



Effects of sludge particles on performance of enhanced backwash for fouling reduction in anaerobic membrane bioreactors

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ABSTRACT

The objectives of this research are to investigate effects of sludge properties in terms of particle size composition on performance of physical and chemical cleaning and to understand fouling mechanisms of the specific feed solutions. The sludge was centrifuged at three rotational speeds to differentiate particle size distributions, and the supernatant and residual flocs were used for microfiltration and cleaning processes. The filtration flux with the flocs was greater than those of the three supernatants, which indicates that fouling was greater with supernatants. Taking into account that the solid concentrations of the raw sludge and the residual flocs were 10 times greater than those of the supernatants, the reduction in flux for the supernatants is significant. The flux was recovered to 64% of the clean water flux at the maximum efficiency by two cleaning of membrane fouled with supernatant treated at 1,000 rpm. In addition, increasing removal of coarse particles by applying higher centrifugal speed (i.e. 1,000 and 1,500 rpm) yielded greater recovery performance by chemical cleaning. Particle composition such as the fine particle concentration played a significant role in fouling and the enhanced backwash cleaning efficiency.

Keywords: Anaerobic MBR; Membrane fouling; Sludge particles; Chemical cleaning; Physical cleaning; NaOCl

1. Introduction

Owing to the possibility of energy generation and efficient separation of treated effluents from sludge, anaerobic membrane bioreactors (AnMBRs) have been received significant attention in wastewater treatment [1]. Compared to aerobic treatments, bio-energy gas

such as methane (CH₄) can be generated and aeration is not required [2]. According to Hu and Stuckey [3], AnMBR not only allows short hydraulic retention times (HRTs) independent to solids retention times, but also can improve effluent water quality and possibly recycle nutrient (nitrogen and phosphorus) as a fertilizer. AnMBR has been proven to provide more

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reliable and more compact anaerobic treatment compared to traditional anaerobic processes.

The critical bottleneck of AnMBR process is membrane fouling, which limits operability of the process. Membrane fouling can decrease the productivity and increase operational cost of the system [1], thus controlling membrane fouling is very important to ensure the successful implementation of membrane processes in long run. The mechanism of membrane fouling has been reported in many studies using Hermia's model. There are in general four fouling mechanisms: pore blocking, complete blocking, intermediate blocking, and cake layer formation. The major membrane fouling mechanisms under constant pressure filtration were found to be cake formation or complete blocking [1,4,5]. According to Meng et al. there are numerous factors that affect membrane fouling, such as hydrodynamic conditions, membrane characteristics, module design, and sludge properties including particle size, extracellular polymeric substances, soluble microbial products (SMP), hydrophobicity, and surface charge [5]. Yan et al. [6] compared fouling properties of two different processes and revealed that the process with higher concentration of SMP and the smaller particle size in the sludge showed greater fouling rate in 0.08 μm pore size of membrane filters. In particular, particle size is one of the key factors that control membrane fouling propensity [7–9]. Liu and Sun [4] reported that 87% of cake resistance is mainly caused by particle sizes of 1.0–2.7 μm . Meng et al. [10] characterized the cake layer formed on the membrane surfaces using several tools including scanning electron microscopy and particle size analyzer and found a strong deposit tendency of the small particles in sludge suspension on the membrane surface, which yielded fouling. Marco [8] showed that increasing particle size through coagulation and sedimentation results in a higher permeability and lower fouling. Lin et al. [11] explained the fouling process in a laboratory scale submerged AnMBR, which was initiated from the attachment of small flocs and progressed to the cake formation.

