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Membrane fouling in thermophilic membrane bioreactor with different sludge retention times

Elif İnce[1]*, Mahir İnce[1] and Alikemal Topaloğlu[2]

[1]Gebze Technical University, Department of Environmental Engineering, Kocaeli, Turkey
[2]Bulent Ecevit University, Department of Environmental Engineering, Zonguldak, Turkey

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Abstract. As membrane fouling is based on various factors, it is a complex phenomenon that is hard to estimate. This study investigated membrane fouling in a thermophilic jet loop membrane bioreactor (JLMBR). With this purpose, four different empirical membrane fouling models with different sludge retention times were applied on the flow data obtained in the system. As a result of the model implementation, it was found for all sludge retention times that, standard blocking is effective in the first 1.5 hours of filtration, while cake filtration was dominant in the remaining duration. Additionally, it was observed that as the sludge retention time increases, membrane fouling rate decreases.

Keywords: EPS; fouling model; filtration; MBR; SMP; thermophilic

1. Introduction

In recent years, there is an increasing tendency in modern wastewater treatment plants for usage of membrane bioreactors (MBR) instead of activated sludge systems with conventional sedimentation tanks. The advantages of this technology are better product quality, higher biomass concentration and reduced need for space. The disadvantage of membrane technology is the issue of fouling, and operating costs are increased due to the necessity of replacing membranes. Membrane filtration is characterized by a periodical change between filtration and filtrate, and backwashing. Backwashing is necessary to remove cake layers and clean blocked membrane pores, while the general filtration performance still drops as complete cleaning is not possible.

In cross-flow filtration systems, the effect of the shear stress on the surface of the membrane is higher than that of the Brownian forces which lead to random displacements of particles (Nataraj *et al*. 2008). Therefore, removal of microorganisms and microbial products accumulated on the membrane surface is based on the relationship between the membrane and these substances. In the beginning of filtration, the colloids and particles in the solution move towards the membrane pores via application of high pressure. As filtration progresses, pores are clogged and sometimes the accumulated particles and colloids cannot be easily separated with cross-flow rate (Wang and Wu 2009). Accumulation of particles leads to formation of a cake layer on the surface by larger molecules. As the cake layer reaches a certain value, the effect of the applied pressure diminishes. These mechanisms are expressed by pore resistance, cake resistance and concentration polarization resistance.

As membrane fouling is based on various factors, it is a complex phenomenon that is hard to estimate. These factors are generally divided into three classes: (i) material membrane properties such as pore size and geometry (Gkotsis *et al*. 2014, Ma *et al*. 2014), porosity and interconnections (Palacio *et al*. 2003, Rezai *et al*. 2014), (ii) wastewater properties such as pH and concentration (Palacio *et al*. 2003, Velasco *et al*. 2003), and (iii) operating parameters such as pressure, cross-flow rate and temperature (Rezaei *et al*. 2011, Ishizaki *et al*. 2016).

There are some fouling models to explain the operation of the filtration mechanism. While some of these models are very simple, others are highly complex and hard to implement with experimental data. Classical models are the simplest ones in explaining fouling mechanisms. However, only one mechanism estimation may be made for the entire filtration process in these models. In many cases, there is a need to combine models to project the behavior of the filtration process. Some models have recently been tried out for this purpose (Rezaei *et al*. 2011). Although these models are able to estimate one or more mechanisms for each flow reduction, they have not been compared to fouling mechanism models. They are also not recommended for various operating conditions. Thus, the best model should be found for each operating condition.

Studies have been conducted to understand the mechanism of blocking. Hermia (1982) developed a model to explain various fouling mechanisms in microfiltration. Although these models are dead-end filtration models, they had been also used to explain dominant blocking mechanism for cross-flow filtration by some researchers (Koltuniewicz *et al*. 1995, Yildiz *et al*. 2003). The model is as the following;

∗Corresponding author, Associate Professor
E-mail: e.senturk@gtu.edu.tr

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

Here, t: filtration time, V: filtrate volume passing through a unit filtration area, n: blocking index and K: coefficient of resistance. The event of blocking is explained by the blocking index (n). For example, n = 2 means "blocking on the surface". n = 1.5 is "standard blocking", where pollutants accumulate inside the pore surface and their diameters are narrowed in time. n = 1 is "partial blocking on the surface" where particles affect each other, whereas n = 0 is "cake filtration." In this model, particles on the membrane surface form a layer along the blockage. The disadvantage of implementing this model developed by Hermia directly is that the n value is highly affected by fluctuations in flow data. Therefore, a time interval is selected and new equations are obtained from the Eq. (1) for constant flow or constant pressure.

