Desalination and Water Treatment
www.deswater.com
doi: 10.1080/19443994.2015.1008576

57 (2016) 6184–6192
March

Taylor & Francis
Taylor & Francis Group

Dead-end filtration of jet loop bioreactor and activated sludge suspensions

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Received 18 April 2014; Accepted 9 January 2015

ABSTRACT

Fouling is the main disadvantage of membrane processes which causes decrease in flux with time. Although there are various mathematical models representing fouling with time, a simplified approach is still needed for predicting flux decline behavior over the course of filtration. Furthermore, a comparative study to determine the effects of floc size and extracellular polymeric substance (EPS)/soluble microbial product (SMP) content for different biological suspension is important in understanding of membrane fouling. The objective of this study was to analyze filtration of two different biological suspensions and to use a simplified model to identify the effects of SMP, EPS, and floc size on fouling. The experimental data were successfully represented by a simplified model with a single fitting parameter. The fouling was independent from membrane material and pore size; however, it depended on either floc size or the EPS_P content depending on the properties of biological suspension. When the floc size of the biological suspension was small (e.g. jet loop membrane bioreactor), neither EPS nor SMP contributed fouling significantly. The fouling was mainly dominated by the small floc size. However, for biological suspension with larger floc sizes (e.g. activated sludge system), the fouling was independent from floc size. For such sludge samples, the fouling was well correlated with EPS_P.

Keywords: Dead-end filtration; Flux modeling; Jet loop bioreactor; Activated sludge

1. Introduction

Separation of microbial suspension by membranes in biological treatment systems has been widely used in recent years. Membrane processes have various advantages including smaller foot print and excellent effluent quality; however, fouling is the major problem for membranes. There have been a number of publications in the literature for modeling of membrane fouling with time. Hermia [1] developed a model to describe the fouling mechanism with different blocking mechanisms.

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

The model describes the complete blocking if $n = 2$, standard blocking if $n = 1.5$, intermediate blocking if $n = 1$, and cake filtration if $n = 0$. The model was applied in various studies including separation of proteins [2–4], micro-organisms [5], and natural organic matters [6]. Hlavacek and Bouchet [2] reported that

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the intermediate blocking well represented the filtration of BSA protein suspension. Bowen et al. [3] reported the occurrence of initial complete blocking or a mixture of complete and standard blocking which was followed by a cake filtration. The initial blocking mechanism was attributed to the wide variation of pore size distribution. If the pore size was smaller than the particle size of the suspension, the initial step was complete blocking; whereas, if the particle size was smaller than the pore size of the membrane, the initial fouling was standard blocking. Wang and Tarabara [7] found that the fouling was a mixture of intermediate, standard blockings, and cake filtration with time. The degree of each mechanism with filtration volume varied significantly. For example, standard blocking followed by intermediate blocking and cake filtration for UF 300 kDa membrane; whereas, intermediate blocking was followed by standard blocking and cake filtration for UF 100 kDa membrane.

Although Hermia model represents the fouling relatively well at different stages of filtration, it does not provide a single mathematical relationship over the course of filtration. Ho and Zydney [8] developed a model in which simultaneous pore blocking and cake formation were represented in a single mathematical relationship. It was assumed that the total flow was contributed by both open and blocked pores and the flow rates were calculated by Darcy's Law. This model was successfully applied for the filtration of humic substances [9]. Bolton et al. [10] developed a new combined model with only two fitting parameters as oppose to three fitting parameters of the model developed by Ho and Zydney [8]. These models represented the experimental data relatively well. However, in all these studies well-defined solutions (i.e. known concentrations of single protein or humic substances) were used. Classical activated sludge systems (CASs) contain a mixture of a very complex media including various proteins, carbohydrates, colloids, and micro-organisms. There have been significant number of studies in the literature that identify the fouling mechanisms of the membranes. However, the results are controversial. One group of researchers identified extracellular polymeric substances (EPSs) and soluble microbial products (SMPs) to be the major compounds causing membrane fouling [11,12]. The other group of researchers identified the colloidal particles as the major foulants in addition to the biopolymers [13,14]. Description of membrane fouling mechanisms in biological systems is usually more complicated and therefore modeling fouling in such systems requires many assumptions. A simplified modeling approach with the effects of fouling parameters can be useful in describing membrane fouling of the filtration of microbial suspensions.

