010), "Impact of temperature seasonal change on sludge characteristics and membrane fouling in a submerged membrane bioreactor", *Separation Sci. Technol.,* **45**(7), 920-927.

Waterborg, J.H. (2002), *The Lowry Method for Protein Quantitation, The Protein Protocols Handbook*, Springer, Berlin, Germany.

Zhang, M., Hong, H., Lin, H., Shen, L., Yu, H., Ma, G., Chen, J. and Liao, B.-Q. (2018), "Mechanistic insights into alginate fouling caused by calcium ions based on terahertz time-domain spectra analyses and DFT calculations", *Water Res.,* **129**, 337-346.

Zhang, X., Wang, Z., Wu, Z., Wei, T., Lu, F., Tong, J. and Mai, S. (2011), "Membrane fouling in an anaerobic dynamic membrane bioreactor (AnDMBR) for municipal wastewater treatment: Characteristics of membrane foulants and bulk sludge", *Process Biochem.,* **46**(8), 1538-1544.

Zhou, Z., Meng, F., Chae, S.-R., Huang, G., Fu, W., Jia, X., Li, S. and Chen, G.-H. (2012), "Microbial transformation of biomacromolecules in a membrane bioreactor: Implications for membrane fouling investigation", *PloS one,* **7**(8), e42270.

Zonoozi, M.H., Moghaddam, M.A. and Maknoon, R. (2015), "Operation of integrated sequencing batch membrane bioreactor treating dye-containing wastewater at different SRTs: Study of overall performance and fouling behavior", *Environ. Sci. Pollut. Res.,* **22**(8), 5931-5942.

Zuriaga-Agustí, E., Bes-Piá, A., Mendoza-Roca, J. and Alonso-Molina, J. (2013), "Influence of extraction methods on proteins and carbohydrates analysis from MBR activated sludge flocs in view of improving EPS determination", *Separation Purif. Technol.,* **112**, 1-10.

CC