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Membrane Fouling and Cleaning of A Thermophilic Anaerobic Membrane Reactor Treating Coffee Grounds and Sewage Sludge in a Long Term Continuous Operation with High Solid

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ABSTRACT

Anaerobic membrane reactor (AnMBR) has many advantages over continuously stirred tank reactor. Nevertheless, the suitability of a membrane reactor with high solid is a problem considering the potential membrane fouling. In this research, a thermophilic AnMBR was continuously operated for 238 days to investigate the membrane performance and to optimize the cleaning procedure by accumulating the total solid up to 75 g/L in the digester. The results showed that the membrane can be used for at least 150 days without cleaning. Flux decreased when changing the feedstock from coffee grounds to a mixture of coffee grounds and sewage sludge. The fine particles and high viscosity of sludge worsen the membrane permeability. Membrane fouling mainly originating from organic pollutants was proved through series cleaning: psychical, oxidation and citric acid. The effectiveness and the optimization of oxidation cleaning (NaClO of 0.1%, for 5 hours) were obtained by dead-end filtration tests.

Keywords: Anaerobic membrane reactor; coffee grounds; sewage sludge; membrane fouling; membrane cleaning

1. INTRODUCTION

Anaerobic digestion is a wieldy recognized technology for organic waste treatment (Digman et al., 2008). Conventional continuously stirred tank reactor (CSTR) requires large volume to accommodate the slow growing methanogenic bacteria and results in the long HRT (Khalid et al., 2011). In 1970s, anaerobic membrane bioreactor (AnMBR) was proposed to treat low-strength wastewaters (Liao et al., 2006). The decoupling of HRT and SRT was achieved by integrating membranes into anaerobic bioreactors. Kanai et al. (2010) reported that the volume of an AnMBR treating food wastewater was scaled down to 1/5 of a CSTR. However, membrane fouling severely limited the application of AnMBR technology (Liao et al., 2006). Over the past 20 years, the mechanism of membrane fouling was intensively investigated for treating wastewaters and sewage sludge (Agarwal et al., 2013; Dagnew et al., 2013; Field et al., 1995; Lin et al., 2009; Meng et al., 2009). The pollutants layer formed on a membrane surface in a submerged AnMBR treating sewage sludge mainly consisted of suspended

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solids (Wu et al., 2012). Jeison et al. (2006) reported that the biomass concentration in a membrane reactor treating wastewater linearly lowered the critical flux. Suspended solids together with extracellular polymeric substances would further worsen the membrane filtration performance (Judd and Judd, 2011). As proposed by Lin et al. (2009), the attachment of small particulates and bacterial clusters would colonize the membrane surface and provide enhanced conditions that allow for cake formation to develop. As a result, the suitability of a membrane reactor treating high solid waste streams is a problem, although a high solid system was preferred to produce more gas and discharge a smaller amount of digestate.

In addition, the characteristics of waste differ from one to another, such as the viscosity, particle size and organic composition. Mechanism of membrane fouling and strategy of membrane cleaning obtained from treating wastewater and sewage sludge cannot be directly adopted for treating other waste (Huang et al., 2001; Marrot et al., 2004).

Coffee grounds are typical food wastes. There are approximately six million tons coffee grounds produced each year in the world (ICO, 2008). Compared to sewage sludge, coffee grounds contain higher dry solids and lipids but its slurry has lower viscosity (Qiao et al., 2013). Methane fermentation of coffee grounds and sewage sludge with a high solid was feasible using an AnMBR, but the membrane performance had not been investigated (Qiao et al., 2013). So far, the application of AnMBR has not been established in a high-solid system. For investigating the mechanism of membrane fouling and cleaning, research for different kinds of waste are meaningful and valuable.

In this research, a thermophilic AnMBR was continuously operated for 238 days and successively feeding sole coffee grounds and mixture of coffee grounds and sewage sludge. Membrane fouling and cleaning were investigated towards the different substrates, wide range of solids concentrations and different cleaning procedure.

2. MATERIALS AND METHODS

2.1 Characteristics of substrates

Characteristics of the coffee grounds and sewage sludge are given in Table 1. Coffee grounds and sewage sludge were provided by an instant coffee manufacturer in Japan. Raw coffee grounds were brown color particles with TS of 34.7% and volatile solid (VS) of 34.4%. The TS and VS of the sewage sludge were 15.8% and 12.4%. Both the coffee grounds and the mixture of coffee grounds and sludge were shredded into slurry using a high-speed blender (LBC-15) at 18,500 rpm for 20 min before feeding into AnMBR. Particle size of the slurry of coffee grounds was 12.7 μm (d_{10}), 118.0 μm (d_{50}), and 397.0 μm (d_{90}), respectively. Particle size of the slurry of mixture of coffee grounds (85%, TS) and sewage sludge (15%, TS) was 9.43 μm (d_{10}), 94.1 μm (d_{50}), and 1081 μm (d_{90}).