There are many studies conducted on the mechanism of blockage in microfiltration processes (Velasco *et al*. 2003, Ansari *et al*. 2006, Bolton *et al*. 2006, Nguyen *et al*. 2010, Rezaei *et al*. 2011). It has been shown that a single model is not sufficient to explain the reduction in flow rate along the process of filtration. Herrero *et al*. (1997) divided blocking into two phases and stated that intra-pore blocking takes place first, and extra-pore blocking happens next. Ho and Zydney (2000) reported that pore blocking and cake filtration happen together. While these models are empirically determined models, they are used in determining how the membrane fouling mechanism takes place by considering the reduction of flow in filtration. Fouling in membrane pores are analyzed based on fouling principles. These principles explain the mechanisms of physical flow through the membrane starting from pore blocking to cake formation. General and linearized equations are used for each fouling model (Koltuniewicz *et al*. 1995, Lee and Clark 1998, Mohammadi *et al*. 2003, Yildiz *et al*. 2003, Judd 2006, Ahsani *et al*. 2017).

Jet loop bioreactors, due to high mass transfer characteristics they have, have been reported to work with high treatment efficiency rates, as they require lower amounts of area and provide higher filtration of organic loads under flexible conditions in comparison to classical treatment systems. Thermophilic aerobic processes in elimination of wastewater with high organic loading rate are considered as advanced treatment technologies. They have various advantages including fast biological decomposition, low sludge production, fast inactivation of pathogenic microorganisms, high organic load rates and as a result, low retention rates and lower requirement of costs. Thus, in JLMBR, treatment of high organic loads is achieved in shorter times and with higher efficiency due to the synergy of the thermophilic biomass.

Thermophilic aerobic treatment has 3 to 10 times the biodegradation rate of mesophilic treatment. However, as its precipitation characteristics are much worse than those in mesophilic systems, the quality of the output water is lower (Shahata and Urase 2016). This problem can be overcome by addition of a membrane filtration system. However, in this case, membrane fouling is higher than that of a mesophilic system operated in the same conditions. Thermophilic aerobic processes produce more EPS than mesophilic aerobic processes (Visvanathan *et al*. 2007, Abeynayaka and Visvanathan 2011a). Similarly, SMP production is also higher in thermophilic aerobic treatment due to the increased microbial activity (Abeynayaka and Visvanathan 2011a, 2011b). The produced EPSs lead to a more intense and less porous cake layer to be formed on the membrane surface of the thermophilic activated sludge. High EPS concentration affects membrane permeability negatively (Drews 2010, Wang *et al*. 2013, Zhang *et al*. 2014, Lin *et al*. 2014, Zhang *et al*. 2015, Hao *et al*. 2017).

Although there are many studies on modelling fouling mechanisms in the literature, no modelling study applied on the data obtained from a thermophilically operated MBR system was found. Therefore, in this study, four different membrane fouling models were applied on flow data obtained from JLMBR thermophilically operated under four different sludge retention times (SRTs).

Table 1 Wastewater characterization

Parameter	Unit	Value	Method (APHA 2005)
COD	mg/L	5600	STM 5220 C
BOD_5	mg/L	4600	STM 5210 B
pH	-	6.8	-
Temperature	°C	18	-
TKN	mg/L	220	STM 4500-Norg B Macro-Kjeldahl
NH_3	mg/L	90	STM 4500-NH_3 C
SO_4^{2-}	mg/L	50	STM 4500-SO_4^{2-}
TP	mg/L	80	STM 4500-P D

COD: Chemical Oxygen Demand; BOD_5: Biochemical Oxygen Demand for 5 days; TKN: Total Kjeldahl Nitrogen; TP: Total Phosphorus; NH_3: Ammonia; $SO_4^{=}$: Sulphate

