Desalination and Water Treatment
www.deswater.com

26(2011)124–130
February

1944-3994/1944-3986
doi: 10/5004/dwt.2011.2121

Fouling characteristics of microfiltration membranes during the filtration of jet loop membrane bioreactor (JLMBR) activated sludge

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Received 25 November 2009; Accepted 22 March 2010

ABSTRACT

The main objective of this study was to characterize the activated sludge of jet loop membrane bioreactor (JLMBR) in order to determine its effects on the membrane biofouling. The sludge characteristics were evaluated in terms of MLSS (mixed liquor suspended solid), EPS (extracellular polymeric substance), SMP (soluble microbial product), relative hydrophicity (RH) and viscosity. The membrane filtration tests were performed at a cross-flow microfiltration system by using cellulose acetate (CA) and cellulose nitrate (CN) filters with 0.2 and 0.45 μm of pore sizes. The jet loop bioreactor was operated at a batch mode for 36 d with an organic load of 3 kg COD $m^{-3}d^{-1}$. The COD treatment efficiency was achieved at 95%. It was found that the sludge properties changed with MLSS concentration. The viscosity of sludge increased and the RH of sludge decreased with increase in MLSS concentration. The EPS concentration of the sludge was much higher than SMP concentration. The carbohydrate contents of EPS and SMP were higher than the protein contents. The flux decline models and the resistance analysis were used to investigate the biofouling mechanism. In the resistance analysis, it was found that the pore blocking resistance (R_p) of CA membrane with pore size of 0.45 μm (CA045) was higher than the cake layer resistance (R_c), (3.56 and 1.02×10^{12} m^{-1}). R_c was more higher than R_p for the other membranes. R_p of membranes were found to be 33.6%, 77.1% , 23.4% and 21.9% of the total resistances (R_t) for CA02, CA045, CN02 and CN045, respectively. It was concluded that the high EPS content in JLMBR was the main fouling parameter for membranes. It was also found that the affinity of membrane types against EPS varied.

Keywords: Jet loop membrane bioreactor; Membrane biofouling; Crossflow microfiltration

1. Introduction

Jet loop bioreactors (JLBR) have gained an increasing popularity in recent years as an efficient technology for wastewater treatment. Different types of JLBRs were successfully applied for treatment of different wastewater including winery [1], cheese whey [2], olive oil [3] and leachate [4]. JLBR has many advantages such as excellent oxygen transfer rates, high mixing capacity, small reactor volume, low energy and maintenance costs. The most important disadvantage of JLBR is the sludge settling problem. The high shear force in nozzle and the high food to microorganism ratio (F/M) cause the different sludge properties than that of the conventional activated sludge systems. The sludge in JLBR has non-settable and slimy characteristic. In addition to the problems in sludge settling, JLBR requires proportionally larger settling tanks since they have high biomass concentrations. The application of membrane separation techniques for JLBR can overcome the disadvantages of settling tank. The micro and ultrafiltration can be used for the separation of solid and liquid.

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Presented at the International workshop on urbanisation, land use, land degradation and environment (ULE 2009), 28 September – 1 October 2009, Denizli, Turkey

