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Enhancing filterability of flat-sheet membrane by addition of cationic polymer for sludge thickening system

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ABSTRACT

The efficiency of coagulation-coupled membrane filtration as an alternative process for sludge thickening was evaluated using various parameters that could influence the thickened sludge, such as mixed liquor suspended solids (MLSS) concentration, viscosity, critical flux and soluble microbial products. Dead-end filtration experiment and determination of critical flux were conducted to investigate the change in permeation across the membrane after addition of coagulant. PVDF flat-sheet membrane with 0.08 μm pore size was used in the experiment. Two separate submerged systems were run in parallel, one with coagulant and the other served as the control. A significant difference between the transmembrane pressures (TMPs) of the two systems was observed. It was found that an increase in soluble microbial products (SMPs) concentration decreases filtration flux, which in turn increases TMP. PSD analysis confirmed that larger flocs were formed after addition of coagulant. Confocal laser scanning microscopy (CLSM) and scanning electron microscopy (SEM) images revealed that the cake resistance was the major contributor in membrane fouling and the coagulated sludge has less potential of pore blocking compared with the normal sludge. In general, deposition of small particles that causes membrane fouling can be decreased by the addition of coagulant, which increases the size of the particle, thus preventing flux decline and TMP increase.

Keywords: Flat-sheet membrane; Sludge thickening; Coagulation; Cationic polymer; Membrane fouling

1. Introduction

Membrane technology has recently established a strong foundation in wastewater treatment. But the popularity of this technology is tailed with the problem concerning sludge treatment and disposal. An estimated 60% of the total operating cost of a wastewater treatment plant is allotted to sludge handling. This involves thickening and dewatering which are the two common processes that decrease sludge volume. Between the two, sludge thickening plays the major role in sludge reduction [1]. In the dewatering process, sludge loading depends on the thickening efficacy and water removal effectivity. However, there are several problems

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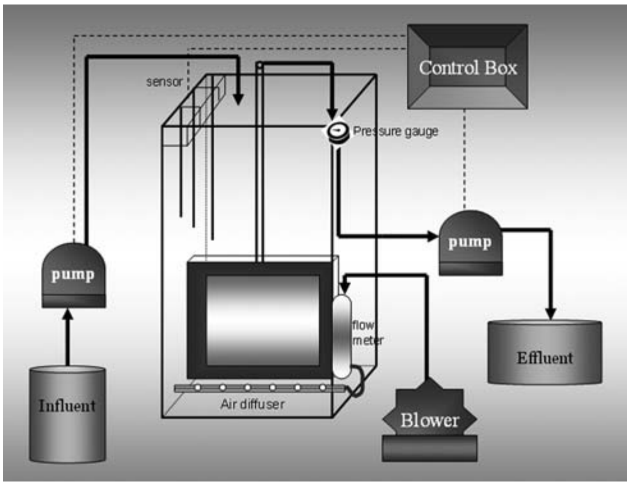

Fig. 1. Schematic of sludge thickening system.

involved in the conventional sludge thickening processes such as inefficient dewatering performance and low effluent quality. Another essential factor that must be considered in sludge handling is the huge amount of money that should be invested to handle, store and dispose this material [2,3]. Therefore, there is a need to decrease the operational cost allotted to sludge handling. In order to do so, the efficiency of the thickening process should be heightened. Wang et al. [1] demonstrated an efficient thickening process with membrane application for waste activated sludge.

However, there are some limitations of membrane application on activated sludge [4]. The main concern evolves on the membrane fouling that is basically caused by biopolymers present in the sludge, which greatly affect filtration flux. Other concerns are related to high capital investment and operating cost. Furthermore, membrane having high fouling potential needs to be cleaned frequently, which results to higher operational expenses.

Polymer addition has been one of the most widely used techniques applied in coagulation process. Polymers create large dense flocs resulting to high settleability of biomass, enhancing liquid and solid separation [5].

One of the recent technologies developed in the field of polymer science is the modification of cationic polymer. A liquid coagulant that is composed of cationic polymers was produced for the purpose of enhancing the permeability of the membrane [4]. Its main objective is to prevent membrane fouling by forming polymer–biopolymer complexes to form a larger structure of bio-flocs. Advantages of this product include high adsorption on biological materials, negligible polymer residue on effluent and wide compatibility to all kinds of commercial membranes. Hence, application of cationic polymer has a great potential in improving the thickening process, and eventually reduce the cost for sludge handling. Supporting this concept is the study conducted by Song et al. [6], which proved that injection of coagulant minimized membrane fouling. Furthermore, it was reported that addition of cationic polymer enhanced filtration flux significantly.

