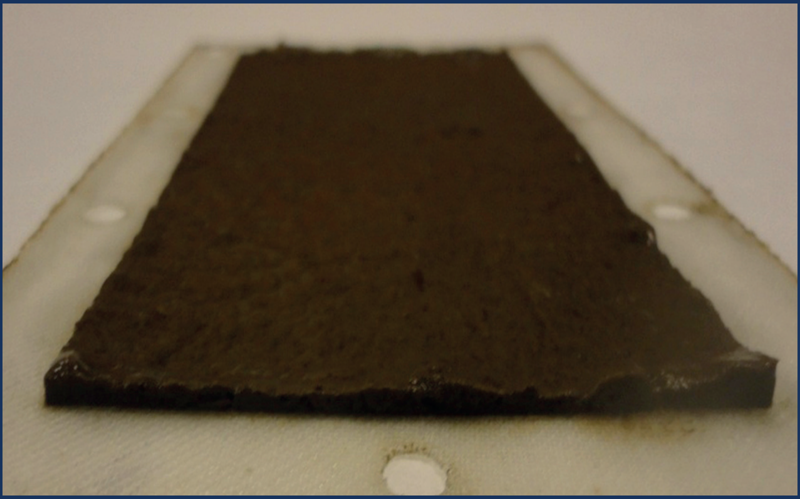
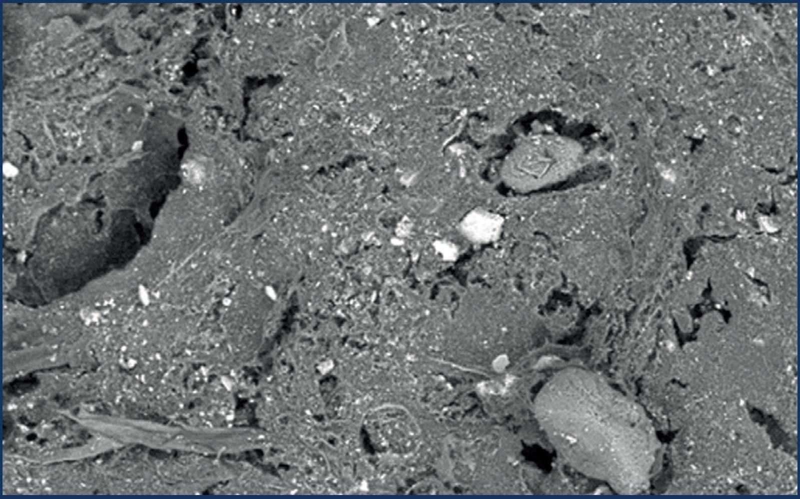
APPLICATION OF DYNAMIC MEMBRANES IN ANAEROBIC MEMBRANE BIOREACTOR SYSTEMS





Mustafa Evren ERŞAHİN

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APPLICATION OF DYNAMIC MEMBRANES IN ANAEROBIC MEMBRANE BIOREACTOR SYSTEMS

Proefschrift

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To my beloved father Mehmet Erşahin and sister Aynur Erşahin,

Çok değerli babam Mehmet Erşahin ve ablam Aynur Erşahin'e,

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Mustafa Evren Erşahin
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SUMMARY

Anaerobic membrane bioreactors (AnMBRs) physically ensure biomass retention by the application of a membrane filtration process. With growing application experiences from aerobic membrane bioreactors (MBRs), the combination of membrane and anaerobic processes has received much attention and become more attractive and feasible, due to advantages provided by the combination with regard to developments for energy-efficient wastewater treatment.

The major drawbacks of MBR technology are related with membrane costs, especially for the full-scale applications, fouling and low flux. Dynamic membrane (DM) technology may be a promising approach to resolve the drawbacks encountered in MBR processes. One of the most important potential benefits of DMs is that the membrane itself may be no longer necessary, because solids rejection is accomplished by the secondary membrane layer that can be formed and re-formed as a self-forming DM in situ.

Different kinds of materials such as mesh, woven or nonwoven fabric instead of microfiltration and ultrafiltration membranes can be used as the support layer for creating DM. In this way, the replacement of the membrane by a low cost filter material is possible. By decreasing membrane cost and generating energy, dynamic AnMBRs (AnDMBRs) would be attractive for waste(water) treatment.

The main aim of this study was to investigate the applicability of DM technology for the treatment of concentrated wastewaters in AnMBRs. Moreover, this thesis provides additional information and understanding of DM technology, including assessment of DM formation and filtration characteristics under different conditions. Submerged and external membrane module configurations were used in order to determine the effect of the configuration on removal efficiency and DM filterability. Synthetic concentrated wastewater with an average COD concentration of 20 g/L was used as the substrate. Determination of an optimal support material and investigations about its structure were achieved by testing various types of support materials including monofilament, multifilament and staple yarn types. Besides, different operating conditions were tested at low fluxes under mesophilic conditions to determine the optimal operation conditions enabling the optimal removal efficiency and permeate quality. Moreover, cost estimation in terms of support material acquisition was also presented.

The results show that support material properties were critical for the formation of an effective dynamic membrane (cake) layer over the filter surface. The critical fluxes obtained with the staple and monofilament filter cloths were higher than those obtained with multifilament material. The results indicate that staple filter cloth was more suitable for depth

filtration, whereas mono-monofilament filter was more suitable for surface (cake) filtration. Thus, mono-monofilament filter was considered more appropriate for DM technology.

The results presented in this thesis show that the DM filtration concept can turn one of the most important disadvantages of MBRs, membrane fouling, into an advantage. Polypropylene mono-monofilament filter cloth was used to form a dynamic membrane (cake) layer and to provide filtration by this self-forming layer as an alternative to microfiltration or ultrafiltration membranes. The AnDMBR achieved over 99% organic matter removal and particulate matter retention. Moreover, over 60% soluble COD removal and over 50% VFA removal were obtained by the DM layer. Considering the results of this research, it was shown that a stable operation with AnDMBRs could be possible for a long period.

Sludge retention time (SRT) was found an important factor in AnDMBRs that had a significant effect on soluble microbial products (SMP) and extracellular polymeric substances (EPS) production, protein/carbohydrate ratio, particle size of the sludge, DM layer formation and bulk sludge filterability. Bound EPS is mainly composed of cell surface materials, including proteins, polysaccharides, lipids, nucleic acids and humic acids. EPS keeps the sludge flocs together on the membrane surface by surrounding them. EPS had a significant positive effect on particle flocculation and thus, particle size distribution in the bulk sludge. Prolonged SRT resulted in lower EPS concentrations in the bulk sludge compared to short SRTs.

A combination of backwashing and biogas sparging enabled the control of DM layer thickness, which is of great importance to obtain a stable operation and high quality permeate. A combined effect of biomass activity and physical retention capacity through the cake layer might be responsible for the removal of organic matter and retention of particulate matter by the DM layer. Pyrosequencing analyses showed that diversity and richness of the microbial communities including bacteria and archaea in the DM layer were high and microbial population composition in the DM layer was different compared to the bulk sludge in the AnDMBR. Following the DM layer morphological analyses results, the DM layer was formed by both organic and inorganic materials, such as sludge particles, SMP, EPS, Ca, N, P, and Mg precipitates. Moreover, a partial gel layer formation under the cake layer was detected. Accumulation of SMP and bound EPS in the DM layer in high amounts led the formation of a dense cake layer and effective retention. Accumulation of organic matters is also related with operating conditions such as SRT.

This research also showed that although slightly better permeate quality in terms of COD concentration was obtained by submerged AnDMBR, high COD removal efficiencies were achieved in both submerged and external AnDMBR configurations. Comparison of the effects of membrane configuration on treatment and filterability performance showed that more time was needed in the external AnDMBR in order to form an effective DM layer enabling a stable removal efficiency and low soluble COD concentration in the permeate. Therefore, submerged AnDMBR configuration appears more suitable when a short start-up period is necessary. Higher methane production rate and methane yield were obtained in the submerged configuration compared to the external configuration reflecting the negative effect of sludge recirculation in the external DM configuration. Conversely, sludge recirculation in the external configuration was more effective in decreasing DM thickness, thus transmembrane pressure, than the bottom biogas sparging in the submerged configuration.

Considering the tested different gas sparging velocities (GSVs), over 99% organic removal was obtained with the external AnDMBR configuration for high strength wastewater treatment irrespective of the GSV, although total filtration resistance increased with decreasing GSV. Total filtration resistance mainly consisted of the resistance by the DM layer that provided effective and stable treatment. Following the organic loading rate study, the AnDMBR achieved high COD removal efficiency at 3.6 kg COD/m^3.d.

In conclusion, following the results obtained in this study, DM technology achieved a stable and high quality permeate. Thus, AnDMBRs can be used as a reliable and satisfactory treatment technology for treatment of high strength wastewaters. Low capital costs of support material and energy generation can make AnDMBRs feasible for those situations in which a high flux is not necessary, such as sludge and slurry treatment or highly concentrated industrial wastewater treatment. However, research on AnDMBRs is still very limited. Long-term applicability and reliability of the DM applications need further research, focusing on cake layer control methods to allow satisfactory DM layer formation as well as on the effect of sludge properties on DM filtration characteristics for large-scale applications.

ÖZET

Anaerobik membran biyoreaktörler (AnMBR), membran filtrasyon prosesi sayesinde biyokütlenin fiziksel olarak reaktör içerisinde tutulmasını sağlamaktadır. Aerobik membran biyoreaktör (MBR) uygulamalarındaki artışla birlikte, özellikle enerji verimli atıksu arıtımı konusunda sağladığı avantajlar dikkate alındığında, membran ve anaerobik proseslerin bir arada kullanılması konsepti her geçen günü daha çok ilgi çekmekte ve fizibil hale gelmektedir.

MBR prosesinde karşılaşılan en önemli zorluklar özellikle tam ölçekli sistemlerde öne çıkan membran maliyetleri, tıkanma ve düşük akı eldesidir. Dinamik membran (DM) teknolojisi MBR proseslerde görülen sorunların ortadan kaldırılmasını sağlayacak yenilikçi bir yaklaşım olarak kabul edilmektedir. DM teknolojisinin sağlayacağı en önemli faydalardan biri katı madde gideriminin uygun bir destek malzemesi üzerinde kendiliğinden oluşabilen ve ikincil membran olarak da adlandırılan DM tabakası ile gerçekleştirilmesi ve bunun sonucunda destek malzemesi olarak kullanılan membranın filtrasyonda rol almamasıdır.

DM tabakasının oluşturulmasında destek malzemesi olarak mikrofiltrasyon ve ultrafiltrasyon membranları yerine çeşitli tipte ve yapıda tel örgü şeklinde, dokunmuş veya dokunmamış kumaş malzemeler kullanılabilmektedir. Bu sayede, membran maliyetleri düşük seviyelere çekilebilecektir. Bununla birlikte biyogaz üretimi de dikkate alındığında, dinamik AnMBR (AnDMBR) prosesi atık(su) arıtımında cazip hale gelecektir.

Bu çalışmanın temel amacı, AnMBR prosesinde DM teknolojisinin konsantre atıksu arıtımına uygulanabilirliğinin incelenmesidir. Ayrıca bu çalışma, DM tabakası oluşumunun ve farklı koşullar altındaki filtrasyon karakteristiklerinin değerlendirilmesi yoluyla DM teknolojisi hakkındaki bilgi birikimine önemli katkılar sağlayacaktır. Bu çalışmada, membran konfigürasyonunun giderim verimi ve DM filtrasyonu üzerindeki etkilerini belirleyebilmek amacıyla batık ve harici membran modülleri kullanılmıştır. Substrat olarak KOİ konsantrasyonu 20 g/L olan sentetik konsantre atıksu kullanılmıştır. Tekli filament (monofilament), çoklu filament (multifilament) ve kısa iplik (staple) yapısına sahip destek malzemesi (filtresi) kullanılarak, DM oluşumuna en uygun destek malzemesi ve bu malzemenin özellikleri bulunmuştur. Bunun yanı sıra, en iyi giderim veriminin ve süzüntü kalitesinin elde edilebileceği işletme koşullarının tespiti amacıyla düşük akıda ve mezofilik şartlarda farklı işletme koşulları test edilmiştir. Ayrıca, destek malzemesini maliyet değerleri de arıtılan atıksu hacmi başına hesaplanarak verimiştir.

Bu çalışmada elde edilen sonuçlara göre destek malzemesinin özellikleri, fitre üzerinde etkili bir DM (kek) tabakasının oluşumu açısından kritik rol oynamaktadır. Staple ve monofilament filtreler kullanılarak multifilament malzemeye göre daha yüksek kritik akılar elde edilmiştir. Farklı destek malzemelerin kıyaslanması sonucunda staple filtrenin derin filtrasyona,

monofilament filtrenin ise yüzeysel filtrasyona daha uygun olduğu tespit edilmiştir. Bu nedenle, monofilament filtre DM teknolojisinde kullanım açısından daha uygundur.

Bu çalışma sonucunda DM filtrasyon teknolojinin MBR'ler için en önemli dezavantajlardan biri olan tıkanma problemini bir avantaja çevirebileceği görülmüştür. Mikrofiltrasyon veya ultrafiltrasyon membranlarına alternatif olarak, polipropilen monofilament filtre malzemesinin DM tabakası oluşumu vasıtasıyla filtrasyon amaçlı kullanılabileceği görülmüştür. AnDMBR ile % 99'un üzerinde organik madde giderimi ve partiküler madde tutulması gerçekleştirilmiştir. Bunun yanında, DM tabakasıyla % 66'nın üzerinde çözünmüş KOİ giderimi ve % 55-87 aralığında uçucu yağ asiti giderimi sağlanmıştır. Bu sonuçlar dikkate alındığında, AnDMBR'lerin uzun dönemde stabil olarak işletilebileceği ortaya konmuştur.

Çamur bekletme zamanı (SRT), AnDMBR'lerin işletilmesinde önemli bir faktör olarak tespit edilmiştir. SRT, çözünmüş mikrobiyal ürünler (SMP) ve hücre dışı polimerik madde (EPS) konsantrasyonu, protein/karbonhidrat oranı, çamur partikül boyutu, DM tabakası oluşumu ve çamurun filtre edilebilirliği üzerinde önemli etkilere sahiptir. EPS; protein, polisakkarit, lipid, nükleik asit ve hümik asit gibi başlıca hücre yüzey maddelerinden oluşmakta ve membran yüzeyini kaplayarak çamur floklarının bir arada tutulmasını sağlamaktadır. EPS kompozisyonunun partikül flokülasyonu üzerinde önemli bir pozitif etkisi olduğu görülmüştür. Bu etki direkt olarak reaktör içindeki çamurun partikül boyut dağılımını belirlemektedir. Kısa SRT'lere kıyasla daha uzun SRT'lerde, biyoreaktör içinde daha düşük EPS konsantrasyonu beklenmektedir.

Bu çalışmada, DM tabakasının kalınlığının kontrol edilmesi amacıyla geri yıkama ve biyogaz sıyırma yöntemleri birlikte kullanılmıştır. DM tabakasının kalınlığının kontrolü, stabil bir işletme ve yüksek süzüntü suyu kalitesi eldesi için hayati önem taşımaktadır. DM tabakasıyla gerçekleştirilen organik madde ve partiküler madde gideriminde, DM tabakasındaki biyokütle aktivitesinin ve DM tabakasının fiziksel tutma kapasitesinin birlikte rol aldığı düşünülmektedir. Mikrobiyal analiz sonuçları incelendiğinde, bakteri ve arkea için mikrobiyal çeşitlilik ve zenginliğin DM tabakasında yüksek olduğu ve DM'deki mikrobiyal popülasyonun biyoreaktör içerisindeki çamurdan farklı olduğu tespit edilmiştir. DM üzerinde yapılan morfolojik analizlerin sonuçları göz önünde tutulduğunda, DM tabakasının organik ve inorganik maddelerden oluştuğu görülmektedir. Bu maddelerin başlıcaları; çamur partikülleri, SMP, EPS, Ca, N, P ve Mg çökeltileridir. Ayrıca, kek tabakası altında kısmi bir jel tabakası oluşumu tespit edilmiştir. SMP ve bağlı EPS'nin DM tabakası içinde yüksek miktarda birikmesi, sıkı bir kek tabakası oluşumu ve yüksek giderim verimi sağlamaktadır. Organik madde birikimi, SRT gibi işletme koşulları ile yakından ilgilidir.