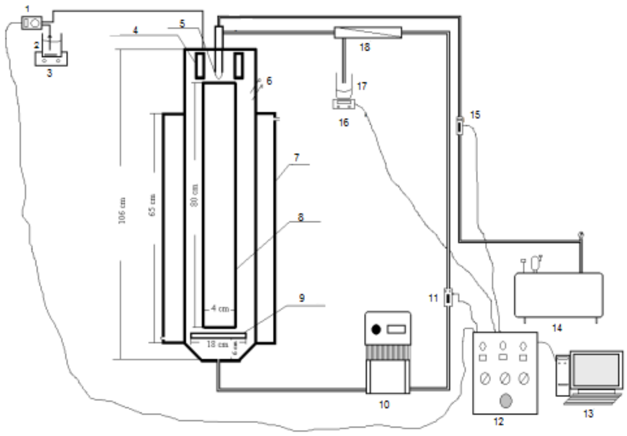


Fig. 1 Schematic depiction of the JLMBR system and its dimensions (1: peristaltic pump, 2: wastewater, 3: stirrer, 4: viewing window, 5: nozzle, 6: pH and DO probes, 7: jacket, 8: draft tube, 9: impact plate, 10: pump, 11: liquid flowmeter, 12: control panel, 13: computer, 14: compressor, 15: gas flowmeter, 16: analytical balance, 17: treated wastewater, 18: membrane)

2. Experimental design

2.1 Wastewater

The wastewater used in this study was obtained from a factory producing potato and corn chips, as well as corn snacks. In the process, approximately 33 m^3 of water is used per 1 ton of potato chip production. The potato processing wastewater used in the study was taken from the facilities after peeling, washing and slicing, before it mixed with other processes (corn processing). The characteristics of the wastewater taken from the factory are given in Table 1.

The wastewater used in the study was collected with an immersion pump before it went through any treatment units and brought to the laboratory in 100-L containers. For the wastewater used in feeding, wastewater samples taken from each container were analyzed for all necessary parameters before usage. Wastewater containers were stored in a cold room at a +4°C temperature.

2.2 JLMBR system

The reactor consisted of two jacketed cylindrical stainless steel tubes with conic bottoms. Two glass windows were added to monitor the loop occurring in the reactor. Fig. 1 shows the schematic representation of the reactor. In cases where the temperature of the reactor run in thermophilic conditions went below or above 45±2ºC, a constant temperature water circulator automatically started working. However, it was seen in the experiments that the heat energy provided to the reactor by the jet pump was sufficient to create thermophilic conditions. Therefore, no additional energy was needed for heating in cases of running the reactor in constant pump motor speeds.

The potato processing industry wastewater was fed to the reactor by a controlled peristaltic pump (Heidolph 5201). Hydraulic retention time (HRT) was fitted to 2.4 day due to provide constant OLR (2 kg COD/m^3·day) at different SRT. Other important operating parameters such as dissolved oxygen, temperature and pH were continuously monitored using a multi-parameter measurement device (WTW).

Table 2 Model equations for different filtration mechanisms under constant pressure

	ΔP=Constant, $\vartheta_0 = \frac{\Delta P.A}{\mu R_M}$		Equation No
	Equation	Constant	
Cake Filtration	$\frac{\frac{\vartheta_0}{\vartheta(t)}-1}{V(t)} = k_d - k_d k_p \frac{t}{V(t)}$	$k_d = \frac{\alpha X_0}{AR_M}$, $k_p = \frac{\vartheta_r}{X_0}$	(2)
Pore Blocking	$\vartheta(t) = \vartheta_0 - K_b V(t)$	$K_b = \frac{\Delta P \sigma}{\mu R_M}$	(3)
Partial Blocking on Surface	$\frac{1}{\vartheta(t)} = \frac{1}{\vartheta_0} + K_i t$	$K_i = \frac{\Delta P \sigma}{\mu R_M \vartheta_0}$	(4)
Standard Blocking	$\frac{t}{V(t)} = \frac{1}{\vartheta_0} + \frac{K_s t}{2}$	$K_s = \frac{2X_P}{LA_0}$	(5)

