Investigation on the performance of hybrid anaerobic membrane bioreactors for fouling control and biogas production in palm oil mill effluent treatment

Choon Aun Ng, Ling Yong Wong, Huey Yee Chai, Mohammed J. K. Bashir, Chii-Dong Ho, Humaira Nisar and Po Kim Lo

ABSTRACT

Three different sizes of powdered activated carbon (PAC) were added in hybrid anaerobic membrane bioreactors (AnMBRs) and their performance was compared with a conventional AnMBR without PAC in treating palm oil mill effluent. Their working volume was 1 L each. From the result, AnMBRs with PAC performed better than the AnMBR without PAC. It was also found that adding a relatively smaller size of PAC (approximately 100 μm) enhanced the chemical oxygen demand removal efficiency to 78.53 $\pm$ 0.66%, while the concentration of mixed liquor suspended solid and mixed liquor volatile suspended solid were 8,050 and 6,850 mg/L, respectively. The smaller size of PAC could also enhance the biofloc formation and biogas production. In addition, the smaller particle sizes of PAC incorporated into polyethersulfone membrane resulted in higher performance of membrane fouling control and produced better quality of effluent as compared to the membrane without the addition of PAC.

Key words | hybrid anaerobic membrane bioreactor (AnMBR), membrane fouling control, palm oil mill effluent (POME), polyethersulfone (PES) membrane, powdered activated carbon (PAC)

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INTRODUCTION

Malaysia is the second largest palm oil producer in the world contributing a large portion of the edible oil globally and increasing the economic growth of present oil market (Ding *et al.* 2016). The increasing popularity of palm oil in Malaysia is due to its wide applications in many areas such as food manufacturing and fuel for cars. As of February 2016, palm oil mill industries in Malaysia had produced 2,168,798 tonnes of palm oil and a total of 1,085,254 tonnes was exported in that month (Malaysian Palm Oil Board 2016). However, such a large amount of palm oil production has generated a relatively large amount of wastewater which is known as palm oil mill effluent (POME). Basically, POME consists of high concentrations of chemical oxygen demand (COD), biological oxygen demand, and suspended solids, which would lead to pollution of natural water resources if it is not treated properly before being discharged (Ahmed *et al.* 2015). Examples of some conventional methods designed to treat POME are adsorption, coagulation,

membrane technologies, and aerobic and anaerobic biodegradation (Tabassum *et al.* 2015).

The membrane bioreactor (MBR) is widely used in municipal and industrial wastewater treatment due to its combined processes of biological degradation and membrane filtration (Lin *et al.* 2013; Chaiprapat *et al.* 2016). The MBR is an alternative solution used to replace conventional activated sludge (CAS) treatment systems by including membrane filtration and utilising suspended growth of biomass to remove contaminants without using a clarifier (Mutanim *et al.* 2012; Woo *et al.* 2016). An MBR system provides various advantages such as (i) minimised excess sludge production, (ii) high rate of organic matter removal, (iii) reduced aeration cost for energy saving, (iv) smaller footprint, and (v) generation of superior effluent quality to achieve a more economical wastewater treatment system (Basset *et al.* 2016).

Membrane filtration applies both separation and purification processes in treating contaminated water, and is

able to retain unwanted materials on the filter by controlling permeation rate effectively (Hong *et al.* 2002). However, membrane fouling is one of the major problems plaguing anaerobic membrane bioreactors (AnMBRs). It was found that fouling is usually caused by accumulation of macromolecules on the surface of the membrane or blocking of membrane's pores completely, which subsequently prevents the membrane from functioning properly (Trzcinski & Stucky 2016). Therefore, in order to solve this problem, solutions such as (i) gas sparging (Hong *et al.* 2002), (ii) backwashing, (iii) membrane brushing, (iv) chemical cleaning, (v) membrane configuration modification (Mutanim *et al.* 2012), (vi) new membrane materials development (Woo *et al.* 2016), and (vii) hybrid MBR with porous and flexible suspended carriers (Cho & Fane 2002) were implemented to reduce the fouling rate of the membrane.