The objectives of this study were to investigate the filtration behavior of biological suspensions obtained from two different types of reactors with different floc sizes and to use a simplified single model to describe the flux decline behavior over the course of filtration and to identify the effects of SMP, EPS, and floc size on fouling. Two different activated sludge samples, obtained from an CAS and a jet loop membrane bioreactor (JLBR), were used in dead-end filtration experiments. Sludge samples were taken at different periods at which the reactors were operated at different solid retention times (SRT). JLBR sludge had substantially smaller floc size than that of CAS. Dead-end filtration experiments were conducted at constant pressure with various membrane materials and different pore sizes. mixed liquor suspended solids (MLSS), SMP_p, SMP_c, EPS_p, and EPS_c analysis were conducted for each sludge samples. As a result, a comparative study was conducted to identify the effects of floc sizes as well as the influences of EPS and SMP contents on fouling.

2. Materials and methods

2.1. Dead-end filtration experiments

The sludge samples were obtained from a pilot JLBR and a pilot CAS reactors operated in our laboratories. These two pilot plant reactors were operated for long periods at different SRTs. A dead-end filtration module with 35.24 cm^2 filtration area and without mixing was used in the experiments. A constant transmembrane pressure of 0.8 bar was provided with a pressurized N_2 gas tank. Outflow was collected in a beaker which was placed on top of a balance, and the data from the balance were collected in a computer at one-minute intervals for the flux calculations. The filtration experiments were conducted using three different membranes with various pore sizes.

2.2. Membranes

Three types of membranes were used in the experiments each with various pore sizes. All membranes were obtained from GE Osmonics. The properties of membranes used in the experiments are given in Table 1. Nitrocellular mixed ester (ME) membranes are composed of a mixture of inert cellulose nitrate and cellulose acetate polymers. They are hydrophilic membranes with interconnected pores. Polyethersulfone (PES) membranes are also hydrophilic. The pore structure and the roughness of the surface looked similar to ME membranes. Cellulose acetate membranes (SA) had a very rough surface with irregular pores.

Table 1
Properties of membranes used in the experiment

Membrane type	Pore size (μm)	Thickness[a] (μm)	R_m[b] (m^{-1})	Contact angle[c] (°)	Roughness[d] (nm)
PES	0.45	110–150	5.1×10^{10}	<5	23.0
	0.22	110–150	7.3×10^{10}	<5	41.9
	0.10	110–150	14.4×10^{10}	<5	41.9
ME	0.45	150	6.4×10^{10}	<5	13.8
	0.22	150	6.7×10^{10}	<5	54.9
	0.10	150	9.4×10^{10}	<5	61.7
SA	0.45	65–110	5.1×10^{10}	<5	89.1
	0.22	65–110	7.5×10^{10}	<5	138.6

[a]Provided from manufacturer.
[b]Measured in the laboratory.
[c]Measured by Goniometer.
[d]Measured by AFM.

The size of pores in surface was more than the nominal pore size of the membrane.

2.3. Analytical methods

Analytical methods from the "Standard Methods for the Examination of Water and Wastewater" were adopted for the measurement of MLSS [15]. The EPS extraction method followed that reported by Li et al. [16]. The measurement of protein content was carried out according to Lowry method [17]. BSA was used as a standard and the results expressed in mg equivalent of BSA per liter. Protein concentrations were determined using a UV–vis spectrophotometer (GBC-Cintra-20) at the wavelength of 660 nm. Polysaccharides were determined by Dubois method [18]. All the analyses were conducted in duplicate and their average values were reported. The viscosity of mixed liquor was measured by a viscosity meter (Brookfield DV-E Viscometer). Zeta potential of each sludge sample was measured using a Zetameter (ZetaMastersize). The floc sizes of the sludge samples were analyzed with a laser particle size analyzer (MasterSizer 2000).