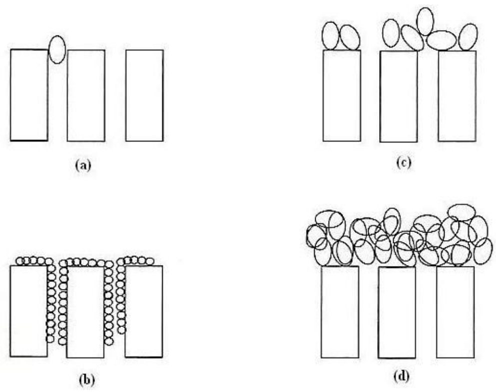


Fig. 2 Membrane blocking mechanisms: (a) Pore blocking, (b) Standard blocking, (c) Partial blocking on the surface, (d) Cake filtration (Judd 2006)

2.3 The membrane and its properties

In order to achieve filtration, a tubular microfiltration membrane unit with 0.2 μm pore diameter of Microdyn-Nadir (MD 063 TP 2N) brand was placed into the circulation line of the reactor externally. The constantly operated polypropylene membrane in the system had an effective filtration area of 0.20 m^2. Membrane flow was measured with a precision scale. The data obtained from the precision scale were monitored for 24 hours via a card automation system. In each SRT value, when the JLMBR reached a stable point, a new membrane module with 0.036 m^2 of filtration area was placed and filtration experiments were conducted.

2.4 EPS extraction and analysis

EPS (extracellular polymeric substance) was detected using the method of formaldehyde extraction (Li *et al*. 2008). More specifically, with the help of this protocol, soluble microbial products (SMP) represents the soluble part of EPS and related EPS were also measured. Especially the total carbohydrate (c) and protein (p) was considered as total EPS (Total EPS = EPS_p + EPS_c + SMP_p + SMP_c). In order to determine the carbohydrate in total EPS, the phenol-sulfuric acid method (Dubois 1956) was used in a modified way. In the analysis, an 80% phenol solution and concentrated 95-97% H_2SO_4 were used. A 1 mL sample was added 25 μL 80% phenol and 2.5 mL H_2SO_4, and left in a water bath at 30°C for 15 minutes. In detection of protein, the Folin method was applied, using bovine serum albumin (Lowry *et al*. 1951).

2.5 Membrane fouling models

While these models are empirically determined models, they are used to determine how the membrane fouling mechanism takes place in the framework of flow reduction basics in filtration. Fouling in membrane pores are analyzed based on rules of blockage. These rules explain physical mechanisms of flow through the membrane in the process from pore blocking to cake formation, and are generally expressed by an equation developed by Hermia (Lee and

Clark, 1998, Yildiz *et al*. 2003, Lazaridis *et al*. 2004, Abbasi *et al*. 2011, Chang *et al*. 2011, Saha and Das 2015). The equation is generally used to define the filtration mechanism using experimental data belonging to membrane filtration. Table 2 shows the linearized equations for each fouling model (Koltuniewicz *et al*. 1995, Lee and Clark 1998, Mohammadi *et al*. 2003, Yildiz *et al*. 2003, Judd 2006, Hosseinzadeh *et al*. 2013, Saha and Das 2015). Additionally, Fig. 2 provides the schematic representation of fouling mechanisms.

Thus, it is indispensable to investigate the membrane fouling mechanism and establish strategies to control membrane fouling. The models provided in Table 2 light the way for determining how the phenomenon of membrane fouling takes place (Hu and Scott 1997). The membrane fouling experiments were conducted under constant pressure (TMP: 190 kpa, and the cross-flow rate: 4.5 m/s) in this study.

3. Results and discussion

The system was run under 4 different SRTs (10, 30, 60 and 100 days) for approximately 7 months. Total COD loading was constant at different sludge retention times as 2 kg COD/m^3·day. Wastewater loadings in the system were made with the help of the peristaltic pump connected to the system. In each SRT, the researchers waited for the system to become stable, and the system was run for at least 10 days after becoming stable. As the reactor was operated thermophilically (Shahata and Urase 2016) and because of low floc sizes due to the characteristics of the jet loop reactor, contact surface increased and the reactor became homogenous because of high turbulence (Yildiz *et al*. 2003). Therefore, the system reached stability very quickly after changing the SRT in the JLMBR.