Bu çalışma sonucunda batık ve harici AnDMBR sistemlerinde yüksek KOİ giderme verimleri elde edilmiştir. Bununla beraber, batık AnDMBR konfigürasyonu ile daha yüksek süzüntü suyu kalitesi elde edilmiştir. Membran konfigürasyonunun giderim verimi ve filtrasyon performansı üzerine etkisi incelendiğinde, stabil bir giderim verimi ve süzüntü suyunda düşük çözünmüş KOİ konsantrasyonu elde etmek amaçlı etkin bir DM tabakası oluşturmak için harici AnDMBR ile, batık AnDMBR'ye göre, daha uzun süreye ihtiyaç olduğu görülmüştür. Bu nedenle, sistemi devreye alma süresinin kısa tutulması gerektiği durumlarda batık AnDMBR uygulanması daha uygundur. Batık konfigürasyonda, harici konfigürasyona göre daha yüksek metan üretim hızı ve metan verimi elde edilmiştir. Bu durum harici AnDMBR sisteminde yapılan çamur sirkülasyonunun olumsuz etkisini göstermektedir. Buna karşın, DM kalınlığının ve dolayısıyla transmembran basıncının azaltılmasında, batık AnDMBR sisteminde tabandan uygulanan biyogaz sıyırma işlemine nazaran harici konfigürasyonda uygulanan çamur sirkülasyonunun daha etkili olduğu görülmüştür.

Harici AnDMBR ile konsantre atıksu arıtımında farklı gaz sıyırma hızlarında (GSV) yapılan testler sonucunda, her ne kadar toplam filtrasyon direnci azalan GSV ile artsa da, GSV'den bağımsız olarak %99'un üzerinde organik madde giderim verimi elde edilmiştir. Toplam filtrasyon direnci başlıca DM direncinden kaynaklanmaktadır. Farklı organik yükleme hızlarında yapılan testler sonucunda, 3,6 kg KOİ/m^3.d yükleme hızında AnDMBR ile yüksek KOİ giderim verimi elde edildiği görülmüştür.

Bu tez kapsamında elde edilen sonuçlar değerlendirildiğinde, DM teknolojisinin stabil ve yüksek kalitede süzüntü suyu kalitesi elde etmek amacıyla başarıyla kullanılabileceği görülmüştür. Konsantre atıksu arıtımında AnDMBR'ler güvenilir ve yeterli bir arıtım sağlama potansiyeline sahiptir. Destek malzemesi açısından düşük ilk yatırım maliyeti ve biyogaz yoluyla enerji üretimi dikkate alındığında, yüksek akı gerekmeyen durumlar için, örneğin çamur veya konsantre endüstriyel atıksu arıtımı gibi, AnDMBR'ler fizibil bir arıtma teknolojisi olarak kullanılabilecektir. Bu sonuçlara rağmen, AnDMBR'ler üzerine yapılmış olan çalışmalar halen sınırlıdır. DM teknolojisi için uzun dönemli uygulanabilirlik çalışmalarına daha fazla ihtiyaç vardır. Özellikle tam ölçekli sistemlerde sürdürülebilir bir filtrasyon eldesi için DM tabakası kontrol metotları ve biyoreaktör içi çamur özelliklerinin DM filtrasyon karakteristikleri üzerine etkileri konularında çalışmalar yapılması faydalı olacaktır.

ABBREVIATIONS

A	filtration area (m^2)
AFM	atomic force microscopy
AnDMBR	anaerobic dynamic membrane bioreactor
AnMBR	anaerobic membrane bioreactor
BOD	biochemical oxygen demand
C	TSS concentration (kg/m^3)
COD	chemical oxygen demand
CSTR	completely stirred tank reactor
CST	capillary suction time
CST_n	normalized capillary suction time
DM	dynamic membrane
DMBR	aerobic dynamic membrane bioreactor
DOC	dissolved organic carbon
EDX	energy dispersive X-ray
EGSB	expanded granular sludge bed
EPS	extracellular polymeric substances
ESEM	environmental scanning electron microscopy
F/M	food/mass
FIP	formed-in-place
FTIR	fourier transform infrared spectroscopy
GSV	gas sparging velocity
HRT	hydraulic retention time
J	flux, ($m^3/m^2.h$)
MBR	membrane bioreactor
MF	microfiltration
MLSS	mixed liquor suspended solids
NF	nanofiltration
OLR	organic loading rate
P/C	protein/carbohydrate
PAA	poly(acrylic acid)
PAC	powdered activated carbon
PBS	phosphate buffered saline
PET	polythylene terephthalate
PSD	particle size distribution
PTFE	poly-tetrafluoroethylene
PVDF	polyvinylidene fluoride
RO	reverse osmosis
RE	external anaerobic dynamic membrane bioreactor
RS	submerged anaerobic dynamic membrane bioreactor

R_T	total filtration resistance, (m^{-1})
SFDM	self-forming dynamic membrane
SMA	specific methanogenic activity
SMP	soluble microbial products
SRF	specific resistance to filtration
SRT	sludge retention time
SS	suspended solids
t	time of filtration, (s)
TMP	transmembrane pressure
TN	total nitrogen
TOC	total organic carbon
TP	total phosphorus
TS	total solids
TSS	total suspended solids
UASB	upflow anaerobic sludge bed
UF	ultrafiltration
V	filtrate volume, (m^3)
VFA	volatile fatty acid
VS	volatile solids
VSS	volatile suspended solids
WW	wastewater
μ	dynamic viscosity, (Pa.s)
ΔP	applied pressure, (kPa)

CHAPTER 1

INTRODUCTION

1 INTRODUCTION

1.1 Background

Anaerobic technology has improved significantly in the last few decades with the applications of differently configured high rate treatment processes, especially for the treatment of industrial wastewaters. High organic loading rates (OLRs) can be achieved at smaller footprints by using high rate anaerobic reactors. Biomass retention is a necessary feature for high rate anaerobic treatment of wastewaters due to the low growth rate of anaerobic microorganisms, particularly at sub-mesophilic conditions when the degradation rate of suspended solids and colloidal particles is the rate limiting step. High rate anaerobic processes generally use biofilm or granular sludge to obtain a high biomass concentration inside the bioreactor (Lettinga et al., 1980; Rittmann and McCarty, 2001). When biofilm formation or granulation cannot be easily achieved, membrane filtration may represent an alternative way to provide biomass retention. Membrane assisted sludge retention also ensures the accumulation of the very slowly growing organisms that are frequently needed for the treatment of toxic and recalcitrant wastewaters. In this way, aggregation property of the biomass is not important anymore for substrate degradation capacity, and cell washout risk can be avoided.

There is a growing interest in combining membranes with aerobic biological wastewater treatment processes, called membrane bioreactors (MBRs), where the membrane is used as the main solids-liquid separation device. MBRs ensure complete biomass retention by the application of microfiltration (MF) or ultrafiltration (UF) enabling an operation at high sludge concentrations. MBR technology offers the complete separation of hydraulic retention time (HRT) and sludge retention time (SRT), which facilitates a more flexible control of operating parameters. Today, MBR technology has been proven for municipal and industrial wastewater treatment. MBRs are increasingly replacing conventional activated sludge processes for treatment of different kinds of wastewater (Wu et al., 2005; Judd, 2006; Lesjean and Huisjes, 2008).

In recent years, with growing application experiences from aerobic MBRs, anaerobic membrane bioreactors (AnMBRs) have received much attention, due to their advantages with regard to developments for energy-efficient wastewater treatment. AnMBRs combine the advantages of MBR and anaerobic technology. In AnMBRs, biomass and particulate organic matter are physically retained inside the bioreactor, providing optimal conditions for the degradation of organic matter. As a consequence, a potential increase in digester organic loading capacity, an improved effluent quality and a decreased excess sludge production can be achieved (Ghyoot and Verstraete, 1997; Abdullah et al., 2005). The applicability of the AnMBR technology for treatment of different kinds of wastewater is summarized in Figure 1.1 (Liao et al., 2006). AnMBR technology can also be applied for the treatment of more concentrated wastes, like excess domestic sewage sludge. Although AnMBRs have been mainly applied for treatment of wastewaters, a few studies for treatment of wastewater sludge are reported in the literature (Ghyoot and Verstraete, 1997; Park et al., 2004; Abdullah et al., 2005).

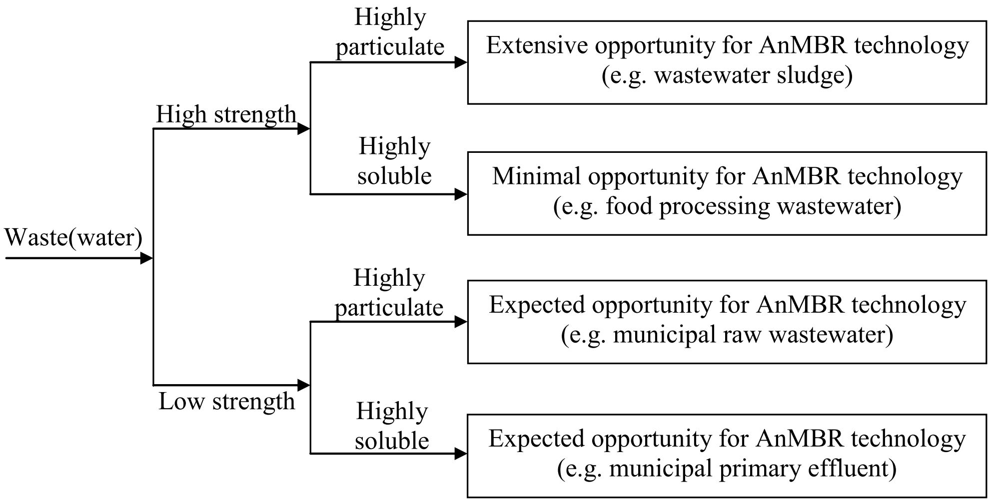

Figure 1.1. Applicability of AnMBRs (Liao et al., 2006).

In a membrane coupled bioreactor system, the membrane can be located either inside or outside the bioreactor, which are called submerged or side-stream configuration, respectively. The layouts of different MBR configurations are presented in Figure 1.2. Most of the reported researches about AnMBRs have used a side-stream/cross-flow configuration that employs a membrane externally connected to the reactor. In this configuration, a pump pushes the effluent of bioreactor into the external membrane unit (Figure 1.2. (a)). The removal of cake layer is brought about by sufficiently high cross-flow liquid velocity along the membrane surface (Liao et al., 2006). Cross-flow membrane modules have some advantages such as the ease of membrane replacement and cleaning. However, rapid development of fouling became an obstacle for cross-flow AnMBRs for large-scale applications (Choo and Lee, 1996; Ince et al., 1997; Kang et al., 2002; Fuchs et al., 2003; He et al., 2005). In the submerged configuration, vacuum is applied at the permeate side to obtain the permeate instead of direct pressure at the feed side. While air bubbling is used to remove the cake layer in aerobic MBR applications, for anaerobic MBRs, biogas recirculation can be used for this purpose. The membrane can be submerged inside the bioreactor (Figure 1.2. (b)) or externally submerged (Figure 1.2. (c)) in a separate chamber that is located outside the bioreactor. For side-stream configuration, the pump is located before the membrane and the operation is done under pressure, whereas for external configuration the pump is located after the membrane and the operation is done under vacuum. Compared to side-stream, submerged AnMBR configuration has attracted more interest recently due to large amount of comparable knowledge from aerobic MBR operations and fouling research (Jeison, 2007, Huang et al., 2008, Lin et al., 2010). Energy and membrane costs of the submerged configuration may be close to one third of the side-stream configuration for a given flux (Jeison and van Lier, 2008a).

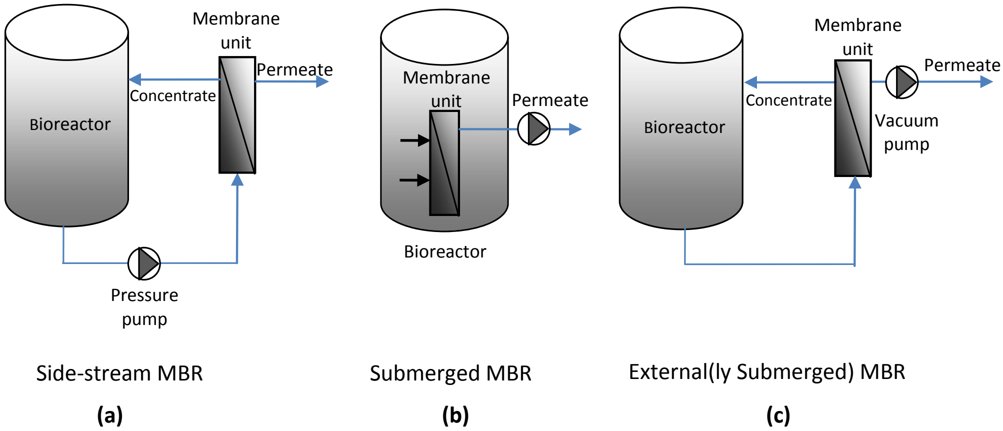

Figure 1.2. Different MBR configurations.

1.2 Statement of Topic

The major drawbacks of the MBR technology are related with membrane costs, especially for the full-scale applications, fouling, and low flux (Fan and Huang, 2002; Jeison et al., 2008; Satyawali and Balakrishnan, 2008; Zhang et al., 2010). Many factors have been reported that might influence the fouling in MBRs such as floc size, mixed liquor suspended solids (MLSS) concentration, viscosity of mixed liquor, pH and soluble and bound extracellular polymeric substances (EPS) (Ahmed et al., 2007; Lin et al., 2009; Gao et al., 2010). In addition, membrane characteristics such as pore size, porosity, surface charge, roughness, and hydrophilicity/hydrophobicity may play a significant role in membrane fouling (Gao et al., 2011). The operating parameters such as HRT, SRT and food/mass (F/M) ratio have no direct effect on membrane fouling; instead, they affect the sludge characteristics and thus the sludge filterability (Meng et al., 2009). Organic fouling, in comparison to inorganic fouling, has been reported as the main reason of membrane fouling during the filtration of activated sludge (An et al., 2009; Meng et al., 2009). Recent studies have shown that cake layer formation is the key factor limiting the flux when operating AnMBRs, irrespective of the applied substrate, configuration (submerged or side-stream) or temperature (Jeison and van Lier, 2008b; Lin et al., 2009; Waeger et al., 2010). Meng et al. (2007) reported that the clean membrane, the cake, and the pore resistance contributed to 9%, 84%, and 7% of the total resistance of an aerobic submerged MBR, respectively.

Considering the fact that the fluxes in AnMBRs are determined by cake filtration (Jeison, 2007), indicates that formation of a controlled cake or a dynamic membrane (DM) on an underlying support material could give similar effluent qualities compared to purchased membranes. In anaerobic reactors, the filter solution always contains suspended solids, indicating that DM application may indeed provide a promising approach to resolve the problems encountered in MBR processes. Different kinds of low-cost materials can be used to serve as the supporting layer instead of UF or MF membranes to form a DM layer. The possibility of operating an AnMBR with a self-forming DM generated by the substances present in the reactor liquor would result in an important saving in costs. By decreasing the membrane material cost and generating energy, anaerobic dynamic membrane bioreactors (AnDMBRs) are expected to receive much attention in achieving a cost-effective operation with a high permeate quality.