3. Results and discussion

3.1. Properties of sludge samples

Sludge samples were obtained on different days from two pilot plants running in the laboratory. Before each filtration experiments, MLSS, SMP_c, SMP_p, EPS_p, and EPS_c analysis were conducted and the results are presented in Table 2. JLBR was operated at two different SRT modes. The first was infinite SRT for which no sludge was wasted and MLSS was about 2,000 mg/L in the reactor. In this period, the reactor was fed with a synthetic wastewater with COD of 1,000 mg/L. In the second mode, the SRT was adjusted to 3 d, and MLSS was approximately 10,000 mg/L with influent COD of 5,000 mg/L. CAS system was operated at 10 and 30 d SRTs for which MLSS values were about 3,000 and 5,800 mg/L, respectively. The influent COD was about 1,000 mg/L at both SRT in CAS. The reactors were operated at least two months before the filtration experiments conducted. Both SMP_p and SMP_c values ranged from 7 to 27 mg/L and did not show apparent differences at different SRT as well as in different reactors. EPS_c and especially EPS_p were greater when SRT was infinity in JLBR. When no sludge was wasted, the excretion of intracellular polymers and cell lyses likely occurred with slower degradation rate due to low MLSS concentration. As a result, total protein and carbohydrate concentration increased. These constituents existed bounded to the micro-organisms. Opposite behavior was observed in CAS; as the SRT increased from 10 to 30 d, both EPS_p and EPS_c decreased. Various results have been presented in the literature. Ng and Hermanowicz [19] observed an increase in EPS when SRT increased from 0.25 to 5 d. Reid et al. [20] observed almost no change in EPS content at SRTs of 20, 40, and 60 d of operation. However, in another study, total bound EPS content decreases from 45–70 to 20–40 mg/g VSS when SRT increased from 10 to 53 d [11] which was similar to the results obtained in this study. It was concluded that the EPS content decreased only when SRT increased above a critical value [11].

One of the main factors influencing membrane fouling is the particle sizes of the colloids and the flocs. The floc size distribution of each sludge samples is presented in Fig. 1. The average floc sizes for JLBR sludge were about 13 and 6 μm and zeta potentials were −12.0 and −10.2 mV for SRT of infinity and three days, respectively. Large number of flocs at the range of 0.2–1 μm were observed in JLBR sludge with SRT

Table 2
Properties of sludge samples for each filtration experiment

Membrane	Reactor	SRT d	MLSS mg/L	SMP_p mg/L	SMP_c mg/L	EPS_p mg/g MLSS	EPS_c mg/g MLSS
PES 045	JLBR	∞	2,040	8.3	24.0	41.4	101.9
PES 02			2,090	17.6	26.8	63.4	88.0
PES 01			1,970	8.3	23.6	45.9	107.6
SA 045			1,966	26.5	21.6	31.5	99.2
SA 02			2,090	17.6	26.8	63.4	88.0
ME 045			1,950	7.7	23.6	31.0	95.9
ME 02			1,966	26.5	21.6	31.5	99.2
ME 010			1,970	8.3	23.6	45.9	107.6
PES 045	JLBR	3	10,200	24.4	19.6	29.9	17.6
PES 02			9,700	26.4	24.9	27.1	35.5
PES 01			9,800	19.5	20.8	21.5	22.8
SA 045			9,750	20.7	21.2	21.0	22.6
SA 02			9,700	26.4	24.9	27.1	35.5
ME 02			9,750	20.7	21.2	21.0	22.6
ME 010			10,200	24.4	19.6	29.9	17.6
PES 045	CAS	10	3,020	15.7	13.5	55.9	61.3
PES 02			2,800	27.2	27.4	68.3	47.8
PES01			2,810	17.0	20.6	60.7	126.3
SA 0.45			3,000	13.8	13.2	63.9	79.0
SA 0.2			3,070	16.6	28.1	85.6	98.7
PES045	CAS	30	5,800	7.7	17.0	34.2	59.0
PES02			5,730	6.9	14.2	33.6	44.7
PES01			6,020	9.0	9.7	26.6	44.7
SA 0.45			5,800	7.7	17.0	34.2	59.0
SA 0.2			6,050	7.8	7.2	38.5	41.3