The jet loop bioreactor integrated with a membrane system is referred as jet loop membrane bioreactor (JLMBR). The membrane filtration in the JLBR can offer great advantages like an excellent effluent quality, good biomass-water separation, small footprint demand, better operational control of sludge amount and other biological conditions [5]. However, the membrane biofouling is the major limitation for the application of the membrane process. The membrane biofouling can be defined as the undesirable deposition and accumulation of microorganisms, solutes and cell debris within/on membranes [6]. The activated sludge in the treatment system has many different biological components, such as extracellular polymeric substances (EPS), soluble microbial products (SMP) and colloids, which can interact with the membrane in different ways [7]. Various MBR publications indicated that the characteristics of the mixed liquor affected the membrane filterability, such as viscosity, EPS, SMP, flock size, hydrophobicity [8–11]. However, current understanding of the membrane biofouling is still insufficient. The membrane properties are also important factors for the membrane biofouling as well the properties of the sludge. These properties can be classified as physical (pore size and distribution, porosity/roughness and membrane configuration) and chemical parameters (hydrophobicity and type of material) [8]. The mixed liquor conveyed toward the membrane is rejected by the membrane and accumulated on the membrane surface. The pore size and the material of membrane specifically influence the initial attachment of the biological components. The types of membranes show different affinity against biological components (EPS, SMP, colloids). Therefore, the membrane fouling mechanism for different MBR system changes with the properties of mixed liquor and the membrane. The main objective of this study was to determine the membrane fouling mechanism of JLMBR activated sludge. For this purpose, the activated sludge in JLMBR was systematically analyzed and the sludge-membrane affinity was determined for different membrane types in a cross-flow microfiltration system. Cellulose Acetate (CA) and Cellulose Nitrate (CN) membranes with pore sizes of 0.45 and 0.2 μm were used in the experiments.

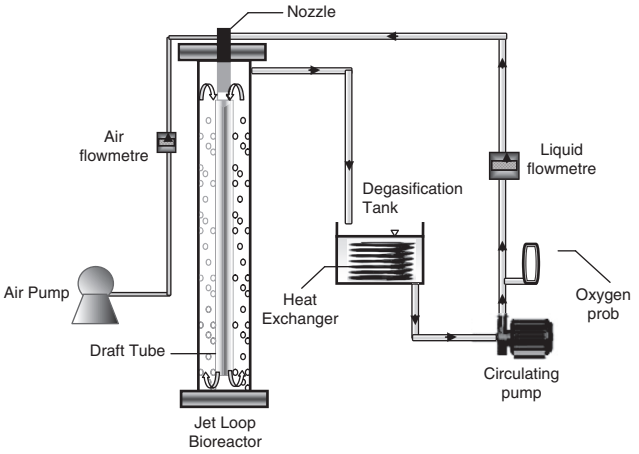

Fig. 1. Jet Loop Membrane Bioreactor experimental setup.

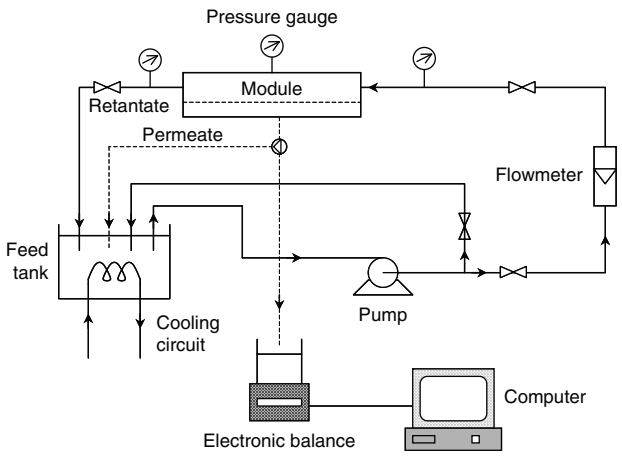

Fig. 2. Cross flow microfiltration system.

2. Materials and methods

2.1. JLMBR experimental system

The experimental setup of the JLMBR used in this study is presented in Fig. 1. JLMBR consists of two concentric tubes which are known as a draft tube (height 100 cm; inner diameter 7 cm) and a reactor (outer tube) (height 113.5 cm; inner diameter 15 cm). The two-phase (air and mixed liquor) coaxial nozzles positioned at the top of reactor create a downward directed two-phase flow inside the draft tube. The mixed liquor to the reactor provided from annular nozzle (1.8 cm and 1.2 cm in diameter at the inlet and the exit sections, respectively) and the air are drawn into the liquid through inner nozzle (0.64 cm diameter). The air to the reactor was provided from the compressor through a gas flowmeter. The aerated mixed liquor passes from the reactor to the degassing tank from where it is recycled, again through the nozzle of the ejector venturi, into the column reactor. The working volume of reactor was 30 l. The reactor was operated under batch conditions for 36 d. The sludge age was set to 3 d. The membrane filtration experiments were conducted when reached to steady state in the bioreactor. In the last day (36th d), the activated sludge was taken from the degassing tank of JLMBR and used for the membrane filtration. An external crossflow microfiltration system (Fig. 2) was used for the membrane experiments. The working volume of