1.3 Aim of the Thesis

The aim of this thesis was to investigate the applicability of DM technology for the treatment of concentrated wastewaters in AnMBRs. The research was oriented to AnMBRs using a mono-monofilament filter cloth instead of a “conventional" membrane. The biological capacity and the filtration performance of two AnDMBRs at low fluxes were investigated under mesophilic conditions (35 ^{o}C). Besides, the determination of an optimal support material and investigations about its structure were achieved by testing various types of support materials. Within this concept, two AnDMBR configurations, submerged and external, were tested. Because there is quite limited information about the potential and applicability of DM technology for treatment of high-strength/concentrated waste(water)s in AnMBRs, the results obtained from this thesis provided a comprehensive view on the role of DM in filtration and treatment. The aims were met by achievement of the following objectives, that is to:

- identify the optimum support material and its optimum pore size, enabling the formation of a coherent DM layer and thus effective particle retention enabling, producing a high permeate quality.

- understand the effects of various reactor operational conditions such as SRT, HRT, OLR, and gas sparging rate on the biological removal efficiency and filtration characteristics of the DM.

- determine the characteristics of the DM (cake) layer formed on the supporting layer and its variation under different operating conditions.

- compare the bulk sludge and cake layer characteristics in order to understand the role and formation mechanism of the DM layer.

- show the impact of membrane configuration on the treatment and compare the biological removal capacities, filtration performances, bulk sludge characteristics of submerged and external AnDMBR configurations.

- determine the advantages and weaknesses of the AnDMBR technology in terms of biological removal efficiency and filtration performance.

1.4 Outline of the Thesis

The objectives to meet the aims mentioned above have been addressed in eight chapters and the chapters are structured as follows:

Chapter 2 presents a comprehensive evaluation of the current status of DM technology as an alternative to conventional MBR systems. A review of the state-of-art of both DM materials and configurations is presented. Factors affecting DM performance in physical and biological, both aerobic and anaerobic, applications are discussed in order to determine the optimum and critical approaches for membrane operation.

Chapter 3 addresses the effects of support material properties including pore size and structure of the material on DM formation and performance in AnDMBR systems. A comparative evaluation between support materials that have different yarn types is presented. An optimum support material and its pore size that provide the formation of DM layer and effective retention are identified.

Chapter 4 deals with the applicability of DM technology in AnMBRs for the treatment of high strength wastewaters, using a mono-monofilament woven fabric as the support material. This chapter discusses the effects of SRT on the removal efficiency and filtration characteristics of the DM in a submerged AnDMBR.

Chapter 5 focuses on the characterization of the DM layer and its role in AnDMBRs. The role of the DM layer in biological removal performance in terms of particulate and soluble organic matter removal is elucidated. This chapter discusses the different aspects of the DM structure in order to obtain a better understanding of the formation mechanisms. Besides, pyrosequencing was used to compare the microbial community structure including both archaeal and bacterial communities and the relative abundance of microbial species in the bulk sludge and in the cake layer.

Chapter 6 provides a comparison of two different membrane configurations, including submerged and external AnDMBRs, for their removal capacities and filtration performances under mesophilic conditions. Impact of the membrane configuration on long-term operation is identified and evaluated. Moreover, microbial community structure including both bacterial and archaeal communities and the relative abundance of microbial species in the bulk sludge of submerged and external AnDMBRs were compared.

Chapter 7 describes the effects of biogas sparging rate and HRT on the removal efficiency and filtration characteristics in an external AnDMBR. For this purpose, long-term operation of an external AnDMBR for the treatment of high strength wastewater under mesophilic conditions was evaluated. In addition, a cost estimation of membrane acquisition/replacement is made.

Chapter 8 concludes the overall results obtained in the different sub-studies and presents a general discussion. In particular, this chapter focuses on the contribution of the results obtained in this thesis to a better understanding of DM technology and formation mechanism in AnDMBRs. In addition, problems encountered, perspectives and recommendations for future research directions are provided to enhance the applicability and functionality of DM technology.

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CHAPTER 2

DYNAMIC MEMBRANE FILTRATION: MATERIALS AND APPLICATIONS

Abstract

This chapter presents a comprehensive evaluation of the current status of DM technology as an alternative to MBR systems. DM filtration makes use of a physical barrier (e.g. cloth or mesh) on which a cake layer is formed. It is already used in traditional filtration systems, but applications in biological wastewater treatment are still at its infancy. Dynamic filtration of sludge has lower risk of fouling and requires less energy and lower capital costs compared to MBR. A review of the state-of-art in both DM materials and configurations is presented. Factors affecting DM performance are discussed in order to determine the optimum and critical approaches for membrane operation. Future perspectives to enhance the applicability and functionality of the technology regarding the treatment and membrane performance are presented.

This chapter is based on:

Ersahin, M.E., Ozgun, H., Dereli, R.K., Ozturk, I., Roest, K., van Lier, J.B., 2012. A review on dynamic membrane filtration: Materials, applications and future perspectives. Bioresource Technology, 122, 196-206.

2 DYNAMIC MEMBRANE FILTRATION: MATERIALS AND APPLICATIONS

2.1 Introduction

Membranes have been used as solid-liquid separation devices in biological treatment (aerobic and anaerobic) and physical applications for many years. There has been a growing interest in combining membranes with biological wastewater treatment in so called MBRs, giving striking advantages such as improved effluent quality and low system footprint (Judd, 2006) . The major constraints of MBR processes are related to membrane costs, energy demand, fouling control, and low flux. DM technology may be a promising approach to resolve problems encountered in MBR processes (Fan and Huang, 2002; Wu et al., 2005; Ye et al., 2006). A DM, which is also called secondary membrane, is formed on an underlying support material, e.g. a membrane, mesh, or a filter cloth, when the filtered solution contains suspended solid particles such as microbial cells and flocs. Organics and colloidal particles which normally result in fouling of the membrane will be entrapped in the biomass filtration layer, preventing fouling of the support material (Kiso et al., 2005; Jeison and van Lier, 2007a, 2007b). An illustration adapted from Lee et al. (2001) is given in Figure 2.1 to demonstrate the dynamic cake layer formation. Formation of this cake layer over the membrane surface can determine rejection properties of the system, since the deposited layer will act as a "secondary" membrane prior the "real" membrane or support material (Kiso et al., 2000; Park et al., 2004; Fuchs et al., 2005; Jeison et al., 2008; Zhang et al., 2010). Water backwash, air backwash, or brushing can be enough for DM cleaning without using chemical reagents (Chu et al., 2008). However, depending on the support material, cleaning obviously might be accompanied by a temporary loss of effluent quality.

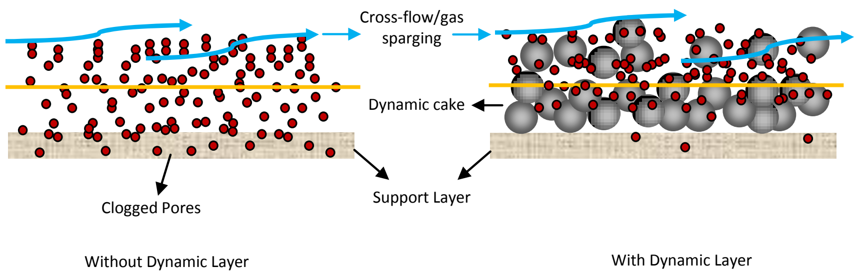

Figure 2.1. Demonstration of the dynamic cake layer.

One of the most important potential benefits of DM is that the membrane itself may be no longer necessary, since solids rejection is accomplished by the secondary membrane layer which can be formed and re-formed as a self-forming dynamic membrane (SFDM) in situ. Repeated processes of DM formation and removal may reduce membrane permeability losses as encountered in conventional MBRs (Lee et al., 2001).

Different kinds of cheap materials such as mesh, non-woven fabric and woven filter-cloth can be used as the supporting layer instead of MF or UF membranes for creating a DM layer (Wu et al., 2005; Chu and Li, 2006; Jeison et al., 2008; Zhang et al., 2010). Substituting the traditional membranes by cheaper filtration materials potentially offers higher flux rates at lower transmembrane pressures (TMPs) in a cost-effective manner (Seo et al., 2002; Fuchs et al., 2005; Satyawali and Balakrishnan, 2008).

Since 1960s, many DM studies have been conducted extending from physical filtration trials to MBR applications. Due to the variability of DM formation mechanisms and DM applications, a comprehensive study is needed to give direction to future studies on DM technology. This chapter summarizes DM studies and evaluates the results in many aspects, trying to better understand the DM formation mechanisms. Challenges encountered and future perspectives are discussed to enhance the functionality of DM technology.

2.2 Materials, Configurations and Historical Development

2.2.1 Materials

2.2.1.1 Dynamic Layer Forming Materials

DMs can be mainly classified into two groups, i.e. self-forming and pre-coated. SFDM is generated by the substances present in the filtered liquor, such as suspended solids (SS) in wastewaters, whereas pre-coated DMs, also denominated formed-in-place (FIP) membranes, are produced by passing a solution of one or more specific colloidal components over the surface of a porous material (Al-Malack and Anderson, 1996; Ye et al., 2006). The main disadvantage of this approach over SFDM is the requirement of an external material. The pre-coated DMs can also be subdivided into two groups, namely single additive and composite (bi-layer) membranes. The single additive pre-coated membranes are generally formed by only one material in a single step. Ye et al. (2006) used powdered activated carbon (PAC) as a single additive to form DM. Composite membranes are generally produced by a two-step formation process (Ip, 2005).

The concept of SFDM formation by microbial flocs has been applied to aerobic MBRs for wastewater treatment with promising results (Fuchs et al., 2005; Kiso et al., 2005; Wu et al., 2005; Chu and Li, 2006). Also the pre-coating method has been used to form a pre-coated DM layer in aerobic dynamic membrane bioreactors (DMBRs). PAC (Ye et al., 2006), kaolinite (Li et al., 2006) and bio-diatomite (Chu et al., 2008; Cao et al., 2010) are some of the ingredients that have been used as pre-coating materials. For anaerobic applications, SFDM method was applied by Jeison et al. (2008); whereas an example of surface modification with poly-tetrafluoroethylene (PTFE) can be found in study of Ho et al. (2007).

Hydrous metal oxide, especially zirconium (Zr(IV)) oxide, is one of the most commonly used and most successful material to form a DM layer in physical dynamic filtration

(Marcinkowsky et al., 1966; Freilich and Tanny, 1978; Ohtani et al., 1991; Rumyantsev et al., 2000). Moreover, modification of Zr(IV) oxide with polymers, generally with poly(acrylic acid) (PAA), was also applied in order to improve the filtration properties of the dynamic layer (Altman et al., 1999). Other materials including MnO_2 (Al-Malack and Anderson, 1996; Cai et al., 2000), TiO_2 (Horng et al., 2009), $Mg(OH)_2$ (Zhao et al., 2006), gelatin (Tsapiuk, 1996), ovalbumin (Matsuyama et al., 1994), solid particles present in pineapple juice (Jiraratananon et al., 1997), kaolin (Wang et al., 1998; Noor et al., 2002), kaolin/MnO_2 bi-layer (Yang et al., 2011), poly(vinyl alcohol) (Na et al., 2000), dextran (Wang et al., 1999), non-coagulating and hydrophylized coagulating polymer (Knyazkova and Kavitskaya, 2000), and clay minerals (Kryvoruchko et al., 2004) have also been tested as forming materials of DMs.

2.1.1.2 Support Materials

Research on DMs, especially for wastewater treatment has been generally focused on the use of meshes, woven and non-woven fabrics as the support material. A mesh consists of a permeable barrier made of connected strands of metal, fiber or other flexible/ductile material. The disadvantage of a mesh filter material may be related to the inefficient sludge accumulation due to its flat structure (Kiso et al., 2005). A woven cloth is based on monofilament and/or multifilament yarn. Monofilament yarns are single extruded synthetic filaments and have smooth surfaces. A multifilament fiber consists of several fine monofilament fibers spun together to form the individual yarns that are eventually woven together. A non-woven cloth is defined as a sheet or web of natural and/or man-made fibers or filaments, excluding paper, that have not been converted into yarns, and that are bonded to each other (Hutten, 2007). Although the non-woven fabric is very thin, attachment of sludge particles has been observed in the pores among the fiber matrix which made the removal of the attached sludge from the filter interstices difficult in the long-term operation (Kiso et al., 2005).

To date, meshes (Kiso et al., 2000; Fan and Huang, 2002; Kiso et al., 2005; Chu and Li, 2006; Satyawali and Balakrishnan, 2008; Jeison et al., 2008; Walker et al., 2009; Zhang et al., 2010), non-woven fabrics (Seo et al., 2002; Wu et al., 2005; Seo et al., 2007; An et al., 2009; Ren et al., 2010), woven fabrics (Pillay et al., 1994; Fuchs et al., 2005; Liu et al., 2009) and ceramic membranes (Li et al., 2006) have been reported as possible support materials for solid–liquid separation in both aerobic and anaerobic dynamic MBRs.

In physical applications, DMs have been successfully formed on a variety of organic and inorganic support materials, such as ceramic tube (Nakao et al., 1986; Ohtani et al., 1991; Tien and Chiang, 1999; Yang et al., 2011), stainless steel tube (Groves et al., 1983; Wang et al., 1999); polymeric membrane (Turkson et al., 1989; Cai et al., 2000); MF membrane (Igawa et al., 1977; Jiraratananon et al., 1997; Na et al., 2000; Hwang and Cheng, 2003), UF membrane (Tsapiuk, 1996; Na et al., 2000; Kryvoruchko et al., 2004), reverse osmosis (RO) membrane (Knyazkova and Kavitskaya, 2000; Kryvoruchko et al., 2004), and woven or non-woven fabrics (Al-Malack and Anderson, 1996; Altman et al., 1999; Rumyantsev et al., 2000;

Horng et al., 2009). Stainless steel and ceramic tubes have been generally used in physical DM applications, especially in the early studies. High cost of these materials is the main disadvantage of using them. Thus, cheaper materials such as woven or non-woven fabrics have also been tested by various researchers.

2.2.2 Configurations

Generally, submerged flat sheet membrane modules have been used in DMBRs. This is probably due to the operational simplicity and practical easiness of constructing a module equipped with flat sheet support materials (Kiso et al., 2000; Seo et al., 2002; Fan and Huang, 2002; Liu et al., 2009). However, Li et al. (2006) and Seo et al. (2007) tested tubular modules in DMBRs at external and submerged modes, respectively. Both flat sheet (Jeison et al., 2008; Zhang et al., 2010) and tubular (Pillay et al., 1994; Ho et al., 2007; An et al., 2009) configurations have been applied to determine the feasibility of AnDMBR applications. Biogas can be recirculated in both configurations for mixing and controlling cake layer thickness.

2.2.3 Historical Development

The first study on physical DMs was reported by Marcinkowsky et al. (1966) who utilized a zirconium oxychloride ($ZrOCl_2$) DM for the rejection of salts in a RO process. After this study, DM research has generally focused on the salt rejection performance of RO processes (Igawa et al., 1977; Tanny and Johnson, 1978; Freilich and Tanny, 1978).

DM applications in UF processes began in 1980s. The main purposes of those investigations were wastewater treatment, dye and protein removal (Gaddis et al., 1979; Groves et al., 1983). Some researchers have also tested dynamic UF membranes in food industry (Kishihara et al., 1984; Jiraratananon et al., 1997). In spite of the high retention capacity of UF processes, high capital costs of support materials and low permeability potential prevented the further applications of DM systems on a large-scale.

DM applications in MF processes have been tested since 1990s, especially for the treatment of wastewaters (Al-Malack and Anderson, 1996; Noor et al., 2002; Hwang and Cheng, 2003; Zhao et al., 2006; Horng et al., 2009). High performance values obtained in recent studies showed that dynamic MF membranes can be a viable option for the separation of oil from water (Zhao et al., 2005; Yang et al., 2011).

First application of aerobic wastewater treatment utilizing DM filtration dates to mid-1990s (Yamagiwa et al., 1994; Al-Malack et al., 1998) and ever since this concept is receiving growing interest from the scientific community. Most researchers presented satisfactory removal efficiencies for SS, biochemical oxygen demand (BOD) and COD comparable to conventional UF/MF membranes (Kiso et al., 2000; Seo et al., 2002). Therefore, dynamic filtration seems to be a promising technique especially for small wastewater treatment systems where minimum investment and operational costs and simplicity are required.