2.2 AnMBR configuration

The set-up of the AnMBR system is illustrated in Fig. 1. A flat sheet membrane with a normal pore size of 0.2 μm and 0.116 m^2 surface area was immersed and centralized within the digester. The AnMBR had a total volume of 15 liters and working volume of 7 liters. The permeate was draw using a vacuum pump and partially omitted as effluent. Biogas were constantly circulated by a gas pump (Iwaki Air Pump APN-085LVX-1) and used to flush the membrane surface through a coarse tube diffuser located below the membrane sheet. The circulating gas flow rate was 5 L/min (43.1 L/min per m^2 membrane). The initial flux of the membrane was at the rate of 4.58

L/m^2 ·h according to a literature (Takahashi et al., 2012). Trans-membrane pressure (TMP) was measured by a pressure sensor (Keyence, AP-V85). The digester was maintained at 55-57˚C by a water bath. A wet gas meter (Shinagawa, 0.1 L/Rev) recorded the daily biogas volume. The digester was fed 4 times per day by an automatically controlled peristaltic pump. The substrate was stored in a 4˚C tank. The digester was inoculated with matured thermophilic sludge (TS, 3.1%) sampled from a full-scale digester treating food waste.

In this research, two membrane sheets were successively used. The initial membrane sheet was used only for 30 days due to accident pipe knot. The second membrane sheet was used from day 31 to 180 and then proceeded cleaning. The cleaned membrane was then put back into the digester until the end of the total experiment.

2.3 Procedure of anaerobic digestion

The AnMBR was continuously operated for 238 days. The total experiment was divided into two stages according to the feed stock. In stage I, the AnMBR was fed with coffee grounds. Suspended solids were entirely retained in the AnMBR to shorten the time for increasing solid concentration. In the same time, the influent TS stepwise increased from 25 g/L to 100 g/L. In stage II, the AnMBR was fed with a mixture of coffee grounds and waste activated sludge. The parameters of the anaerobic digestion experiment and the system performance were shown in Table 2.

Table 1 Characteristics of coffee ground and sludge

	Coffee grounds	**Sludge**
TS (%), n = 10	34.7 ± 0.29	15.8 ± 0.45
VS (%), n = 10	34.4 ± 0.46	12.4 ± 0.57
VS/TS (%)	99.1	79.5
COD/TS (g/g)	1.60	0.98
Carbohydrate (g/g-TS)	0.59	0.31
Protein (g/g-TS)	0.24	0.69
Lipid (g/g-TS)	0.24	0.02
C (%), n = 3	55.2 ± 3.71	34.0 ± 0.22
H (%), n = 3	7.07 ± 0.39	5.47 ± 0.07
O (%), n = 3	34.4 ± 4.54	25.7 ± 0.42
N (%), n = 3	2.33 ± 0.24	5.93 ± 0.09
S (%), n = 3	0.30 ± 0.19	0.70 ± 0.02