Activated carbon was used to act as a bio-fouling reducer to prolong membrane lifespan (Mutanim *et al.* 2012). Adding powdered activated carbon (PAC) into the MBR can enhance membrane fouling control by reducing fine organic matter through simultaneous processes of adsorption and biodegradation (Shao *et al.* 2014). Incorporation of PAC into the bioreactor allows better biofloc formation by converting PAC to become biological activated carbon (BAC) to promote attached growth biomass which can carry out a better biodegradation process (Ng *et al.* 2013). Li *et al.* (2005) revealed that the MBR with activated carbon had 32% higher critical-flux compared to the conventional MBR without activated carbon. Cost assessment reported by Yang *et al.* (2010) shows that the operating cost can be reduced by 25% for membrane cleaning and/or membrane replacement by PAC dosing, as it can significantly improve the fouling control.

In addition, instead of putting PAC into an AnMBR to improve its filtration process, by incorporating additives such as (i) crystalline silicotitanate and ferrihydrite (Weerasekara & Choo 2015), (ii) sulfated TiO_2 and SiO_2 nanotubes (Yuqing & Pingli 2015), and (iii) nanosilica (Lin *et al.* 2016) directly into the membrane could produce higher flux by having better membrane fouling control.

The MBR provides many benefits in treating wastewater compared to the CAS treatment system. However, membrane fouling is still an on-going problem. It would increase operation cost due to membrane cleaning, maintenance and reduction in filtration performance. It is agreed that the membrane can be physically or chemically cleaned for fouling control. However, its total resistance will be decreased and membrane service lifetime is reduced and it incurs high membrane replacement cost (Meng *et al.* 2009; Woo *et al.* 2016).

In this study, PAC with different sizes was added into several anaerobic bioreactors to investigate their performance based on contaminants removal and biogas production under controlled temperature. In addition, instead of putting the PAC into bioreactors, various particle sizes of PAC were incorporated directly into a polyethersulfone (PES) hybrid membrane to study their effects on membrane fouling control.

MATERIALS AND METHODS

AnMBRs set-up

Four 1 L anaerobic bioreactors (operating in batch system) were set up. First one anaerobic bioreactor (namely B_{blank}) was designed to treat POME without addition of PAC. The other three bioreactors (namely B_{coarse}, B_{medium}, and B_{fine}) were designed to be hybrid bioreactors with addition of different particle sizes of PAC with equal dosages. B_{coarse} is the anaerobic bioreactor with added PAC of relatively coarser size; B_{medium} is the anaerobic bioreactor with added PAC of medium size and B_{fine} is the anaerobic bioreactor with added PAC of relatively finer size. All anaerobic bioreactors were equipped with biogas probe, and supernatant and sludge collectors. A rubber pipe was connected between each bioreactor and a measuring cylinder was used to determine volume of biogas production. The bioreactors were placed in a water bath with the temperature set at 45 °C. The sludge retention time (SRT) and hydraulic retention time (HRT) of these anaerobic bioreactors were fixed at 30 days and 6 days respectively. Concentration of PAC added into the three bioreactors was 5 g/L. The supernatant from the anaerobic bioreactors was further treated using an external cross-flow membrane filtration system. The membrane filtration system for this AnMBR set-up is located externally from the bioreactor. New hybrid PAC-PES membranes were also fabricated. PAC of 5 wt% was used for preparation of new hybrid PAC-PES membranes. Their performance in terms of membrane fouling control was assessed using both the cross-flow and dead-end filtration systems. The supernatant from the bioreactor (B_{fine}) was diluted 10 times to increase its volume and used as feed for the different new PAC-PES membranes to test their membrane fouling control.

Materials

Cellulose acetate membrane was used to test the filtration performance of the different AnMBRs. PAC used in this

study was originally in granular form and supplied by GENE Chem Company. It was ground into desired sizes as per Figures 1 and 2 using a Panasonic blender from Bendosen Company. The anaerobic sludge seed and POME were obtained from a POME treatment plant owned by Tian Siang Group which is located in Perak, Malaysia. The dope used to cast new PAC-PES membranes was obtained by heating the mixture of PES and 1-methyl-2-pyrrolidone (NMP, 99%), which were supplied by Friendemann Schmidt Chemical, at the temperature in between 60 °C and 70 °C.

Dope preparation and fabrication of hybrid PES membrane

Mass ratio used for chemical dosages calculation of NMP and PES was 87:13. One hundred grams of dope was required for every membrane fabrication; 87 g of NMP and 13 g of PES were used to produce the dope without addition of PAC. Concentration percentage of PAC used was 5% based on weight of PES. The calculated formula for dope preparation using PAC with different particle sizes is tabulated in Table 1. Dry–wet phase technique was

used to fabricate membranes by using a semi-auto membrane casting machine.