The COD removal efficiency of the system in different sludge retention times stayed at approximately constant values (98-99%) independently of SRT. The most important reason for high removal efficiency is the type of the reactor and the thermophilic operating temperature. As a typical characteristic of jet loop reactors, biodegradation is much faster due to the increased surface area provided by small microorganism flocs as opposed to classical systems. Secondly, organic substances are able to stay in the activated sludge longer due to the circulation in the system (Farizoglu and Keskinler 2006). Ke and Junxin (2009) also reported that COD removal is independent of SRT. Hao *et al*. (2017) said that SRT had only limited influence on COD removal efficiency, under steady-state operation. However, some researchers reported better treatment with higher SRT. Grelier *et al*. (2006) reported that an SRT of longer than 40 days achieved better biodegradation of organic and nutrient substances. Palmarin and Young (2016) also reported that the removal efficiency of COD increased with increasing MLSS concentration owing to the direct increase in biodegradation capacity.

For each SRT, four different models (equations 2 to 5) were applied on the flow data obtained after the system reached stability, and the obtained graphs are shown in Fig. 3.

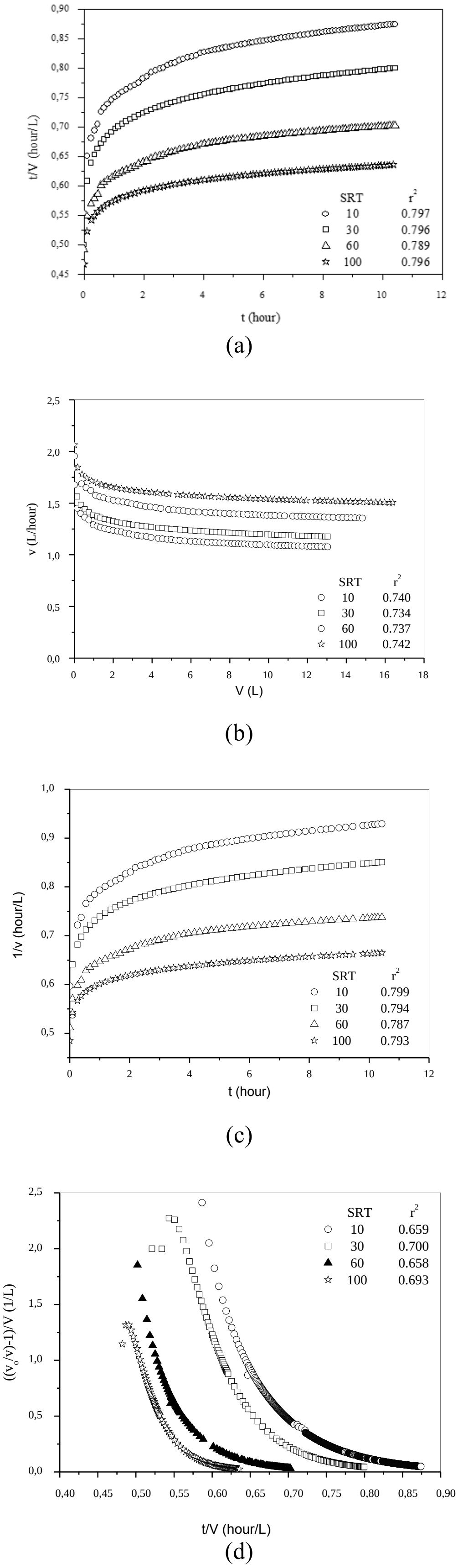
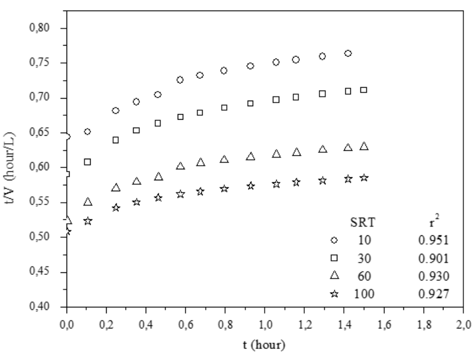
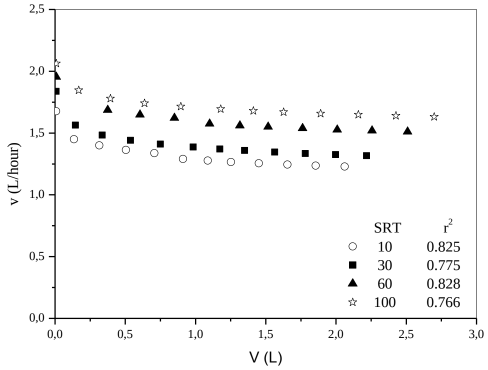


