



The influence of operating conditions on the filtration behavior of actual extracellular polymeric substances (EPS) using dead-end membrane filtration cell

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ABSTRACT

The EPS solution extracted from the activated sludge of sequencing batch reactors (SBRs) by the formaldehyde–NaOH extraction method was filtered in dead-end cell with 0.1 μm PVDF micro-filtration membrane under various operating conditions, and the filtration behaviors of actual EPS solution were investigated. The experimental results show that: firstly, the membrane filtration mechanism is governed by cake filtration, and the cake is compressible; secondly, the cake specific resistance increased with the increase of transmembrane pressure (TMP) and decreased as the concentration increased; thirdly, all operating conditions had a significant influence on the cumulative filtrate volume (CFV) of actual EPS solution; finally, the CFV increased with the rise of temperature and TMP, but decreased as EPS concentration increased. The sequence of influence degree of operating conditions is the temperature (38.1%) > the TMP (34.8%) > the EPS concentration (27.1%). A quantitative regression relationship between the CFV and the temperature (T), TMP (ΔP) and EPS concentration (C) was obtained as follows:

$$\text{CFV} = -3.7 \times 10^{-8}C + 5.79 \times 10^{-7}T + 1.27 \times 10^{-4}\Delta P$$

Keywords: Microfiltration; Dead-end; Cumulative filtrate volume; Regression method; Actual EPS solution; Influence degree

1. Introduction

As the shortage of water resource worldwide, the demand for potable water becomes more and more urgent, consequently, the wastewater reclamation becomes increasingly widespread. Thus, many methods, such as ion exchange, resin adsorption and biological treatment, are selected to treat wastewater for reuse [1,2].

The membrane bioreactor (MBR) owing to combination of membrane filtration and biological treatment is becoming one of the most attractive technologies, and it has been used widely in sewage treatment and reclamation [3]. However, in practical applications, the major obstacle to MBRs is membrane fouling, which leads to a decline in membrane flux and shortens the longevity of membrane modules in MBR systems. Therefore, how to ascertain the main substance causing membrane fouling in MBR, and determinate the influence degree of

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various operating conditions on the membrane filtration behaviors of the main substance are very important.

Many researchers take extracellular polymeric substances (EPS), which are mainly composed of polysaccharide, protein, humic substances and uronic acid, as major foulants in the MBRs [4–7]. Shang-Hsin Ou [8] investigated the characteristics of actual EPS and the mechanism of fouling formation in detail. As the extracted EPS has variability in composition, concentration, and complexity due to various MBR systems and different extraction methods, many researchers selected some model solutions, such as alginate [9,10], dextran [11], bovine serum albumin (BSA) [9], β -lactoglobulin [12], lysozyme, myoglobin, cytochrome C [11] or their mixture for example, BSA + alginate [9] which was investigated as model EPS solutions, to study its fouling mechanism and filtration behavior. However, up to present, the filtration behavior of the model EPS solutions can't exactly describe the filtration behavior of actual EPS solution; hence, it is necessary to investigate the filtration behavior of actual EPS solution.

The aim of this paper is to study the filtration behaviors of actual EPS solutions under different operating conditions by using a self-made microfiltration cell, and to ascertain the influence degree of these operating conditions on the cumulative filtrate volume (CFV).