This study evaluated the effectiveness of coagulant addition using cationic polymer in the performance efficiency of sludge thickening system using flat-sheet membrane. In addition, fouling mechanism on the membrane surface was examined to further explore the influence of sludge characteristics on filtration flux during thickening process.

2. Materials and methods

2.1. Experimental set-up

Sludge thickening system is composed of three portions: the influent sludge, the reactor and the effluent. The sludge was aerated in the container and then fed to the reactor using a peristaltic pump. The reactor has a total working volume of 2 L with a dimension of 16L × 19W × 45H (cm). Sensors were installed inside the reactor to monitor the level of the sludge. Sensors are designed to detect the upper limit which prevents sludge overflow, and the lower limit which signals the influent pump to supply sludge. To ensure sufficient oxygen uptake for microorganisms, air was provided by an air pump, which also scoured the membrane surface to help minimize membrane fouling.

Water suction was done using a flat-sheet membrane submerged inside the reactor. The water that passed through the membrane was collected in an effluent container with another peristaltic pump. In addition, intermittent suction was applied in the system to improve membrane filterability, which was monitored by a control system. A schematic diagram of the sludge thickening system is presented in Fig. 1.

2.2. Membrane and coagulant

PVDF flat-sheet membrane (TORAY, Japan) was used in the experiment. The membrane has a pore size of 0.08 μm with an effective membrane area of 0.03 m^2. Additional specifications of the membrane are presented in Table 1.

The modified cationic polymer was used as a coagulant. The liquid-type coagulant has a specific gravity of 1.018–1.058 and a viscosity range of 1,500–3,200 cps at 25°C. It is completely soluble in water and has an average density of 1.03 g/mL. The pH of the undiluted liquid coagulant is 6.5.

Table 1
Membrane specifications.

Specification	Description
Material	PVDF
Filtration flux	0.2–1.5 m/d
pH	5–10
Temperature	5–40°C
Scouring air	1–2 L/min/element
Membrane area	0.03 m^2
Dimension (mm)	130W130H × 7T

2.3. Experimental procedure

Two separate sludge thickening systems were run in parallel. One system was added with 100 ppm coagulant while the other served as the control. The concentration of coagulant used in the experiment was based on the preliminary investigation of the pH drop occurred during addition of coagulant. Significant drop in pH was observed upon addition of more than 100 ppm of coagulant. Thus, 100 ppm was chosen to be the working coagulant concentration in this study. Also, this is to minimize the effect of pH drop caused by coagulant in the microbial community inside the reactor.

A membrane was submerged in each reactor and was continuously aerated. The experiment was conducted at constant flux of 0.3 m/d with continuous monitoring of transmembrane pressure (TMP). The biological activity as well as the changes in the system condition were monitored by measuring the temperature, pH and dissolved oxygen (DO) inside the reactors. Sampling was done at the top of the reactor every 4 h to evaluate the increase of mixed liquor suspended solids (MLSS) as well as the change in viscosity of the biomass. Also, TMP was monitored for the entire operation. At the end of the experiment, membrane samples were collected and were analyzed using confocal laser scanning microscopy (CLSM) and scanning electron microscopy (SEM). The system's operating condition is summarized in Table 2.

Investigation on the effect of coagulant addition to membrane fouling was conducted by subjecting the sludge samples, with and without coagulant, to various analyses. These were done specifically to determine the change in critical flux, to have an overview of the particle size distribution inside the reactor and to evaluate the effect on fouling resistances. Polymeric substances, which are believed to be the primary contributor in membrane fouling, were also quantified.

Table 2
Operating condition of sludge thickening system.

Parameter	Target value
Hydraulic retention time (h)	5
Aeration intensity (L/min) permeate	2
Total filtration volume (L/day)	9
Flux (m/d)	0.3
Intermittent suction time: On/Off (min)	8/2

2.4. Analytical methods

Following the standard method, sludge concentration was determined by measuring the amount of suspended solids and volatile components of the mixed liquor. Brookfield DV-2 Viscometer was used to obtain the viscosity of the thickened sludge. SMC Digital Pressure Switch monitored the pressure increase across the membrane, whereas LabVIEW 8.5 (National Instrument, Co.) gathered and recorded these values in a computer to visualize the change in TMP through time.