cross-flow microfiltration tank was 10 l. The effective area of membrane module was 28 cm^2. The membrane filtration experiments were performed at constant pressure (at 1 bar) and the temperature was kept constant at 30 °C by means of a water jackets. The flux was determined by weighing the permeate on a top-loading balance at timed intervals. Two membranes (CA, CN) with two pore sizes (0.45 and 0.2 μm) were tested consecutively with the same activated sludge. Membranes were purchased from Whatman Industry. The filtration time was set to 2 h.

2.2. Resistance analysis

The membrane flux was determined by weighing the permeate on a top-loading balance at timed intervals and then calculating the average flux. Filtration experiments continued for 120 min, which allowed the permeation flow rate to become stable. According to the resistance-in-series model, the fouling resistance was estimated by Darcy's equation as follows:

$$R_t = R_m + R_c + R_p = \frac{\Delta P}{\mu \times J_{ss}} \quad (1)$$

where R_t is the total hydraulic resistance, R_m the membrane resistance, R_p the pore blocking resistance, R_c the cake layer resistance, μ is dynamic viscosity, and J_{ss} is the steady state flux.

The experimental procedure to get each resistance value was as follows [12]: (1) R_m was estimated by measuring the water flux of de-ionized (DI) water; (2) R_t was evaluated by the steady state flux from the filtration of the sludge; (3) the membrane surface was then flushed with water and cleaned with a sponge to remove the cake layer. After that, the DI water flux was measured again to obtain the resistance of $R_m + R_p$. The pore blocking resistance (R_p) was calculated from steps (1) and (3) and the cake resistance (R_c) from (2) and (3).

2.3. Synthetic wastewater

The JLMBR was fed with a synthetic wastewater having COD of 3000 mg/l (COD:TN:TP ratio of 360:10.6:1). The synthetic wastewater contained 202 mg/l NH_4CI, 106 mg/l urea, 235 mg/l KH_2PO_4, 15 mg/l K_2HPO_4, 0.5 mg/l $FeCl_3$, 100 mg/l $MgSO_4 \cdot 7H_2O$, 7.5 mg/l $CaCI_2$, 294 mg/l $NaHCO_3$. The seed microorganism from activated sludge system of domestic wastewater treatment plant at Kocaeli, Turkey was used for the JLMBR.

2.4. Analytical methods

Measurements of COD and MLSS were performed as defined in Standard Method [13]. The viscosity of the sludge was measured by Brookfield DV-A viscometer (speed of 30 rpm with spindle No. 1). The relative hydrophobicity of the mixed liquor was determined according to MATH test [14]. In order to keep possible errors that may originate from electrostatic effects to a minimum, bacterial solutions were washed three times with Tris Buffer (pH 7.2) at 3000 rpm for 20 min. After washing, three ml of bacterial suspension was transferred to a UV tube and the initial turbidity ($OD_{initial}$) of the solution was determined. Bacterial suspension was again taken into to the tube and 0.3 ml of n-Hexadecane was added. The mixture was vortexed for 2 min. UV was set time scan mode for 15 min (settlement time) and the last turbidity (OD_{final}) of the solution were determined at 600 nm. Results are given in percentages calculated from the following relation:

$$Hydrophobicity\ (\%) = 100\left(1 - \frac{OD_{final}}{OD_{initial}}\right) \quad (2)$$