Fig. 3 Implementation of four different models for different sludge retention times (a: Standard blocking, b: Pore blocking, c: Partial blocking on the surface, d: Cake filtration)



(a)



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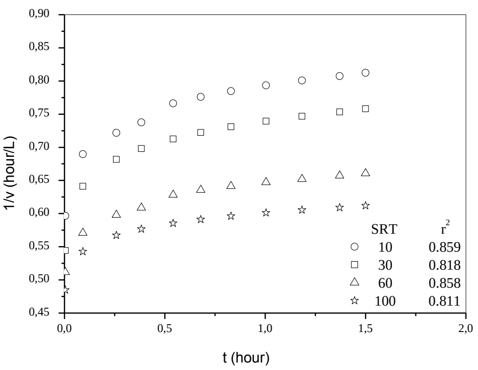

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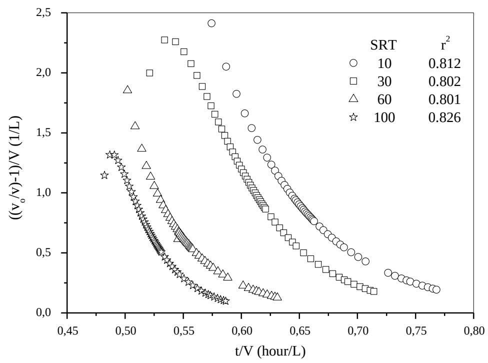


(d)

Fig. 4 Application of the models in the first 1.5 hours for different sludge retention times (a: Standard blocking, b: Pore blocking, c: Partial blocking on the surface, d: Cake filtration)

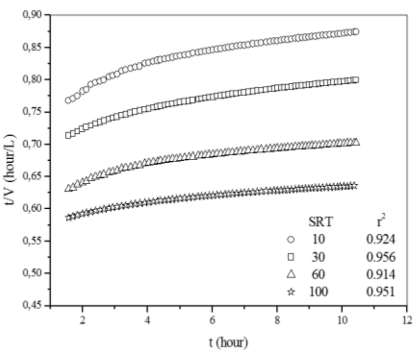
As it may be clearly seen in Fig. 3, none of the models was suitable by itself for all data. The r^2 value for even the best regression was only 0.799. Therefore, the flow data obtained in each SRT were divided into certain time intervals and many model applications were made. As a result of the trials, in all four different SRTs, it was found that the standard blocking model was suitable for the first 1.5 hours, and the cake filtration model was suitable for the rest of the process. Fig. 4 shows the data in the first 1.5 hours and Fig. 5 shows the resulting graphs of model applications after the first 1.5 hours.

Standard blocking happens in cases where particle size is lower than the size of the membrane pores. When suspensions permeate into the membrane, the membrane wall is covered with a partially uniform layer due to the adsorption or trapping of particles on the wall of pores or the membrane support material. This situation may affect membrane rejection significantly. The standard pore blocking model assumes that the membrane has straight cylindrical pores. According to this model, the rate of change in pore volume is directly proportional to the flow rate and bulk concentration.