Bound-extracellular polymeric substances (bound-EPS) and soluble microbial products (SMP) were analyzed by measuring the protein and carbohydrate contents of the sludge. For the extraction of the total polymeric substances, a modified steaming extraction was used [7], while the soluble portion of EPS was obtained by adopting the method used by Nuengjamnong [8]. Total carbohydrate concentration was determined by Anthrone method [9] while Bradford protein assay was used for protein analysis.

Dead-end filtration was conducted using Amicon Cell (Amicon™) to determine the specific fouling resistances of the fouled membrane [6,10]. Two- and three-dimensional images of the membrane surface were captured by Hitachi S-3500 and Olympus OLS 3000, respectively. Distribution of the sludge particle size was determined by Particle Size Analyzer - Mastersizer S.

3. Results and discussion

3.1. MLSS and viscosity

One of the main goals of thickening process is to reduce bulk of the solids by removing excess water making it more acceptable upon dewatering [3]. Two parameters that influence the concentration of sludge were evaluated to assess the effectiveness of coagulant addition in sludge thickening. First is the biomass concentration, which is a primary index for determining the thickening efficiency, and another is the sludge viscosity that has a linear relationship with the thickened sludge [11].

The concentrations of MLSS and MLVSS during the entire operation are shown in Fig. 2. The concentration

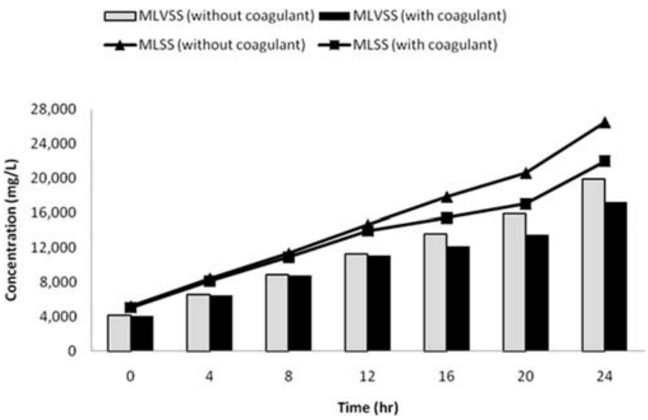

Fig. 2. Increase in MLSS and MLVSS concentrations for the control and the system with coagulant during thickening process.

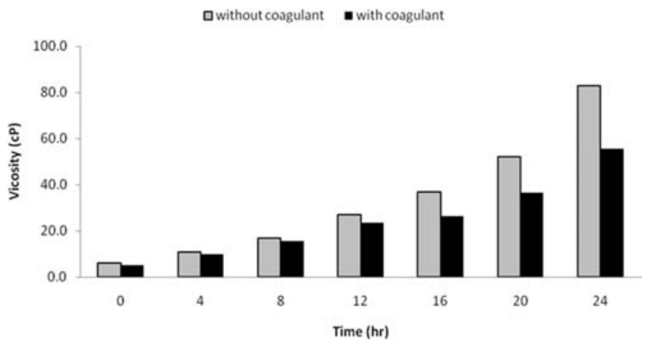

Fig. 3. Increase in the viscosity of the normal sludge and coagulated sludge during sludge thickening operation.

reached a final value of 27 and 22 g/L for the system without and with coagulant, respectively. Higher MLSS concentration for the system without coagulant compared with the system with coagulant was observed. It was previously mentioned that higher concentration of biomass indicates better performance for thickening process. This phenomenon can be attributed from the sampling procedure used. Increase in the concentration of MLVSS is observed as presented in the bar graph. The control system achieved an MLVSS concentration of 20 g/L while the system added with coagulant attained only 17 g/L at the end of the operation.

Theoretically, MLSS concentration inside the reactors should be the same given that the flux used on the two systems were the same. This is where the addition of coagulant will interfere. After addition of coagulant, the density of the biomass increased, and the majority of the MLSS was dragged to the bottom of the reactor by gravity resulting to a better separation between solid and liquid as compared to the system without coagulant. The reactor was designed with an air diffuser slightly elevated to provide an extra space for the settling of the sludge. It should be noted that the sludge sampling was conducted on the upper part of the mixed liquor, where in the case of the system with coagulant, this portion has lower MLSS concentration. This suggests that the biomass in the control was more dispersed and well distributed throughout the reactor resulting to higher concentration as compared to the other system where most biomass clustered at the bottom. Therefore, it was concluded that a much thickened sludge was present in the system with coagulant. Moreover, addition of coagulant proved that flocculation of particles occur providing lower concentration at the top and higher concentration at the bottom.

