UNIVERSITI PUTRA MALAYSIA

PERFORMANCE OF TWO-STAGE DYNAMIC ANAEROBIC MEMBRANE BIOREACTOR FOR TREATING HIGH STRENGTH FOOD PROCESSING WASTEWATER

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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Food processing wastewater (FPW) contains a high level of oil and grease, requiring extensive treatment. The submerged dynamic membrane in anaerobic digestion (AD) treatment offers cheap and complete biological and physical separation of solid-liquid. Dynamic membrane (DM) developed onto cheap support material can replace the expensive conventional membranes. Although promising DM utilization in AD treatment has been reported, scarce research focused on the DM formation to explain its performance and fouling control. Thus, this study's objectives were to evaluate the submerged dynamic membrane two-stage anaerobic bioreactor (DAnMBR) performance in treating FPW and assess the DM characteristics and development mechanism.

The batch biodegradability test assays were used to determine the best performance of substrate (FPW) to inoculum (anaerobic digester sludge) ratio (S/I) at 1.0, 1.5 and 2.0. A two-stage anaerobic digester (named acidogenic and methanogenic reactors) with two submerged 20 µm woven filter cloth as the supporting material (DAnMBR) in the second tank was used in this study. Successful start-up using synthetic wastewater and then acclimatization by adding FPW in steps up to 100% FPW was achieved when 90% of chemical oxygen demand (COD) were removed. Treatment using support material commenced thereafter. The treatment performance utilizing APHA methods was evaluated at different hydraulic retention time (HRT) of 0.4-1.3 days and organic loading rates (OLR) of 3.5, 5.0, 6.5, and 7.0 g COD/L.d.

Best OLR was used to assess the DM formation until fouling occurred, and the cake layer samples were taken for characterization periodically. S/I 1.0 ratio performed best with COD, biochemical oxygen demand, total solids (TS) and volatile solids (VS) removals of 96.9, 96.6, 75.8, and 65.2%, respectively. The bioreactor presented a good

performance at OLR 5.0 g COD/L.d with removals of 97.5% COD and 99% total suspended solids at HRT of 0.5 day. The methane gas production yield achieved a maximum of 0.40 L methane/g COD added at OLR 3.5 and 5.0 g COD/L.d with the same HRT 0.5 day on both OLRs. The average permeate flux in these studies was around 60 L/m^2 h. Fouling occurred at 35 days during the DM development and characterization study with a final flux of 2.5 L/m^2.hr and transmembrane pressure of 0.7 bar. The cake layer thickness increased slightly from day 14 to 28 but sharply at the fouled stage, agreeing with the treatment performance. Protein to polysaccharide ratio (PN/PS) of the extracellular polymeric substances (EPS) increased significantly compared to soluble microbial product PN/PS ratio; thus, it is the main contributor to the membrane fouling. High-through-put 454 pyrosequencing of total DNA revealed that *Proteobacteria*, *Bacteroidetes* and *Methanosaeta* were abundant in bacterial and archaeal communities, which played an important role in the DAnMBR system. In conclusion, following the results obtained in this study, DM technology achieved a stable and high-quality permeate. Thus, DAnMBRs can be used as a reliable and satisfactory treatment technology to treat high strength wastewaters.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PRESTASI DUA PERINGKAT BIOREAKTOR ANAEROBIK DINAMIK MEMBRAN UNTUK MERAWAT AIR SISA INDUSTRI PEMBUATAN MAKANAN

Oleh

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Air buangan pemprosesan makanan (FPW) mengandungi tahap tinggi minyak dan gris yang memerlukan rawatan yang intensif. Membran dinamik terendam dalam rawatan pencernaan anaerobik (AD) menawarkan pemisahan biologi dan fizikal pepejal-cecair yang murah dan lengkap. Membran dinamik (DM) yang dikembangkan ke bahan sokongan yang murah dapat menggantikan membran konvensional yang mahal. Walaupun menjanjikan, penggunaan DM dalam perawatan AD, penyelidikan yang jarang difokuskan pada pembentukan DM untuk menjelaskan prestasi dan pengotorannya. Oleh itu, objektif kajian ini adalah untuk menilai prestasi bioreaktor anaerobik dua peringkat (DAnMBR) membran dinamik tenggelam dalam merawat FPW dan menilai ciri DM dan mekanisme pengembangan.