of 3 d. On the other hand, much greater floc sizes were observed in CAS suspension. JLBR sludge had much smaller floc size than CAS suspension because of the excess sheer that occurred by circulating the sludge within the reactor to form jet effect. The average floc size was about 90 μm at SRT of 10 d; whereas, it was about 55 μm at SRT of 30 d. The floc size decreased as the SRT increased in CAS. That was attributed to the reduction in EPS and increase in MLSS with increasing SRT. Floc size reduction due to decrease in EPS has been reported in the literature [21,22]. Defrance and Jaffrin [23] also reported the floc size reduction from 200 to 50 μm when MLSS content increased from 3,500 to 10,000 mg/L.

3.2. Simplified filtration model and effects of sludge properties on fouling

In order to investigate the filtration behavior of biological suspension, first, Hermia model was used. The cake filtration is the dominant mechanism when $n = 0$ and a linear relationship is observed between dt/dV and V. The change of dt/dV with V for CAS and JLBR samples at different MLSS concentrations for PES 0.22 μm membrane is presented in Fig. 2. A linear relationship was observed after a few minutes of filtration suggesting that the filtration behavior of biological suspension exhibited the cake filtration. A nonlinear behavior was observed in the first five minutes of filtration that was likely due to the other blocking mechanisms suggested by the model. However, the initial blocking mechanism was not investigated separately since the transition was fast and there were only a few data points. A slightly concave down behavior was observed then reaching linearity at longer filtration volumes for activated sludge with MLSS concentration of 5,800 mg/L. Since the dominant fouling was the formation of cake layer a different approach was followed to model the filtration of biological suspension with time. Darcy's Law was assumed to represent the flux through the blocked pores by

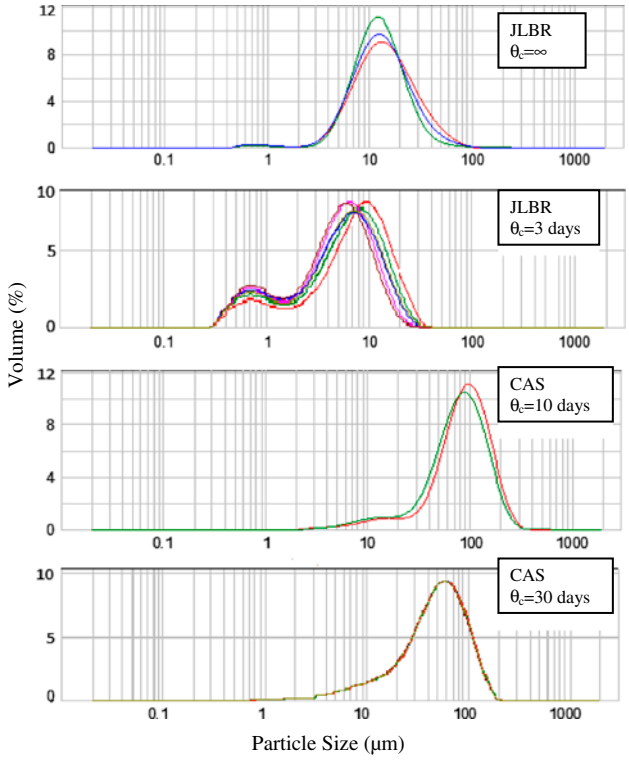

Fig. 1. Floc size distribution of sludge suspensions of JLBR and CAS at different sludge retention times.