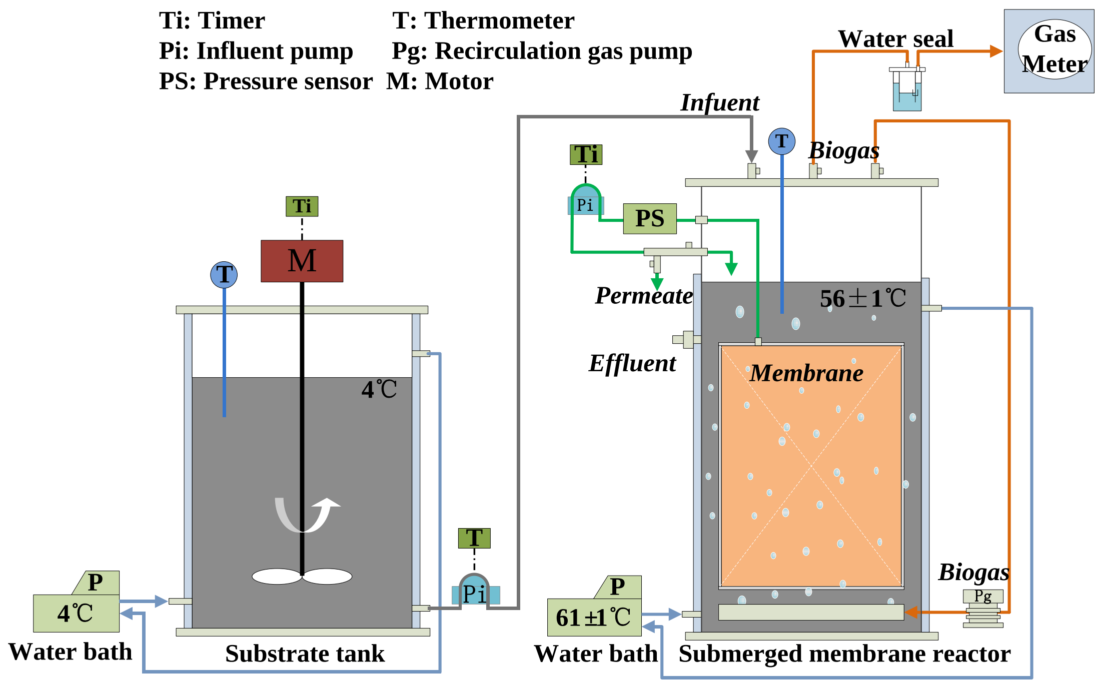


Figure 1 Configuration of AnMBR

Table 2 Operation parameters of anaerobic system

	Stage I (phase I~IV)				Stage II (phase V~VII)			
Phase	I (1-16)	II (17-32)	III (33-43)	IV (44-82)	V (83-114)	VI (115-184)	VII (185-216)	VIII (217-238)
HRT, days	20	30	30	30	30,40	70	30	20
SRT, days	/	/	/	/	/	100	30	20
Influent TS, g/L	25	50	75	100	100	100	150	150
Influent coffee, g/L	25	50	75	100	90	85	127.5	127.5
Influent sludge, g/L	0	0	0	0	10	15	22.5	22.5
SCOD in MBR, g/L	2.51	2.33	7.02	11.22	9.17	7.63	13.9	14.0
TS in MBR, g/L	15.1	17.5	26.0	31.3	48.9	52.8	65.4	75.4
SS in MBR, g/L	10.4	12.9	17.9	26.3	42.5	42.3	52.9	58.5
TVFA in MBR, g/L	0.39	0.26	0.38	4.43	3.33	2.93-2.21	1.90	3.41
Max. TMP, kPa	4.26	/	3.33	3.34	3.50	3.42	3.74	3.80
Min. TMP, kPa	2.81	/	3.07	3.10	3.07	3.07	3.32	3.45
Flux, $L/m^2 \cdot h$	4.92	/	4.92	5.25	4.96	2.63	/	3.21

Note: "nd": Not detected. "/": Data not available

2.4 Procedure of membrane cleaning

The used membrane was firstly flushed by tap water and then dipped in NaClO of 0.1% for 15 hours. Furthermore, the membrane was immersed in citric acid of 2% for 3 hours. A test of TMP, flux and permeability at 35 °C and 55°C was conducted. At the end of the total experiment, the membrane was taken out and was cut into 12 circles. Series dead-end filtration was carried out to optimize cleaning procedure. The filtrate flow rate was calculated by passing 150 mL water through the circles.

2.5 Analytical methods

The concentration of TS, SS and COD was determined according to the Japan Standard Testing Method for Wastewater (JSWA, 1997). The VFA was determined by an Agilent-6890 gas chromatograph equipped with a DB-WAXetr capillary column and a FID detector. The elemental compositions of C, H, O, N, and S were analyzed by an elemental analyzer (DKSH Elementar). Carbohydrate was measured using H_2SO_4/phenol oxidation and a calorimeter method. Protein was measured using the Folin/Ciocalteu method. Lipid was determined by marginal/chloroform extraction and weighting method. The micro-shape image of the membrane surface was observed by scanning electron microscope (SEM).

3. RESULTS AND DISCCUSION

3.1 Performance of the anaerobic digestion process

A successful process was not established in stage I due to the deficiency of nitrogen and trace metals (Qiao et al., 2013). Soluble COD in permeate gradually increased but did not significantly affect the membrane filtration. In stage II, the digester was fed with a mixture of coffee grounds and sewage sludge. The unstable problem thus was overcome as shown in Fig. 2a. The efficiency of the co-digestion process was investigated by shortening the HRT and stepwise increasing the feed stock TS concentration as listed in Table 2. Under the OLR of 23.6 kg-COD/m^3 d, HRT of 10 days and influent TS of 150 g/L, the COD removal efficiency was 44.5%. Under stable phases, the total VFA in digester did not greatly fluctuate. The variation of biogas production rate, pH, SCOD and solid concentration were provided in Fig. 2.

3.2 Variations of flux and TMP

There was no sludge layer attached on the first membrane surface. TS and SS in the AnMBR and permeate SCOD was 20.8, 17.3 and 2.5 g/L. The permeate flux stabled around 4.9 L/m^2 ·h as showed in Fig. 2b. As listed in Table 3, the filtration resistance of the membrane increased from -0.079 kPa/(L/m^2 ·h) (blank) to -0.225 kPa/(L/m^2 ·h) after 30 days. The used membrane was then treated by NaClO and citric acid. The filtration resistance of the membrane decreased to -0.022 kPa/(L/m^2 ·h) (NaClO of 0.1%, 15 hours) and -0.021 kPa/(L/m^2 ·h) (citric acid of 2%, 3 hours). The close filtration resistance with and without citric acid cleaning indicated that the inorganic pollutants did not form during the short term operation (30 days) and using coffee grounds as sole-substrate. From phase II to phase VI (day 33 to 184), the second membrane was put into the digester. From day 44 to day 82 (phase IV, stage I), the influent TS increased to 100 g/L. TS and SS in the digester was 40.2 and 29.5 g/L. The SCOD and VFA significantly increased to 15.3 and 10.4 g/L due to the unstable operation of the anaerobic system. As showed in Fig. 3a and 3b, flux and Max.TMP stabled at 5.25 L/m^2 ·h and -3.34 kPa. The increment of the solid, SCOD and VFA did not significantly and negatively affect the membrane filtration in this period.