Analytical parameters methods

Mixed liquor suspended solids (MLSS), mixed liquor volatile suspended solids (MLVSS), and COD were analyzed based on the procedures in *Standard Methods for the Examination of Water and Wastewater* (2005). Polysaccharide concentration was measured with the phenolsulfuric acid method (Dubois *et al.* 1956) and the concentration of protein was measured using Bradford reagent with bovine serum albumin as standard. pH measurement was done using a pH meter (Hanna HI 2550, USA). Particles size distribution measurement was carried out using a particle size analyzer (Malvern Mastersizer 2000, UK). Total biogas production the anaerobic bioreactors was collected and measured using the water displacement method. Content of biogas was measured using a RASI 700 biogas analyser. Performance of AnMBRs and hybrid PAC-PES membranes was measured using cross-flow and dead-end filtration systems respectively.
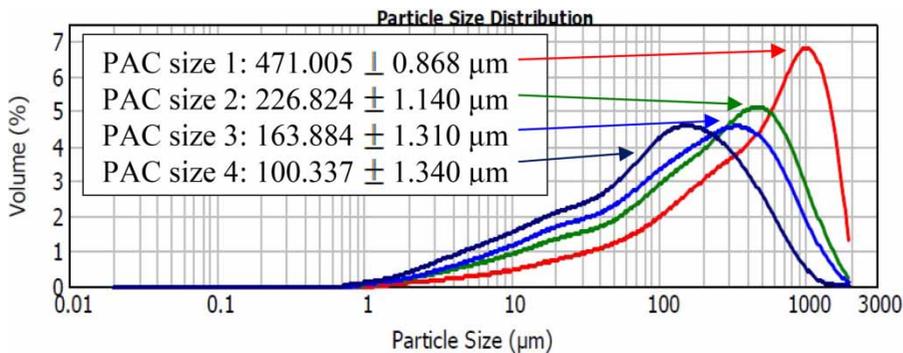

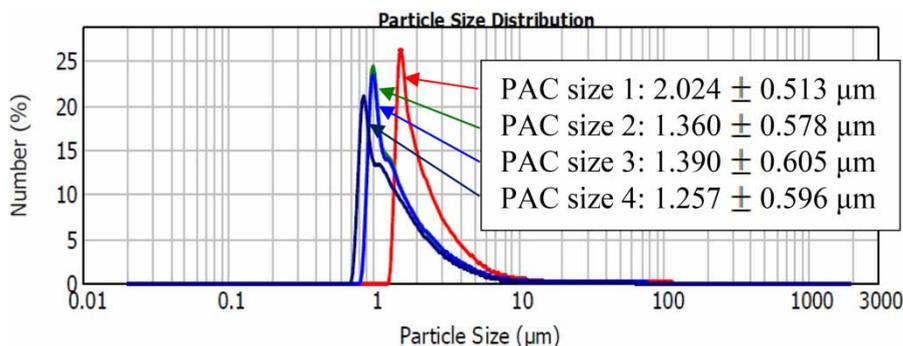**Figure 1** | Different PAC sizes (in volume) prepared from granular activated carbon.

Figure 2 | Different PAC sizes (in number) prepared from granular activated carbon.

Table 1 | Formula for dope preparation

Samples	NMP (g)	PES (g)	PAC (g)	Particle sizes of PAC used (µm)
M_{blank}	87.00	13.000	–	–
M_{coarse}	87.00	12.350	0.650	471.005
M_{medium}	87.00	12.350	0.650	226.824
M_{fine}	87.00	12.350	0.650	100.337

RESULTS AND DISCUSSION

Performance of different AnMBRs without PAC and with different sizes of PAC, in terms of COD removal, production of protein and polysaccharide, MLSS and MLVSS concentrations, biogas production and membrane fouling control, was investigated. In addition, performance of new membranes, namely hybrid PAC-PES membranes incorporated with different sizes of PAC, in terms of membrane fouling control was also investigated.

Performance of anaerobic bioreactors without PAC and with different sizes of PAC

The performance of four different anaerobic bioreactors was investigated and is discussed. Their overall POME treatment performance is shown in Table 2. pH value of each bioreactor was controlled and maintained at approximately 7.8 throughout the study.