To further understand this phenomenon, Bolto et al. [5] elaborated the mechanism involved between the biomass and the added coagulant, which in this case is a cationic polymer. Sludge particles are mostly negatively charged, and these charged particles are strongly attracted to cations. The aim of the cationic polymers is to bind as many negatively charged particles as possible to create flocs. The electrostatic interaction between the sludge and the polymer is the force responsible for the negatively charged particle to attract the positively charged polymer. After this attraction, they will undergo charge neutralization that happens in the surface of the negatively charged particle. In this stage, combination of polymer and particle occurs to form flocs. These flocs are bridged together creating bigger flocs. This was proven by the experiment conducted by Song et al. [6] confirmed that small particles became bigger after addition of coagulant, which eventually helped in lessening the tendency of membrane fouling. In the study conducted by Xing et al. [11], it was concluded that there is a direct realationship existing between membrane fouling and MLSS concentration. Thus, it can be assumed that as the concentration of MLSS increases, a corresponding increase in the tendency of membrane fouling occurs.

As illustrated in Fig. 3, an increase in operation time increases the viscosity of the thickened sludge. Correlating Figs. 2 and 3, it can be seen that as the MLSS concentration increases, a corresponding increase in viscosity occurs. According to Xing et al. [11], MLSS concentration has a direct impact on viscosity. The viscosity of the system with coagulant reached 56 cP from an initial viscosity of 5 cP, whereas the control obtained 83 cP from an initial value of 6 cP. As the value of the viscosity becomes higher, the greater is the tendency of the sludge to be thickened. Moreover, the same trend was observed from the results obtained from MLSS analysis suggesting that the same concept can be applied as previously explained. It was evident

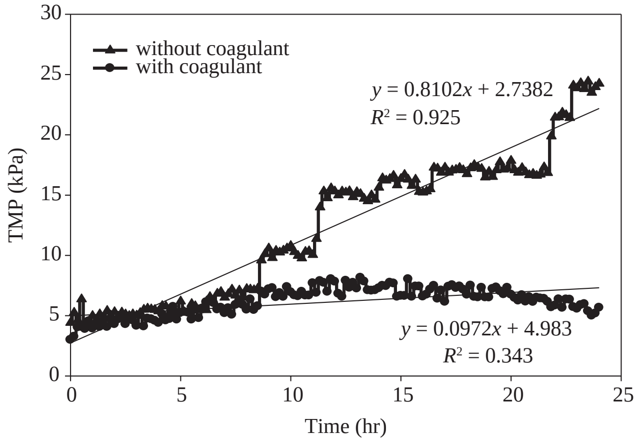

Fig. 4. TMP profile of the two systems, with and without coagulant, operated at constant flux of 0.3 m^3/m^2d.

that the reactor with coagulant has a less viscous sludge, indicative of a good solid and liquid separation. Addition of coagulant helped the particles to destabilize and produced bigger flocs. Gravity pulled these bigger flocs towards the bottom resulting to a more viscous sludge at the lower portion of the reactor. Hence, the system with coagulant still holds to be an efficient process for sludge thickening.

Wang et al. [2] found that the increase in viscosity of the mixed liquor greatly contributed in membrane fouling, while Meng et al. [12] reported that at higher viscosity, more small particles were accumulated on the membrane surface. Therefore, the high viscosity of the control system could result to a more severe fouling compared to the system with coagulant. Several researchers supported this observation proving that viscosity significantly influences membrane fouling as well as membrane filtration efficiency [13,14]. In addition, Chu et al. [15] reported that an increase in vicosity occurs when the amount of bio-flocs increases.

3.2. TMP and critical flux

As shown in Fig. 4, the TMP of the system without coagulant was observed to increase rapidly. Comparing the two systems, the slope of the system with coagulant was much lower than the slope of the control. The control system obtained a slope of 0.81 which is almost incomparable with the slope of the system with coagulant, which obtained a value of 0.10. TMP of the control started from 4.4 kPa and reached 24.3 kPa at the end of the experiment. The system with coagulant obtained 5.7 kPa from the initial TMP of 3.0 kPa. At the beginning of the operation, the TMPs of both systems were almost the same, ranging from 3.3 to 8.1 kPa. However, after 8 h, the TMP of the control abruptly increased from 7.3 to 10.1 kPa. This is due to the decrease in the filtration volume caused by the formation of cake layer in the membrane surface. Therefore, to maintain the filtration flux of 0.3 m/d, the rate of suction by peristaltic pump was adjusted resulting to the sudden increase of TMP. This also explains the succeeding TMP increase for the system without coagulant. It was also reported that a sudden rise of TMP can also be attributed to the deposition of biopolymers [3]. In contrast, the system with coagulant did not show abrupt increase in TMP, rather a roughly constant TMP was observed. This indicates that addition of coagulant improves filtration efficiency during sludge thickening.