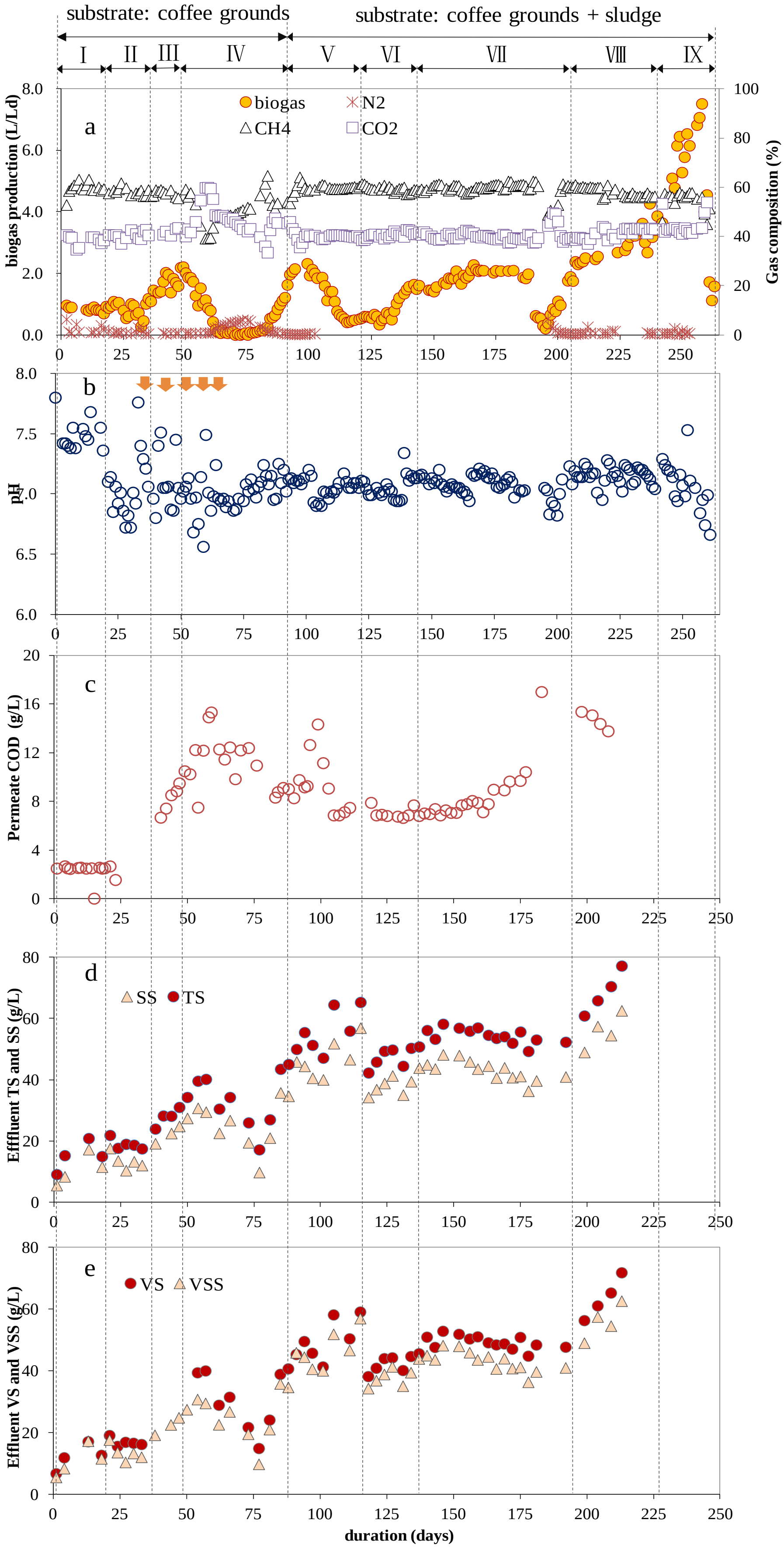


Figure 2 Variations of AnMBR system

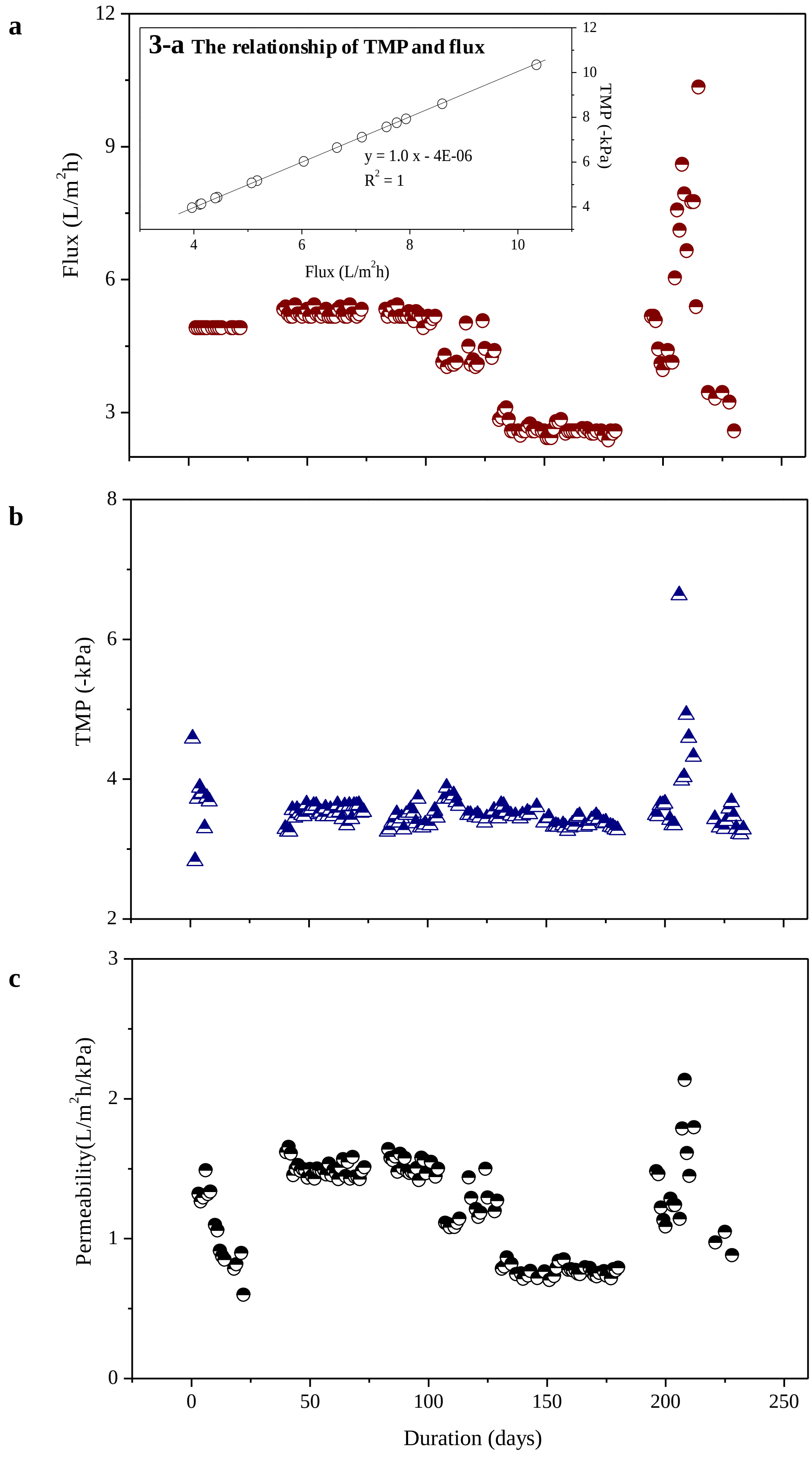


Figure 3 Variations of flux, TMP and permeability

Table 3 Filtration resistance of the first membrane

New membrane		**Membrane used for 30 days**		**NaClO cleaned membrane**		**Citric acid cleaned membrane**	
Flux (L/m^2 ·h)	TMP (-kPa)	Flux (L/m^2 ·h)	TMP (-kPa)	Flux (L/m^2 ·h)	TMP (-kPa)	Flux (L/m^2 ·h)	TMP (-kPa)
19.7	3.65	5.17	5.08	4.53	4.49	4.533	4.52
31.9	4.27	19.4	7.45	18.1	4.80	18.9	4.87
47.9	5.38	33.6	10.4	34.5	5.30	33.6	5.25
62.7	6.64	46.6	13.4	49.1	5.50	49.1	5.62
77.6	8.22	60.9	17.7	65.9	5.86	67.2	5.81
Resistance = 0.079 (n = 5, R^2 = 0.984)		Resistance = 0.225 (n = 5, R^2 = 0.988)		Resistance = 0.022 (n = 5, R^2 = 0.988)		Resistance = 0.021 (n = 5, R^2 = 0.977)	

The sewage sludge was mixed into coffee grounds at TS of 10% from 83 to 114 (phase V, stage II) and further to 15% from 115 to 238 (phase VI to VII, stage II). The particle size (d50) of coffee grounds slurry and the