The results showed that bioreactor without addition of PAC (B_{blank}) had the lowest treating efficiency compared

to the rest of bioreactors added with PAC of different particle sizes. It was found that the bioreactors added with relatively smaller particle size of PAC had better removal efficiency compared to the bioreactors added with larger PAC sizes. By comparison of COD removal efficiencies, B_{fine} had the best performance in POME treatment as PAC acts as a colony to allow microbes to attach on it and they are synergistic and mutual in bioreactors (Satyawali & Balakrishnan 2009). In anaerobic bioreactors, PAC was developed to become BAC and carried out a better biodegradation process to decompose natural organic matter (NOM) in POME (Ng et al. 2013). The presence of PAC in smaller sizes had contributed larger surface area that allowed more microbes and organic matter to adsorb on it, which resulted in an enriched environment for microbes' metabolism. Thus, increase in surface area of PAC combined with its famous adsorption characteristics can relatively improve the COD removal efficiency and treating performance.

Assessment of MLSS and MLVSS in various anaerobic bioreactors without PAC and with different sizes of PAC

Ratio of MLVSS to MLSS is used to determine sludge activities as it will significantly affect the treating performance of bioreactors (Fan et al. 2015). In this study, biomass concentrations of four different anaerobic bioreactors were measured and analysed as shown in Table 3.

Throughout the experiment, the ratios of MLVSS to MLSS in the bioreactors were in stable condition and

Table 2 | Performance of anaerobic bioreactors without PAC and with different sizes of PAC

Parameter	B_{blank}	B_{coarse}	B_{medium}	B_{fine}
Temperature, °C	45	45	45	45
SRT, days	30	30	30	30
HRT, days	6	6	6	6
Raw POME pH	4.16 ± 0.01	4.16 ± 0.01	4.16 ± 0.01	4.16 ± 0.01
Supernatant pH	7.79 ± 0.01	7.81 ± 0.02	7.73 ± 0.07	7.78 ± 0.04
PAC dosage, g/L	NA	5	5	5
Raw PAC size, D_{50} (volume), µm	NA	471.005 ± 0.868	226.824 ± 1.140	163.884 ± 1.310
Raw PAC size, D_{50} (number), µm	NA	2.024 ± 0.513	1.360 ± 0.578	1.390 ± 0.605
Raw POME COD, mg/L	$95,729 \pm 5,115$	$95,729 \pm 5,115$	$95,729 \pm 5,115$	$95,729 \pm 5,115$
Organic loading rate (feed in COD, mg/(L D))	$7,658 \pm 408$	$7,658 \pm 408$	$7,658 \pm 408$	$7,658 \pm 408$
COD of supernatant, mg/L	$2,695 \pm 337$	$2,075 \pm 305$	$1,937 \pm 322$	$1,647 \pm 175$
Protein of supernatant, mg/L	$2,407 \pm 230$	$1,677 \pm 124$	$1,194 \pm 122$	887 ± 247
Polysaccharide of supernatant, mg/L	71 ± 1.827	66 ± 0.435	57 ± 1.481	52 ± 1.073
Removal efficiency of COD, %	64.90 ± 1.46	72.99 ± 1.47	74.80 ± 1.66	78.53 ± 0.66

Table 3 | Comparison of MLSS and MLVSS in anaerobic bioreactors without PAC and with different sizes of PAC

Parameter	B_{blank}	B_{coarse}	B_{medium}	B_{fine}
MLSS, mg/L	$10,900 \pm 1,200$	$13,800 \pm 100$	$17,800 \pm 3,100$	$18,950 \pm 3,850$
MLVSS, mg/L	$9,250 \pm 1,250$	$12,700 \pm 700$	$15,450 \pm 2,450$	$16,100 \pm 3,200$
MLVSS/MLSS	0.85 ± 0.01	0.92 ± 0.03	0.87 ± 0.01	0.85 ± 0.002

maintained at approximately 0.85, which is an allowable condition for bacteria survival (Fan et al. 2015). The condition shows that there was enough food for growth of microbes, which were able to degrade NOM effectively. Based on Table 3, B_{fine} had the highest biomass whereas B_{blank} had the lowest biomass concentrations. This indicated that addition of PAC with smaller particle sizes could benefit biomass activities and growth rate in bioreactors. Higher biomass rate can help to remove NOM in POME, subsequently increasing contaminant removal efficiencies.