Ujian biodegradabiliti kumpulan digunakan untuk menentukan prestasi terbaik nisbah substrat (FPW) hingga inokulum (enapcemar pencernaan anaerob) (S/I) pada 1.0, 1.5 dan 2.0. Pencernaan anaerobik dua peringkat (dinamakan reaktor asidogenik dan metanogenik) dengan dua kain penapis tenunan 20 µm yang tenggelam sebagai bahan sokongan (DAnMBR) dalam tangki kedua digunakan dalam kajian ini. Permulaan yang berjaya menggunakan air sisa sintetik dan kemudian aklimatisasi dengan menambahkan FPW dalam langkah hingga 90% FPW dicapai apabila 90% permintaan oksigen kimia (COD) dikeluarkan. Rawatan menggunakan bahan sokongan dimulakan selepas itu. Prestasi rawatan yang menggunakan kaedah APHA dinilai pada masa sewa hidraulik (HRT) yang berbeza dari 0.4-1.3 hari dan kadar pemuatan organik (OLR) 3.5, 5.0, 6.5, dan 7.0 g COD/L.hari.

OLR terbaik digunakan untuk menilai pembentukan DM sehingga pencemaran berlaku dan sampel lapisan kek diambil untuk pencirian secara berkala. Nisbah S/I 1.0 menunjukkan prestasi terbaik dengan COD, permintaan oksigen biokimia, penyingkiran pepejal total (TS) dan pepejal mudah alih (VS) masing-masing 96.9, 96.6, 75.8, dan 65.2%. Bioreaktor menunjukkan prestasi yang baik pada OLR 5.0 g COD/L.hari dengan penyingkiran 97.5% COD dan 99% pepejal terampai. Hasil pengeluaran gas metana mencapai maksimum 0.40 L metana/g.COD yang ditambahkan pada OLR 3.5 dan 5.0 g COD / L hari. Fluks meresap purata dalam kajian ini adalah sekitar 60 L / m^2.jam. Fouling berlaku pada 35 hari semasa kajian pengembangan dan pencirian DM dengan fluks akhir 2.5 L / m^2.jam dan tekanan transmembran 0.7 bar. Ketebalan lapisan kek meningkat sedikit dari hari ke-14 hingga ke-28, tetapi secara tajam pada tahap kekotoran setuju dengan prestasi rawatan. Nisbah protein ke polisakarida (PN/PS) bahan polimer ekstraselular meningkat dengan ketara berbanding dengan nisbah PN / PS produk mikrob larut; oleh itu, ia adalah penyumbang utama pembuangan kotoran. *Pyrosequencing* 454 dari jumlah DNA yang tinggi menunjukkan bahawa *Proteobacteria*, *Bacteroidetes* dan *Methanosaeta* banyak terdapat dalam komuniti bakteria dan archaeal, yang memainkan peranan penting dalam sistem DAnMBR. Kesimpulannya, berikutan hasil yang diperoleh dalam kajian ini, teknologi DM mencapai permeate yang stabil dan berkualiti tinggi. Oleh itu, DAnMBR dapat digunakan sebagai teknologi rawatan yang boleh dipercayai dan memuaskan untuk rawatan air buangan berkekuatan tinggi.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) were adhered to.