mixture slurry was 118μm and 94μm. The smaller particles could worsen the filtration resistance. The SCOD concentration gradually increased during these phases in stage II. A decrease of flux to 2.63 L/m^2 ·h was observed in phase V after changing the substrate. The permeability of the membrane decreased as the same trend with flux. Compared to stage I, the mixture substrate worsen the membrane performance by lowering the membrane flux with a stable TMP.

On day 184, the second membrane was taken out. The cleaned membrane was then put back into the digester. From day 217 to 238, TMP was intentionally adjusted between -2.63 and -10.42 kPa. The relationship of TMP and flux was plotted and embed in Fig. 3a. At the final phase, TS in digester reached 75 g/L. The high TS concentration did not significantly affect the membrane filtration.

Through the long term experiment, the feasibility of using an AnMBR with high solid was proved.

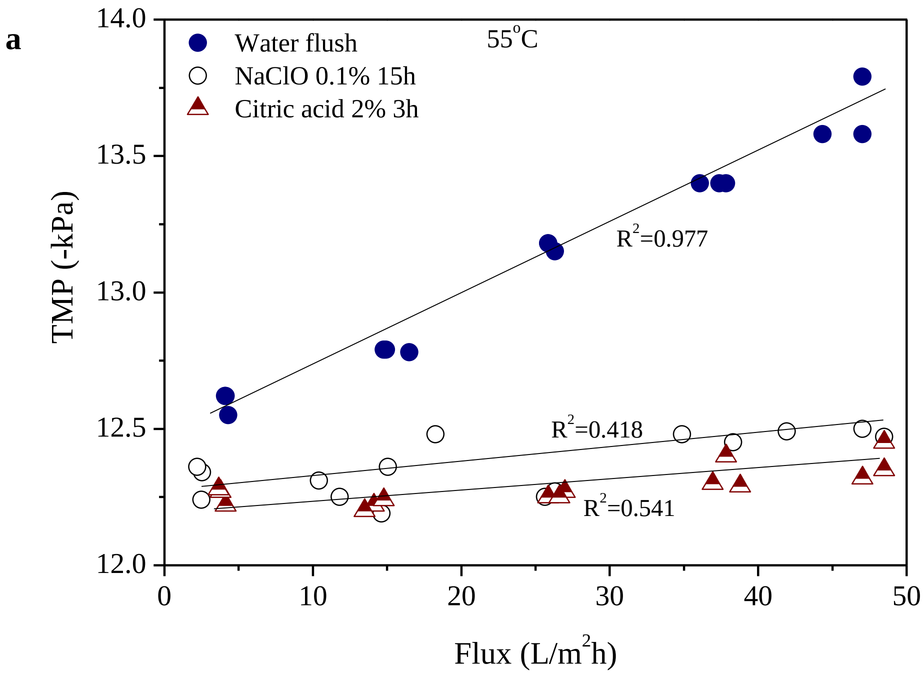
3.3 Membrane cleaning after long-term operation