Moreover, the increase in TMP for the control system can also be attributed to the increase in MLSS concentration presented previously. According to Fan et al. [16], MLSS concentration alone has the most negative effect on the filtration flux. As mentioned earlier, the distrubution of biomass at the top of the reactor was higher for the system without coagulant. And it is worth mentioning that the membrane is placed slightly higher from the bottom of the reactor exposing a larger membrane surface area to the upper portion of the mixed liquor. Therefore, the system without coagulant has a higher tendency for the particle-membrane interaction that will eventually cause membrane fouling.

Flux-step method was used in determining the critical flux of the submerged membrane bioreactor [17]. For each flux-step, with 15-min interval, two TMP values were obtained, the initial TMP and the final TMP. An increase of 6 LMH was employed in the succeding steps. The rate of TMP increase was calculated by dividing the change in TMP with the operating time in each step.

As shown in Fig. 5, after addition of coagulant, the value of the critical flux became higher. Initially, the normal sludge has a critical flux of 48 LMH. However, after coagulant addition, the value rose to 78 LMH, which is almost twice of its initial value. This serves as an evidence for the unchanged TMP observed in the system with coagulant during the thickening process. Critical flux could also be affected by the characteristic of sludge and could be controlled predominantly by colloidal particles [16,18]. Change in the concentration of MLSS and quantity of SMP are the two properties of sludge that could influence critical flux. Coagulated sludge having larger flocs has lower surface area of contact to the membrane surface compared to sludge that is not yet coagulated. This enhances the filtration flux which is directly related to the improvement of critical flux in the system added with coagulant.

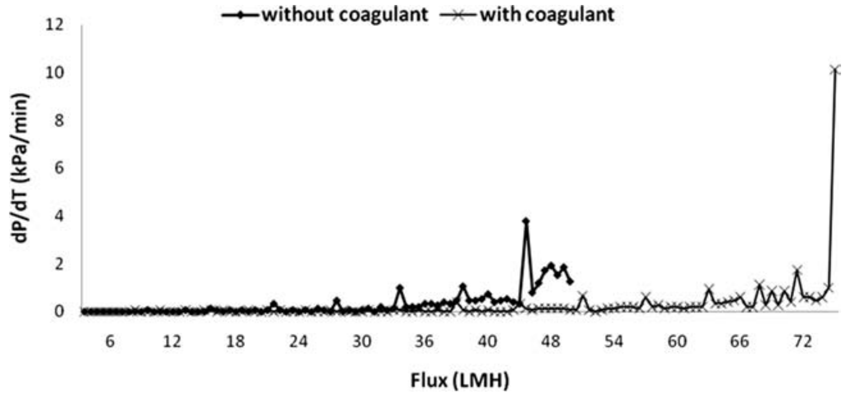

Fig. 5. Change in critical flux after addition of coagulant.

3.3. EPS

Currently, researchers have high interest on the effect of EPS on membrane fouling. It was believed that the most significant factor that should be considered in fouling mechanism is the polymeric substances produced during microbial metabolism [19,20]. Particularly, soluble EPS, also known as SMP, is said to play the main role in membrane fouling [21].

During the system operation, it was observed that the pH of the system was decreasing with time indicating that some polymeric substances were released during microbial degradation. These polymeric products, particularly SMP, were reported to be the primary contributor for severe fouling in the membrane [13,22]. The microbial products present in the sludge affect the fouling mechanism by accumulating and forming a thin layer in the membrane's surface [20].

The differences on the concentrations of SMP and bound-EPS are summarized in Table 3. Bound-EPS was calculated by subtracting SMP from the total EPS obtained by steaming extraction. Protein and carbohydrate concentrations presented in the table comprised mostly of microbial products. It was reported that carbohydrates and protein are considered as irreversible foulants [1]. For SMP, the total concentration of polymeric substances was greater in the system without coagulant that contributes to the prospective foulants that could adhere on the membrane. These results were also observed by Meng et al. [20] after comparing the deflocculated sludge with bulking sludge. It was found that for the bulking sludge, the bound-EPS was higher than the deflocculated sludge, while an opposite result was observed for the free EPS concentration. Moreover, the higher the free EPS concentration, the severe the fouling will be observed.