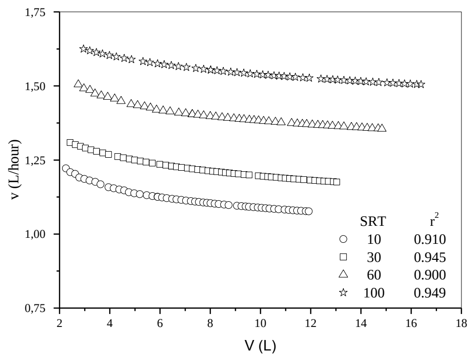
In cases where particle size is higher than the size of the pores, pore blocking is no longer in question. Instead, formation of a cake layer takes place in relation to accumulation of particles on the membrane surface. In this case, membrane pore structure is not affected. That is, the membrane fouling is not permanent but temporary. The cake filtration model assumes that a uniform cake layer forms on the entire membrane surface, and this layer is permeable although it has resistance. It states that the rate of change in the resistance of the formed cake layer is directly proportional to the convection of particles towards the membrane surface.

While only one model of membrane pore blocking may be able to define the reason for decreased flow rate, considering the change in the flow rate reduction (from the beginning to a short time after the beginning, fast flow reduction and slow flow reduction periods), a different model in every different time period may represent the flow reduction better.

All trials for the model application show that no mechanism is effective in membrane fouling by itself. As the system is operated thermophilically and due to the high turbulence as the typical characteristics of jet loop reactors, particle size is very small. Therefore, in the period of the first 1.5 hours, particles smaller than the membrane pore size and suspensions may enter the pores of the membrane and form a partially uniform pollutant layer by getting adsorbed or trapped by the membrane wall (Hu and Scott 1997). Thus, in this process, standard blocking is dominant for all sludge retention times. After this process, the dominant mechanism is cake filtration. As known, cake filtration takes place in cases where particle sizes are larger than the membrane pore size. While other models are still applicable after the first 1.5 hours, the most effective one is the cake filtration model. In extended operation, the cake layer, formed on the surface of membrane, develops a dynamic biofilm layer. Its structure changes biologically due to underneath anoxic layer (Hosseinzadeh *et al*. 2013).



(a)



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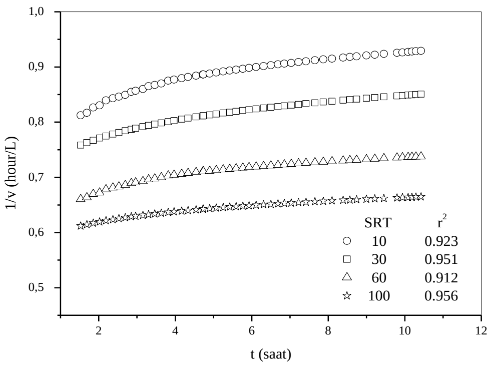

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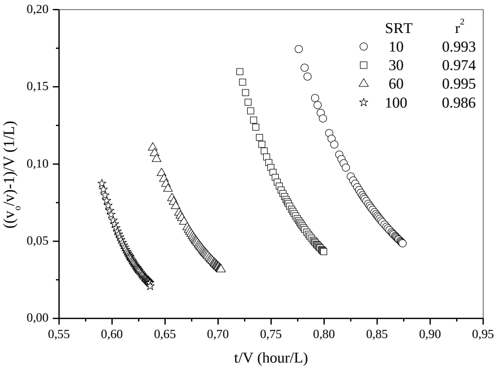

(d)

Fig. 5 Application of the models after the first 1.5 hours for different sludge retention times (a: Standard blocking, b: Pore blocking, c: Partial blocking on the surface, d: Cake filtration)

Table 3 Model constants obtained for suitable models

Model and Time Interval			SRT (days)			
			10	30	60	100
First 1.5 hours	Standard blocking	K_s	0.043	0.039	0.029	0.022
		r^2	0.918	0.901	0.930	0.927
After 1.5 hours	Cake filtration	k_d	1.130	1.070	0.850	0.820
		k_p	1.040	1.280	1.300	1.600
		r^2	0.993	0.974	0.995	0.986

Additionally, a new biofilm layer forms the existing deposited biofilm. Thus, the opening pores on the membrane surface area change through the filtration process, resulting in change of k_d and k_p. Table 3 shows the model constants and correlation coefficients obtained for suitable models.