$$J = \frac{\Delta P}{\mu(R_m + R_c)} = \frac{\Delta P}{\mu R_T} \tag{2}$$

where ΔP is the transmembrane pressure, μ is the viscosity of solution passing through the filter, R_m is the membrane resistance, R_c is the cake resistance, and R_T is the total resistance. It was assumed that the total filtration resistance was proportional to the mass accumulation rate and specific filtration resistance as follow:

$$\frac{dR_T}{dt} = \alpha \frac{dM}{dt} \tag{3}$$

where α is the filtration resistance, M is the mass of the biological suspension, and t is the time. A similar relationship was used for the cake resistance in other studies [8,24]. By mass balance approach, the accumulation of the mass can be expressed as follow:

$$\frac{dM}{dt} = f\ C_B\ A_o\ J \tag{4}$$

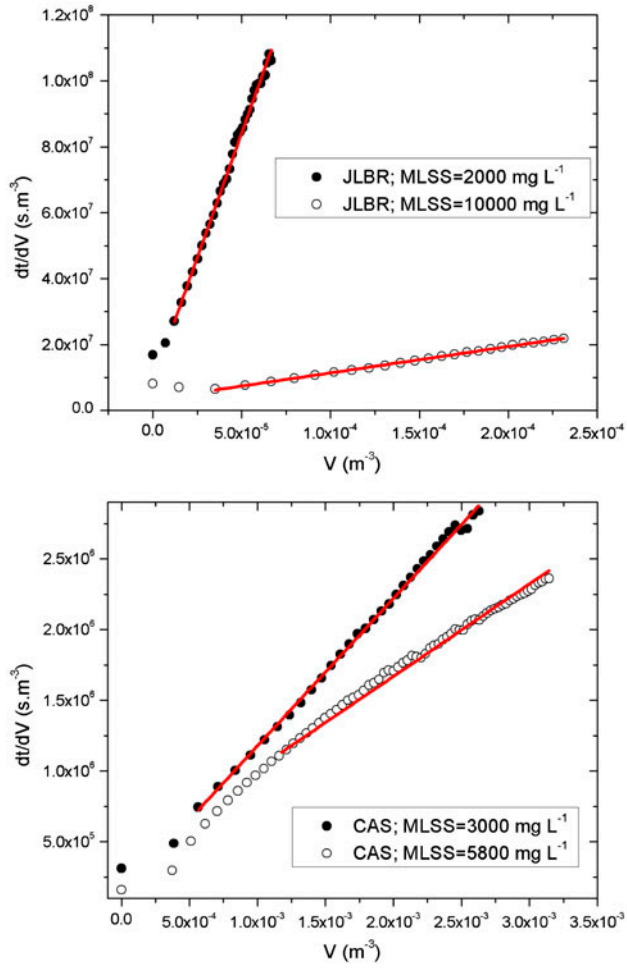

Fig. 2. Hermia relationship for cake filtration (PES 0.22 μm membranes).

where f is the fraction of the micro-organisms contribute to the cake formation, C_B is the concentration of the micro-organisms, A_o is the filtration area, and J is the permeate flux. Substituting Eq. (3) into Eq. (2), the change of total filtration resistance can be expressed as

$$\frac{dR_T}{dt} = \alpha\ f\ C_B\ A_o\ J = \alpha^*\ C_B\ \frac{\Delta P}{\mu R_T} \tag{5}$$

$$\int_{R_m}^{R_T} R_T\, dR_T = \int_0^t \alpha^*\ \frac{\Delta P}{\mu} C_B\ dt \tag{6}$$

An integration yields,

$$R_T = R_m \sqrt{2\,\alpha^* \frac{\Delta P}{\mu} \frac{C_B}{R_m^2} t + 1} \tag{7}$$

where R_m is the membrane resistance. The total filtration resistance can be substituted into Eq. (2) to predict the flux with a single fitting parameter (α^*). This simplified modeling approach was used to investigate the fouling behavior in this study.