The EPS and SMP analysis methods were made through a physical-chemical extraction method [15]. The 5mL activated sludge samples were taken from degassing tank. The suspension was centrifuged (4000 × g, 10 min, 4 °C) and the supernatant was decanted into another sterile tube and re-centrifuged (13,200 × g, 20 min, 4 °C) to ensure complete removal of the suspended solids. The resultant supernatant by this physical extraction contained soluble polysaccharide and soluble protein. The supernatant was analyzed for SMP content. The sediment in the tube was re-suspended with distilled water to obtain another 5 ml of suspension. Then, 6 μl formaldehyde (37%) was added into the suspension and was left at 4 °C for 1 h, followed by 500 μl NaOH (1N) for another 3 h at 4 °C. The suspension was centrifuged (13,200 × g, 20 min, 4 °C) and the supernatant, containing bound polysaccharide and bound protein, was analyzed for EPS content. Polysaccharide concentrations were quantified by the phenol-sulfuric acid method with glucose as a standard [16]. Protein concentrations were determined using the Bradford coomassie Blue method with Bovine Serum Albumin (BSA) as a standard [17]. All samples were determined the concentrations using a UV-visible spectrophotometer (GBC Instrument) at the wavelength of 490 nm for polysaccharide or at the wavelength of 595 nm for protein.

3. Results and discussion

3.1. Mixed liquor properties in JLMBR

The variations of JLMBR sludge properties during 36 d of batch operation are shown in Fig. 3(a)–(c). The samples were taken daily from JLMBR degassing tank. The removal of COD was quite successful, reaching up to 95% COD treatment efficiency. It can be seen

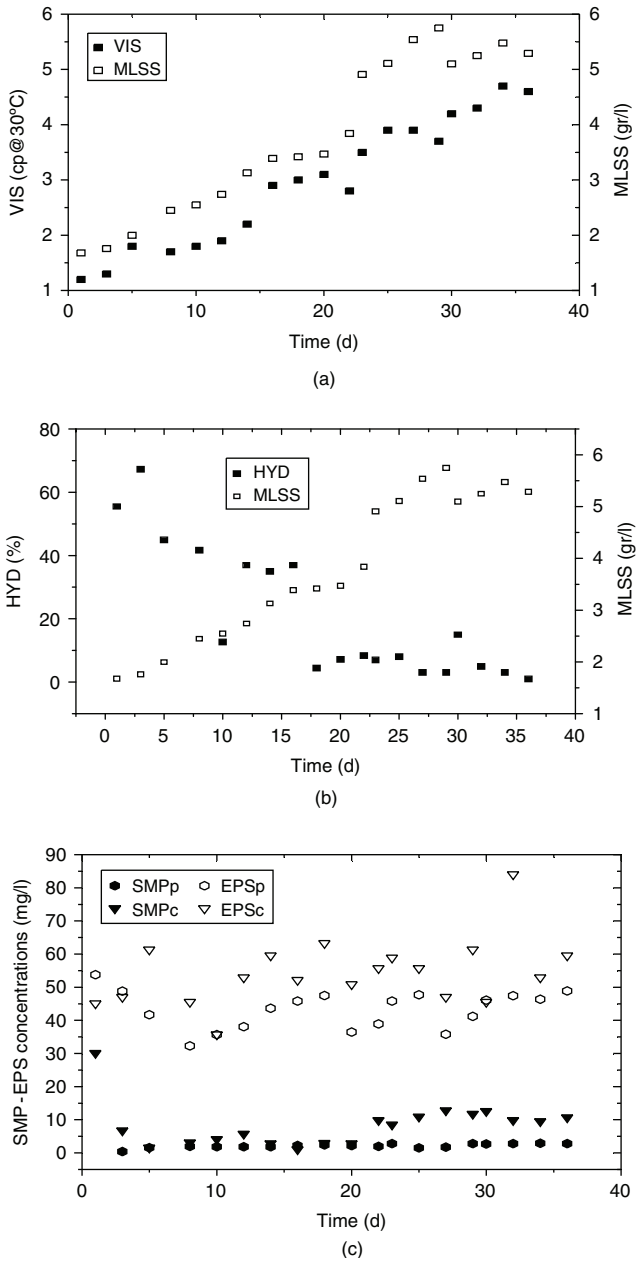

Fig. 3. Variations of sludge characteristics during the operation. (a) Variations of viscosity with MLSS; (b) Variations of hydrophicity with MLSS; (c) Variations of carbohydrate, protein of SMP-EPS in the JLMBR.