As seen in Table 3, the standard blocking model constant of K_s, which is an indicator of membrane fouling rate, decreased while SRT increased. Increases in sludge retention time lead to a decrease in the amounts of EPS and SMP (Malamis and Andreadakis 2009, Hao *et al*. 2017). Therefore, it may be stated that as the concentration of microbial products decreases, so does the model constant. Therefore, a decrease in the value of K_s indicates a decrease in fouling. Some researchers showed that EPS concentration increased in line with the increase in SRT (Chang and Lee 1998, Cho *et al*. 2005). However, some other researchers reported that the exact opposite of this happened (Ng and Hermanowicz 2005) or the change was not significant (Liao *et al*. 2001, Lee *et al*. 2003). The MLSS concentration increased with increasing SRT, which were 3200, 5100, 7400, and 12500 mg/L for SRT of 10, 30, 60 and 100 day, respectively. When F/M (food/microorganism) ratio decreased, result of increasing MLSS concentration, EPS and SMP concentrations were lowered (Table 4) due to microorganisms consuming these substances as substrates (Massé *et al*. 2006, Li and Wu 2014). Additionally, the amounts of protein and carbohydrate in the reactor were lowered by the increase in SRT. Increased SRT also decreased the P/C ratio. Soluble P/C ratio is an indicator of microbial activity. This ratio increases with increased temperature. Protein concentration is reduced under thermophilic conditions. However, this reduction is reported to be very small (Abeynayaka and Visvanathan 2011a). The P/C ratio in EPS under different SRTs (10, 30, 60 and 100 days) were found respectively as 2.35, 2.07, 1.58 and 1.03. The ratio in the SMP measured in the reactor was found respectively as 2.38, 2.21, 1.72 and 1.56. Likewise, among the constants of the cake filtration model, k_d also decreased with the increase in SRT. This reduction in the value of k_d may be explained by the reduction in the specific cake resistance. As flocs have more compact and porous structure in low EPS concentrations, specific cake resistance is lower (Feng *et al*. 2012). Another model constant k_p increases in line with SRT. This increase is caused by the increase in flow rate. The flow rate of the filtrate passing through the membrane decreased with increased membrane fouling. As the membrane fouling decreased in line with SRT, the k_p value also increased. As

Table 4 EPS and SMP concentrations under different sludge retention times

SRT	EPS (mg/L)		SMP (mg/L)	
	Carbohydrate	Protein	Carbohydrate	Protein
10	194.02	455.95	201.08	478.13
30	183.48	379.80	193.26	427.05
60	171.01	270.75	182.27	313.50
100	163.84	168.76	176.35	275.10

mentioned before, the k_p value changes with EPS and SMP because membrane fouling is influenced by microbial product types and concentrations.

4. Conclusions

4 different empirical models were applied on the flow data obtained in the thermophilic JLMBR operated under different sludge retention times. It was found in all SRTs that standard blocking was dominant in the first 1.5 hours of filtration, while cake filtration was dominant after the first 1.5 hours. However, it was observed that membrane fouling decreased as SRT increased. The reason for this was that increased SRT decreased the concentrations of EPS and SMP in the reactor. Additionally, the amounts of protein and carbohydrate in both the reactor and the output were also reduced by the increased SRT. Similarly, the P/C rate also decreased as SRT increased. In order to operate thermophilic JLMBR for longer time without membrane backwashing and/or replacement, the system should be run with longer sludge retention times.

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Symbols

ΔP	Transmembrane pressure
A	Membrane area
μ	Viscosity
R_M	Membrane resistance
t	time
ϑ_0	Flow rate, t=0
ϑ	Flow rate
V	Filtrate volume
K_i, K_s, K_b, k_d, k_p	Model constants
α	Specific cake resistance
X_0	Volume taken by particles
σ	Blocked area per unit filtrate
X_p	Volume of accumulated solid particles per unit filtrate
L	Membrane thickness
n	blocking index
K	resistance coefficient

Abbreviations

HRT	Hydraulic Retention Time
SRT	Membrane area
TMP	Viscosity
BOD_5	Membrane resistance
c	carbohydrate
EPS	Extracellular Polymeric Substance
F/M	Food/microorganism
JLMBR	Jet Loop Membrane Bioreactor
COD	Chemical Oxygen Demand
MBR	Membrane Bioreactor
NH_3	Ammonia
p	protein
SMP	Soluble Microbial Product
$SO_4^{=}$	Sulphate
TKN	Total Kjeldahl Nitrogen
TP	Total Phosphorus