There are several ways to control fouling in membrane processes, such as development of anti-fouling membranes, use of good quality feed water, and optimization of operations to reduce fouling. Stuckey [12] categorized the fouling control strategies during operation into three approaches: (1) scheduled continuous cleaning by both relaxing/backflushing and chemical cleaning while maintaining a high flux, (2) less chemical cleaning than in the first approach and operating below a critical flux, and (3) using efficient operational procedures such as employing hydrodynamic tools or adsorbent additions to the AnMBR reactor. The most

frequently applied practice for fouling control in both MBRs and AnMBRs is periodic backwash with permeate [13–15]. Backwash can alleviate fouling, but for only a short period of time. The study by Jeison and Lier [16] also pointed out that the cake was mainly reverse on a short-term basis; however, the consolidated cake by the long-term operation was not removed by back flush and needed more extensive cleaning methods. Compared to regular backwash, enhanced backwash has been proposed as a more efficient backwash method and is popular in membrane processes in water and wastewater treatment plants. During enhanced backwash, low doses of one or a mixture of chemical agents such as NaOCl and NaOH, are intermittently introduced. For example, operations could consist of the usual filtration, regular backwash every 30 min, and enhanced backwash every 12 h. Enhanced backwash is effective for flux recovery in high-strength organic feed solutions due to the use of oxidizing chemicals. However, little research has been conducted on the effect of sludge properties on enhanced backwash efficiency in AnMBRs.

Therefore, in this study, sludge properties were differed by particle compositions, more specifically particle size distributions. The sludge was centrifuged at three rotational speeds to yield different particle size distributions. The supernatant, the residual flocs, and the raw sludge were used for microfiltration and cleaning procedures. The cleaning consisted of physical and chemical cleanings to distinguish mechanical and chemical effects during enhanced backwash in real plants. Results from the three types of feed waters were evaluated for performance of cleaning processes and for better understanding of fouling mechanisms.

2. Theoretical models

2.1. Resistances-in-series model

A flux model is useful for obtaining membrane operating parameters such as membrane resistances. The resistances-in-series model has been used to understand flux and fouling characteristics in microfiltration as shown in Eq. 1. The permeate flux can be expressed in terms of resistances and the transmembrane pressure [17]. Cake resistance and specific cake resistances can be obtained from the resistance-in-series model evaluated using flux measurements for a solution. In addition, resistances incorporate the operational process of microfiltration.

$$J = \frac{\Delta P}{\mu \cdot R_T} = \frac{\Delta P}{\mu \cdot (R_M + R_{ph} + R_{ch} + R_{ir})} \quad (1)$$

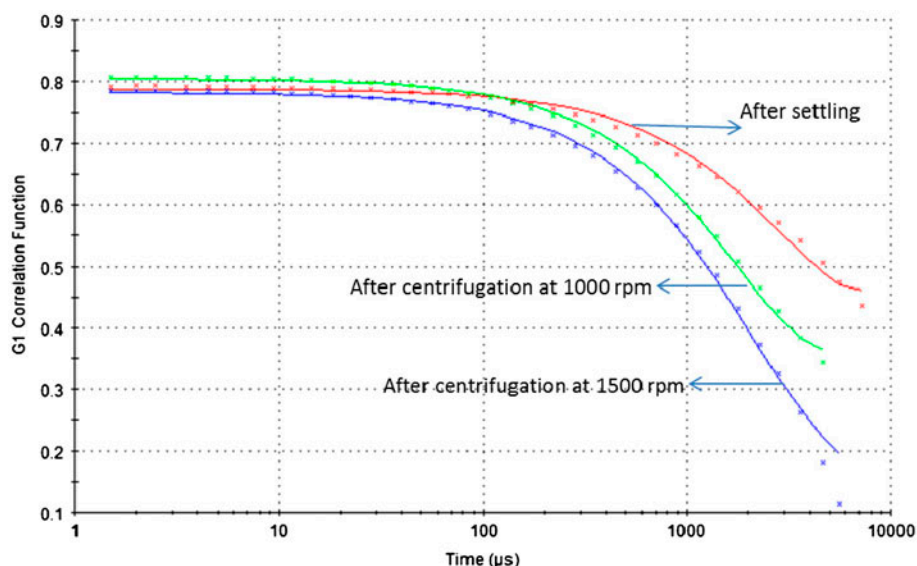


Fig. 1. Correlation function of supernatants from centrifuge with different rotational speed.

where J is the permeate flux through the membrane ($L/m^2/s$); ΔP , the transmembrane pressure (kPa); μ , the viscosity of permeate (Pa-s); R_T , the total resistance (m^{-1}); R_M , the intrinsic membrane resistance (m^{-1}); R_{ph} , the cake resistance (m^{-1}), which can be recovered by physical cleaning; R_{ch} , the foulant resistance (m^{-1}), which can be recovered by chemical cleaning; and R_{ir} , the irreversible fouling resistance (m^{-1}), which is not recovered by the cleaning procedures applied in this study. The clean water flux of the new membrane was used to obtain R_M . The flux at the end of the sludge filtration, after physical cleaning and NaOCl cleaning, was used to calculate R_T , R_{ph} , and R_{ch} , respectively. The irreversible fouling resistance was calculated from the difference between the total resistance and the sum of the resistances recovered by physical and chemical cleaning.