Assessment of microbial floc size in various anaerobic bioreactors without PAC and with different sizes of PAC

Sludge floc is one of the parameters which would affect membrane fouling control. The size of the microbial floc can be increased by adding PAC into the bioreactors (Lee & Kim 2013). Transformation of PAC to BAC in bioreactors can help to enhance membrane filtration performance by reducing and preventing NOM reaching the membrane surface (Ng et al. 2013). Microbial floc sizes from different bioreactors were determined in terms of volume and number and are shown in Figures 3 and 4 respectively. B_{blank} was found to have the smallest particle sizes in biofloc among the reactors. From Figures 3 and 4, it is proven that a larger biofloc can be formed by adding PAC into bioreactors. B_{fine} was observed to be the largest biofloc size due to the

greatest surface area that allowed the microbe population to gather together.

Membrane fouling control of various anaerobic membrane bioreactors without PAC and with different sizes of PAC

Cross-flow filtration test was carried out to determine overall efficiencies of AnMBRs by using a cellulose acetate membrane. The supernatant of each anaerobic bioreactor was subjected to a filtration process and the quality of its permeate was analysed as per Table 4. It could be observed that B_{blank} had relatively poorer COD removal efficiency than the others. The same trend was obtained by comparing removal efficiencies of protein and polysaccharide, which indicated that a higher microbe population could result in better treating efficiency. The removal efficiency was calculated based on the initial concentration of COD, protein, and polysaccharide before treatment as presented in Table 2.

Extracellular polymeric substances (EPS) such as protein and polysaccharide are major metabolic products of bacteria that lead to membrane fouling (Shao et al. 2014). As shown in Figure 5, AnMBRs with addition of PAC performed better compared to AnMBR without PAC. The membrane used to treat the supernatant from B_{blank} was fouled easily in a short time and B_{fine} performed the best in membrane fouling control. Membrane fouling was
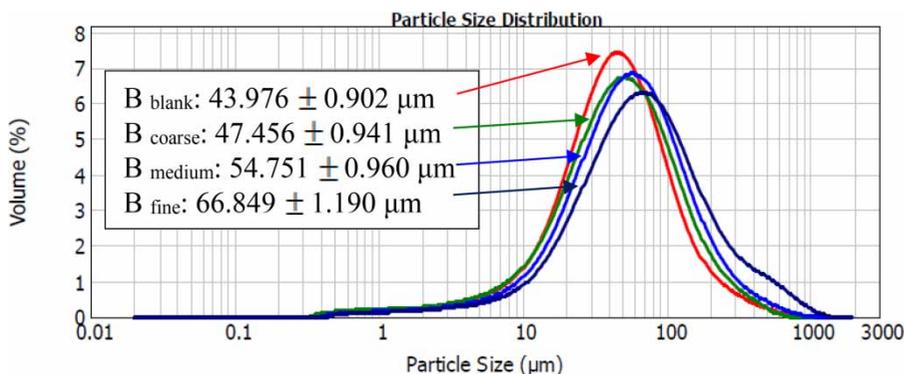
Figure 3 | Microbial floc size distribution of different anaerobic bioreactors in terms of volume.
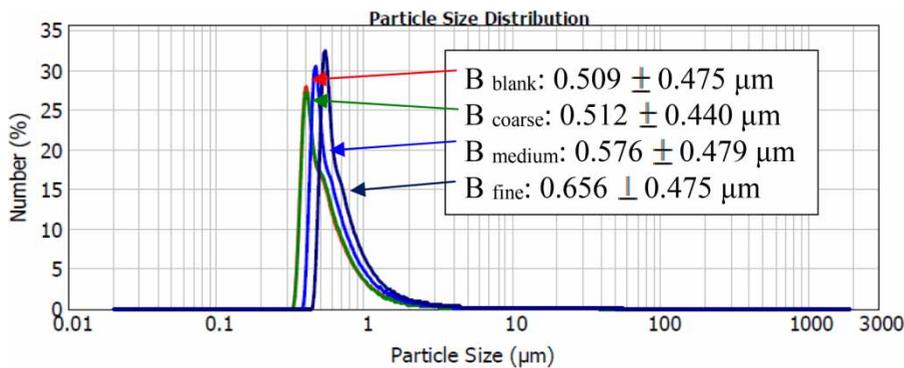
Figure 4 | Microbial floc size distribution of different anaerobic bioreactors in terms of number.