Fan et al. [16] mentioned that there is an existing correlation between critical flux and SMP. The higher the amount of SMP in the sludge, the greater the amount of particles that could be deposited on the membrane, especially in the membrane pores, during permeation. Also, SMP exhibited a low back transport, which is potential for irreversible fouling [3]. Higher concentration of bound-EPS for coagulated sludge was observed compared with the normal sludge. This indicates that portions of microbial products from the bulk phase were incorporated in the flocs during coagulation process. A study conducted by Hwang et al. [23] supports this observation confirming that after addition of cationic polymer, a large portion of SMP were entrapped in the coagulated flocs during flocculation and coagulation of sludge.

Table 3
Composition of microbial products with specific concentrations for SMP and bound-EPS for both systems, with and without coagulant.

	SMP		Bound-EPS	
	Without coagulant	With coagulant	Without coagulant	With coagulant
Protein (ug/ml BSA)	14.3	10.1	38.8	49.8
Carbohydrates (ug/ml glucose)	88.8	77.8	215	230
Total concentration (mg/g VSS)	25.8	19.5	63.6	69.8

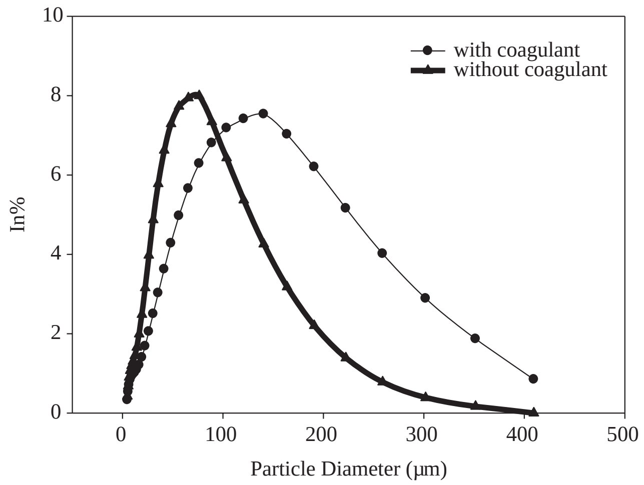

Fig. 6. Comparison of the particle size distribution of the normal sludge and coagulated sludge.

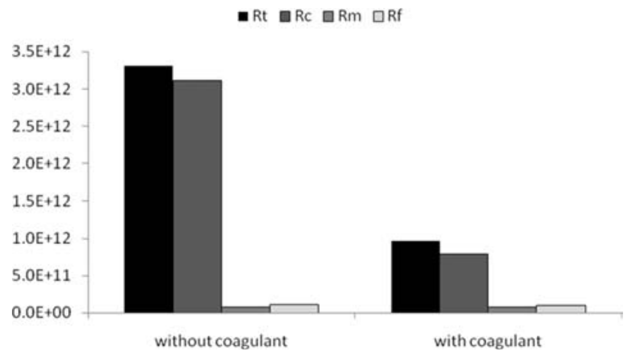

Fig. 7. Specific resistances of the fouled membrane using coagulated and normal sludge.

As previously mentioned, membrane fouling was attributed to the increase of sludge concentration which resulted in high viscosity. However, according to Wang et al. [2], change in sludge properties, such as particle size, also plays an important role in membrane fouling. Particle size is one of the factors that affect the cake formation in the membrane surface and a key parameter in lowering hydraulic resistance [24]. Addition of cationic polymer as coagulant could neutralize negatively charged particles by electrostatic attraction, where flocculation happens. According to Hwang et al. [23], during flocculation, small particles with approximately 5 μm in size disappeared in the system added with cationic polymer. Particle sizes of both kinds of sludge were compared. A distinct difference was observed as depicted in Fig. 6. The graph of the system without coagulant was more positively skewed indicating that a large number of smaller particles were present. Whereas coagulated sludge has a higher position of its tail in the graph compared with the normal sludge, which suggests that larger particle size exists. It was concluded that the effect of coagulant addition is dependent on the degree of difference of the tails of the graphs. Moreover, efficiency of coagulation could be directly related on the location of the tail, specifically when the graph is negatively skewed.