from Fig. 3(a) and (b) that the MLSS concentration played an important role in the evolution of viscosity and hydrophobicity of sludge. The viscosity of sludge linearly increased with increasing the MLSS concentration (Fig. 3a). It was reported that the biomass viscosity is closely related to its concentration [8,18,19]. At steady state, the viscosity of JLMBR sludge changed between 4 and 5 cp while the MLSS concentration was ranging from 5 to 6 g/l. The viscosity values measured in several submerged MBR studies were found to be between 5 and 10 cp for the MLSS concentration ranging from 15 to 25 g/l [18] and between 2 and 4 cp for the MLSS concentration of 18 to 25 g/l [20]. Compared to these results, the viscosity of JLMBR sludge was observed to be high for 6 g/l of MLSS concentration.

The variation of relative hydrophobicity (RH) of sludge with the MLSS concentration is shown in Fig.3b. The RH values of JLMBR were ranging from 5% to 10% when the MLSS concentration was between 5 and 6 g/l. These RH values were very low when compared to the other activated sludge systems and MBR studies [11,21,22]. In these studies, the RH values of activated sludge varied from 45% to 85%. Arabi and Nakhia [23] measured the RH of EPS and SMP as carbonhydrate and protein fractions and found that the RH was ranging from 3 to 7% for EPSc, 21 to 38% for EPSp, 13 to 17% for SMPc, and 18 to 47% for SMPp. These values showed that the RH of sludge changed with the biomass polymer products. It was reported that the EPS level and filamentous index (parameter related to the relative presence of filamentous bacteria in sludge) had a direct influence on the relative hydrophobicity [24]. Microscopic observation was made in some studies by using JLBR sludge and it was found that no filamentous bacteria was present in the flocks [2,3]. Up to now, there is no study in the literature at which RH of JLMBR sludge was measured. Therefore, the hydrophobicity values of JLBR sludge could not be compared. However, the low RH in JLBR could be explained that the mechanical stress (aeration, turbulence, shear force etc.) imposed by JLBR eliminated the filamentous bacteria and changed the surface characteristics of bacteria.

The variations of SMP and EPS concentrations during 36 d of operation are shown in Fig. 3c. The ratios of protein/carbohydrate for SMP and EPS were below 1.0, thus the carbohydrate contents were higher than the protein contents. The SMP showed lower concentrations compared with the EPS concentration. SMP can be defined as soluble cellular components released during substrate metabolism and cell lysis [25]. Some SMP can be utilized by active biomass flocks as recycled electron donors; and some can be adsorbed by the biomass flocks and by the EPS matrix [6]. In JLMBR, the SMP concentrations had low values because the some part of SMP could be used by the bacteria which had the high growth rate. Another important factor for that would be the fact that the particle size of the flocs was much smaller in JLBR than that of the conventional activated sludge system, resulting higher surface area per mass of bacteria. That may cause accelerated sorption of SMP to microbial community, causing lower SMP content in solution.

The SMP values in the reactor remained the same during operation; however, the EPS values showed variations throughout the experiment. This may be due to the high stress conditions in JLMBR. The EPS are the polymers bound to active or inert biomass [18]. The formation of EPS is growth-related and is produced in direct proportion to substrate utilization. In addition, the aeration intensity, the dissolved oxygen and the feed substrates have been proven as important parameters affecting EPS production [6]. The synthetically fed JLMBR caused to the high growth rates for the bacteria. The substrate utilization rate was very high at JLMBR, this caused increase in active bacteria concentration. Therefore the EPS concentration in JLMBR increased with increasing the MLSS concentration. As a result, high MLSS concentration, high viscosity, low hydrophobicity, high EPS concentration and low SMP concentration was observed in JLMBR system.