3. Experimental

3.1. Raw sludge and feed solutions

Primary sludge was collected from an anaerobic digester (AD) in the Ansan wastewater treatment plant (Gyeonggi Province, Korea). The AD was operated at a temperature of 35°C and a HRT of 39 d. The primary sludge from the digester, which had a total solid (TS) concentration of approximately 21,440 ($\pm 1,450$) mg/L, was sampled and moved to the laboratory for membrane filtration tests on that day, so that the sludge activity was maintained at the same level as in real operations. The sludge was diluted to a concentration

of 3,000 mg TS/L for further experiments on microfiltration and cleaning efficiency. The concentration of 3,000 mg/L was determined considering pumping the sludge from upper part of the digester out to external microfiltration. The raw sludge with 3,000 mg/L suspended solid was then centrifuged for 10 min (VS-6000N, Vision Scientific Co. Korea) at three rotational speeds, i.e. 500, 1,000 and 1,500 rpm, to produce solutions with different particle properties such as particle size distribution. Centrifugal sedimentation is useful to separate submicron particles [18]. A particle in a centrifugal field settles with a velocity which established by two forces in opposition, a centrifugal force and a drag force. The settling velocity from centrifugal sedimentation is increased proportional to the square of particle diameters and of rotational speeds. The smaller particles settle at the higher rotational speed [19]. In addition, gravity settling was also applied for comparison. The average particle sizes by gravitational settling alone and centrifugal settling at 500, 1,000, and 1,500 rpm were measured as z-average sizes of particles by Zetasizer (Malvern zetasizer Nano, UK) and the z-average sizes were 2.2, 2.0, 1.0, and 0.88 μm , respectively. Supernatant from centrifuged by the rotational speed of 500 rpm seemed similar to the supernatant from gravitational settling. The correlation curves of the cumulants analyses of different supernatants are also presented in Fig. 1. The curves showed the measured data by the analyzer using dynamic light scattering experiments. The curve clearly indicated that the centrifugation produced the supernatants with different particle characteristics.

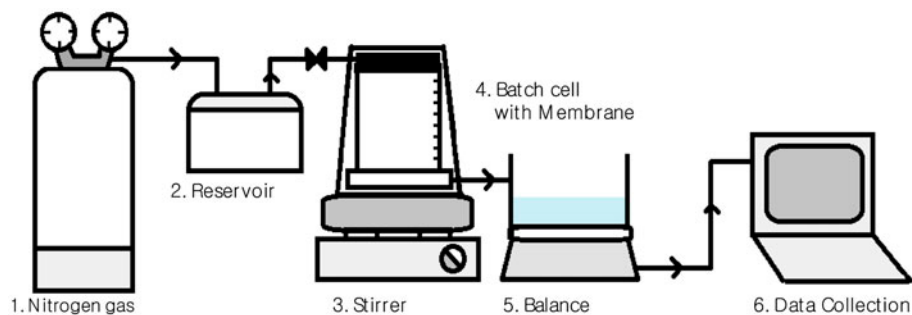


Fig. 2. Schematic of a bench-scale membrane setup.

After the supernatants were removed, the remaining flocs were completely mixed with distilled water of the same volume as that of the liquid withdrawn. The supernatants from centrifugation and the remaining flocs were used for microfiltration and the cleaning processes. Characteristics of the key water quality parameters of the feed waters are shown in Table 1.