The first application of DM technology in anaerobic systems was reported by Pillay et al. (1994). Research on AnDMBR systems has been increasing since 2007 with several attempts in order to optimize the operational conditions of the DMs (Jeison et al., 2008; Walker et al., 2009; An et al., 2009; Zhang et al., 2010).

2.3 Applications

2.3.1 Physical

Research on physical DM applications generally has been focused on the membrane forming materials and conditions of formation. By adjusting both factors, filtration performance similar to that of MF, UF, RO or nanofiltration (NF) membranes can be achieved by DMs. Sharp and Escobar (2006) found that DM filtration could provide higher steady state flux values than UF and improved the rejection of dissolved organic carbon (DOC), hardness and UV-254 values as compared to conventional UF treatment. They concluded that DM technology has a potential to decrease the membrane cost. Tsapiuk (1996) determined that a DM layer formed by gelatin increased the retention capacity of poly(ethylene glycol)s in a UF process. It was also stated that this positive effect depends on DM formation conditions. Al-Malack and Anderson (1996) compared the pore sizes of a MF membrane and a MnO_2 dynamic membrane layer, which was formed on the MF surface. They determined that the pore size of dynamic layer (2 μm) was much less than the pore size of the primary membrane, which provided an enhanced retention capacity. With respect to the performance of DMs, similar separation efficiencies, i.e. 85% ovalbumin retention at a concentration of 1000 ppm and similar permeabilities (10-50 $L/m^2.h.bar$) can be achieved in comparison to commercial UF membranes (Altman et al., 1999). Knyazkova and Kavitskaya (2000) showed that a dynamically modified RO membrane with a coagulating polymer provided an enhancement in salt rejection in contrast to uncoated membrane. Also the DM formed by non-coagulating polymers increased the flux in comparison with the uncoated membrane. Table 2.1 presents the formation condition and filtration performance of different DM applications described in literature.

Table 2.1. Formation condition and filtration performance of different non-biological dynamic membrane applications.

Dynamic Membrane Material	Support Material (Pore Size, μm)	Application Type	Formation Pressure (bars)	Formation Cross-flow velocity (m/s)	Flux	Removal Efficiency (%)	Reference
Hydrous Zr(IV) oxide-PAA	Porous carbon, ceramic, sintered glass, and metals (5)	RO	8-70	n.a[a]	n.a.	72-97% (Salt removal) 68 (NaCl removal) 90 ($MgCl_2$ removal)	Marcinkowsky et al. (1966) Shor et al. (1968) Johnson et al. (1972)
PAA, Al(III)-PAA, methylcellulose	MF membrane (0.025)	RO	78.5	n.a.	0.122 cm/min	90 (NaCl removal)	Igawa et al. (1977)
Hydrous Zr(IV) oxide-PAA	Filter (Millipore/Acropor) (0.025-0.4)	RO	65.5	4.6	1.8-2.0 $L/m^2.h.bar$	83-94 (NaCl removal)	Tanny and Johnson (1978)
Hydrous Zr(IV) oxide	Filter (Millipore) (0.1)	RO	3.4	n.a.	n.a.	10-50 (NaCl removal)	Freilich and Tanny (1978)
Hydrous Zr(IV) Oxide-PAA	Stainless steel Tube	UF	52	2.1	n.a.	(96-99) (Color removal)	Gaddis et al. (1979)
Hydrous Zr(IV) oxide-PAA	Stainless steel, fibre glass tubes	UF	n.a.	n.a.	30-200 $L/m^2.h$	48-62 (Salt removal) 66-97 (TOC[b] removal) 85-92 (Total solids removal)	Groves et al. (1983)
Colloid solutions of Zr(IV), Al (III), Fe(III)	Ceramic tube (0.5-1.0)	UF	8	3.3	n.a.	n.a.	Nakao et al. (1986)
Ca-oleate, CdS, ZrO_2	Polymeric membrane (0.2)	UF	n.a.	n.a.	0.005-0.012 cm/s	>80 (Bovine serum albumin (BSA) removal)	Turkson et al. (1989)
Hydrous Zr oxide	Ceramic tube (0.5)	UF	5	1	n.a.	90 (Dextran removal)	Ohtani et al. (1991)
Ovalbumin, γ-globulin	Ceramic Tube (0.05)	UF	6-10	0.15-0.78	n.a.	>80 (Protein removal)	Matsuyama et al. (1994)
Gelatin	UF membrane (0.81)	UF	0.05-5	n.a.	n.a.	100 (Poly(ethylene glycol) removal)	Tsapiuk (1996)
MnO_2	Tubular polyester yarn woven fabric (20-40)	MF	1	2	100 $L/m^2.h$	99 (Turbidity removal)	Al-Malack and Anderson (1996)
Pineapple juice (solids particles)	Monolith alumina MF membrane (0.1)	UF	1-3	1.30-3.95	6.37 $m^3/m^2.h$	84-87 (macromolecules removal)	Jiraratananon et al. (1997)
Kaolin	Stainless steel (4.7)	MF	100	n.a.	n.a.	100 (CH_3COONa removal)	Wang et al. (1998)
Hydrous Zr oxychloride-	Ceramic tube (0.2)	UF	8	10 L/min	16.5-34.2 $L/m^2.h.bar$	1.6-5.8 (glucose removal) 96.4-98.3 (glucose removal)	Chen and Chiang (1998)
Zr(IV) colloid, glutaraldehyde	Ceramic tube	UF	8	10 L/min	n.a.	n.a.	Tien and Chiang (1999)
Hydrous Zr(IV) oxide-PAA	Polypropylene polyethylene non-woven fibers	UF	4-5	10-12 L/min	10-50 $L/m^2.h.bar$	85-95 (Ovalbumin removal)	Altman et al. (1999)

Table 2.1-continued. Formation condition and filtration performance of different dynamic membrane applications.

Dynamic Membrane Material	Support Material (Pore Size, μm)	Application Type	Formation Pressure (bars)	Formation Cross-flow velocity (m/s)	Flux	Removal Efficiency (%)	Reference
Zr-dextran	Steinless steel tube (0.5-5)	UF	1	0.23	7.7 $L/m^2.h.bar$	60-100 (Hemoglobin removal)	Wang et al. (1999)
Zr hydroxide colloids	non-woven fabric, polysulfone MF membrane (3)	UF	4	n.a.	n.a.	75-95 (Protein removal)	Rumyantsev et al. (2000)
MnO_2	Polyethylene tube (5-20)	MF-UF	<20	n.a.	n.a.	98 (Turbidity removal)	Cai et al. (2000)
Non-coagulating and hydrophylized coagulating polymers	Cellulose acetate RO membrane	RO	40	0.4-2.7	21.6-23 $L/m^2.h$	95-98 (Salt removal)	Knyazkova and Kavitskaya (2000)
Poly (vinyl alcohol)	Polyvinylidene fluoride, nylon, polyacrylonitrile MF and UF membranes	UF	2	n.a.	n.a.	97.2-99.9 (Protein removal)	Na et al. (2000)
Kaolin	Tubular MF fabric membrane	MF	n.a.	1.6-2.5	n.a.	96 (Turbidity removal)	Noor et al. (2002)
Polymethyl methacrylate particles	Polyvinylidene fluoride MF membrane (0.1)	MF	0.2-1.4	0.1-0.4	n.a.	7-42 (Dextran removal)	Hwang and Cheng (2003)
Clay mineral montmorillonite, cation-exchange resin	UF and RO membranes	UF-RO	2	n.a.	n.a.	8-98 (Co(II) removal)	Kryvoruchko et al. (2004)
$Mg(OH)_2$	Al_2O_3 ceramic tubes (5)	MF	1	1	100 $L/m^2.h$	98 (Oil removal)	Zhao et al. (2005)
$Mg(OH)_2$	Al_2O_3 ceramic tubes (5)	MF	1	1	1 $L/m^2.h.kPa$	>98 (TOC removal)	Zhao et al. (2006)
TiO_2	Non-woven filter (0.2, 2, 20)	MF-UF	0.005-0.02	0.25-2 m/d	125 $L/m^2.h$	99 (Turbidity removal)	Horng et al. (2009)
Kaolin- MnO_2	Al_2O_3 porous ceramic tubes (1)	MF	2	1	120.1-153.2 $L/m^2.h$	98.2-99.9 (Oil removal)	Yang et al. (2011)

[a] n.a.: Not available; [b] TOC: Total organic carbon

The first attempt on DM application in RO processes has not provided satisfactory results in terms of salt rejection for desalination systems (Shor et al., 1968; Igawa et al., 1977; Freilich and Tanny, 1978). The main problems faced in the earliest studies were low and non-stable fluxes and difficulties encountered in the control of membrane forming conditions.

DM technology was used for different purposes in UF processes, especially for the treatment of wastewaters. Treatment of textile industry effluents was successfully achieved by DMs with high dye removal efficiencies (96-99%) (Groves et al., 1983, Gaddis et al., 1979; Townsend et al., 1989). One of the other main application areas of dynamic UF membranes was protein removal (Turkson et al., 1989; Matsuyama et al., 1994; Chen and Chiang, 1998; Altman et al., 1999; Wang et al., 1999; Na et al., 2000).

Recent research on DM-MF applications was mainly focused on wastewater treatment. The deposition of MnO_2 particles onto the surface of a kaolin dynamic layer was found to be effective for oily wastewater treatment in a DM-MF process (Yang et al., 2011). Oil concentration, pH and temperature were identified as the most effective parameters affecting DM performance. Al-Malack and Anderson (1996) studied the treatability of secondary effluent from a domestic wastewater treatment plant by a DM layer on a woven fabric and obtained a flux of nearly 100 $L/m^2.h$ and turbidity removal of 99% in short-term experiments (~10 h). The improvement in process performance by DM application was attributed to the narrowing of the pore size and surface modification of the primary support membrane. The mass of the DM layer was reported as the most critical factor in the rejection capacity of Dextran by Hwang and Cheng (2003). They concluded that the cake resistance played a major role in the filtration rate and an increase in the filtration pressure augmented cake resistance. This conclusion is compatible with the results of Zhao et al. (2006). Hwang and Cheng (2003) determined that the filtration rate has increased with the increase in the cross-flow velocity, resulting in a thinner cake, leading to a lower rejection of Dextran. Recently, Horng et al. (2009) found that cake formation is the dominant factor controlling the filtration rate, rather than pore blocking in a dynamic MF process. The filtration resistance increased with the decrease in the aeration intensity due to the accumulation of a cake layer on the non-woven filter. Authors stated that the aeration intensity should be increased up to a certain value in order to prevent an excess cake layer thickness.

DMs can also potentially benefit from recent developments in nano-technology applications (Srivastava et al., 2004; Wang et al., 2005). Brady-Estevez et al. (2008) developed a composite DM filter composed of a polyvinylidene fluoride (PVDF) based microporous support layer and a thin carbon nanotube layer. They demonstrated that the thickness of carbon nanotube layer plays an important role in virus removal. Results showed that it removed 3.2-7 log10 virus particles by in-depth filtration. This observation supports the idea that DM filters remove contaminants by size exclusion, adsorption, and depth filtration as opposed to UF and MF which perform separation mainly by size exclusion.

Dynamic filtration was also found feasible for wastewater sludge thickening. It could be possible to get filtrates with SS concentration of less than 1 mg/L after 10 minutes of filtration by using mesh filter (Park et al., 2004). They also reported that mesh opening size had a little influence on the filtration rate which is not consistent with the results of Hwang and Cheng (2003).

2.3.2 Biological

2.3.2.1 Aerobic Dynamic Membrane Bioreactors (DMBRs)

SFDMs and cake layer filtration for wastewater treatment were mostly investigated in DMBR systems as an effective and economical alternative to conventional MBR systems. Table 2.2 and Table 2.3 present the performance of DMBR applications in literature, listed with regard to both biological treatment and membrane aspects, respectively.

One of the most important advantages of DMBR is that filtration can be carried out by only gravity. Thus, a suction pump is not necessary to achieve high flux values, even up to 80 $L/m^2.h$ (Wu et al., 2005), which makes DMBR an important alternative for small wastewater treatment systems in rural areas, where low cost is required (Ren et al., 2010). Although Kiso et al. (2005) applied higher initial water heads, i.e. 0.5-2 m, many researchers were able to operate DMBRs at much lower water heads (<0.5 m). Fan and Huang (2002) even reported a DMBR operating at less than 0.05 m water head.

The operation period of DMBRs can be divided into three stages such as DM layer formation, filtration and backwash (Chu et al., 2008). Although the DM layer can easily retain sludge particles inside the reactor and achieve high SS removal, the effluent quality obtained at the initial stages of filtration is generally poor and the effluent can contain high SS concentrations due to the passage of sludge flocs through the relatively large filter pores. However, once the DM layer is formed, a very high effluent quality comparable to MF/UF membranes can be achieved. In most of the studies, a DM layer was generally formed rapidly in the initial stage of filtration ensuring a high SS removal efficiency for the rest of the filtration period. Therefore, as a practical solution, initial filtrates can be returned back to the reactor in order to ensure a high effluent quality (Fan and Huang, 2002; Kiso et al., 2005; Wang et al., 2006; Chu et al., 2008).

Table 2.2. Biological treatment performance of DMBRs for waste(water) treatment.

Operation Mode/Membrane Configuration	Volume (L)/ Temperature (°C)	Substrate	MLSS (g SS/L)	Organic Loading Rate (kg COD/m^3.d)	COD Removal (%)	Reference
Submerged/Flat sheet	17/n.a[a]	Synthetic Municipal WW [b]	6-8	n.a.	>80 (BOD)	Kiso et al. (2000)
Submerged/Flat sheet	140/27	Municipal WW	7.5	0.7-2.6	84.2 34.3 (by dynamic membrane)	Fan and Huang (2002)
Submerged/n.a.	470/n.a.	Municipal WW + Glucose	1.8	0.7	91.6	Seo et al. (2002)
Submerged/Flat sheet	10.5/20	Synthetic	3.5-5	295 (g TOC/m^3.d)	83-93 (TOC)	Alavi Moghaddam et al. (2002)
Submerged/Flat sheet	15/n.a.	Activated sludge (Municipal)	3-9	n.a.	Sludge volume reduction ratio: 85-95	Park et al. (2004)
Submerged/Flat sheet	20/n.a.	Synthetic + Municipal WW	6-9	10	87.4 12.6 (by dynamic membrane)	Wu et al. (2005)
Submerged/Flat sheet	30/14-17	Municipal WW	4-7	0.2-0.35 (kg BOD$_5$/m^3.d)	>80	Fuchs et al. (2005)
Submerged/Flat sheet	16/25	Synthetic	3-5.5	n.a.	n.a.	Kiso et al. (2005)
Submerged/n.a.	28/n.a.	Municipal WW	n.a.	14-25	>95	Zhi-Guo et al. (2005)
Submerged/Flat sheet [a]	14/9-13	Municipal WW	6	0.8-1.4	81	Chu and Li (2006)
Submerged/Flat sheet (Pre-coated membrane)	12/25	Municipal WW	n.a.	n.a.	97.09 10.59 (by dynamic membrane)	Ye et al. (2006)
Side-stream/Tubular (Pre-coated membrane)	20/30	Synthetic Textile WW	4.5	n.a.	>98	Li et al. (2006)
Submerged/Flat sheet	10/ambient	Municipal Sludge	25-32	n.a.	93.5	Wang et al. (2006)
Submerged/Flat sheet and tubular	12/20	Synthetic Municipal WW	2.5-3.5	8.3	93-98 (TOC)	Seo et al. (2007)
Submerged/n.a.	8/n.a.	Distillery WW [c]	10-12	3-5.7	23-41	Satyawali and Balakrishnan (2008)
Submerged/Plate frame (Pre-coated membrane)	35/16-33	Municipal WW	11	3.2-16.6	>90 <40 (by dynamic membrane)	Chu et al. (2008)
Submerged/Plate frame	7.5/n.a.	Municipal WW	3	10.4-16.8	78	Liu et al. (2009)
Submerged/Filter bag	15/ambient	Household WW	4-12	0.36-0.39 (kg BOD$_5$/m^3.d)	85.5-89.3	Ren et al. (2010)

[a] n.a.: Not available; [b] WW: Wastewater; [c] Anaerobic treatment was applied before the MBR

Table 2.3. Membrane performance of DMBRs for waste(water) treatment.