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AD	Anaerobic digestion
AnMBR	Anaerobic membrane bioreactor
BOD	Biological oxygen demand
COD	Chemical oxygen demand
DM	Dynamic membrane
DMBR	Dynamic aerobic membrane bioreactor
DO	Dissolved oxygen
EM	External membrane
EPS	Extracellular polymeric substances
MBR	Membrane bioreactor
MF	Microfiltration
MLSS	Mixed liquor suspended solids
NF	Nanofiltration
PAC	Powdered activated carbon
SBR	Sequencing batch reactor
SEM	Scanning electron microscopy
SFDM	Self-forming dynamic membrane
SM	Submerged membrane
SMP	Soluble microbial product
SND	Simultaneous nitrification-denitrification
SRT	Sludge retention time
TMP	Trans-membrane pressure
TN	Total nitrogen
TOC	Total organic carbon
UASB	Up-flow anaerobic sludge beds
UF	Ultrafiltration
WAS	Waste activated sludge

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Malaysia is one of the rapidly developing countries in Asia, and its industrialization program has dramatically increased the amount of wastewater need to dispose of and suitable treatments are required. However, the ability to receive water to accept the increasing inorganic and organic loads remains the same, resulting in a rapid deterioration of surface water quality. The emerging problems have prompted concerned government agencies to introduce and implement more stringent legislation. Industries are searching for the least cost options to reduce their pollution load and the latest wastewater treatment technology (Chernicharo, 2007). One or more effective treatment processes would be required for effluent consistency, land availability, construction, operating costs, and operational simplicity.

Malaysia has a population of 28.3 million based on the Report of Census 2010 by the Department of Statistics (Department of Statistics, 2010). The estimated volume of wastewater generated by municipal and industrial sectors is 2.97 billion cubic meters per year. The large volume of domestic and industrial wastewater produced in Malaysia must be treated to prevent pollution to the environment and protect public health by safeguarding water supplies. Aerobic treatment systems were the primary biological treatment methods of wastewater until the 1970s. The aerobic process needs oxygen to degrade pollutants before discharging them into the water stream (Seow et al., 2016).

However, the environmental debate and rise in energy prices in the 1980s have dramatically changed this scenario. Reusing and recycling waste have created considerable interest, and methane gas as an energy-produced anaerobic process has become a highly potential alternative (Gerardi, 2003). The anaerobic system is currently used worldwide for a broad spectrum of industrial wastewater treatment since the development of high rate anaerobic processes wastewater such as food processing effluents (Tedjani et al., 2012), textile wastewater (Yurtsever et al., 2020), landfill leachate (Jasni et al., 2020), paper mill wastewater (Chelliapan et al., 2012) and high strength lipid wastewater (Ramos et al., 2014).

Conservation of energy in industrial processes has become an important issue, and anaerobic processes have quickly become an appropriate alternative. As a result, several reactor designs were developed to handle low, medium, and high-strength wastewater. In addition to being high-energy intensive, the introduction of aerobic processes as treatment options requires high capital investments alone. On the opposite, a low investment technology in the anaerobic system requires no aeration systems, reduced sludge disposal facilities, and the key benefit of methane gas recovery (Alkarimiah et al., 2011). The most cost-effective approach for organically contaminated industrial waste streams is through anaerobic wastewater treatment. The development of high-quality systems, in which hydraulic retention times are uncoupled to solid retention times, in particular, contributed to a global acceptance of anaerobic treatment. However, anaerobic biomass growth during start-up makes the process control fragile as the system recovery is prolonged when exposed to adverse environmental conditions.

Nevertheless, with the expansion of research, high-rate anaerobic treatment systems have been developed to retain high amounts of biomass, even at low hydraulic retention times. Accordingly, high solid retention time is maintained at high hydraulic loads to the anaerobic system. The result is reactors with a lower volume than the conventional anaerobic digesters with a high degree of sludge stabilization (Chernicharo, 2007; Yee et al., 2019).