3.2. Membrane filtration

A bench-scale membrane apparatus (Millipore Co., USA) as shown in Fig. 2 was set up to conduct short-term filtration tests. Feed solutions were raw sludge, supernatants from centrifugal sedimentation and re-suspended flocs. The polytetrafluoroethylene (PTFE) membrane (Millipore Co, USA) used in the batch cell had an effective area of 28.7 cm² with its properties listed in Table 2. The clean water flux of each filter was measured for 10 min after one day of soaking in distilled/deionized water.

The feed for microfiltration was introduced into a 3-L reservoir before delivering to a dead-end permeation cell at 14.5 psi by nitrogen gas. Permeate from the cell was measured with an electronic balance (AND GF-2000, A&D Engineering Inc., San Jose,

USA), which was connected to a data acquisition system to record the mass of water at every 20 s. In addition, the flux was measured at each step of cleaning procedure with this bench-scale apparatus for 15 min. Therefore, four values of the flux were gathered for one operation: the pure water flux, the flux at the end of the operation, the flux after physical cleaning, and the flux after chemical cleaning. These flux values were used to calculate the resistances recovered by physical cleaning and chemical cleaning.

3.3. Membrane cleaning procedures

For chemical cleaning, sodium hypochlorite (NaOCl) was selected due to its frequent use in water and wastewater treatment. Several NaOCl concentrations were experimented to learn their efficiencies on enhanced backwash cleaning with the raw sludge. The concentration of 100 mg Cl₂/L was chosen for further experiments since the greatest flux recovery was detected. In addition, anaerobic toxic assay was performed with the NaOCl solution to prevent adverse effects of the chemicals on anaerobic microbes as shown in Fig. 3. The assay data showed little change of cumulative gas production compared to the blank

Table 1
Characteristics of feed solution before microfiltration

Parameters	SS (mg/L)	Turbidity (NTU)	pH	UV ₂₅₄ (cm ⁻¹)	SCOD (mg/L)	z-Average particle size (μm)**
Raw sludge	3,000	580	7.79	0.256	116	–
Supernatants from centrifuge*	SC500	–	63.9	0.241	26	1.985
	SC1000	–	46.7	0.135	8	1.040
	SC1500	–	38.2	0.197	4	0.876
Flocs	19,067	343	6.77	0.018	–	–

*SC500, SC1000, and SC1500 are supernatants centrifuged at a speed of 500 rpm, 1,000 rpm, and 1,500 rpm, respectively.

**Measured with the Zetasizer.

Table 2
Properties of PTFE membrane filter used in this study

Material	Pore size (μm)	Thickness (μm)	Flow time* (mL/min/cm ²)	Porosity (%)
PTFE	0.1	30	100	80

*100-mL water, 20°C, 47-mm disk, 8.97''-Hg vacuum conditions were used (provided by the manufacturer).

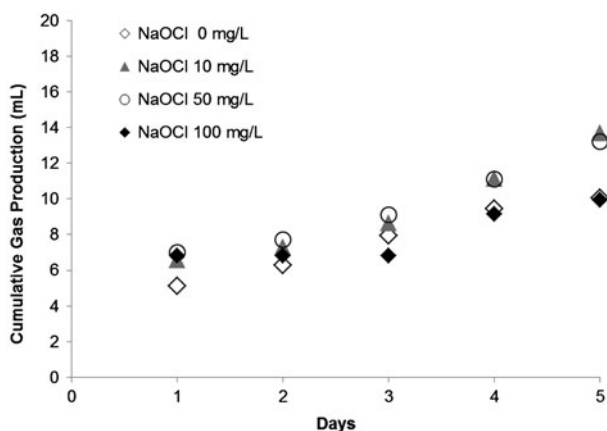


Fig. 3. Microbial reactions with different doses of NaOCl.

sample (which contained only substrate and activated sludge) during the 5 d of operation with 100 mg Cl₂/L of the chemical agent. The sludge was added to obtain 0.5 of the feed to microbe concentration ratio. The washing procedure involves 10 min of physical cleaning followed by another 10 min of chemical cleaning with NaOCl solution. Between two cleaning, the flux was measured for 15 min to evaluate recovery of permeability. The physical cleaning was done with a stirring bar. The clean water flux with distilled water (PURELAB classic, ELGA LabWater, Lane end, UK) was measured after each cleaning procedure.