Table 4 | Performance of various anaerobic bioreactors without PAC and with different PAC particle sizes towards membrane fouling control

| Parameter	B_{blank}	B_{coarse}	B_{medium}	B_{fine}
COD of permeate, mg/L	$1{,}718 \pm 82$	$1{,}354 \pm 27$	$1{,}233 \pm 55$	948 ± 56
Protein of permeate, mg/L	$1{,}229 \pm 41$	654 ± 13	203 ± 20	108 ± 6
Polysaccharide of permeate, mg/L	19.98 ± 1.42	13.34 ± 0.27	11.43 ± 1.39	10.59 ± 1.59
Removal efficiency of COD, %	77.56 ± 0.07	82.32 ± 0.34	83.90 ± 0.08	87.62 ± 0.04
Removal efficiency of protein, %	48.76 ± 1.85	60.92 ± 1.23	83.03 ± 0.05	87.28 ± 1.74
Removal efficiency of polysaccharide, %	72.03 ± 0.74	79.91 ± 0.16	79.86 ± 1.12	80.36 ± 1.29
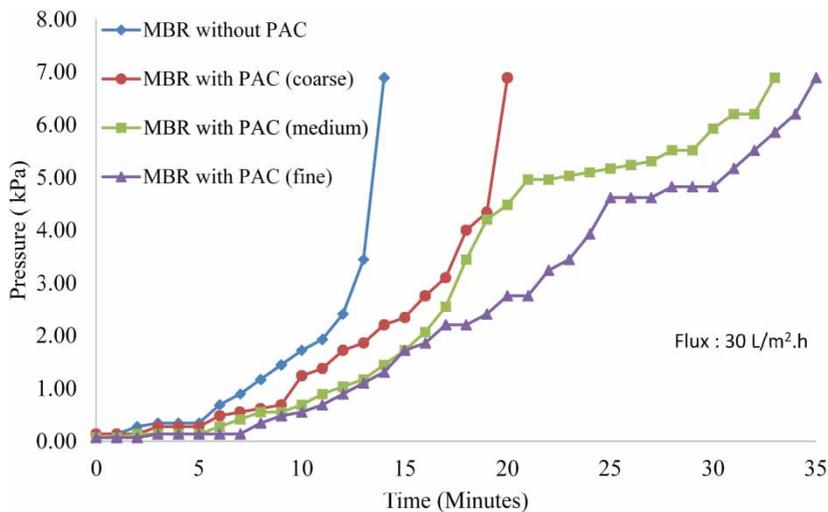
Figure 5 | Comparison of different anaerobic bioreactors towards membrane fouling control.

caused by untreated EPS and NOM from bioreactors reaching the membrane surface, blocking membrane pores and promoting cake formation, which resulted in increased transmembrane pressure (TMP) in a short period. For the best performer, B_{fine}, reduction in EPS and fine pollutants in its bioreactor had successfully reduced the amount of them reaching the membrane surface and relatively extended the time of TMP increase.

Effects of biogas production in various anaerobic bioreactors without PAC and with different sizes of PAC

Bacteria have ability to convert organic matter into biogas during the anaerobic biodegradation process (Chen et al. 2016). In this study, biogas production from the various anaerobic bioreactors was measured and is shown in Figure 6 and Table 5. There was an increasing trend in that B_{blank} had the lowest biogas production and B_{fine} produced the highest volume of biogas.

BAC which was formed from PAC in the anaerobic bioreactors could help to enhance the anaerobic digestion process and promote methane-forming bacteria in NOM biodegradation and subsequently produced biogas effectively (Chen et al. 2016). Generated methane (CH_4) in biogas is one of the renewable energy sources that can be harvested and converted into electricity, which is a way of saving operation cost and is an environmentally friendly concept (Yan et al. 2016). Based on Table 5, B_{fine} had the best methane yield, which may be due to biomass richness in the anaerobic bioreactors.

Performance of new hybrid PAC-PES membrane incorporated with different sizes of PAC towards membrane fouling control

It was found that adding PAC into the MBR can help to improve filtration performance (Satyawali & Balakrishnan 2009; Ng et al. 2013). As a result, different new hybrid PAC-PES membranes were fabricated by incorporating different sizes of PAC directly into PES membranes to test their filtration performance in treating supernatant from an anaerobic bioreactor.