3.4. Membrane fouling mechanism

Dead-end filtration experiment was conducted to determine the specific fouling resistances and to study the fouling mechanism in the membrane surface. Fig. 7 shows the specific resistances – Rc (cake resistance), Rm (membrane resistance), Rf (fouling resistance), and Rt (total resistance) – of the fouled membrane using coagulated and normal sludge. This shows that addition of coagulant decreased the total resistance from 3.3×10^{12} m^{-1} to 9.7×10^{11} m^{-1}. This result could explain the unchanged TMP observed in the system with coagulant, whereas in the control, having higher total resistance, passage of water through the membrane was prevented that caused the flux to decrease and the TMP to increase. Increase in hydraulic resistance was mainly due to the cake formation in the membrane surface, which was evident in the increase of TMP. Cho et al. [25] also investigated this phenomenon and concluded that specific cake resistance plays an essential role in assessing hydraulic resistance.

Low concentration of microbial products observed from the coagulated sludge could be a possible reason for the decrease in total resistance of the sludge with coagulant. Colloidal particles that caused bio-fouling were directly proportional to the specific resistances [26]. Cake resistance was the predominant factor that hindered water permeation. Approximately 94% of the total resistance for the normal sludge caused cake layering. However, after addition of coagulant, this value was decreased to 81%, enhancing the permeability across the membrane.

The membrane morphology was investigated by examining the three-dimensional images captured by CLSM. According to Ferrando et al. [27], visualization for the adsorption-deposition processes in the membrane surface is possible through CLSM analysis. The morphology of the cake layer and the depth of the fouling layer are the two important parameters that should be considered in examining a fouled membrane. It was observed that the membrane surface of the system with coagulant was covered with more sludge as depicted by the white regions as seen in Fig. 8. As previously illustrated, the coagulated sludge with low viscosity could be assumed to be less sticky. Chu et al. [15] mentioned that the sludge viscosity has a direct

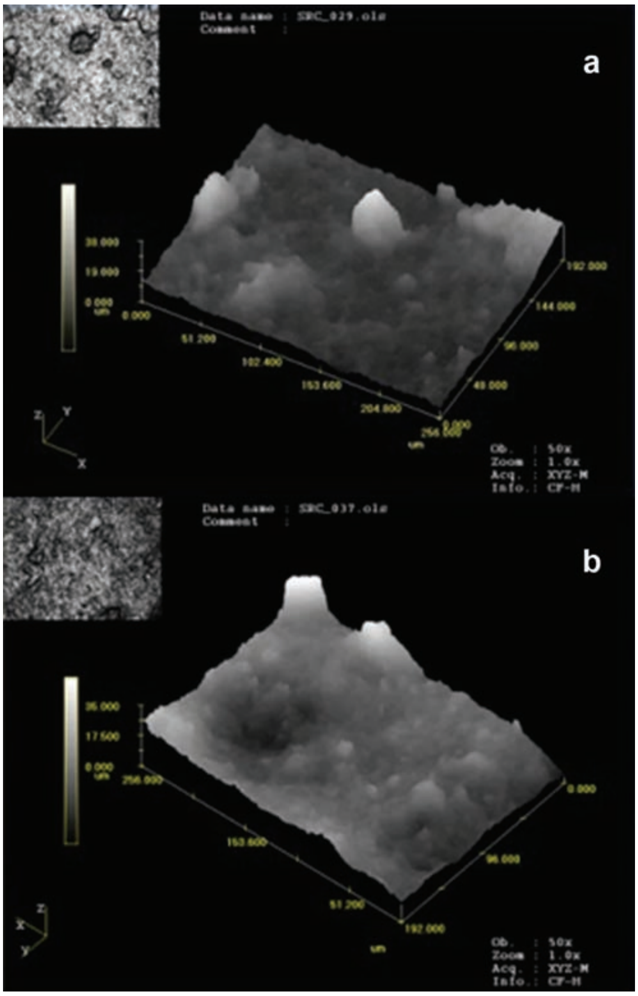

Fig. 8. Three-dimensional CLSM images of biofilm formation on the membrane surface: (a) without coagulant and (b) with coagulant.

relationship to biomass stickiness. Thus, as the viscosity increases, the stickiness also increases resulting to a difficulty in removing the adhered foulants on the membrane. This could explain the characteristic of the coagulated sludge having a greater potential to be removed from the membrane surface compared to the normal sludge. Furthermore, normal sludge was composed of smaller particles that penetrated through the membrane pores that caused irreversible fouling. This is unlike the coagulated sludge which was simply removed through air scouring.

SEM was conducted to investigate the membrane surface before and after addition of coagulant. The fouled membrane was washed with dI water then dried at room temperature. To examine the surface of the membrane for any signs of fouling, the images were magnified to 10K as seen in Fig. 9.