Operation Mode/Membrane Configuration	Support Material	Pore Size (µm)	TMP (kPa)	Flux ($L/m^2.h$)	Gas sparging rate (L/h)	Effective filtration area (m^2)	Reference
Submerged/Flat sheet	Nylon mesh	100	Driven by water level difference	21-32	120-480	0.11	Kiso et al. (2000)
Submerged/Flat sheet	Dacron mesh	100	Driven by water level difference	14.8-33.3	120 (m/h)	2.7	Fan and Huang (2002)
Submerged/Flat sheet	Fabric filter	n.a[a]	Driven by water level difference	16.7	n.a.	2	Seo et al. (2002)
Submerged/Flat sheet	Non-woven	50-200	3-6	42-125	120-600	n.a.	Alavi Moghaddam et al. (2002)
Submerged/Flat sheet	Mesh	100, 200, 500	Driven by water level difference	n.a.	Without gas sparging	1.078	Park et al. (2004)
Submerged/Flat sheet	Non-woven fabric	n.a.	Driven by water level difference	50-80	n.a.	n.a.	Wu et al. (2005)
Submerged/Flat sheet	Woven nylon fabric	30	0.3-1	50-150	120-480	0.1	Fuchs et al. (2005)
Submerged/Flat sheet	Mesh	100	Driven by water level difference	42-625	n.a.	0.016	Kiso et al. (2005)
Submerged/n.a.	Non-woven	3-5	-	20	1200	n.a.	Zhi-Guo et al. (2005)
Submerged/Flat sheet	Filter-cloth	n.a.	Driven by water level difference	17-21	< 150	0.066	Chu and Li (2006)
Submerged/Flat sheet (Pre-coated membrane)	Terylene filter cloth	56	0-42	18.6	100	0.09	Ye et al. (2006)
Side-stream/Tubular (Pre-coated membrane)	Ceramic	2	1000-2000	6.3-12.6	Cross-flow velocity: 2 m/s	0.0672	Li et al. (2006)
Submerged/Flat sheet	Nylon mesh	100	Driven by level difference	9.7-31.3	n.a.	0.016-0.0518	Wang et al. (2006)
Submerged/Flat sheet Submerged/Tubular	Non-woven Non-woven	n.a. n.a.	up to 20 up to 15	20.8-72 20.8-41.6	600 600	0.052 0.035	Seo et al. (2007)
Submerged/n.a.	Nylon mesh	30	0.6-8.8	0.8-0.9	156-270	0.05	Satyawali and Balakrishnan (2008)
Submerged/Plate frame (Pre-coated membrane)	Steel mesh	74	40	8.8-130	18.58 (m/h)	0.084	Chu et al. (2008)
Submerged/Plate frame	Silk	100	n.a.	4-10	n.a.	0.01	Liu et al. (2009)
Submerged/Filter bag	Non-woven fabric	100	Driven by water level difference	~ 5	n.a.	0.29	Ren et al. (2010)

[a] n.a.: Not available

Most of the investigations on DMBR were focused on the treatment of municipal sewage or low to medium strength synthetic wastewater. DMBRs were reported to exhibit a similar biological removal performance of pollutants compared to their counterparts equipped with MF/UF membranes. Kiso et al. (2005) obtained high COD, BOD, TOC and total nitrogen removal efficiencies in a sequencing batch reactor equipped with mesh filter. Fan and Huang (2002) achieved 84% and 93% average removal efficiencies for COD and ammonia, respectively. The effectiveness of DMBR to retain and enrich the slow growing nitrifiers was reported by Chu and Li (2006). Similarly, Kiso et al. (2000) achieved complete nitrification in a mesh filtration bioreactor.

It is also possible to obtain high phosphorus removal efficiency in DMBRs, although biological phosphorus removal depends on the substrate composition and system configuration. Ren et al. (2010) obtained satisfactory phosphate removal efficiency by using an innovative design MBR equipped with non-woven fabric filter. Moreover, Seo et al. (2007) could also achieve high total phosphorus removal efficiency (85%) by adding 20 mg/L poly-aluminum chloride in a DMBR.

The rejection of some high molecular weight organic matter by the DM layer in an DMBR was reported by Li et al. (2006). They concluded that the SMP accumulated in the reactor at the start-up period and were degraded into low molecular weight compounds after acclimatization of the biomass during long-term operation. Wu et al. (2005) reported that the supernatant, which was obtained by centrifugation of reactor mixed liquor, of a submerged DMBR mainly consisted of hardly biodegradable organic material such as SMP, small particles, and colloids.

The biological processes taking place in the DM layer cannot be ignored since the mass of biomass accumulated on the cake layer can be considerably high. Sludge accumulated on the cake layer can be regarded as a biofilm in which hydrolysis, carbon removal, ammonification, nitrification and denitrification processes can occur depending on the environmental conditions such as availability of oxygen, substrate and nutrients. Fan and Huang (2002) attributed the decrease in DOC concentration in the permeate of a DMBR system to the biological degradation process in the DM layer. Wu et al. (2005) reported elevated concentrations of ammonia in the permeate in comparison to the bulk liquid in a DMBR indicating that the organic nitrogen was degraded to ammonia while passing through the cake layer. They have shown that oxygen was depleted in the first 1.5-2.5 mm of the DM layer.

Virus removal is one of the important advantages of MBR technology over conventional treatment systems. By using MF/UF membrane separation systems, virus particles can be physically retained (Sano et al., 2006; Sima et al., 2011). Sima et al. (2011) determined that high virus removal efficiency (3.3-6.8 log10 units) could be obtained by a full-scale submerged MBR process treating municipal wastewater. Although the pore size of the used membrane was larger than the diameter of virus particles (~30 to 40 nm), high virus removal efficiencies could be achieved (Sano et al., 2006; Sima et al., 2011). This may be explained by the formation of a dynamic cake or gel layer on the membrane surface with a smaller pore size than the real membrane.

Only one application for the treatment of industrial wastewaters by DMBRs was reported so far. Satyawali and Balakrishnan (2008) investigated the treatability of anaerobically treated distillery wastewater in a DMBR equipped with a nylon mesh filter. Although the DMBR provided excellent SS removal, the COD removal efficiency was significantly lower (22-41%) compared to other studies conducted with DMBRs. This was explained by the highly recalcitrant characteristics (BOD/COD: 0.14) of the treated wastewater. DMBR technology has been used also for aerobic digestion of excess municipal wastewater sludge (Wang et al., 2006). High SS degradation performance, up to 84%, with a low SS concentration in the effluent (<60 mg/L) was obtained in comparison to the conventional aerobic digesters.

2.3.2.2 Anaerobic Dynamic Membrane Bioreactors (AnDMBRs)

The use of AnDMBR technology has been tested for the treatment of wastewater sludge, solid waste and wastewater. Table 2.4 and Table 2.5 present the performance results of AnDMBR applications in the literature both regarding biological treatment and membrane aspects, respectively.

The utilization of DM for primary sludge treatment was first applied by Pillay et al. (1994) by using woven fiber in a side-stream AnDMBR. They observed a significant enhancement in the performance of an anaerobic digester by decoupling HRT from SRT. An economical evaluation confirmed the feasibility of the AnDMBR system over the conventional digester system (Pillay et al., 1994).

Walker et al. (2009) used an MBR including a nylon woven mesh membrane as the first stage of a two-stage (AnDMBR+Anaerobic Filter) anaerobic process for the digestion of a synthetic municipal waste. Continuous filtration was sustained without the replacement of meshes for 85 days during the study. Ho et al. (2007) reported that the AnDMBR system could be operated at low TMP and cross-flow velocity in order to maintain a DM layer for efficient particle removal from municipal wastewater. It was also concluded that a non-woven filter could be an alternative for MF.

SFDM was applied in AnMBR with submerged and side-stream filtration modules by Jeison et al. (2008) under thermophilic and mesophilic conditions for the treatment of synthetic wastewater composed of a mixture of volatile fatty acids and nutrients. They used woven and non-woven materials as the support layer. However, low flux and unstable operation under both temperature conditions were obtained, which was in contrast with those reported for DMBRs. This difference was attributed to the different floc morphology and particle size distribution of anaerobic sludge and thus to the high filtration resistance even for a very thin cake layer.

An et al. (2009) determined that EPS extracted from the cake layer in an AnDMBR treating municipal wastewater consisted mainly of protein-like and humic acid-like substances. The results showed that the supporting layer (non-woven fabric) surface was covered with a rough and dense layer consisting of mainly protein and inorganic elements such as Mg, Al, Ca, Si, and Fe which could function as a bridge between biopolymers and deposited cells to form a dense cake layer. Similar observations were made by Zhang et al. (2011).

Ho et al. (2007), Jeison et al. (2008) and An et al. (2009) could only achieved low fluxes (≤5 $L/m^2.h$) which limit practical engineering applications of AnDMBRs. However, Zhang et al. (2010) investigated the formation process of the DM layer at the upper part of a UASB reactor and reported a high flux of 65 $L/m^2.h$ in an AnMBR treating municipal wastewater at low temperatures (10-15 ^oC). They mentioned that the filtration resistance of the cake layer was much higher than the intrinsic resistance of the mesh and the resistance of pore-clogging which is consistent with the studies of Jeison et al. (2008) and Waeger et al. (2010). The cake layer played a major role in the filtration resistance increase due to both thickness increase and compaction. Zhang et al. (2011) further characterized the cake layer and identified a double-layered structure, i.e. a loosely bound outer layer and a tightly bound internal layer. It was shown that especially fine particles in the bulk sludge attached to the support material surface in comparison to the large particles since larger particles are more subjected to shear induced diffusion and inertial lift. Microbial activity in the membrane fouling layer was found lower in comparison to the bulk sludge and different communities were observed in the fouling layer and bulk sludge. This result was attributed to the suppressed mass transfer in the cake layer.

Table 2.4. Biological treatment performance of AnDMBRs for waste(water) treatment.

Operation Mode/ Membrane Configuration	**Volume (L)/ Temperature (°C)**	**Substrate**	**MLSS (g SS/L)**	**Organic Loading Rate (kg COD/m^3.d)**	**COD Removal (%)**	**Reference**
Submerged/Flat sheet	3/30	Synthetic	17.5	n.a.	n.a.	Jeison et al. (2008)
Side-stream/Tubular	1800/n.a.[a]	Municipal Sludge	55	n.a.	n.a.	Pillay et al. (1994)
Side-stream/Tubular (Pre-coated membrane)	-/25	Synthetic Municipal WW[b]	9.6-12.5	n.a.	>90	Ho et al. (2007)
Submerged/Flat sheet	3/50	Synthetic	7.2	n.a.	n.a.	Jeison et al. (2008)
Side-stream/Flat sheet	3/30	Synthetic	25.6	n.a.	n.a.	Jeison et al. (2008)
Submerged/Cylindrical	1.5/n.a.	Synthetic Municipal Waste	n.a.	3.75 (g VS[d]/L.d)	n.a.	Walker et al. (2009)
Submerged/Tubular	12.9/15-20	Municipal WW	n.a.	2.36	70	An et al. (2009)
Submerged[c]/Flat sheet	45/10-15	Municipal WW	n.a.	n.a.	57.3	Zhang et al. (2010)

[a] n.a.: Not available; [b] WW: Wastewater; [c] Submerged on the top of an upflow anaerobic sludge bed (UASB) reactor; [d] VS: Volatile solids

Table 2.5. Membrane performance of AnDMBRs for waste(water) treatment.

Operation Mode/ Membrane Configuration	Support Material	Pore Size (µm)	TMP (kPa)	Flux ($L/m^2.h$)	Gas sparging rate (L/h)	Effective filtration area (m^2)	Reference
Side-stream/Tubular	Woven fiber	n.a[a]	200	50	Cross-flow velocity: 2 (m/s)	n.a.	Pillay et al. (1994)
Side-stream/Tubular (Pre-coated membrane)	Non-woven (Polypropylene)	12	6.9-20	4-12	Cross-flow velocity: 0.2 (m/s)	0.015	Ho et al. (2007)
Submerged/Flat sheet	Mesh	20	n.a.	0.5-3	n.a.	0.0188	Jeison et al. (2008)
Submerged/Flat sheet	Non-woven	30	n.a.	3	n.a.	0.0188	Jeison et al. (2008)
Side-stream/Flat sheet	Mesh	15	n.a.	0.5-3	n.a.	0.0292	Jeison et al. (2008)
Submerged/Cylindrical	Woven nylon mesh	30, 100, 140	n.a.	44	Without gas sparging	n.a.	Walker et al. (2009)
Submerged/Tubular	Non-woven fabric[b]	0.64	up to 30	5	Without gas sparging	0.98	An et al. (2009)
Submerged[c]/Flat sheet	Dacron mesh	61	up to 25	65	Without gas sparging	n.a.	Zhang et al. (2010)

[a] n.a.: Not available; [b] Polythylene terephthalate (PET); [c] Submerged on the top of a UASB reactor

2.4 Factors Affecting the Performance of DMs

2.4.1 Materials

Selection of an appropriate support material enabling the formation of a dynamic layer is a critical step for DM applications. The support material should have an appropriate pore size in order to form and retain the membrane forming material on its surface (Igawa et al., 1977); it should be strong enough to withstand the required pressures for a long time and should be cheap.

The pore size affects dynamic layer formation rate and permeate flux. Therefore, the pore size of support material and particle size of DM forming material should be considered together in order to get the best separation performance. Since the pore size of the support materials is wider than the conventional MF/UF membranes, the effluent quality at the first stages of filtration will be lower due to the passage of particles through the material pores. Kiso et al. (2000) investigated the filtration properties and effluent quality of mesh support material at different pore sizes (100, 200, 500 μm). They found that meshes having a pore size of 100 μm provided the best results in terms effluent quality and effectively rejected activated sludge flocs.

Jeison et al. (2008) showed the impossibility to build a cake layer on meshes with pore sizes over 60-70 μm for anaerobic applications using volatile fatty acids as feed. Zhang et al. (2010) achieved to have a DM over a Dacron mesh with a pore size of 61 μm. The filtration performances of non-woven material and a conventional hollow fiber membrane as support layer in submerged DMBRs treating municipal wastewater were compared by Zhi-Guo et al. (2005). They obtained similar effluent qualities for both filtration processes. They determined that the pore size of non-woven filters had little effects on the organic carbon removal, probably due to the formation of a dynamic layer on the non-woven filter surface. They concluded that fouling of non-woven filter was mainly caused by internal fouling. As a result, non-woven material with a smaller pore size might exhibit a greater advantage in comparison to the one with a larger pore size.

Specific weight representing the density of the fibers in non-woven materials can play an important factor on the filtration properties of these materials due to its effect on material porosity. A light fabric filter (e.g. 35 g/cm^2) and low pressures were found desirable for long time filtration and high flux with non-woven fabric filter (Seo et al., 2002). However, sludge accumulation due to the penetration and entrapment of the fine particles in the non-woven fabric filters should always be taken into consideration on the long-term operation. Once entrapped in the fiber matrix, it is difficult to remove the particles from the filter, subsequently increasing membrane resistance. Therefore, mesh filters with larger pore size and regular pore distribution can represent an alternative material for non-wovens (Kiso et al., 2005).

Support material is also important in physical DM applications. Diaper et al. (1996) compared different support materials and found that stable DM layers can only be obtained on ceramic, carbon, and extruded polymer supports. Besides support materials, forming material is also one of the significant factors that affects the performance of DMs. The research on physical DM filtration has commonly focused on the properties of the dynamic layer forming materials and dynamic layer formation conditions. Pore size (Nakao et al., 1986; Al-Malack and Anderson, 1996; Chen and Chiang, 1998; Yang et al., 2011), formation pressure (Igawa et al., 1977; Zhao et al., 2006), cross-flow velocity (Horng et al., 2009; Knyazkova and Kavitskaya, 2000; Zhao et al., 2006), pH (Nakao et al., 1986; Ohtani et al., 1991; Matsuyama et al., 1994; Rumyantsev et al., 2000), and concentration of forming material (Matsuyama et al., 1994; Al-Malack and Anderson, 1997a; Na et al., 2000; Noor et al., 2002; Zhao et al., 2005; Horng et al., 2009; Yang et al., 2011) have been identified as effective parameters on DM properties and performance.