In this study, B_{fine} was identified as having the best quality of supernatant out of the four anaerobic bioreactors. Thus, the various fabricated new hybrid PAC-PES membranes were tested for their fouling control by using the supernatant produced from B_{fine}. Their filtration performance results were analysed and are shown in Figure 7. M_{blank}, which is PES membrane without addition of PAC, had the lowest performance as it fouled relatively easily compared to others. The contaminants could easily block the pores of M_{blank} and caused the rise of TMP at a relatively
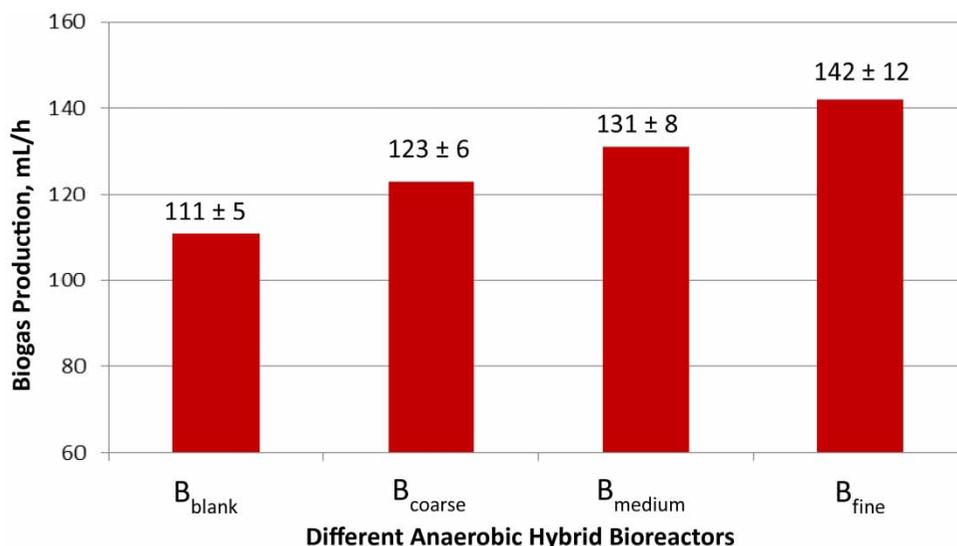

Figure 6 | Biogas production in various anaerobic bioreactors without PAC and with different sizes of PAC.

Table 5 | Methane gas concentrations in the biogas produced from various anaerobic bioreactors without PAC and with different PAC particle sizes

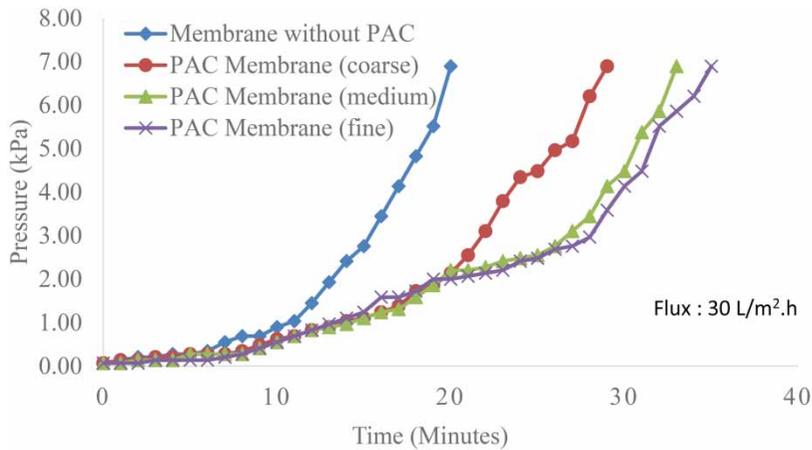| Biogas Content	B_{blank}	B_{coarse}	B_{medium}	B_{fine}
Biogas production, mL/h	111 ± 5	123 ± 6	131 ± 8	142 ± 12
CH_4, %	56.79 ± 0.33	57.52 ± 0.11	57.22 ± 0.71	57.59 ± 0.15
CH_4, mL/h	62.85 ± 1.60	70.56 ± 1.99	75.15 ± 2.51	81.77 ± 4.01
CH_4 mL/g COD	310.94 ± 66.01	308.35 ± 55.69	320.48 ± 57.83	318.63 ± 29.72

Figure 7 | Comparison of various PES hybrid membrane incorporated with different sizes of PAC towards membrane fouling control.

faster rate. Instead, hybrid PAC-PES membrane added with the smallest size of PAC, M_{fine}, had the highest fouling resistance performance followed by M_{medium} and M_{coarse} at the operating flux of 30 L/(m² h).