3.4. Analytical methods

Chlorine solutions with a concentration of 100 mg/L were formulated from 8% NaOCl stock solution (DukSan, Seoul, Korea). The chlorine concentration was standardized by titrating the solution with 0.01 N Na₂S₂O₃ which in accordance to standard method 4500 B.2c. The dose specified for the NaOCl cleaning was then diluted from the stock solution. The TS were also measured using 10 mL of the sludge according to standard method 2540D. A Hach 2100N turbidimeter (Hach, Loveland, Co, USA) was used to measure turbidity and a StablCal[®] calibration set was used for calibration. pH was measured using an Orion Model 410 + ApH meter (Thermo Electron Co, Beverly, MA, USA). Chemical oxygen demand (COD) was measured using a reactor digestion method with

a UV–visible spectrophotometer (DR2500, Hach Co, USA). COD tests were carried out after filtering the samples through the prerinsed 0.45- μm membrane syringe filters.

4. Results and discussion

4.1. Water quality from microfiltration

The diluted AD sludge and solutions treated at different centrifuge speeds underwent microfiltration. The water quality of the microfiltration permeate was relatively high in terms of turbidity as shown in Table 3. The turbidity of the permeate was decreased to less than 0.51 NTU for the entire experiment. The great solid removal has been one of the main reasons for consideration of AnMBRs application.

The organic removal by microfiltration was analyzed using UV254 absorbance instead of COD values. The absorbance reduction was in the range of 36.5–66.7%. Stuckey reviewed recent developments in AnMBRs and noted that high removal of COD (~98%) could be maintained at even HRTs as low as 3 h [12]. Lew et al. [14] also mentioned that the great COD removal (>96% COD) was remained throughout the six-month operational period. However, the membrane filters in the literatures had molecular weights of 30 kDa and 100 kDa, which was much smaller pore sizes than the membrane filters in this study (i.e. 0.1 μm). Dagnew et al. [20] fractionated anaerobic sludge to supernatant and cake solids and found that solids contributed more than 84% for the whole sludge. Since the UV analyses were conducted after passing through by 0.45- μm filter, the UV absorbance accounted only soluble fractions of the sludge, which might be one of causes for the small reduction in the UV absorbance in addition to the relative great pore size of the microfilter used in this research. In usual, the organic matter removal of microfiltration has reported in the range of 20–50% for treating surface water [8,17].

4.2. Flux reduction of feed solutions with different particle compositions

The pure water flux measured in this work is in the range of 40–43 L/m²/h/kPa. After the feed

Table 3
Water quality of microfiltration permeates

Permeate	Raw sludge	SC500	SC1000	SC1500	Flocs
Turbidity (NTU)	0.389	0.228	0.511	0.367	0.128
% Removal	99.9	99.6	98.9	99.0	99.9
UV ₂₅₄ (cm ⁻¹)	0.119	0.087	0.086	0.089	0.006
% Removal	53.8	63.9	36.5	54.9	66.7

solutions entered the batch permeation cell, the flux decreased immediately to 2.5–11.7 L/m²/h/kPa. As filtration continued, the flux fell to 0.55 L/m²/h/kPa, as shown in Fig. 4.

The rapid fouling at the beginning of membrane operation was frequently observed with AnMBR sludge [20]. The TMP was increased to 5–15 kPa as soon as the filtration was started. The specific flux of a feed solution with 18,000 mg/L of TS was dropped from 6 to 2.5 L/m²/h/kPa in 30 min of operations with a bench scale apparatus. Amine et al. [21] studied fouling mechanisms responsible for flux decline of microfiltration and ultrafiltration of AnMBR sludge. They investigated 44 cases of experimental data from numerous literatures to characterize fouling into pore constriction, cake formation, complete blocking, and intermediate blocking. They concluded that the most appropriate fouling model in AnMBRs was cake layer formation which was fitted to 61% of studied cases while 25% of the cases were due to complete blocking. The most rapid flux decline was observed when the cake layer formation was built on the membrane surfaces.