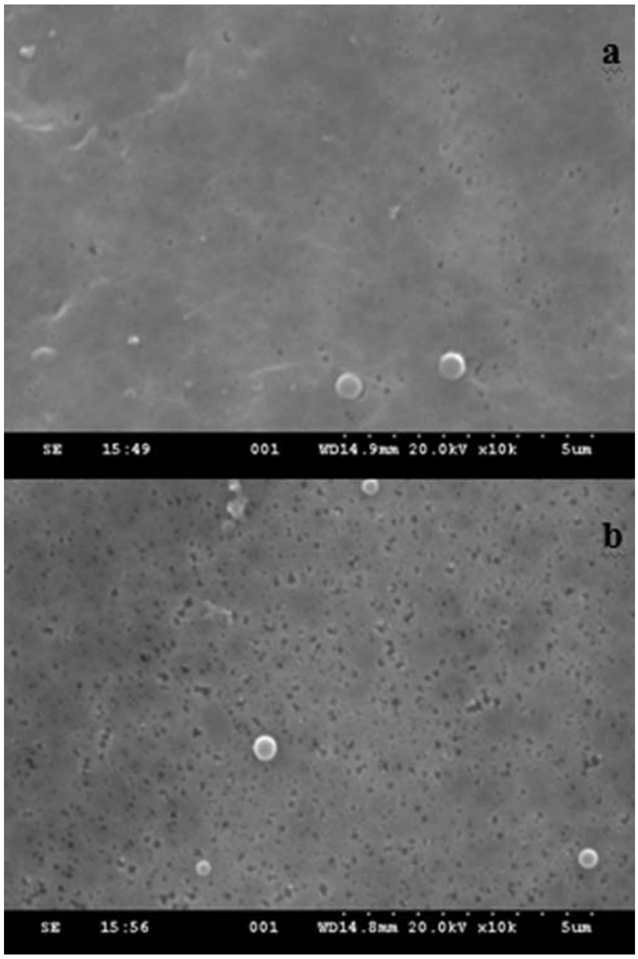

Fig. 9. SEM images of the membrane surface after physical washing: (a) without coagulant and (b) with coagulant.

For comparison, the membrane of the system with coagulant (Fig. 9b) has more holes compared with the membrane of the control (Fig. 9a). Some particles were deposited in the membrane causing pore blocking, thus less and smaller pores were observed in the system without coagulant. After coagulant addition, pores of the membrane were observed to be bigger as compared to the control. This can be attributed to the bigger flocs formed during addition of coagulant. Smaller particles were incorporated in the flocs causing the smaller particles to decrease in the bulk phase, thus, less pore fouling occurred.

The mechanism of particle adhesion on the membrane surface by biopolymer and other smaller particles is initiated by surface adsorption caused by the dragging force applied during filtration. Biopolymers are then introduced into the membrane pores resulting to difficulty in cleaning by any physical means [15]. Furthermore, the decrease in permeability cannot be attributed solely to the structural change of the cake layer but also on the rate of cake growth on the membrane surface and the deposition of microbial flocs in the membrane pores.

4. Conclusions

It was found that cationic polymer is an effective coagulant that improves the efficiency of the thickening process. Addition of coagulant caused the particles to form complexes creating bigger flocs. Increase in the density of the flocs made the particles moved downward producing a thickened sludge at the bottom of the reactor. MLSS concentration was found to have a direct correlation to the sludge viscosity. Critical flux was increased after addition of coagulant resulting to the unchanged TMP observed throughout the operation of the system with coagulant. In contrast, the rapid increase in the system without coagulant could be attributed to the accumulation of SMP on the membrane surface forming a thin layer that contributed in membrane fouling.

Total fouling resistance on the membrane surface was decreased after addition of coagulant, which enhanced the permeation across the membrane. The slope of TMP increase and the skewness of the PSD graph are two good indices for evaluating the effectiveness of coagulant addition.

PSD analysis confirmed that coagulant addition increased the size of the particles preventing small particles to deposit in the membrane pores. The morphology of the fouled membrane was visualized using CLSM and SEM analysis. Images captured elucidated the fouling mechanism occurred in the membrane surface. It was revealed that cake resistance was the primary contributor in membrane fouling. However, foulants were easily removed after physical cleaning suggesting that irreversible fouling was the main reason for the flux decline in the system without coagulant. Moreover, decrease in the number of open pores of the membrane where observed with the application of the normal sludge, as compared with the coagulated sludge.

Accordingly, application of coagulant to sludge thickening by membrane filtration was proven to be effective for the enhancement of the thickening process.

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