Reactor configuration is vital in controlling the effluent quality. Staging the phases in AD will improve effluent quality and may be essential to produce anaerobic effluent that meets discharge quality standards. Furthermore, a staged reactor can accommodate toxic sludge more efficiently by slowly passing through the reactor system. The staged reactor will result in a much-abbreviated exposure of the biomass to the undiluted toxicants. Physical separation of the two anaerobic digestion phases, namely acidogenesis and methanogenesis, aims to satisfy the optimum environmental conditions for each type of microbial population in two separate reactors (Demirel et al., 2002). This so-called two-stage process supports greater archaeal diversity's growth and performance than a single-stage process (Srisowmeya et al., 2019). A significant limitation of anaerobic digestion of solid wastes in a single-stage system is during the first phase (acidogenesis phase) where rapid production of volatile fatty acids (VFA) occurs. Such acids will reduce the pH, which stresses and inhibits methanogenic bacteria’s activity. Conditions favourable to the growth of acid-forming bacteria, such as short hydraulic retention time (HRT) and low pH, are inhibitory to the methanogens (Maspolim et al., 2015). A two-stage reactor can optimize both species of bacteria's condition in the acidogenic and methanogenic groups (Ibrahim et al., 2013). During the acidogenic phase, the pH is usually maintained between 5.5 and 6.0 and HRT of less than five days. Anaerobic membrane reactors (AnMBRs) were developed based on aerobic MBRs concept within the last decade, with either external membrane or submerged in the reactor. Thus, coupling MBR with anaerobic digestion obviates the need for a sedimentation/clarifier tank. The advantages of MBR are complete biomass retention, low sludge production, increase treatment capacity, and lower operational cost (Mike & Shannon, 2014). In recent years, considerable attention has focused on developing a novel anaerobic process in which a membrane separation is incorporated in place of a settling system. To date, several investigators have studied two-phase anaerobic membrane processes for the treatment of wastewaters such as cheese whey (Saddoud et al., 2007), sugarcane vinasse (Mota et al., 2013), sewage sludge (Joo et al., 2016), synthetic molasses-based wastewater (Wijekoon et al., 2011), vinasse wastewater (Silva et al., 2020), synthetic wastewater (Chaikasem et al., 2014), municipal solid waste (Trzcinski & Stuckey, 2011), biodegradable municipal solid waste (Walker et al., 2009b), piggery wastewater (Lee et al., 2001), and starch wastewater (Yu et al., 2016). Based on their results, it can be inferred that in a two-stage anaerobic membrane bioreactor (2-AnMBR) where the methanogenic reactor is coupled with a membrane module or the membrane is installed via side-stream configuration, the occurrence of acidogenesis in a preceding reactor could prevent acidogenic biomass growth in the methanogenic reactor, thereby enhancing sludge properties and filtration performance (Mota et al., 2013).

Generally, cake layer formation on the membrane surface regulates aerobic and anaerobic MBRs membrane resistance (termed fouling). However, high membrane capital costs such as microfiltration (MF) and ultra-filtration (UF), high energy requirement and fouling problem become significant MBR technology problems. Its replacement with a low-cost macroporous material, starting in the middle of the 1990s, seems a more promising technology. Correspondingly, applying cheaper materials such as macroporous material including mesh, non-woven fabric and filter cloth as the filter instead of the expensive MF and UF is more promising in dynamic membrane (DM) technology (Ersahin et al., 2016b). The cake layer itself function as the filtration instead of just contributing to fouling. In most of the dynamic membrane bioreactors (DMBRs) experiments, if the transmembrane pressure (TMP) or the water head reached a certain level, the DM layer could easily be scoured off with air. DM formation involves multiple physicochemical and microbiological processes, such as the formation of gel layers and cake. The structure of DM in MBRs have not yet been fully understood (Liu et al., 2009). Limited information on the cake layer's characteristics on the supporting layers, such as fabric or mesh is available (Ersahin et al., 2012).

Since 2008, the extensive use of anaerobic dynamic membrane bioreactors (DAnMBRs) is still in the early stage. The key issues include the development of DAnMBRs for the treatment of various wastewaters, process efficiency and system optimization, and sludge properties (Alibardi et al., 2014, 2016; An et al., 2009; Quek et al., 2017; Xie et al., 2014; Zhang et al., 2010, 2011). DM formation and mechanism, DM layer characterization, and the production and collection of biogas have received little attention (Hu et al., 2018; Saleem et al., 2016).