3.2. Membrane filtration performance of the JLMBR system

Crossflow microfiltration is an increasingly important technique for processing particulate suspensions in areas such as biotechnology, water and wastewater treatment. The crossflow microfiltration processes are usually operated at constant transmembrane pressure. The initial flux decline depends on the movement of particles towards the membrane surface. During the filtration, the membrane foulants (the biopolymers-SMP, EPS and the other substances-colloids, solutes) could block membrane pores and/or deposit on membrane surface to form a fouling layer. Accumulation and detachment of foulants are determined by the particle convection towards the membrane surface and the back transport rate of the deposited particles from membrane surface. The back transport of foulant from membrane surface depends on the specific particle-membrane interactions. The particle and membrane properties play a significant role in determining the foulant layer onto membrane surface. It was reported that the deposition of SMP or EPS on membranes strongly depended on its affinity with membranes [6].

The flux decline graphics are shown in Fig. 4 as a function of the membrane type. The permeate fluxes reached to the steady state after the permeate fluxes of membranes rapidly decreased within 10 min. This can mainly be attributed to a rapid sorption of colloids to the membranes since the SMP content of the sludge was low as discussed in the previous section. It can be seen that the permeate flux for cellulose acetate membranes decreased much faster then the other membranes. The initial fluxes were 204, 204, 171 and 132 $l/m^{-1}/h^{-1}$ for CA045, CA02, CN045 and CN02, respectively. The resistance-in-series model results are

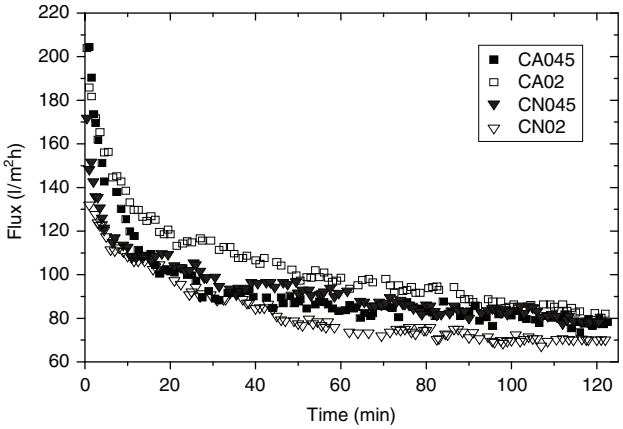

Fig. 4. Variation of fluxes with time at different membrane types (ΔP = 1 bar, filtration area = 28 cm^2).

Table 1
Results of resistance in series approach (Darcy' Law)

Membrane type	R_t (×10¹²) (m^{-1})	R_m (×10¹⁰) (m^{-1}) (%)	R_p (×10¹²) (m^{-1}) (%)	R_c (×10¹²) (m^{-1}) (%)
CA045	4.62	3.57 (0.77)	3.56 (77.1)	1.02 (22.1)
CA02	4.33	5.88 (1.36)	1.45 (33.6)	2.81 (65)
CN045	4.56	4.55 (1.00)	0.99 (21.9)	3.52 (77.1)
CN02	5.13	5.00 (0.97)	1.20 (23.4)	3.88 (75.6)

presented in Table 1. The total resistances (R_t) of 0.45 μm membranes were very similar with 4.62 × 1012 and 4.56 × 1012 m^{-1} for CA and CN, respectively. However, the pore blocking resistances (R_p) were quite different with 3.56 × 1012 and 0.99 × 1012 m^{-1} for CA and CN, respectively. Different fouling behaviors were observed for different membrane types. The pore blocking was the main mechanism for biofouling for CA membrane while the cake layer was dominant mechanism for CN membrane. The difference in pore resistance showed that CA membranes had a greater affinity for SMPs. For the smaller pore sized membranes (0.2 mm), CA and CN membrane showed similar result in terms of total resistances (R_t). However, Rp of membranes were found to be 1.45 × 1012 and 1.20 × 1012 m^{-1} for CA and CN, respectively. The Rc of membranes were found to be 2.81 × 1012 and 3.88 × 1012 m^{-1} for CA and CN, respectively. Pore resistance decreased substantially for CA membranes when the pore size decreased. Cake formation became the dominant fouling mechanism for both CA and CN membranes.