Various formation pressures between 0.005-100 bars were applied for different purposes in various DM studies (Table 2.1). For RO applications, the applied formation pressure is generally more than 10 bars, and some researchers reported up to 90 bars (Igawa et al., 1977). For UF and MF applications, the formation pressure is at low values, i.e. below 10 bars. Zhao et al. (2006) determined that an increase in formation pressure enhanced the convective flow of particles to the membrane, thus enhanced the polarization, deposition of particles, and mass of dynamic layer on the support layer. This resulted in an increase in TOC rejection efficiency.

The thickness of the dynamic cake layer on the support material is related to the cross-flow velocity and applied flux (Horng et al., 2009). Cross-flow velocities between 0.006-9.1 m/s have been applied during DM layer formation in various applications (Table 2.1). Knyazkova and Kavitskaya (2000) determined that water flux in a coated membrane layer increased linearly with an increase in the cross-flow velocity from 0.4 to 2.7 m/s. In contrast, Zhao et al. (2006) found that forming a satisfactory DM layer is difficult at high cross-flow velocities and increasing the cross-flow velocity resulted in erosion of the dynamic layer and decrease in TOC rejection.

pH is generally reported as a parameter that affects the particle size of the DM forming material, thus dynamic layer formation time. Rumyantsev et al. (2000) determined that the particle size of Zr particles increases with the pH level of the suspension. The zeta-potential of the Zr particles shows a negative charge in alkaline solution, and a positive charge in acid solution. Electrical repulsion is the weakest and the cohesion forces between the particles are the strongest at the isoelectric point (Ohtani et al., 1991). Therefore, the DM layer obtained at the isoelectric point had the smallest porosity and thus the lowest flux (Nakao et al., 1986; Matsuyama et al., 1994).

Concentration of the forming material affects the separation efficiency especially by changing the dynamic layer thickness. It was determined that the thickness of dynamic layer increased at higher concentration of the membrane forming material (Matsuyama et al.,1994; Al-Malack and Anderson, 1997a; Na et al., 2000; Horng et al., 2009; Yang et al., 2011). Zhao et al. (2005) showed that the dynamic cake mass increased from 26 to 33 g/m^2 with a magnesium hydroxide concentration from 250 to 1000 mg/L. Non-stable dynamic layers were obtained using hydroxide concentrations below 250 mg/L. A thicker DM layer may provide a better rejection capacity (Yang et al. 2011; Noor et al., 2002).

2.4.2 Sludge Properties

2.4.2.1 Bulk Sludge

Microbial floc characteristic is an important parameter affecting both the permeability and effluent quality of DM systems. The filterability properties of the microbial flocs in membrane systems is a function of the operation conditions such as MLSS concentration, SRT, F/M and applied shear rate (aeration intensity, cross-flow velocity, etc.) (Judd, 2006).

Chu and Li (2006) postulated that higher sludge concentrations could positively contribute to DM formation, however, elevated permeate SS concentrations and lower flux were observed. Alavi Moghaddam et al. (2002) obtained good effluent quality at both MLSS concentrations of 5000 mg/L and 3500 mg/L in 4 months experiments. However, the flux was significantly lower (42 L/m^2.h) at high MLSS concentration compared to the lower MLSS concentration (125 L/m^2.h). Pillay et al. (1994) also determined that an increase in sludge concentration resulted in a significant decrease in flux following a semi-log relationship.

Liu et al. (2009) found that a DMBR could be continuously operated for several months at low MLSS concentration (3000 mg/L) without membrane cleaning. Interestingly, the time needed for the complete formation of the DM layer at high MLSS concentrations (7540 mg/L) was much longer than that needed at low MLSS concentration. Based on the flux data obtained under constant pressure, they explained the formation mechanism of DM by four classic filtration laws (cake filtration, complete blocking, intermediate blocking and standard blocking).

Specific cake resistances of mesophilic and thermophilic sludge were found to be 6.3×10^{14} and 3.7×10^{14} m/kg (Jeison et al., 2008) for AnDMBR systems, respectively, which are one to two orders of magnitude higher than those observed for aerobic MBRs (Ahmed et al., 2007; Wang et al., 2007). Stable operation at moderate to high fluxes that were reported for DMBRs (Kiso et al. 2000; Fan and Huang, 2002) contradicted with the unstable operation results with low fluxes obtained in AnDMBRs (Jeison et al., 2008). This can be attributed to different floc morphology and particle size distribution of the bulk sludge between DMBRs and AnDMBRs.

Zhang et al. (2010) implied the importance of EPS and SMP accumulation in DM formation as polymeric interactions played an important role in the enhancement of sludge adhesion. SMP and EPS macromolecules are readily attached to the support material by permeation drag. The sludge particles with higher SMP and EPS content preferentially adhere to the surface, and then other particles can be retained by the DM permeation drag. An et al. (2009) also implied the role of EPS in blocking the membrane pores and depositing on membrane surfaces to form a fouling layer. On the contrary, Zhang et al. (2011) compared the SMP and EPS contents in the dynamic layer and the bulk sludge and found significantly lower values in the dynamic layer.

2.4.2.2 Dynamic Membrane Layer

The DM layer plays an essential role in the rejection of particulate matter in DMBRs (Chu and Li, 2006). According to the findings of Fan and Huang (2002), DM layers consist of two sub-layers, a cake layer and an underlying gel layer. The cake layer is mainly composed of sludge flocs that are attached loosely. Therefore, it could be easily removed by air scouring. However, the gel layer, which was mainly composed of EPS, sticking tightly to the filter surface, could hardly be flushed. They reported that a balanced DM layer could be formed more rapidly after a physical cleaning event in comparison to its initial formation. After cleaning, the gel layer which adhered tightly to the support material surface created an optimum initial surface for the cake layer accumulation. Moreover, the gel layer was reported to play an important role in the dynamic MBR rejection capability of the fine particles by its similar structure to the conventional membranes. On the other side, the cake layer achieved two important functions: to improve the effluent quality by rejecting most of the coarse flocs and to prevent the gel layer from direct interaction of the large particles. Moreover, microorganisms in the cake layer may contribute to organic carbon conversion during permeation through the cake layer. Overall results showed that the cake layer comprised most of the filtration resistance of the DMBR and periodical bottom aeration was adequate for cleaning the dynamic layer (Fan and Huang, 2002; Kiso et al., 2005).

The structural properties such as density, porosity and compaction of the dynamic layer play a key role on the achievable fluxes and the pressure losses. Sludge cake density is directly related to the sludge cake resistance. A cake layer with a low density can break up due to the insufficient durability, whereas at higher sludge cake densities rapid increase in filtration resistance can be observed.

2.4.3 Operation Conditions

Alavi Moghaddam et al. (2002) examined the filtration characteristics and effluent quality of a DMBR at different SRTs of 10 days, 30 days and infinite (no sludge wasting except for sampling). The reactor operated at infinite sludge age showed the lowest performance in terms of TOC removal and filtration. The authors mentioned that biomass developed in this reactor was sticky and resulted in a thick biomass layer on the filter surface. As a result of the average sludge concentration being 2-3 times higher than the other reactors, the F/M ratio was very low. Therefore, the poor filtration characteristics may originate from accumulating bacterial decay products at the high sludge age. Fuchs et al. (2005) also indicate that increasing the F/M ratio results in an increased number of intermediate and large size flocs, whereas small flocs decrease. This resulted in a better effluent quality in terms of SS concentration. On the other hand, Ahmed et al. (2007) determined that specific cake resistance decreased as SRT increased (from 20 to 60 days) and they suggested that a lower ratio of F/M provided a reduction in membrane bio-fouling. They also observed that bound EPS content, one of the most important factors related to the membrane fouling, decreased at longer SRTs (above 60 days) when MLSS concentration became higher than 5000 mg/L. They attributed the reduction of bound EPS to a low formation rate of microbial substances or an increase in EPS degradation as substrate by microorganisms at a low F/M condition.

High aeration intensity can sometimes disturb the DM layer, which is indispensible for effective solids separation, and impair the effluent quality. Kiso et al. (2000) determined that an increase in the aeration intensity led to higher effluent turbidity in the mesh filtration. On the other hand, Alavi Moghaddam et al. (2002) reported that aeration intensity had no significant effect on the effluent SS concentration and turbidity and claimed that thin biomass layers on the filter surface could not be affected by shear stress supplied by the increase of aeration intensity.

In contrast to the study of Chu and Li (2006), Fuchs et al. (2005) reported that sludge accumulation on the membrane surface was not affected by MLSS concentration, whereas aeration intensity played a significant role on it. Higher shear stress by increased aeration intensity reduced the thickness of the secondary filter layer and thus, made the retention of SS less effective. Moreover, intensive aeration and high shear rate can manipulate the particle size distribution in the bioreactor by disturbing the structure of large flocs producing fine flocs. Kiso et al. (2000) operated the DMBR under continuous aeration conditions without clogging for 2-5 months. On the other hand, Satyawali and Balakrishnan (2008) determined the critical flux of a DMBR equipped with 30 μm nylon mesh as 3.9 $L/m^2.h$ which is significantly lower than the conventional membranes used in aerobic MBRs. This result was attributed to the low aeration intensity used in the study.

Satyawali and Balakrishnan (2008) showed that the average floc size in a DMBR decreased from 178 μm to 47.1 μm during the operation. This phenomenon was similar to the MBRs where the floc size tends to decrease due to the high shear rate applied by the aeration and recirculation pumps. The accumulation of fine material on the support material can produce a less porous DM layer, decreasing the attainable flux. Chu and Li (2006) reported that the average particle size on a filter cloth surface was much lower than that of bulk liquid in a bioreactor, which indicates that smaller flocs are more likely to accumulate in the cake layer, which is similar to conventional MBRs.

2.4.4 Configuration and Operation Mode

Different membrane configurations of submerged non-woven fabric filters in a DMBR were compared by Seo et al. (2007). They used flat sheet (vertical) and tubular (vertical and horizontal) membrane modules. The thickness of sludge layer formed on the tubular filter was found more than twice of that formed on the flat sheet filter. Although there was not much difference in particle size and shape of the sludge flocs, the pressure increase in the tubular module was more stable compared to the flat sheet module. Similar filtration pressures were observed with horizontally and vertically positioned tubular modules. Stable and high organic pollutants removal was achieved for all different modules used in the study. Jeison et al. (2008) did not observe any significant difference between the trials conducted with submerged and external AnDMBR configurations.

2.5 Cleaning Methods for Dynamic Membrane Applications

Fouling is one of the most important menaces plaguing any filtration process. Different cleaning methods can be applied to control fouling. Cleaning of a fouled membrane is still a problem for conventional MBRs, and it is often costly and a troublesome task, particularly for full-scale submerged MBRs (Fan and Huang, 2002).

Flux decline due to fouling, and membrane cleaning or replacement play a key role in the overall economics of membrane processes. Thus far, only limited studies in literature are available that directly focus on DM cleaning processes. DM forming material, and chemical resistance of the filters determine the required cleaning process and its frequency. In submerged systems, the removal of cake layer is generally done by bottom aeration or biogas sparging (Fan and Huang, 2002; Jeison et al., 2008).

Al-Malack and Anderson (1997b) investigated various cleaning techniques including acid cleaning, cleaning with tap water and air scouring for physical DM processes. They used multifilament polyester yarn woven in the form of interleaved fabric as a support material and a DM of MnO_2. Results showed that none of these methods provided a feasible cleaning without altering the DM performance. Brushing was suggested as the best way of cleaning the DM layer. Cai et al. (2000) suggested HCl solutions to regenerate MnO_2 DMs. At low pH, MnO_2 is reduced to Mn^{2+}, facilitating removal of the MnO_2 dynamic layer. Increase in HCl concentration decreased regeneration time.

2.6 Conclusions

A porous and compressible layer formation, which can serve as a barrier that limits the passage of fine particles through the support layer, is the most important factor for achieving optimal performance in DM processes. The investment and operational costs are expected to be substantially lower than the conventional membrane filtration and competitive with settling tanks, including sand filtration due to the lower costs of the filter modules and the potentially higher fluxes with energetically favorable flux control of dynamic filtration.

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CHAPTER 3

EFFECT OF SUPPORT MATERIAL PROPERTIES ON DYNAMIC MEMBRANE FILTRATION PERFORMANCE

Abstract

A dynamic membrane is defined as a cake layer that forms on a support material, e.g. filter cloth or mesh when the liquid to be filtered includes suspended particles. Therefore, support material properties are considered of prime importance in the performance of dynamic membrane treatment systems. This chapter investigates the effect of support material properties including pore size and structure of the material on dynamic membrane formation and performance. In this concept, a comparative evaluation was made between support materials which have different yarn types. The results showed that high total suspended solids removal efficiency (>98%) could be achieved by using dynamic membrane filtration technology. Mono-monofilament and staple filter cloths were determined as the most appropriate materials in terms of the critical fluxes which were 9.2 L/m^2.h and 17-19 L/m^2.h for mono-monofilament and staple materials, respectively. However, considering the results of more long-term experiments, mono-monofilament filter cloth was found more suitable for cake layer accumulation. Therefore, we postulate that mono-monofilament cloth can be used in dynamic membrane filtration systems as an alternative to conventional membranes in anaerobic membrane bioreactors.

This chapter is based on:

Ersahin, M.E., Ozgun, H., van Lier, J.B., 2013. Effect of support material properties on dynamic membrane filtration performance. Separation Science and Technology, 48(15), 2263-2269.

3 EFFECT OF SUPPORT MATERIAL PROPERTIES ON DYNAMIC MEMBRANE FILTRATION PERFORMANCE

3.1 Introduction

AnMBRs combine the advantages of both membrane separation and anaerobic technology enabling high-quality effluents. Biomass and particulate organic matter can be physically retained inside the AnMBRs providing optimal conditions for further degradation of the organic matter. However, fouling is one of the most important drawbacks of this technology. Cake layer formation on membrane surface is indicated as the key factor limiting the flux in AnMBRs, irrespective of the applied substrate, configuration (submerged or side-stream) or temperature (Jeison and van Lier, 2008; Lin et al., 2009; Waeger et al., 2010; Xie et al., 2010). However, this cake layer, which is also referred to as secondary or DM layer (Ersahin et al., 2012), can also be used as a filter for filtration and retention of particulate material in AnMBRs.

The DM concept and its benefit can be explained by the formation of a cake and/or gel layer over a support material surface, e.g. a mesh or a filter cloth, since the deposited layer can act as a "secondary" membrane prior to the support material (Ersahin et al., 2012). Large suspended solids particles inside the liquor to be filtered can easily accumulate on the surface of a support material and protect it from a rapid pore fouling by decreasing its interaction possibility with small particles like soluble and colloidal organics. The dynamic characteristic of this phenomenon allows the formation and removal of DM layer easily and extends the sustainable filtration time by alleviating the permeability loss (Kiso et al., 2000; Lee et al., 2001; Fuchs et al., 2005; Jeison et al., 2008; Zhang et al., 2010). Different kinds of cheap support materials can be used to create a DM layer and in this way, a low cost filtration process may be possible (Seo et al., 2002; Wu et al., 2005; Chu and Li, 2006). By decreasing filter material costs and generating biogas energy, AnDMBRs may have interesting potentials as a cost effective alternative for waste(water) flows.