Theoretically, membrane filtration resistance gradually increases with operation time (Tay et al., 2005). The second membrane was continuously used for 150 days and maintained a stable permeability. AnMBR generally operated under a mesophilic or thermophilic temperature. Fig. 4a and 4b represented the relationship of TMP and flux at 55˚C and 35˚C. Close filtration resistance was observed under the two temperatures. Resistance of the used membrane after water flushing, NaClO cleaned membrane and citric acid treated membrane were 2.57×10^{-2}, 0.39×10^{-2} and 0.31×10^{-2} -kPa/(L/m^2 ·h) as listed in Table 4. That indicated the significant inorganic pollutants did not form within the 150 days operation. The similar results of TMP and flux were observed under 35˚C. The resistance of the membrane was 5.10×10^{-2}, 1.26×10^{-2} and 1.25×10^{-2} -kPa/(L/m^2 ·h).

3.4 Optimization of NaClO cleaning

The cleaned second membrane was put back into the digester and was continuously used in the subsequent 75 days experiment. At the end of the experiment, the membrane was taken out. To evaluate the effectiveness of cleaning, the membrane sheet was cut into 12 separate circle species to proceed dead-end filtration. As shown in Fig. 5a, the upper and bottom of the membrane sheets were evaluated. Filtration resistance of the upper membrane was significantly higher than the bottom species. At the bottom of the membrane reactor, the biogas shearing force is stronger than the upper space that facilitates the removal of particular solids. Significant dark areas were ever observed in the side of the membrane near the bottom (Zhang et al., 2006). In the present experiment, fouling in upper membrane was severe than bottom ones.

The initial cleaning procedure was NaClO of 0.1% with 15 hours. The effects of NaClO concentration and cleaning time were evaluated and provided in Fig. 5b and 5c. Filtrate flux at NaClO concentration of 0.01%, 0.03%, 0.05% and 0.1% was 1.17, 1.04, 0.92 and 1.73 L/cm^2/min. That indicated the inconspicuous effects of the reagent concentration. The reduced cleaning reagent would reduce the costs of the process operation and the wastewater treatment. Furthermore, NaClO cleaning time effects were evaluated from 1 to 15 hours and showed in Fig. 5c. The permeate flux after 0.1% NaClO for 1, 3, 5, 10 and 15 hours was 0.45, 0.85, 1.27 1.37 and 1.73 L/cm^2/min. Membrane cleaning time by NaClO solution can be shortened to 5 hours. The relationship of permeate flux with cleaning time was described using a logarithmic function with an R^2 of 0.982 as showed in the embed figure in Fig. 5c. Permeate flux of different NaClO cleaning methods was listed in Table 5.



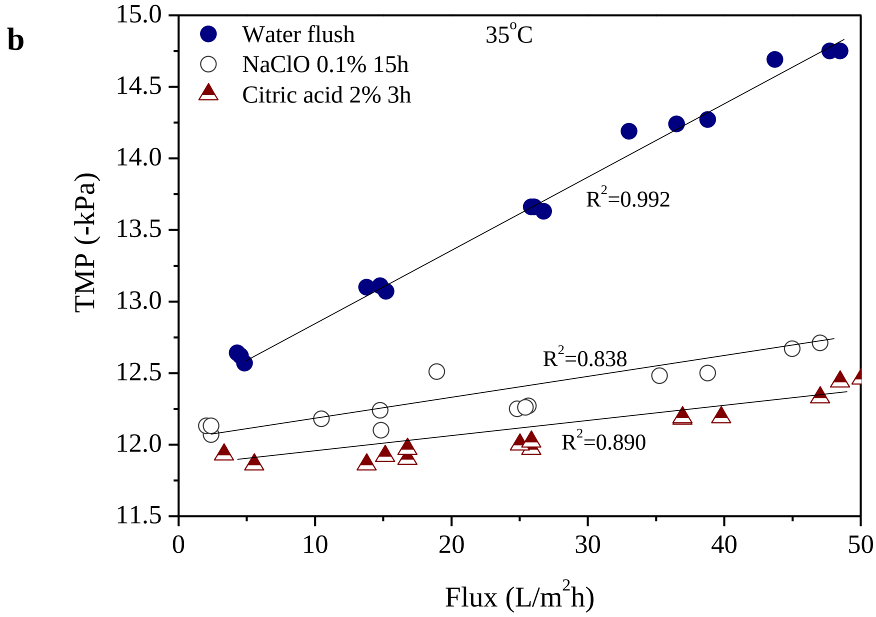


Figure 4 The relationship of TMP and flux (a: cleaning at 55˚C; b: cleaning at 35˚C)

Table 4 Filtration resistance of second membrane by cleaning

	Thermophilic cleaning -kPa/(L/m^2 ·h)	**Mesophilic cleaning** -kPa/(L/m^2 ·h)
Water flushing	2.57×10^{-2}	5.10×10^{-2}
NaClO cleaning	0.39×10^{-2}	1.26×10^{-2}
Citric acid cleaning	0.31×10^{-2}	1.25×10^{-2}