PAC with larger surface area has relatively higher porosity which allows contaminants to be adsorbed onto it before the contaminants block the pores on the membrane surface (Satyawali & Balakrishnan 2009). The smallest size of PAC incorporated into the PES membrane is able to reduce TMP by forming permeate filter cake with the highest porosity. This phenomenon could help to enhance

membrane fouling resistance and maintain service lifetime of the membrane.

In addition to the cross-flow filtration (fixed flux) test, another dead-end filtration test (fixed pressure) was carried out to verify the performance of the PES membranes, without PAC and with different PAC particle sizes, in membrane fouling control using the supernatant from B_{fine}. The performance of the various PES membranes is shown in Tables 6 and 7.

Based on Table 6, it was observed that PES membrane incorporated with PAC had better flux rate compared to the PES membrane without PAC. Among the PES membranes incorporated with PAC, M_{fine} had the highest flux rate compared to M_{medium} and M_{coarse}, as smaller sizes of PAC with higher porosity could allow more permeate to pass through the membrane and reach higher productivity. Results in Table 7 show that PAC incorporated into PES hybrid membrane had effectively retained contaminants on the membrane surface and converted into BAC. BAC has advantages over PAC as it is equipped with simultaneous adsorption and biodegradation processes to produce relatively higher quality effluent.

Table 6 | Flux performance of various PES membranes without PAC and with different sizes of PAC

Types of membrane	Sizes of PAC (μm) incorporated	Flux production, L/(m² h)
Blank (without PAC), M_{blank}	–	16.81 ± 1.25
PAC membrane, M_{coarse}	471.005 ± 0.868	23.68 ± 0.62
PAC membrane, M_{medium}	226.824 ± 1.14	28.27 ± 0.62
PAC membrane, M_{fine}	100.337 ± 1.34	32.09 ± 1.25

Table 7 | Overall treating performance of PES membranes without PAC and with different sizes of PAC

Parameter	M_{blank}	M_{coarse}	M_{medium}	M_{fine}
COD of permeate, mg/L	$1,550 \pm 35$	$1,222 \pm 32$	$1,002 \pm 31$	723 ± 11
Protein of permeate, mg/L	707 ± 27	366 ± 15	162 ± 13	91 ± 3
Polysaccharide of permeate, mg/L	19.06 ± 0.33	12.33 ± 0.12	9.70 ± 0.09	7.89 ± 0.02
Removal efficiency of COD, %	79.73 ± 0.36	84.03 ± 0.25	86.91 ± 0.17	90.55 ± 0.21
Removal efficiency of protein, %	70.53 ± 0.97	78.12 ± 0.42	86.38 ± 0.20	89.24 ± 1.59
Removal efficiency of polysaccharide, %	73.30 ± 0.13	81.42 ± 0.03	82.88 ± 0.17	84.96 ± 0.16

CONCLUSION

Addition of PAC in an AnMBR was proven to be beneficial in terms of POME treatment and membrane fouling control. It was noticed that PAC with relatively smaller size (PAC size of $163.884 \pm 1.31\,\mu m$) helped to increase microbial growth rate and biofloc formation. Transformation of PAC into BAC in anaerobic bioreactors obviously helped in reducing NOM amount by carrying out adsorption and biodegradation processes simultaneously. This could help to reduce membrane fouling rate and prolong the membrane lifespan. Application of PAC in smaller sizes into bioreactors also resulted in higher volume of biogas production as more methanogen bacteria functioned together to convert NOM into biogas at a faster rate during the biodegradation process. Also, PES membrane incorporated with smaller sizes of PAC (PAC size of $100.337 \pm 1.34\,\mu m$) resulted in higher flux, and a cleaner permeate can be obtained. This is due to the fact that incorporated PAC provided greater porosity on the membrane surface. It also helps to adsorb fine pollutants, preventing them from blocking the pores on the membrane surface, to form higher porosity permeate filter cake during the filtration process.

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