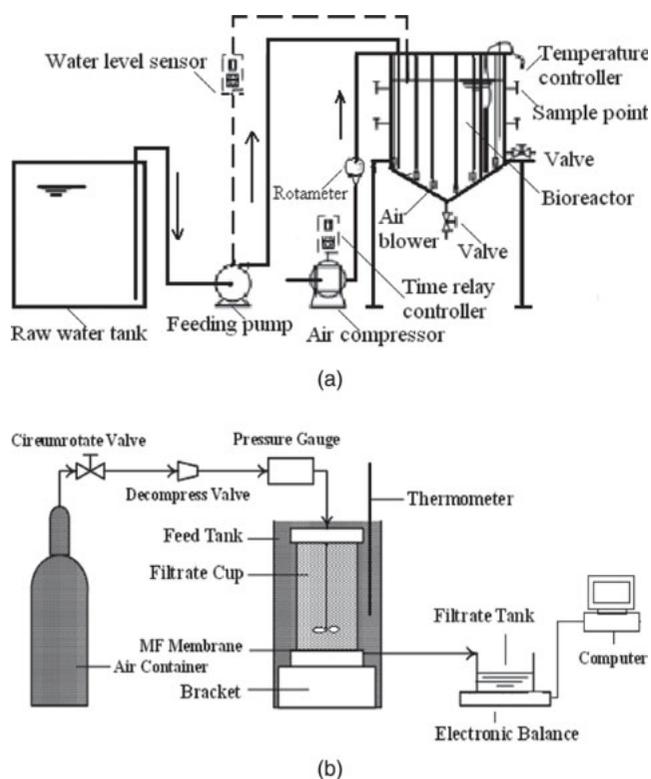


Fig. 1. Schematic diagram of the experimental system.

2. System and methods

2.1. System

The experimental system, as shown in Fig. 1, consists of SBR (a) with an effective volume of 25 L and dead-end filtration set-up (b). Raw wastewater was obtained from the storage tank of domestic sewage. A dead-end microfiltration cell with an effective membrane area of 12.56 cm² was used in this experiment. Before each experiment, 0.1 μ m PVDF hydrophilic membrane which was purchased from Ande Membrane Separation Technology and Engineering (Beijing, China) was soaked in deionized water for 10–12 h to remove glycerin (protectant). Electronic balance was provided by Ohaus Corp Ping Brook, NJ with precision of 0.0001 g. The temperature was controlled by electrical constant temperature boiler (HH SY21-Ni8B).

The operating conditions of different bioreactors are shown in Table 1. When the value of COD and NH₃-N concentrations of influent and the process parameters, such as mixed liquor suspended solid (MLSS), temperature, dissolved oxygen (DO), pH and hydraulic retention time (HRT) were almost stable, the EPS were extracted from the bioreactors using the method as described in 2.2. Then the extracted actual EPS solution was filtered under different TMPs (0.02–0.10 MPa), different temperatures (18–26°C) and different EPS concentrations (35.14–348.75 mg L⁻¹) in a dead-end microfiltration cell as shown in Fig. 1(b), and the experimental data was recorded in the interval of 15 s.

2.2. The extraction and measurement of EPS

The output of actual EPS strongly depends upon the extraction methods [13], compared with other methods, the formaldehyde-NaOH extraction method was chosen in this paper due to the highest amounts of EPS from all the sludges [14–16]. The protein and humic substance contents in EPS are measured by the modified Lowry method [17], while the polysaccharides contents are measured by the anthrone method [18]. The following apparatus were used in the extraction of EPS: LD5-10 centrifuge provided by Beijing Medical Centrifuge Factory Company; HP 8451-A UV spectrophotometer supplied by Beijing optics factory.

The compositions of EPS extracted from different SBRs using the formaldehyde-NaOH extraction method are shown in Table 2. In present paper, the concentration of EPS is the sum of polysaccharides, proteins and humic substances approximately.

2.3. Analytical method

In order to study the influence of various operating conditions on the CFV of actual EPS solution in dead-end

Table 1
Operating conditions of different bioreactors

Operating parameter	Reactor 1	Reactor 2	Reactor 3	Reactor 4	Reactor 5	Reactor 6
MLSS (g L ⁻¹)	1.8	5	4.5	6	7	3
Temperature (°C)	20	27	28	18	27	24
DO (mg L ⁻¹)	5.2	8	4.6	7.5	6	6
pH	7.5	7	6.5	6	6.5	7
HRT (h)	20	21	17	22	26	25

Table 2
Compositions of EPS extracted from activated sludge of SBR (mg L⁻¹)

Reactor	Polysaccharide (mg L ⁻¹)	Protein (mg L ⁻¹)	Humic substance (mg L ⁻¹)	EPS (mg L ⁻¹)
1#	114.0	75.1	10.2	199.3
2#	29.5	39.2	20.2	68.9
3#	178.5	87.8	82.4	348.7
4#	29.0	6.06	0.12	35.1
5#	104.6	62.0	0.30	166.9
6#	204.8	32.7	1.85	239.4

microfiltration cell quantitatively, the experimental data is processed by the regression method as described in literature [19].