Table 5 Filtrate flow rate after NaClO cleaning

Conc. NaClO (%)	**Cleaning time (hours)**	**Filtrate flow rate (L/cm^2/min)**	**Conc. NaClO (%)**	**Cleaning time (hours)**	**Filtrate flow rate (L/cm^2/min)**
0.01	15	1.17	0.1	1	0.45
0.03	15	1.04	0.1	3	0.85
0.05	15	0.92	0.1	5	1.27
0.1	15	1.73	0.1	10	1.47
/	/	/	0.1	15	1.73

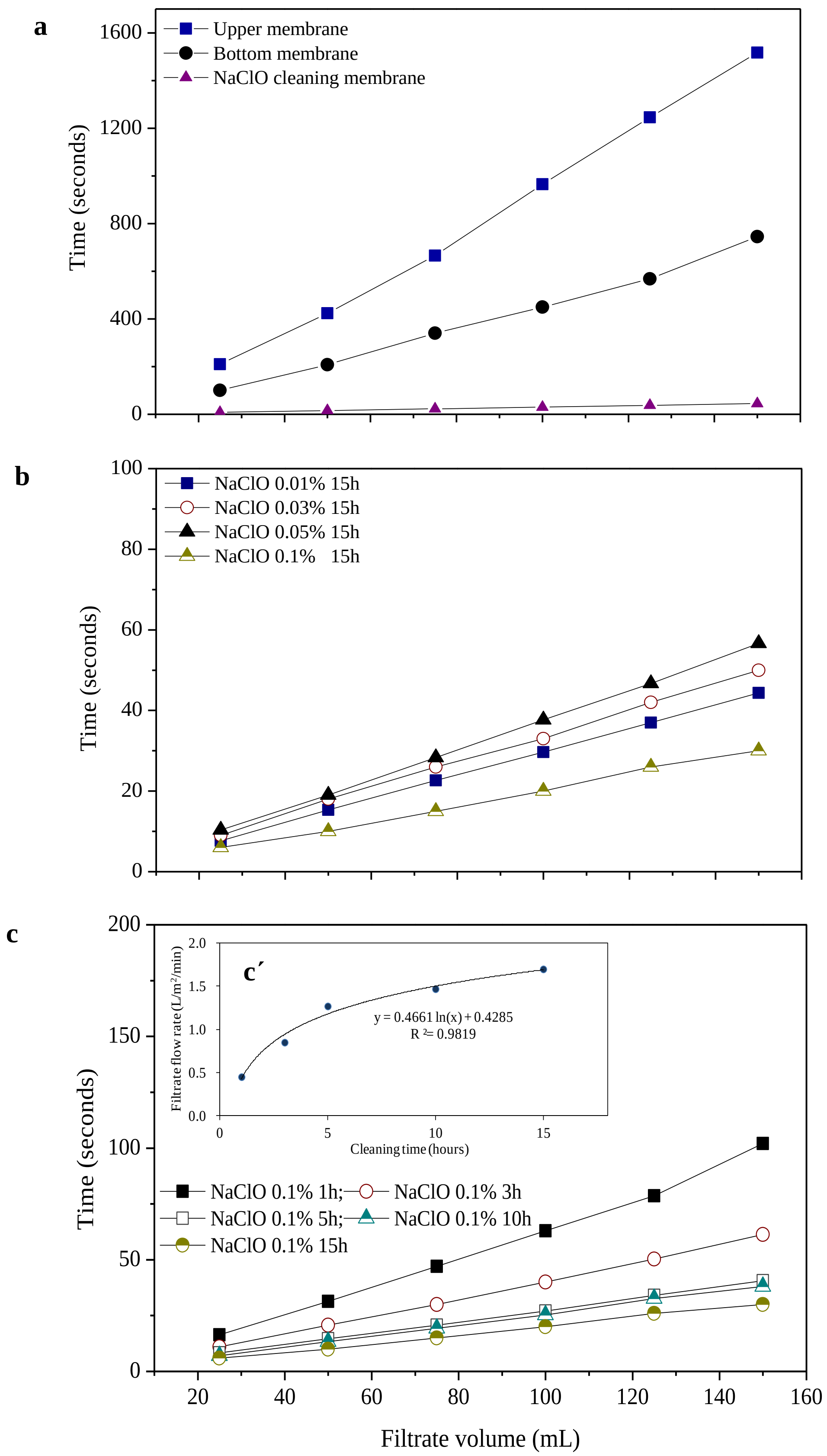


Figure 5 Effects of NaClO concentration and duration on membrane filtration

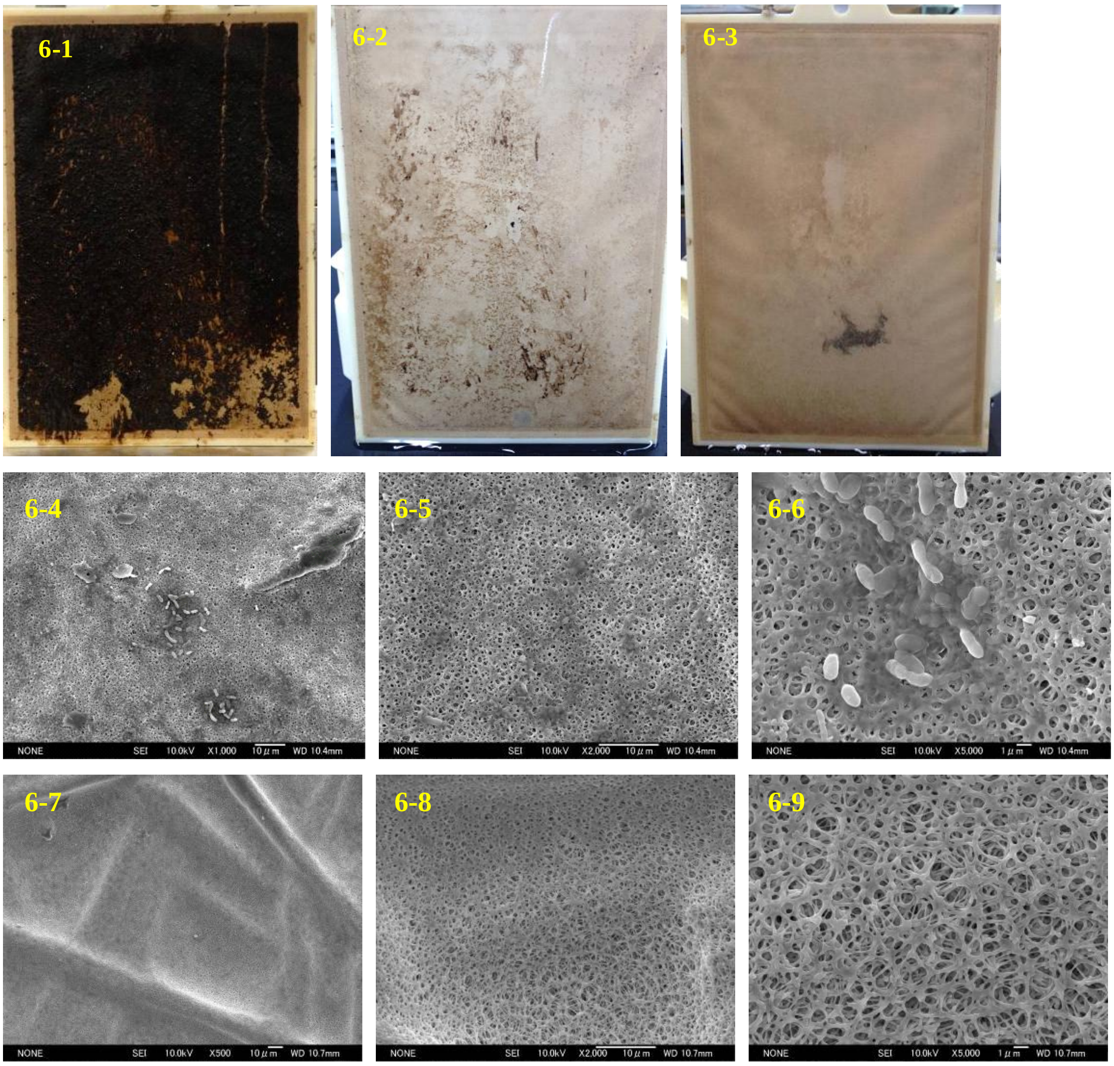


Figure 6 Pictures of 150 days used membrane (6-1: removed from reactor; 6-2: water flush membrane; 6-3: dipping in water 24 h); SEM of membrane before (6-4: ×1000; 6-5: ×2000; 6-6: ×5000) and after NaClO cleaning (6-7: ×1000; 6-8: ×2000; 6-9: ×2000)

3.5 SEM images of membrane with and without cleaning

The loosely attached sludge layer formed on the membrane surfaces as showed in Fig. 6-1. Most particles were removed by gently flushing the membrane using tap water. As reported by Brink et al. (2013), pollutants on the permeate side of the fouled membrane were observed by operating the membrane for more than 1 year. Both the bacteria and EPS covered the permeate side membrane. In this study, the permeate side membrane was clearer than the sludge side.