The total resistance (R_T) change with time for each filtration experiments were found by using Eq. (2) and they were fit into Eq. (7) to determine α^* values. A sample fit for R_T and flux is presented in Fig. 3. The resistance showed an initial rapid increase followed by a steady raise with time. The resistance in JLBR suspension was greater than those obtained for CAS samples, indicating more fouling. The fitted α^* values in all filtration experiments for both JLBR and CAS suspensions are presented in Fig. 4(a) and (b). Only small variations were observed in α^* values for JLBR sludge with 2,000 mg/L of MLSS, which were ranging from 47.4×10^{13} to 70.9×10^{13} with an average value of 58.4×10^{13}. Relatively more variations were observed in α* values when MLSS increased to 9,870 mg/L. The values were in between 28.7×10^{13} and 70.9×10^{13}. Reductions were observed in PES 0.22 and SA 0.22 membranes; whereas, the increases were obtained in four other membranes. However, the variations were not following a specific pattern which could not be attributed as an effect of the membrane properties. Overall, the difference between α^* values at low and high MLSS values were not significant. In Fig. 4(b), results obtained for CAS suspension is presented for two different membrane materials and various pore sizes. α^* values for CAS suspension was about 10fold smaller than those for JLBR suspensions. When MLSS concentration was about 3,000 mg/L, α^* values ranged from 2.5×10^{13} to 3.5×10^{13} with the exception of SA 022 membrane for which α^* was 6.4×10^{13}. When MLSS increased, α^* decreased almost by half in all membranes tested, which was a clear indication of less fouling.

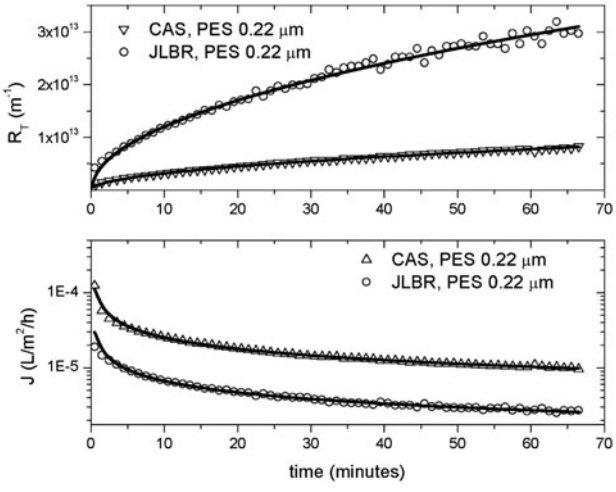

Fig. 3. Variation of R_T and J with time and model fit (MLSS for JLBR = 2,090 mg/L; MLSS for CAS 2,800 mg/L).

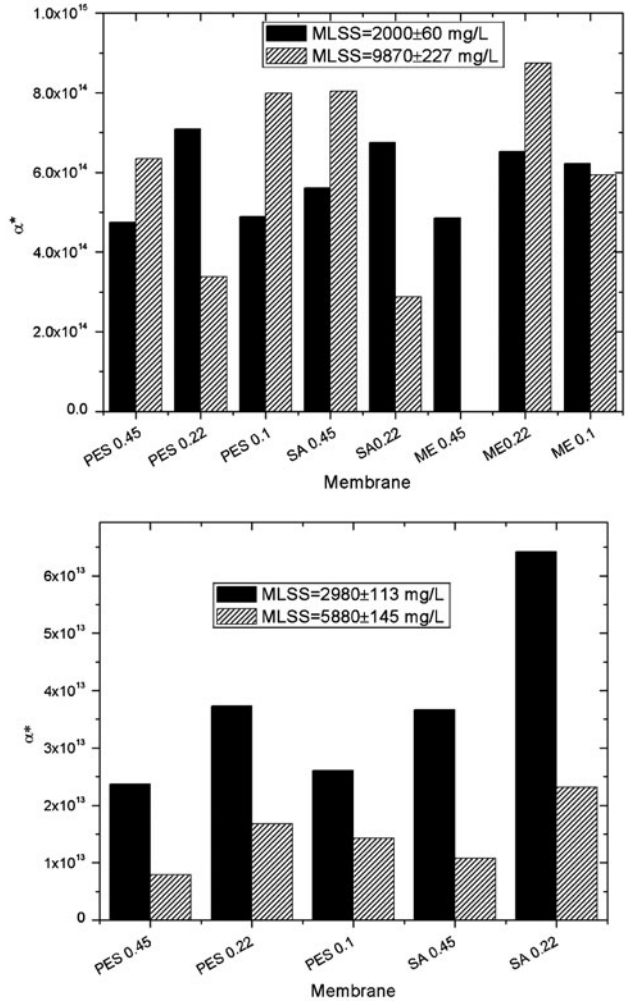

Fig. 4. Variation of α* values for different membrane materials and pore sizes (a) JLBR and (b) CAS.