2.4. Fouling mechanism

The Blocking Law, first put forward by Herman et al. [20] in 1935, was used to study the fouling mechanism of filtration experiments [21]:

$$\frac{d^2t}{dV^2} = k \left[\frac{dt}{dV} \right]^m \quad (1)$$

where, V is the CFV at time t , m³; k is the proportional coefficient. The exponent m characterizes the fouling mechanism, with $m = 0$ for cake filtration, $m = 1$ intermediate blocking, $m = 3/2$ for pore constriction (also called standard blocking) and $m = 2$ for complete pore blocking [22].

2.5. The calculation of the cake resistance

The cake resistance R_c of the EPS solution extracted from activated sludge of SBRs was measured by Eq. (2) [23]:

$$J = \frac{dV}{Adt} = \frac{\Delta P}{\eta(R_c + R_m)} \quad (2)$$

where, J is the permeate flux, m³ m⁻² h⁻¹; ΔP is the TMP, Pa; η is the viscosity, Pa s; R_m and R_c are the intrinsic membrane resistance and the resistance of deposition layer respectively, m⁻¹.

2.6. The calculation of cake mass

The cake mass M of the EPS solution extracted from activated sludge of SBRs was measured by Eq. (3) [24]:

$$R_c = \alpha_c \cdot M \quad (3)$$

where, α_c is the cake specific resistance, m kg⁻¹; R_c is the cake resistance, m⁻¹; M is the cake mass on membrane surface, kg.

2.7. The calculation of specific resistance

The specific resistance α_c of the EPS solution extracted from activated sludge of SBRs was measured by Eq. (4) [9]:

$$\frac{t}{V} = \frac{R_m \mu}{\Delta P A_m} + \alpha_c \left[\frac{\eta C}{2 \Delta P A_m^2} \right] V \quad (4)$$

where V is the CFV at time t , m³; A_m is effective filtration area, m²; α_c is the cake specific resistance, m kg⁻¹. η is the viscosity, Pa s.

Eq. (4) showed that, the slope of the line can be got through plotting t/V versus V , and it can be used to calculate the cake specific resistance α_c . The intercept of the line can be used to calculate the intrinsic membrane resistance R_m .

2.8. Determination of formed cake compressibility

The relationship between the cake specific resistance and the TMP is represented as follows [9]:

$$\alpha_c = \alpha_{c0} \Delta p^n \quad (5)$$

The exponent n can be found from the slope of line between $\lg(\alpha_c)$ and $\lg(\Delta p)$. When $n = 0$, the cake is incompressible, but when $n > 0$, the cake is compressible.

3. Results and discussion

3.1. Membrane fouling mechanism

All experimental data were analyzed using classic filtration laws developed by Hermia [20]. The t/V value within 1290 s was plotted to t as the first part show in Fig. 2(a) and the t/V value after the 1290th s was plotted to V as the second part shown in Fig. 2(b), respectively, filtration met the standard blocking law described as $t/V = at + b$ in Fig. 2(a), then the cake filtration model described as $t/V = aV + b$ could be applied up to the end of the run as shown in Fig. 2(b). The time of standard blocking was very short and the most time of filtration met the cake filtration. In another word, all experimental data can almost be described by the cake filtration model because the major components, such as the polysaccharide and protein were rejected by membrane easily, and cake formed on the membrane surface [25].