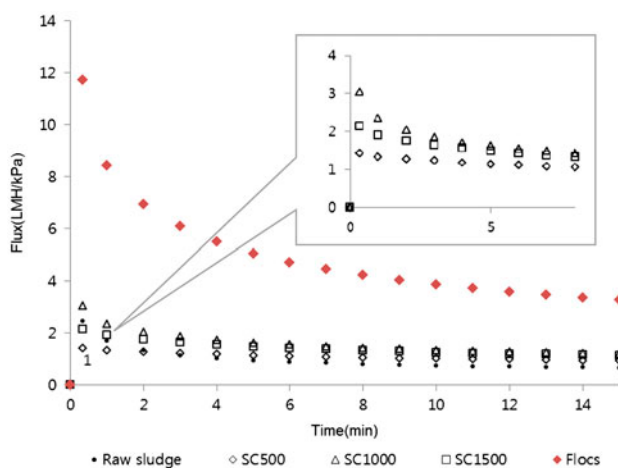


Fig. 4. Flux reduction of feed solutions with different particle compositions.

Since the fouling was severe in AnMBRs operation, filtration under the critical flux was also emphasized in many studies [22,23]. Stuckey reported that maintaining the flux under the critical condition would be very important for stable AnMBR operation [12].

The flux reduction was larger with raw sludge, as expected. Surprisingly, the flux reduction with the centrifuged solutions was similar to the raw sludge, although the solid concentrations of the solutions (38.2–63.9 NTU) were significantly lower than that of raw sludge (580 NTU). In addition, the flocs remaining at the bottom of the centrifuge tubes were resuspended with distilled water and were microfiltered. Flux reduction for the flocs was relatively low, indicating that the effects of large particles on membrane fouling were less detrimental although the flocs solution had the largest solid concentration (19,067 mg/L). It has been reported that supernatant is the main contributor to membrane fouling in MBR reactors [4]. Supernatant caused 50% of specific cake resistance and was the main contributor to membrane fouling [9]. Dagnew et al. [20] fractionated sludge to whole, cake, and supernatant and experimented to evaluate fouling resistance by each fraction. They concluded that 70–84% of the total fouling resistance was contributed by the supernatant fraction. In addition, Shimizu et al. [24] mentioned that certain ranges of particles, i.e. particles with sizes from 8 to 15 μm , caused the lowest lift velocity during cross-flow microfiltration.

4.3. Flux recovery by physical and chemical cleaning

Enhanced backwashing uses chemicals during the backwash process. In this study, enhanced backwash was simulated with physical cleaning by stirring followed by chemical cleaning by NaOCl. Three concentrations of NaOCl were used during the cleaning procedure on sludge with TS concentrations of 3,000 mg/L. The recovered flux is presented in Fig. 5. As the NaOCl concentrations were increased, the flux recovery was increased and reached 54% of the initial pure water flux. For the further experiments, the concentration of 100 mg/L was selected since it showed

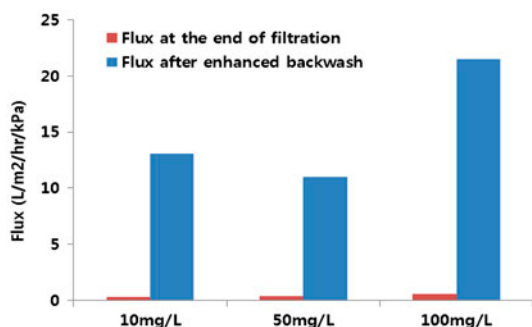


Fig. 5. Flux recovered by chemical cleaning with three NaOCl concentrations.

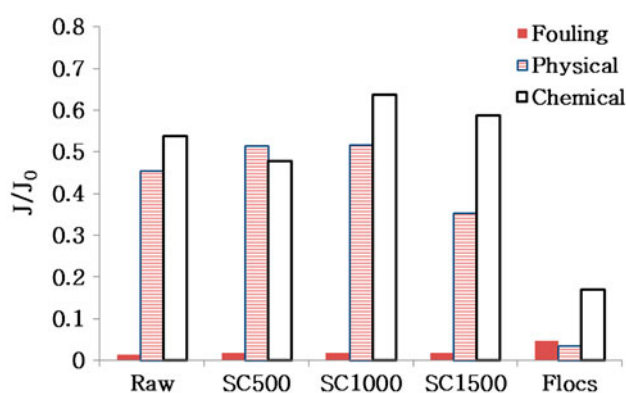


Fig. 6. Flux recovery by each cleaning method. J_0 : initial pure water flux.

the greatest flux recovery and little impact on AD microorganisms. Based on the anaerobic toxic assay results shown in Fig. 3, increasing chemical reagent concentration greater than 100 mg Cl_2/L might yield some reduction of biogas production. The concentration of 100 mg Cl_2/L is, in general, a higher dose than the dose of usual enhanced backwash operation and a

lower dose than the dose during recovery chemical cleaning in water treatment utility.