It is important to keep the sludge (cake) layer stable on an appropriate support material in order to provide an effective DM layer that achieves sufficient biomass retention (Kiso et al., 2005). Therefore, selection of a support material plays a major role in the performance of dynamic membrane filtration systems. Meshes, woven and non-woven fabrics have been reported as the common support materials used for DM formation (Ersahin et al., 2012). Not only the material type but also properties of the material, i.e. different pore sizes, may affect the performance of the system. For instance, a woven fabric may have different retention capabilities based on the use of monofilament, multifilament, combination of mono and multifilaments or staple yarns in its production. Monofilament yarns are single continuous strands with an even texture which provides a good cake release and easy cleaning. By combining and spinning of monofilament yarns, individual multifilament yarns can be produced. Further, monofilament and multifilament yarns can be combined to form mono-multifilament materials. Staple yarns are not continuous fibers like monofilament or multifilament material; instead, they consist of short individual pieces of fibers which are spun to get a single piece of yarn (Ersahin et al., 2012; Kiso et al., 2005).

The aim of this chapter was to investigate the effect of support material properties on dynamic cake layer formation potential. For this purpose, woven filter cloths with different yarn types including mono-monofilament, mono-multifilament and staple yarns which had different pore sizes including 10 and 40 μm were tested and compared based on their filtration characteristics. The selection of an optimal support material was achieved by investigating various types of support materials for the filtration of anaerobic sludge.

3.2 Material and Methods

3.2.1 Experimental Set-up

A laboratory scale submerged AnDMBR set-up was used in this study (Figure 3.1). AnDMBR set-up consisted of a bioreactor with a volume of 6.8 L and a submerged outside/in flat sheet membrane module with a filtration area of 0.018 m^2. Permeate was collected by a peristaltic pump (Watson Marlow 120U/DV). TMP was measured by a pressure sensor (AE Sensors, ATM -800/+600 mbar) placed on the permeate line. Biogas recycling was provided by a diaphragm pump (KNF, N86 KTDCB) in order to provide mixing inside the bioreactor. The applied biogas recycling flow rate was 2.3 m/h during the experiments. Two baffles were included inside the submerged AnDMBR in order to obtain even distributed mixing conditions. Besides reactor mixing, a second diffuser was placed under the membrane module to provide biogas sparging on the filter surfaces (Figure 3.1). This second diffuser was only used for the long-term experiments. The AnDMBR system was connected to a computer equipped with LabVIEW software (LabVIEW 10.0.1, National Instruments) in order to control all the pumps, and collect and store data. Nitrogen gas was sparged into the reactor in the beginning to remove oxygen in the headspace.

3.2.2 Sludge Source

The sludge used in this study was taken from a pilot-scale upflow anaerobic sludge bed reactor treating black water. Total suspended solids (TSS), volatile suspended solids (VSS), total solids (TS) concentrations and VSS/TSS ratio of the sludge were 20.2±0.08 g/L, 16.9±0.2 g/L, 22±0.3 g/L and 0.84, respectively.

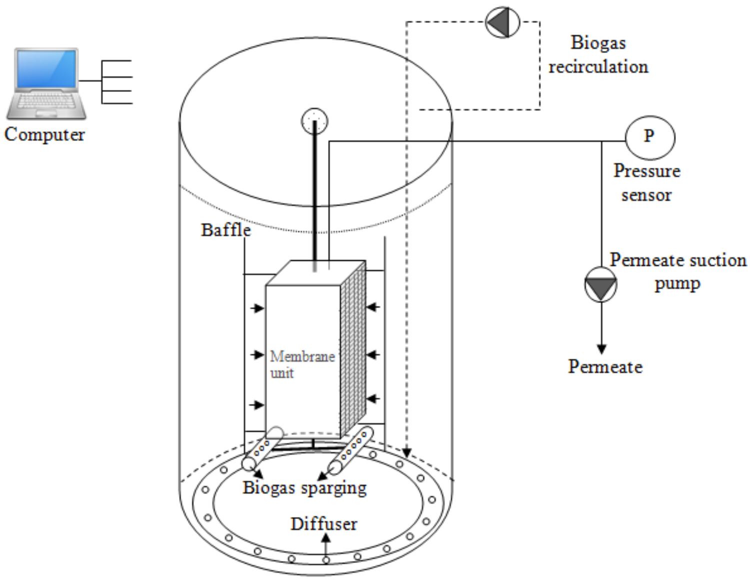

Figure 3.1. Submerged AnDMBR set-up.

3.2.3 Support Materials

Six different support materials (supplied by Lampe BV, the Netherlands) were tested to determine the effects of yarn type and pore size on the filtration performance. The specifications of the materials are given in Table 3.1. All the materials were polypropylene woven fabrics. Mono-monofilament, mono-multifilament and staple filter materials are illustrated in Figure 3.2.

Table 3.1. Characteristics of the support materials.

Support Material Code	Support Material Yarn Type	Average Pore Size (μm)	Tap Water Permeability ($L/m^2.h.bar$)	Resistance (Clean filter) (1/m)	Thickness (mm)	Weight (g/m^2)	Air Permeability ($L/dm^2/min$ at 200Pa)
D1	Mono-monofilament	10	4910	$9.2x10^{10}$	0.6	260	15
D2	Mono-monofilament	40	7830	$5.8x10^{10}$	0.2	260	360
D3	Mono-multifilament	10	5290	$8.5x10^{10}$	0.2	270	60
D4	Mono-multifilament	40	6240	$7.2x10^{10}$	0.4	275	220
D5	Staple	10	5720	$7.9x10^{10}$	0.6	355	12
D6	Staple	40	8660	$5.2x10^{10}$	0.9	370	65

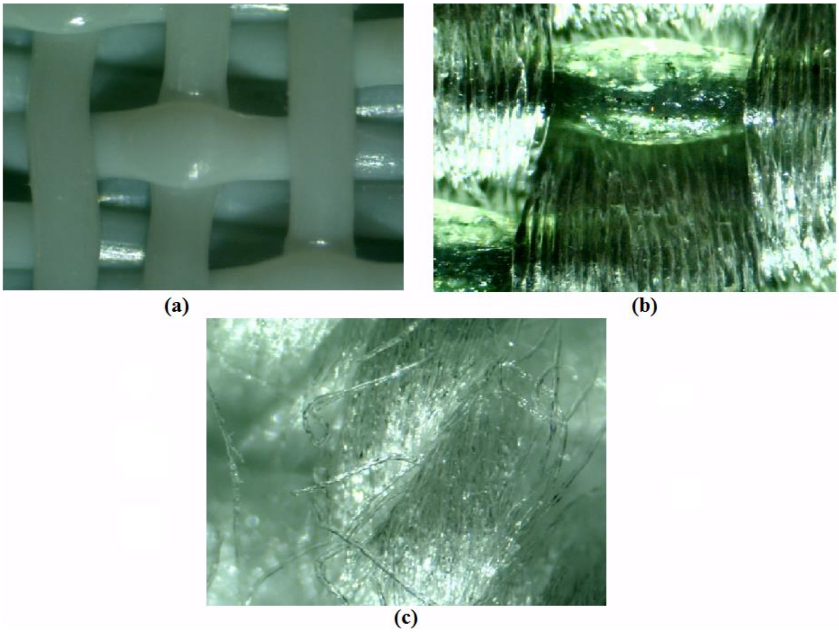

Figure 3.2. Yarn types: (a) mono-monofilament, (b) mono-multifilament, (c) staple filter (magnification 40x).

3.2.4 Experimental Plan

Critical flux test was applied for each support material at the first stage of the study to determine the operational fluxes to be used in the short-term experiments. Following the results of the critical flux analyses, two support materials which had higher critical fluxes in comparison to the other materials were selected. Sub-critical fluxes, i.e. 2 $L/m^2.h$ less than the critical flux of each support material, were applied during the short-term filtration experiments. These experiments were conducted at a TSS concentration of 17.5 g/L in the bioreactor for 2 hours to determine the retention capacity of the dynamic cake layer. TSS concentration in the permeate and TMP were measured during the filtration tests. Following short-term experiments, more long-term experiments (2 weeks) were applied to validate the sustainability of the dynamic membrane filtration with different support materials. In more long-term experiments, the AnDMBR was continuously fed with the same sludge used in short-term experiments. TSS concentration was kept constant during the experiments.

3.2.5 Experimental Analyses

Critical flux was measured according to the step flux method proposed by Le Clech et al. (2003). A flux below which there is no flux decline and no fouling observation over time is defined as critical flux (Field et al., 1995). A flux step height of 2 $L/m^2.h$ and filtration duration of 15 min for each flux step were used in the test. During each step, TMP was recorded with 30 seconds intervals. Filtration resistance was calculated as below (equation 3.1):

$$J = \frac{TMP}{R_T.\mu} \tag{3.1}$$

The flux through the membrane (J) is a function of the TMP, the permeate dynamic viscosity (μ) and the total filtration resistance (R_T).

TSS, VSS and TS parameters were determined following Standard Methods (APHA, 2005). The yarn types of support materials were viewed by an electronic microscope (Bresser Digital LCD Microscope).

3.3 Results and Discussion

3.3.1 Critical Flux Tests

Flux and TMP trends obtained from the critical flux tests are given in Figure 3.3. Comparison of the critical fluxes of different support materials is given in Figure 3.4. For each support material, the TMP value increased tangibly at a certain flux, which was accepted as the critical flux (Cho and Fane, 2002; Satyawali and Balakrishnan, 2008).

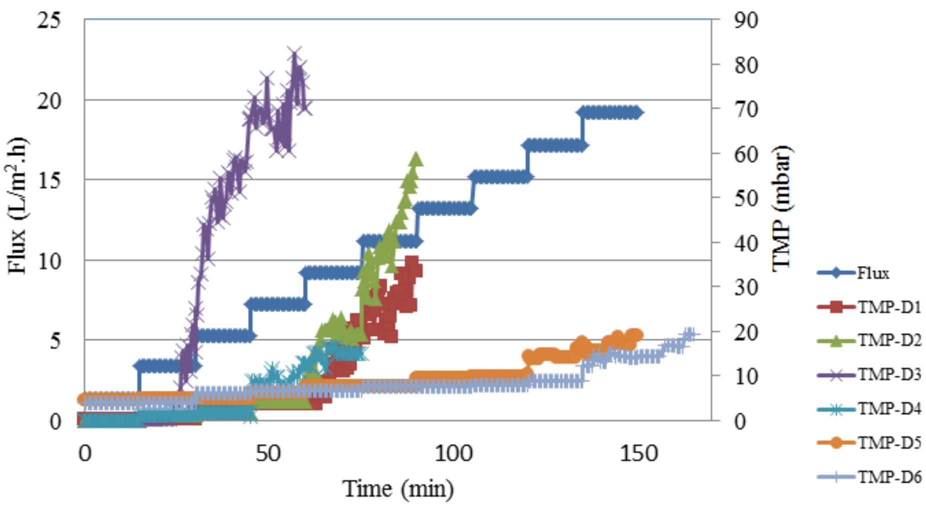

Figure 3.3. Critical flux determination for different support materials.

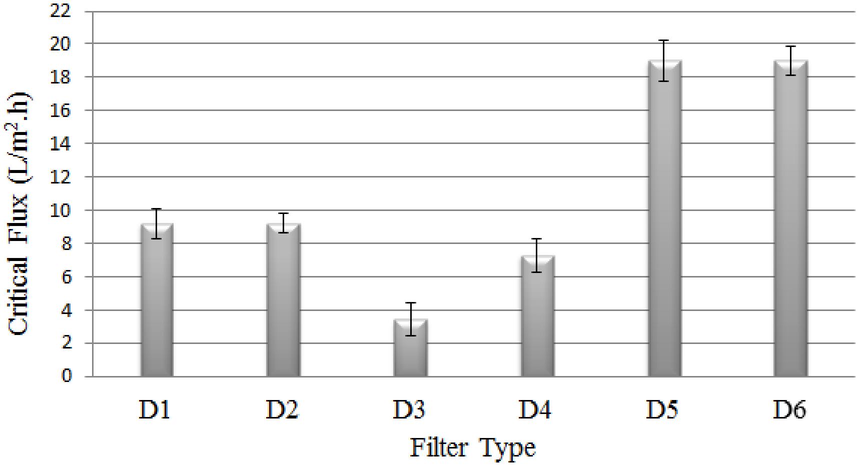

Figure 3.4. Comparison of critical fluxes.

The highest critical flux value was obtained with staple support material for average pore sizes of both 10 and 40 µm. Moreover, differences in critical fluxes based on pore size were smaller in comparison to those based on yarn type. For instance, mono-monofilament and staple support materials have the same critical flux at different pore sizes, whereas the critical flux values of the various yarn types with the same pore size were different. For 10 µm pore size, the critical flux obtained with staple filter was 2.1 times higher than that obtained with mono-monofilament filter, and the critical flux obtained with mono-monofilament filter was 2.7 times higher than that obtained with mono-multifilament filter. Moreover, the critical fluxes obtained with the staple material were higher than those obtained with the mono-monofilament and mono-multifilament filters (Figure 3.4). Our findings confirm the high dependency of critical flux and filterability to yarn type rather than to pore size.

According to the best knowledge of the authors, critical flux data for AnDMBR applications have not been reported till now. However, quite few critical flux data are available for AnMBR and DMBR applications (Table 3.2). Following Table 3.2, the critical fluxes obtained in this study are similar to those obtained with conventional AnMBRs and higher than those obtained in DMBRs. Critical flux data can be used to compare filterability of different support materials and/or to determine a reasonable flux value for the start-up of membrane processes in order to prevent rapid fouling. However, it should be noted that critical flux is not the only parameter determining the long-term stable filtration operation since it is measured in a short period (Cho and Fane, 2002; Martinez-Sosa et al., 2011a). As an example, Satyawali and Balakrishnan (2008) determined that the long-term operational flux could be 76-79% lower than the critical flux in an DMBR treating distillery wastewater.

Table 3.2. Comparison of critical fluxes obtained from different studies.

Application	Filter Material	Critical Flux (L/m^2.h)	Reference
AnMBR	Polysulfone microfiltration membrane (0.2 µm)	5-21	Jeison and van Lier (2006a)
AnMBR	Polysulfone microfiltration membrane	6-17	Jeison and van Lier (2006b)
AnMBR	Polyolefine microfiltration membrane (0.4 µm)	10.5	Achilli et al. (2011)
AnMBR	Polysulfone microfiltration membranes (0.2 µm)	18-21	Vallero et al. (2005)
AnMBR	Polyether sulfone ultrafiltration membrane (0.038 µm)	7	Martinez-Sosa et al. (2011b)
AnMBR	Microfiltration membrane (0.4 µm)	<10	Spagni et al. (2010)
AnMBR	Polyvinylidene fluoride microfiltration membrane (0.3 µm)	13-28	Xie et al. (2010)
ADMBR	Nylon mesh (30 µm)	3.9	Satyawali and Balakrishnan (2008)
ADMBR	Glass fiber filter (n.a.[a])	8.8	Poostchi et al. (2012)
AnDMBR	Polyproplyene woven filter cloth (10-40 µm)	9.2-19	This study

[a] n.a.: Not available.

3.3.2 Short-term Experiments

Following the results of critical flux analyses, filter cloths with mono-monofilament and staple yarn types with 10 µm pore sizes were selected for short term studies since these two materials achieved higher critical fluxes in comparison to mono-multifilament filter cloth. Besides, the small pore size has a higher potential for the development of a cake layer (Jeison et al., 2008). Fluxes of 9 L/m^2.h and 17 L/m^2.h were applied in the short-term experiments for mono-monofilament and staple filter cloths, respectively.

TSS retention capacities of two support materials are given in Figure 3.5. TSS concentrations of the initial permeate obtained from both the mono-monofilament filter cloths were high due to insufficient cake layer formation at the initial period. However, TSS concentration decreased with filtration time. A sharp decrease in permeate TSS concentration with mono-monofilament filter cloth might be an indicator of cake layer formation (Figure 3.5). Following the permeate TSS trend in Figure 3.5, it can be concluded that an efficient dynamic cake layer which retained the particles has been formed within 20 minutes of filtration start. A similar result has been obtained by Seo et al. (2002) with non-woven fabric filter in a submerged DMBR. It may be expected that the dynamic cake layer may be more compact and dense with long-term continuous operation, and therefore, TSS concentration in the permeate would decrease. Considering the short-term data in Figure 3.5, the TSS retention efficiencies were 98.5 and 99.6% for mono-monofilament and staple filters, respectively. There was not a clear breakthrough point in TSS concentration trend observed with the staple material, which indicated that cake layer formation was not the main phenomenon achieving filtration by staple filter cloth. In fact, the staple filter cloth itself played an important role in the retention of TSS instead of the cake layer formation over the filter surface.