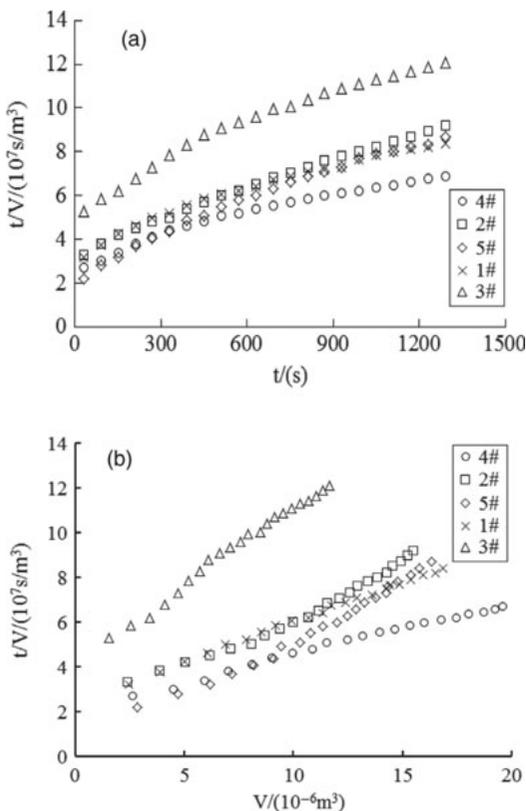


Fig. 2. Standard blocking (first part whose filtration time was from 0 to 1290 s) model (a) and cake filtration (second part whose filtration time was after 1290th s) model (b) of different EPS solutions for 0.1 μm PVDF membrane, at 0.06 MPa.

3.2. Calculation of the cake specific resistance and the compressibility of cake layer

The concentration order of EPS extracted from different reactors is reactor 4#, 2#, 5#, 1# and 3#. The cake resistance R_c can be calculated by using the Eqs. (2) and (4), and the results were shown in Fig. 3. The cake mass M formed during filtration can be calculated by using the Eq. (3) for different bioreactors and the results were shown in Fig. 4. The specific resistance α_c can be calculated by using the Eq. (4), and the results were shown in Fig. 5.

It can be seen from Fig. 5 that the cake specific resistance increased with the increase of TMP in the range of 0.02–0.1 MPa for each reactor. With the increase of filtration time, the large molecules or particles are adsorbed and aggregated gradually in the membrane pores or on the membrane surface, and then the cake layer formed on the membrane surface. Finally, the porosity of compressible cake reduces due to the rearrangement and transmutation of solid particles as TMP increases.

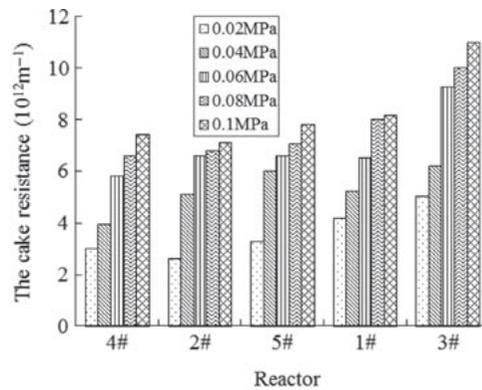


Fig. 3. The cake resistance R_c of actual EPS solution under different operating conditions.

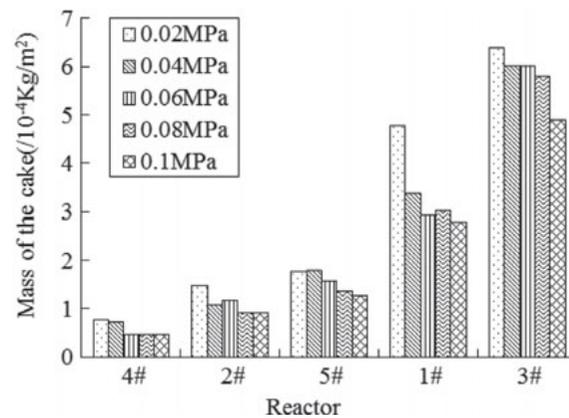


Fig. 4. The mass of the cake M of actual EPS solution under different operating conditions.