1.2 Problem statement

There are scarcity of anaerobic treatment of high strength real wastewater. Industrial wastewater has poor biodegradability and a high level of toxicity, possing difficulties for AnMBR, such as long start-up time and low biogas output (Dereli et al., 2012). There have been few reports based on FOG pilot-scale or bench scale co-digestion in continuous fluid digesters, which is likely to be due to the variety of operational challenges of FOG co-digestion, such as process inhibition, substrate transport restrictions, digester floatation and foaming, and massive problem with digestion pipeline system blocking and clogging (Li et al., 2013).MBRs have wide-scale industrial application but the majority of DM studies were bench-scale applications treating synthetic or municipal streams (low strength wastewater) (Alibardi et al., 2016; Hu et al., 2016; Saleem et al., 2016; Xiong et al., 2016). Although high-strength wastewater (such as industrial wastewater or landfill leachate) can be effectively treated with high biogas production using DAnMBR, potential obstacles have been mostly induced by toxics and other non-biodegradable compounds (Berkessa et al., 2020; Xie et al., 2014).

Due to the option of performing the phases in distinct settings, two-stage AD appears to be particularly well suited for substrates with a high insoluble COD content, such as fruit and vegetable wastes (Colussi et al., 2014) . This method of operation improves the stability, efficiency, and conversion rate. Hydrolysis can be accelerated by inoculating the reactor with microorganisms that produce substrate-specific hydrolytic enzymes (Joo et al., 2016; Maspolim et al., 2015). Despite these advantages, two-stage AD processes are not widely used in commercial applications, most likely because more comprehensive studies are required to justify larger plant investment. Thus, the present research aims at assessing the performance of DAnMBR with phase separation treating FPW at mesophilic temperature (35°C).

The majority of prior research has focussed on the effect of sludge characteristics and operating parameters on membrane fouling. There is scarcity of information on the analysis of cake layer formation. A detailed examination of the cake layer formed on the membrane surface will aid in determining the optimal operating parameters for the DAnMBR. Hence, controlling membrane fouling can be facilitated by knowledge of the cake layer characteristics. One of the most critical challenges in DAnMBR is to keeping DM's thickness within an acceptable range (efficient DM layer) to ensure that the treatment is effective (Ersahin, 2015). The analysis is a prerequisite for achieving stable high permeate quality and preventing an unforeseen increase in TMP. This study aimed to characterize the DM layer and elucidate its role in the biological removal performance of particulate and soluble organic matter. This approach would help to understand the cake layer formation that enables a stable operation in DAnMBR. In addition, most of the studies about cake layer formation (a mechanism) were conducted in conventional AnMBRs rather than DAnMBRs. The formation phases of filtration cake on the micro-membrane surface had been suggested by (Jiang et al., 2003) based on the increasing characteristics of membrane resistance in MBR. However, due to the variable nature of dynamic membranes, the formation mechanism and structure of dynamic membranes in DMBR are still unknown (Liu et al., 2009).

This research’s findings are expected to fill the knowledge gap and provide new insights into staging the anaerobic digestion process into two separate phases for more efficient AD treatment. DM utilization as a cheap method for post-treatment of the AD treatment of high FOG wastewater may overcome the commercialization problem of utilizing membrane in AnMBR processes. It is possible to treat wastewater post anaerobic digestion by replacing the costly microfiltration (MF) or ultrafiltration (UF) membranes, using cheap materials via the developed cake layer on the polypropylene woven filter cloth.

1.3 Research objectives

This study aims to assess the potential of two-stage submerged anaerobic dynamic membrane bioreactor (DAnMBR) in treating food processing wastewater (FPW). Specific objectives to achieve the main aim are listed as follows:

(i) To investigate anaerobic biodegradability of food processing wastewater in a two-stage anaerobic bioreactor based on BMP analysis
(ii) To evaluate the efficiency of the submerged dynamic membrane anaerobic bioreactor (DAnMBR) treating food processing wastewater at different HRT and OLR.
(iii) To assess the characteristics of the dynamic membrane formed on the support material and elucidate its development mechanism.