Table 2
Models for flux decline analysis

Model name	Model	Simplified forms
Complete pore blocking	$J = J_0 \times d^{-k_b} \times t$	$\ln(J^{-1}) = \ln(J_0^{-1}) + k_b \times t$
Standard pore blocking	$J = J_0 \times [1 + 1/2 \times k_s \times (A \times J_0)^{1/2} \times t]^{-2}$	$J^{-1/2} = J^{-1/2} + k_s \times t$
Intermediate blocking	$J = J_0 \times [1 + k_i \times A \times J_0 \times t]^{-1}$	$J^{-1} = J_0^{-1} + k_i \times t$
Cake filtration	$J = J_0 \times [1+2 \times k_c \times (A \times J_0)^2 \times t]^{-1/2}$	$J^{-2} = J_0^{-2} + k_c \times t$

In the MBR studies, generally it was assumed that the SMP was responsible for the pore blockage of the membrane, while EPS was responsible for the cake layer formation onto membrane. The activated sludge formed in the JLBR had dispersed and small flock structure and had also low SMP and high EPS concentrations. The pore blocking mechanism was expected for the membranes with higher pore sized membranes. But the interactions between EPS and membrane changed the mechanism of the fouling. The EPS in the sludge of JLMBR strongly formed the gel layer onto CN membrane. The gel formation onto CN membrane surface prevented the pore blocking. The EPS affinity of the surface of CA membrane was lower than CN membrane.

In addition to resistance-in-series model, the flux decline models [2,26] were also applied to CA02 and CN02 membranes to compare the results with the resistance analysis. These models and their simplified forms are shown in Table 2. J0 is the initial flux rate and ki, kc, ks and kh represent the mass transfer coefficients corresponding to various filtration laws. For constant pressure filtration, the term (A × J0) is constant and thus the simplified forms in Table 2 are obtained. Applying the experimental data to the above filtration models, i.e., plotting the different flux functions ($J^{\frac{-1}{2}}$), (J–1), (J–2) and ln(J–1) against time t, and assuming a linear correlation, the slope for each curve gives constant ki, kc, ks and kh. As seen from Fig. 5, the flux decline in