Based on these observations, several conclusions could be made. It was concluded that the effect of membrane material and pore size was not significant on the filtration of biological suspensions and the filtration was dominated by the cake layer since α* values did not change significantly at each MLSS for the sludge samples obtained from different biological reactors. On the contrary, apparent influence of membrane materials and pore size have been reported in the literature. Dizge et al. [25] reported that in the cross-flow microfiltration of activated sludge ME membranes yielded greater flux than those of polycarbonate, cellulose acetate and PES membranes. They

Table 3
Pearson correlation matrix

	α^*	MLSS mg/L	SMP_p mg/L	SMP_c mg/L	EPS_p mg/g MLSS	EPS_c mg/g MLSS
JLBR						
α^*	1	0.114	0.015	−0.374	−0.034	−0.224
MLSS	0.114	1	0.569	−0.500	−0.694	−0.986
SMP_p	0.015	0.569	1	−0.324	−0.430	−0.581
SMP_c	−0.374	−0.500	−0.324	1	0.727	0.506
EPS_p	−0.034	−0.694	−0.430	0.730	1	0.644
EPS_c	−0.224	−0.986	−0.581	0.510	0.644	1
CAS						
α^*	1	−0.702	0.633	0.629	0.924	0.471
MLSS	−0.702	1	−0.897	−0.610	−0.899	−0.635
SMP_p	0.633	−0.897	1	0.795	0.788	0.329
SMP_c	0.629	−0.610	0.795	1	0.731	0.477
EPS_p	0.924	−0.899	0.788	0.731	1	0.619
EPS_c	0.471	−0.635	0.329	0.477	0.619	1

also reported that the fouling decreased with increasing pores size. Yamato et al. [26] found that Polyvinylidene fluoride membrane was much better than polyethylene membrane in terms of prevention of irremovable fouling. It was reported that the fouling behavior in MBRs was determined by the affinity between EPS/SMP and membrane [27]. The contradiction between these results and the results obtained in this study can be attributed to the mode of filtration. A sheer force exists on the surface of membranes scouring the cake layer and therefore the interaction between the membrane and the foulants are more important. However, in dead-end filtration without mixing accumulation of foulants occur on top of the membranes. As a result, cake layer controls the filtration rather than the initial interaction of foulants and the membranes.

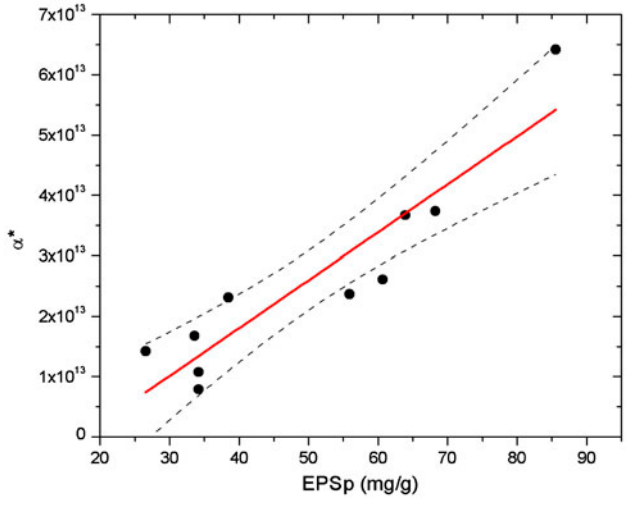

Fig. 5. Variation of α* values with EPS_p content.