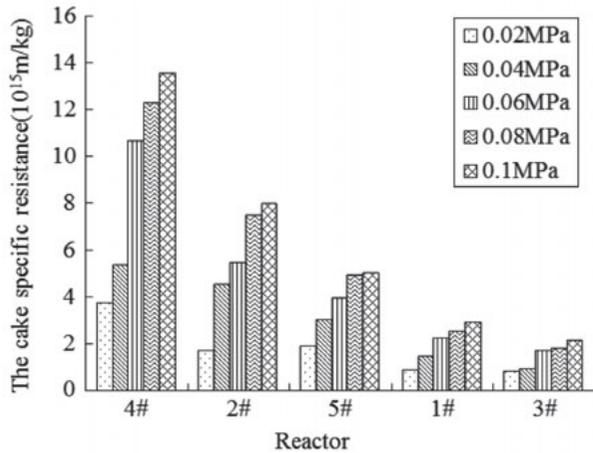


Fig. 5. The cake specific resistance α_c of actual EPS solution under different operating conditions.

It also can be seen from Fig. 5 that the cake specific resistance decreased with the increase of EPS concentration as described in the literatures [26,27]. The mass of the cake layer increased greatly with the increase of EPS concentration in Fig. 4, but the cake resistance increased gently with the increase of EPS concentration as shown in Fig. 3, so the cake specific resistance decreased with the increase of concentration.

Moreover, the cake compressibility can be calculated by using the Eq. (5) for different bioreactors and the results were shown in Fig. 6. It can be seen from Fig. 6, the value of n (the cake compressibility) is between 0 and 1, which means the cake formed on the membrane surface during the filtration is compressible. In addition, the value of n increased with the increase of DO concentration in Fig. 7 as described in the literature [28]. It can be considered that the activated sludge of SBR is much looser with the increase of DO concentration. Therefore, the

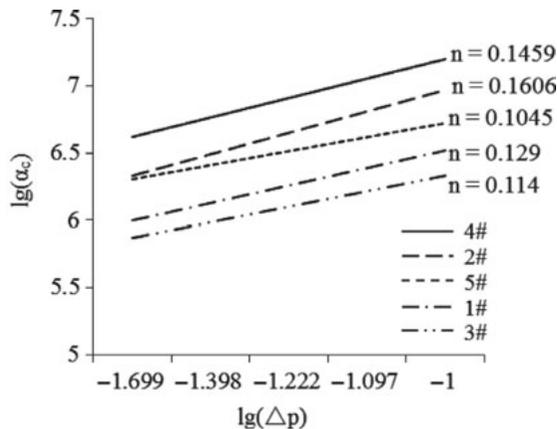


Fig. 6. The compressibility of cake forms during filtration of the actual EPS solution for different bioreactors.

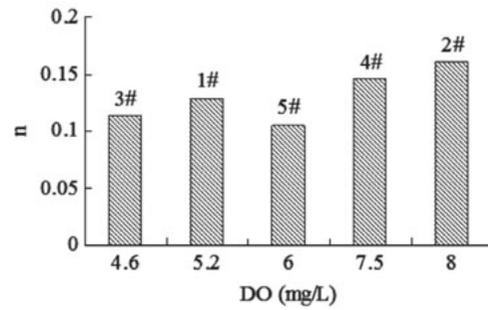


Fig. 7. The cake compressibility n vs. DO of actual EPS solution under different operating conditions.

compressibility (the value of n) of cake increased with the increase of DO concentration, the higher DO concentration caused the higher compressibility, and the n became larger.

3.3. Impact of pressure and concentration on the CFV

The compositions of EPS extracted from different SBRs are shown in Table 2. All actual EPS solutions were carried out at the actual temperature and pH with 0.1 μm PVDF membrane for 1210 s and the results are shown in Fig. 8.