1.4 Scope of study and limitation

This study mainly focuses on the development of a two-stage DAnMBR system and its performance. During the start-up period, synthetic wastewater containing yeast and meat extract was used, and real food processing wastewater was used to investigate the reactor system's full performance. The fed process was done in stages. A polypropylene woven filter cloth with a pore size of 20 µm was used as the supporting materials for DM formation. This pore size was selected based on preliminary study done on different pore sizes of 20, 40 and 60 µm to treat food processing wastewater using DMBR. Based on the results (duration of 4 days), the 20 µm pore size monofilament filter cloth was found most suitable for the cake layer development. The results also demonstrate that the biofilm composed of the cake layer of the DM on smaller pore size significantly concurs with the high treatment efficiency compared with the larger pore size (Mahat et al., 2020). The maximum COD removal achieved was more than 80% for the smaller pore size filter cloth (20 µm), but only 70% for larger pore size (60 µm) filter cloth. The smallest pore size (20 µm) of the supporting material was selected for the cake layer development throughout the study because it showed the best cake layer development and performance (turbidity and ammonia-nitrogen removals of 99.0 and 98.0%, respectively). During the treatment phase, organic loading rate intervals were increased in a 1.5 step increment (3.5 – 7.5 g COD/L.d). The applied increment is not too high or too low in order to get the optimum results for DAnMBR performance. For the 1.5 step increments, it is indicated the addition of 1.5 in each new OLR from 3.5 gCOD/L.d to 5.0 gCOD/L.d and lastly for 7.5 gCOD/L.d. During the last application of the highest OLR (6.0-7.5), we want to test whether the system can survive on the maximum feed COD of 9,000 mg/L (possibility of inhibition) in different HRT. The OLR resulted in the best performance was selected to assess the mechanism of the cake layer development. In this study, the highest OLR applied was 9.0 g COD/L.d but the severe pH fluctuation resulted in reduced performance due to the inhabitation of biomass and accumulation of higher volatile fatty acids (organic shock loading of higher concentration substrate).

Throughout the treatment, although oil and grease (O&G) removal were possible with the DM, an operational problem was observed in the acidogenic reactor (AR) tank. Accumulation of solids (scum) caused pipe clogging required manual cleaning periodically. Although the methanogenic reactor (MR) tank was not obstructed due to the less frequent floating solid material presence, it was also subjected to the same cleaning process, albeit less frequent compared to the AR tank. Hence, it is possible the treatment performance for all parameters (COD, BOD, methane yield, ammoniacal nitrogen etc.) do not entirely reflect the actual result.

1.5 Significant of research

The cheap materials for the dynamic supporting membrane potentially provide improved flux rates cost-effectively at low transmembrane (TMP) pressure (Ersahin et al., 2012). For a two-phase system food processing effluent, Tedjani et al. (2012) observed a COD removal efficiency of 65% when no membranes were used, compared to 80-90% when a membrane was used in the DAnMBR (2nd tank). A number of studies have shown that co-digestion with municipal or food industry waste fats, oil, and grains used as a co-substrate will boost production of methane as FOG requires lower mass loading per unit of methane production and has shown more significant biogas production potential than other organic waste (Li et al., 2013).

1.6 Thesis layout

This thesis consists of five chapters. The introduction in Chapter 1 first gives the background of the study and the problem statement and ends by stating the research's objectives and scope. Chapter 2 covers the literature review with the discussion focusing on supporting material, dynamic membrane application in wastewater treatment to remove organic matters and solids. Chapter 3 covers the preparation of substrates, membranes, types of analytical equipment used, and analytical methods by the Standard Methods for the Examination of Water and Wastewater. Chapter 4 presents the results and discussion on the start-up development and performance of DAnMBR through different HRT and OLR. Additionally, the mechanism of DAnMBR is discussed within this chapter with the in-depth characterization of the DM layer. Finally, Chapter 5 wraps up the thesis with a conclusion and recommendations for future work.

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