The flux recovery of each cleaning during enhanced backwash, i.e. physical stirring and chemical cleaning was investigated for solutions of raw sludge, supernatant from solutions centrifuged at various rotational speeds, and for the remaining flocs, and the results are shown in Fig. 6. The physical cleaning was effective in recovering water flux of AnMBR. The flux recovery by physical cleaning was at most approximately 22 $\text{L}/\text{m}^2/\text{h}/\text{kPa}$. The chemical cleaning showed greater recovery with the feed solution treated at 1,500 rpm. For the solution treated at 500 rpm, no recovery was observed when membrane was subject to chemical cleaning. This result is consistent with the result of the poorest flux reduction being for the solution treated at 500 rpm. Since the greater rotational speed produced supernatants with smaller sizes of particles and greater fractions of organic matter, the chemical cleaning could exhibit better performance. The good recovery achieved by the cleaning methods implies that the fouling layer made by cake formation could be effectively removed by enhanced backwash. However, the low recovery of the flocs solution showed that substantial concentrations of solids might disrupt chemical cleaning, and thus, removal of solids would be beneficial for enhanced backwash.

Resistances after each process as shown in Table 4 were calculated based on the Eq. (1) given in Section 2. The intrinsic membrane resistances, which are considered as characteristics of the PTFE membrane, were similar in all cases. R_T of the raw sludge had a higher value ($73.94 \times 10^{10} \text{ m}^{-1}$) than those of the supernatants. The resistances recovered by physical cleaning, R_{ph} , accounted for the majority of R_T (96–98%). The supernatants exhibited lower recoveries by physical cleaning than raw sludge.

Table 4

Membrane resistances, resistances recovered by physical and by chemical cleaning, and resistance by irreversible fouling of PTFE filters ($R \times 10^{10}, \text{ m}^{-1}$)

Feed water	R_m	R_{ph}	R_{ch}	R_{ir}	R_T
Raw sludge	0.97	73.6	0*	0.47	73.9
SC500	0.99	56.5	0*	1.08	57.4
SC1000	0.96	49.3	0.35	0.55	50.2
SC1500	0.96	49.6	1.08	0.67	51.3
Flocs	0.90	0*	21.40	4.38	18.5

*No effect of cleaning treatment observed.

5. Conclusions

In this study, enhanced backwash was proposed for a promising option for fouling reduction methods for AnMBR. In addition, the effect of sludge properties in terms of particle size composition on the performance of enhanced backwash was investigated. The microfiltration produced great quality of treated water as expected, although the feed concentration was high such as 3,000 mg/L of TS concentration. The particle size composition altered easily by centrifugation. The supernatants from different rotational speeds during centrifugation yield samples with different z-average sizes and scattering patterns. The microfiltration of the supernatants and the remained flocs showed that supernatant contained main foulants presenting the rapid flux decline. The series of physical and chemical cleaning was effective to recover the flux. The greatest recovery, 64% of the initial clean water, was obtained with the supernatants treated at 1,000 rpm when 100 mg/L of NaOCl was applied during chemical wash. When the enhanced backwash investigated by each cleaning step, i.e. the physical cleaning and chemical cleaning, the physical cleaning was the more effective to recover the flux than chemical cleaning. The resistances recovered by physical cleaning were taken up to 98% of the total resistances by the cakes formed on the membrane surfaces. The greater recovery by chemical cleaning with higher rotational speed of centrifugation revealed that sodium hypochlorite was promising to use for anaerobic sludge mainly composed with fine particles and organic matter. The enhanced backwash could be a promising option when anaerobic sludge was entered after certain solid–liquid separation.

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