It can be seen from Fig. 8 that the CFV increased with the increase of TMP in all the range of 0.02–0.1 MPa for each reactor. It can be considered that the driving force of the filtration process increased with the increase of TMP. On the one hand, in the TMP range of 0.02–0.04 MPa, when the TMP is lower, the rising trend of CFV with the increase of TMP is obvious due to the stronger driving force and less pollution of the filtration process; On the other hand, in the range of 0.04–0.08 MPa the rising trend of the CFV is slower than that in the range

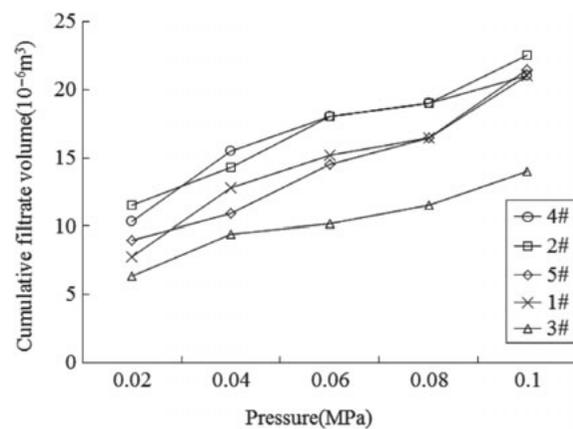


Fig. 8. The CFV vs. the TMP of actual EPS solution under different operating conditions.

of 0.02–0.04 MPa or 0.08–0.10 MPa. The CFV alters non-linearly in all the range of TMP. It can be explained that there are concentration polarization and less pollution on the membrane surface in the low TMP. As TMP increased, the large molecules or colloid particles were adsorbed and aggregated in the membrane pores or on the membrane surface, and the membrane pores diminished gradually. Hence, more foulants on the membrane surface caused the slower increase of CFV with the increase of TMP. However, the CFV increased more quickly at the higher TMP range of 0.08–0.10 MPa than that at the range of 0.04–0.08 MPa, this was because that higher TMP had larger influence on CFV than cake resistance caused by foulants accumulated on the membrane surface or in the membrane pores in filtration process.

In addition, the CFV under different concentrations for the same TMP decreases with the increase of EPS concentration in Fig. 8. This lower CFV was due to thicker cake formation which generated higher total resistance. With the same filtration time of 1210 s, the thicker the cake, the less the CFV. The viscosity term in Darcy’s law refers to the viscosity of pure liquid not the slurry so the higher slurry viscosity does not have an impact on Darcy’s law.

3.4. The effect of operating conditions on the CFV

The influence of operating conditions on the CFV of actual EPS solution was analyzed by using regression method quantitatively and the result was shown in Table 3. The TMP, concentration and temperature are represented by ΔP , C and T , respectively. The basic multivariate linear regression model is described as following [29]:

$$CFV = b_1C + b_2T + b_3\Delta P \tag{6}$$

where, b_1 , b_2 and b_3 are regression coefficients.

If the regression coefficient divides the relative standard error, then F_j is obtained by:

$$F_j = b_j / \sigma_j \tag{7}$$

where, $j = 1, 2, 3$; b is the regression coefficient, and σ is the relative standard error. If $|F_j| > 1$, the factor is

considered as an influence factor, otherwise, the factor has no effect on model parameter or there is a very small influence.

The bigger the absolute value of F_j is, the larger the effect of the factor on CFV is. If the absolute value of $F_j < 1$, it will be regressed again excluding the factor. It can be seen from Table 3, all absolute values of $F > 1$, which indicated that the TMP, concentration and temperature all had effect on the CFV. According to the regression results shown in Table 3, the absolute values of F_j , the sequence of influence degree of operating conditions on CFV was the temperature (38.1%) > the TMP (34.8%) > the concentration (27.1%), and the results are shown in Fig. 9.