SEM images of the membrane surface were presented in Fig. 6. After water flushing, few microbes left on the membrane surface (Figs. 6-4, 6-5 and 6-6). The dark area indicates some organic pollutants deposition. The membrane became clearer after chemical cleaning (0.1% NaClO 15 h), as showed in Figs. 6-7, 6-8 and 6-9. Dark area and microbes were removed. On the other hand, as reported by Qiao et al. (2013), the soluble carbohydrate, lipid and protein were partly retained by the membrane.

In a submerged AnMBR system, high TS could present a burden on the hydrodynamic shear of gas scouring. In this research, biogas recycling ratio was 5 L/min, which is equivalent to 43.1 L/min ·m^2 of the membrane. Similar gas sparging strength was adopted by a submerged AnMBR treating alcohol stillage wastewater (Takahashi et al., 2012). The results of this study revealed that in an AnMBR system, imposed shear from biogas sparging effectively minimizes irreversible attachment, but may not totally eliminate it.

4. CONCLUSIONS

- The AnMBR could work well in a high solid system: 150 g/L TS in substrate and 75 g/L TS in digester.
- The membrane can be consecutively used for at least 150 days without cleaning.
- Mechanism of membrane fouling was identified by successively proceeding physical, oxidation and acid cleaning.
- The procedure of NaClO cleaning can be optimized by dead-end filtration tests.

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