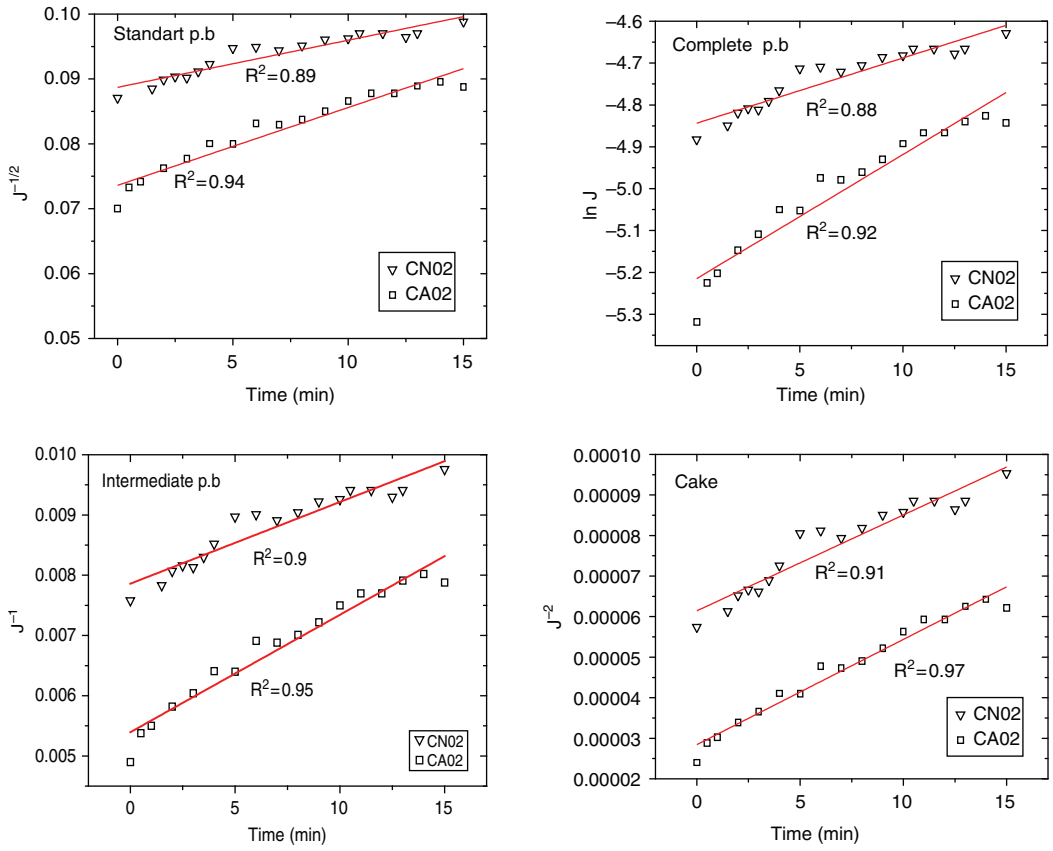

Fig. 5. Flux decline analysis for CA02 and CN02.

15 min is highly compatible with intermediate pore blocking model and particularly cake filtration model for two membranes. These results supported to the result of resistance analysis.

The high EPS concentration affected the retention and variation of the gel layer as related to the membrane type. The characteristic of gel layer changed with membrane type. The gel layer of CA membrane moved towards the membrane pore with the crossflow velocity while the gel layer of CN membrane steadily stayed onto membrane surface.

4. Conclusions

The combination of a JLBR and MF membrane unit is an efficient, reliable and compact process for biological treatment of high strength wastewater. In this study, it was observed that sludge characteristics of JLMBR were quite different than the conventional activated sludge system with low RH, high EPS and low SMP content. It was found that the protein content was lower that carbonhydrate content. Different fouling behaviors were observed for different membrane types. Microfiltration experiments show that there was no distinct difference in terms of total resistances of CA and CN membranes with pore sizes of 0.45 and 0.2 μm. Cake formation fouling was found to be dominant mechanism in CN045, CN02, and CA02, while pore fouling was the main mechanism for CA045. It was concluded that the EPS in the sludge of JLMBR strongly formed the gel layer onto CN membrane. The gel formation onto CN membrane surface prevented the pore blocking. The EPS affinity of the surface of CA membrane was lower than CN membrane. Flux decline analysis suggested cake filtration which was a comparable result with resistance-in series-model.

Acknowledgement

This study was financially supported by the TUBITAK, the Scientific and Technological Research Council of Turkey (Project No: 108Y129).

Symbols

R_t	—	Total hydraulic resistance
R_m	—	Membrane resistance
R_p	—	Pore blocking resistance
R_c	—	Cake layer resistance
μ	—	Dynamic viscosity
J_{ss}	—	Steady state flux
J_0	—	Initial flux
OD	—	Optical density
HYD	—	Hydrophobicity
VIS	—	Viscosity
SMPp	—	The protein content of soluble microbia-products
SMPc	—	The carbohydrate content of solublemicrobial products
EPSp	—	The protein content of extracellular poylmeric substances
EPSc	—	The carbohydrate content of extracellular poylmeric substances

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