Results also showed that as MLSS increased from 3,000 to 5,800 mg/L, α^* values decreased for CAS suspensions causing reductions in the cake resistance. On the other hand, no apparent changes in α^* values were observed for JLBR sludge. Slight increases were observed for four membranes but for the other two membranes decreases were observed. It was also shown that JLBR sludge samples yielded much greater α^* values than CAS samples indicating greater fouling. In order to explore the potential reasons that causes changes in α^* values, Pearson Correlation matrix were determined for α^* and sludge properties. The results are presented in Table 3. There was no correlation among α^* values and the chemical sludge properties for JLBR sludge. At both MLSS, α^* values remained independent from MLSS, SMP, and EPS. On the other hand, strong positive correlations were observed between α* values and EPS_p for CAS samples, indicating that the EPS_p was the major foulant causing greater cake resistance. As MLSS increased EPS_p decreased, and therefore, lower fouling was observed. The change of α^* values with EPS_p showed almost a linear increase, which was presented in Fig. 5. The dotted lines in Fig. 5 is the 95% confidence interval. The role of EPS on fouling was also addressed in the literature with contradicting reports. Cho et al. [28] and Ahmed et al. [29] found that increase in EPS concentration increased the specific cake resistance. On the other hand, Rosenberger and Kraume [12] reported that SMP had more impact on filtration and EPS had no effect.

Strong negative correlations among MLSS, EPS_p, and EPS_c were also observed in JLBR; however, they

did not affect the total resistance as was the case in CAS suspension. The major difference between CAS sludge and JLBR sludge was the particle size. The average particle size of JLBR sludge was much lower than that of CAS samples as discussed above. It was concluded that when the floc size was small (e.g. JLBR sludge), EPS and SMP did not effect the fouling substantially. The dominant factor influencing the fouling was the smaller particle size of the microbial flocs. Whereas, as the floc size became larger (e.g. CAS samples), the cake structure was affected by the EPS content, forming looser cake structure due to the reduced EPS_P concentration. That results lower cake resistance.

4. Conclusions

The conclusions can be summarized as follow.

(1) Cake resistance was dominant in dead-end filtration of biological suspensions with different floc sizes. A simplified model with a single fitting parameter represented the flux decline behavior with time over the course of filtration relatively well.
(2) Sludge samples obtained from conventional activated sludge have larger floc size. As the SRT increased from 10 to 30 d, the average floc size decreased from 90 to 55 μm which was attributed to the reduction in EPS. The membrane fouling decreased with increasing SRT in spite of substantial decrease in floc size. This behavior is well correlated with EPS_P concentration. It was concluded that for larger floc sized biological suspension less permeable cake layer was formed causing smaller cake resistance due to reduced EPS_P content.
(3) The fouling was independent from both EPS and SMP when the floc size was relatively small (e.g. JLBR sludge). Much more compact cake layer formed causing greater cake resistance due to small floc size of suspension. In this case, neither MLSS concentration nor SMP and EPS contents controlled the fouling.

Acknowledgment

This study was financially supported by the TUBITAK, the Scientific and Technological Research Council of Turkey (Project No: 108Y129). Authors also thank to Dr Nadir Dizge and Dr Derya Imer Koseoglu for providing the sludge samples.

Nomenclature

CAS — conventional activated sludge process
JLBR — jet loop bioreactor
COD — chemical oxygen demand (mg/L)
EPS_c — fraction of carbohydrate contained in extracted solution from sludge (mg/g MLSS)
EPS_P — fraction of protein contained in extracted solution from sludge (mg/g MLSS)
EPS — extracellular polymeric substances (mg/g MLSS)
MLSS — mixed liquor suspended solids (mg/L)
R_t — total hydraulic resistance (m^{-1})
SMP — soluble microbial products (mg/L)
SMP_c — fraction of carbohydrate contained in the sludge solution (mg/g MLSS)
SMP_P — fraction of protein contained in the sludge solution (mg/g MLSS)
SRT — solid retention time (d)
ΔP — trans-membrane pressure (Pa)
R_c — cake resistance (m^{-1})
R_T — total resistance (m^{-1})
R_m — membrane resistance (m^{-1})
μ — dynamic viscosity of MLSS (mPa s)
α — filtration resistance (mg^{-1} m^{-1})
M — mass of the biological suspension (mg)
T — time (h)
F — fraction of the micro-organisms contribute to the cake formation (−)
C_B — concentration of the micro-organisms (mg/L)
A_o — filtration area (m^2)
J — permeate flux ($L/m^2/h$)
α^* — Lumped filtration resistance or fitting parameter (mg^{-1} m^{-1})

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