The regression equation between CFV and operating conditions was obtained using the experimental data from reactor 1#, 2#, 3#, 4# and 5#, to predict the CFV under a significant level $\alpha = 0.05$, as follows:

$$CFV = -3.7 \times 10^{-8}C + 5.79 \times 10^{-7}T + 1.27 \times 10^{-4}\Delta P \tag{8}$$

where, CFV is the CFV, ml; T is the temperature, °C; ΔP is the TMP, MPa; and C is the actual EPS concentration, mg L⁻¹.

It can be seen from the Eq. (8), the CFV increased with the increase of temperature and TMP, but decreased with the increase of EPS concentration.

The model prediction is validated by experimental data. The relative error was used as a testing index. The experiment data from reactor 6# which were not used to build the regression formula was performed to validate the availability of the regression formula and the result was shown in Table 4.

It can be seen from Table 4, the experimental results from reactor 6# which were obtained under different operating pressures were used to compare with the experimental values and theoretical values, and the average relative error was less than 1.5%. The results show good agreement between model prediction and experimental observation. It also indicates that the operating conditions have an effect on the CFV. Therefore, the regression formula can predict the CFV of actual

Table 3
Values of regression coefficient, F_j and D_j for cumulative filtration volume

Regression times	j	b_j	σ_j	F_j	D_j (%)
1	C	-3.7×10^{-8}	2.54×10^{-9}	-14.43	27.1
1	T	5.97×10^{-7}	3.38×10^{-8}	17.11	38.1
1	P	1.27×10^{-4}	7.76×10^{-6}	16.36	34.8

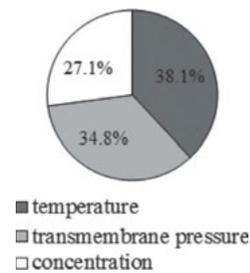


Fig. 9. The influence degree of different operating conditions on the cumulative filtration volume.

Table 4
Comparison of the experimental values with the theoretical values

No.	C (mg ⁻¹ L ⁻¹)	T (°C)	P (MPa)	Experimental value, × 10 ⁻⁶ (m ⁻³)	Theoretical value, × 10 ⁻⁶ (m ⁻³)	Relative error (%)	Arithmetic average value (%)
1	239.3	21	0.02	5.92	5.94	0.40	
2	239.3	21	0.06	11.2	11.0	-1.19	1.5
3	239.3	21	0.1	15.3	16.1	5.34	

EPS solutions extracted from SBRs during the dead-end microfiltration procedure successfully.

4. Conclusions

In this study, the filtration behavior of actual EPS extracted from activated sludge suspension of SBR was investigated detailedly in dead-end filtration cell under different operating conditions. The first part of filtration met the standard blocking, while the following second part satisfied cake filtration model. The cake layer was compressible, and the value of cake compressibility ranged from 0 to 1. The cake specific resistance of actual EPS increased with the increase of the TMP, whereas decreased with the increase of EPS concentration. All operating conditions had significant influences on the CFV of actual EPS solution. The CFV increased with the increase of temperature and the TMP, but decreased with the concentration. The sequence of influence degree of operating conditions is the temperature (38.1%) > the TMP (34.8%) > the concentration (27.1%). In addition, the temperature and the TMP had positive contribution to cumulative filtration volume. EPS concentration had negative contribution to cumulative filtration volume. The effects of operating conditions on the CFV are quantitatively expressed by:

$$CFV = -3.7 \times 10^{-8} C + 5.79 \times 10^{-7} T + 1.27 \times 10^{-4} \Delta P$$

The results show good agreement between model prediction and experimental observation.

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Symbols

CFV	—	cumulative filtrate volume, m ³
V	—	total filtered volume, ml
k	—	proportionality coefficient

J	—	permeate flux, L m ⁻² h ⁻¹
ΔP	—	transmembrane pressure, Pa
H	—	viscosity Pa s
R _m	—	intrinsic membrane resistance, m ⁻¹
R _c	—	resistance of deposition layer, m ⁻¹
T	—	temperature, °C
C	—	concentration of actual EPS, mg L ⁻¹

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