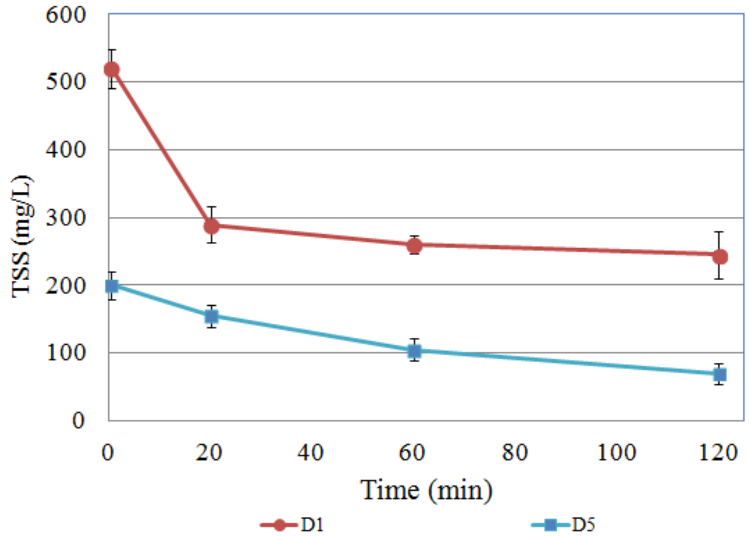

Figure 3.5. Variation in permeate TSS concentration.

Total filtration resistances were monitored during 2 hours experiments (Figure 3.6). As can be seen in Figure 3.6, the filtration resistance obtained by using mono-monofilament material is almost 10 times higher than that obtained by staple material at the end of two hours. Filtration resistance increase can be an indicator to observe the formation of the homogenous cake layer. Meng et al. (2007) determined that cake layer resistance constituted nearly 84%, which was >11 times higher than the contribution of pore fouling resistance, of total filtration resistance in a submerged MBR. A similar result was also reported by Lee et al. (2001). Cake resistances were found between 78-92% of total filtration resistance for sub- and super-critical fluxes with a polyester monofilament filter cloth with a pore size of 30 μm (Poostchi et al., 2012). They reported that the cake layer is the major source of the total resistance for mesh filtration in submerged ADMBRs. Besides, the filtration performance of non-woven material in a submerged ADMBR treating municipal wastewater was investigated by Zhi-Guo et al. (2005). It was found that the filter cloth, which has a nonwoven structure, has a tendency to internal (pore) fouling. Wei Li et al. (2011) determined that cake layer formation on the surface of a nylon mesh filter with a pore size of 90 μm followed a two-stage pattern including a linear increase in filtrate volume over time followed by a nonlinear increase. Cake layer fouling on the mesh filter was found to be reversible in short-term operation in a submerged ADMBR. Considering these results, the observed resistance difference in our study may be attributed to a rapid accumulation of cake layer on the mono-monofilament support material which resulted in a higher filtration resistance mainly consisting of cake layer resistance.

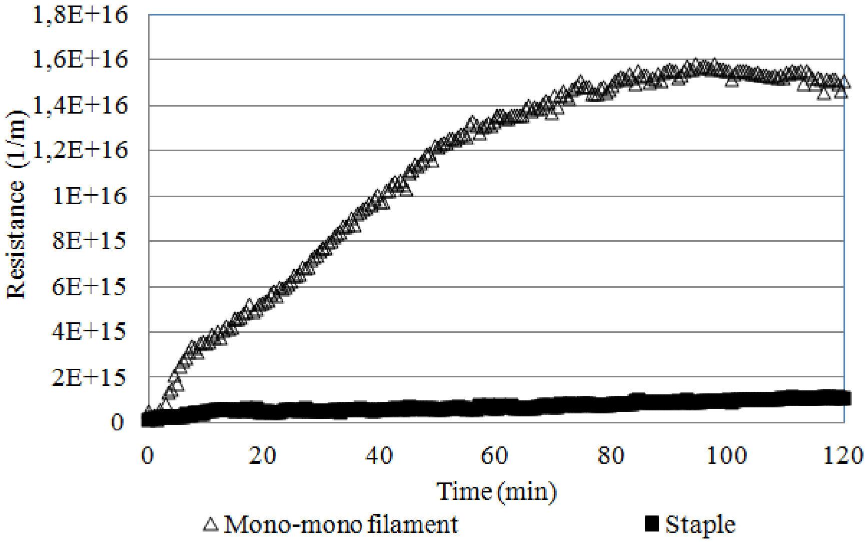

Figure 3.6. Comparison of resistances for mono-monofilament and staple materials.

3.3.3 Long-Term Experiments

In order to further assess a more long-term performance stability of a DM layer on textile cloths, filtration tests using mono-monofilament and staple materials with a pore size of 10 µm were conducted over an extended filtration time of two weeks. With mono-monofilament material, TMP showed a slight increasing trend with concomitant cake layer build-up in the first 10 days and stabilized at about 550 mbar (Figure 3.7). However, stable operation could not be obtained with staple filter cloth. After one day of operation, TMP values exceeded 700 mbar and flux decreased below 1 L/m^2.h. Even after a backwash to recover the permeability, TMP did not stabilize and filtration failed.

The clear breakthrough in permeate TSS concentration (Figure 3.5) and the high filtration resistance (Figure 3.6) observed for the mono-monofilament filters support the usefulness of mono-monofilament filter for a dynamic filtration process. Results obtained from the more long-term experiments also supported this claim. To investigate the reasons for the different behaviors of the two filter cloths with different yarn types, the surfaces of each support material was observed at the end of the filtration operation after physical cleaning. The higher tendency of cake layer accumulation rather than pore blocking on mono-monofilament support material in comparison to staple material is clearly illustrated in Figure 3.8.

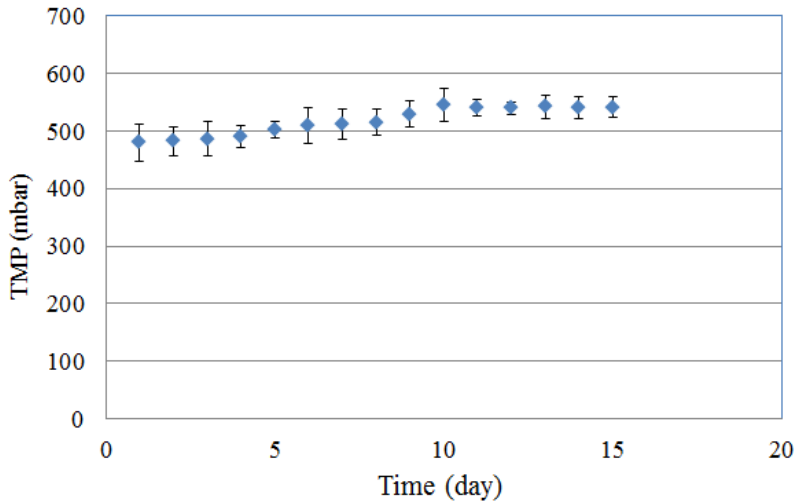

Figure 3.7. TMP profile during long-term filtration with mono-monofilament filter.

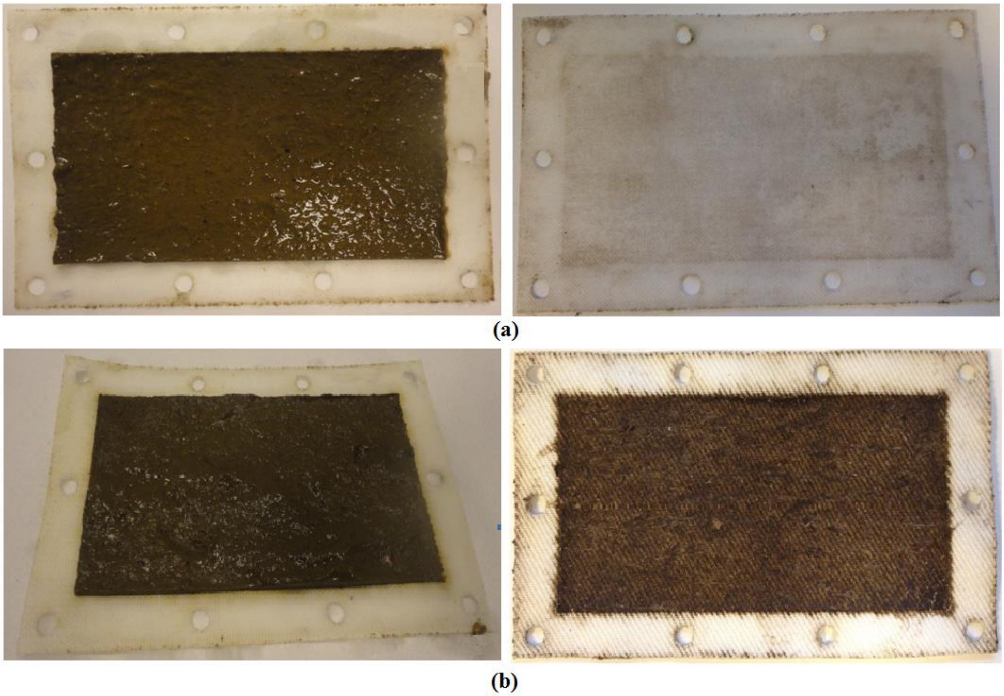

Figure 3.8. Cake layer formation, and pore accumulation after physical cleaning:
(a) mono-monofilament support material, (b) staple support material.

After filtration of anaerobic sludge, cake layer formation was observed on the surface of both support materials (Figure 3.9). However, after physical cleaning with tap water, while there was no pore blocking observation for mono-monofilament filter cloth, an intensive pore accumulation could be clearly seen inside the staple support material (Figure 3.8). This may be attributed to the twisted and hairy structure of the staple yarn types.

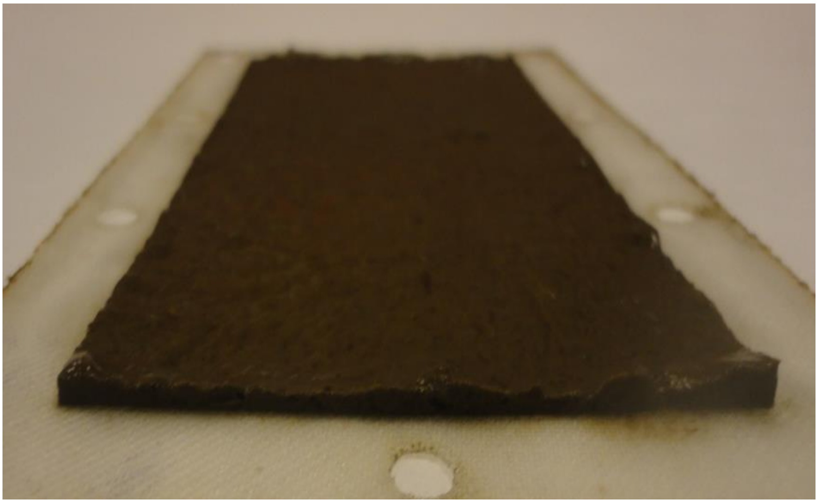

Figure 3.9. Dynamic membrane (cake) layer.

Due to their structure, these materials are, indeed, suitable for depth filtration (Figure 3.10) through which the particles can be retained not only by the cake layer formed on the filter surface but also within the filter pores. The latter is not favorable for DM filtration. In contrast, mono-monofilament filter cloth has a smooth surface without a tortuous path which allows cake layer formation on the filter and retains the particles through the cake layer instead of within the filter cloth.

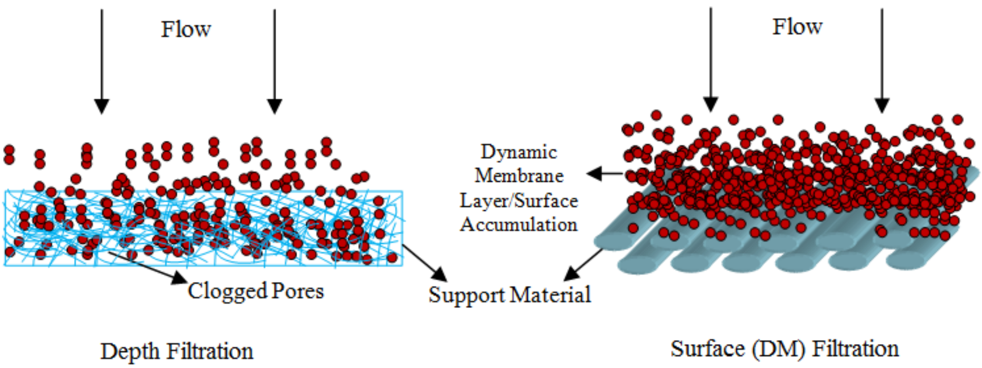

Figure 3.10. Difference between depth and surface filtration.

3.4 Conclusions

High removal efficiencies comparable to AnMBR systems can also be obtained with AnDMBR technology. This can be accomplished by formation of a porous and compressible cake layer on the support material surface. Support material properties are critical for the formation of an effective cake layer over the filter surface in DM filtration technology. An optimal support material was determined by applying various types of filter cloth. Remarkably, the differences in critical fluxes between the filter cloths with different pore sizes were very small. Contrary, the structure of woven support materials, e.g. yarn type, determines to a higher extent the critical flux and filterability than the pore size of the material. However, critical flux itself is not a very useful indicator to determine the long-term filterability of a support material in a DM filtration process. The results of the short and the more long-term experimental studies indicated that staple filter cloth is more suitable for depth filtration, whereas, mono-monofilament filter is more suitable for cake filtration. Therefore, mono-monofilament filter is considered more appropriate for DM filtration systems. Application of staple filter cloths will result in severe pore fouling. Further research should be focused on the applicability of DM technology in AnMBRs in terms of biological removal over long-term operation periods.

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CHAPTER 4

TREATMENT OF CONCENTRATED WASTEWATERS WITH SUBMERGED ANAEROBIC DYNAMIC MEMBRANE BIOREACTORS (AnDMBRs)

Abstract
This chapter investigated the applicability of dynamic membrane technology in anaerobic membrane bioreactors for the treatment of high strength wastewaters. A mono-monofilament woven fabric was used as support material for dynamic membrane formation. An AnDMBR was operated under a variety of operational conditions, including different SRTs of 20 and 40 days in order to determine the effect of SRT on both biological performance and dynamic membrane filtration characteristics. High COD removal efficiencies exceeding 99% were achieved during the operation at both SRTs. Higher filtration resistances were measured during the operation at SRT of 40 days in comparison to SRT of 20 days, applying a stable flux of 2.2 L/m^2.h. The higher filtration resistances coincided with lower extracellular polymeric substances concentration in the bulk sludge at SRT of 40 days, likely resulting in a decreased particle flocculation. Results showed that dynamic membrane technology achieved a stable and high quality permeate and AnDMBRs can be used as a reliable and satisfactory technology for treatment of high strength wastewaters.

This chapter is based on:

Ersahin, M.E., Ozgun, H., Tao, Y., van Lier, J.B., 2014. Applicability of dynamic membrane technology in anaerobic membrane bioreactors. Water Research, 48, 420-429.

4 TREATMENT OF CONCENTRATED WASTEWATERS WITH SUBMERGED ANAEROBIC DYNAMIC MEMBRANE BIOREACTORS (AnDMBRs)

4.1 Introduction

Anaerobic technology for wastewater treatment has evolved into a consolidate alternative for a wide variety of wastewaters. Particularly the avoidance of fossil energy use while converting the chemically stored energy in the organic pollutants into energy-rich biogas, has made anaerobic treatment an attractive alternative in the last few decades. Industrial wastewater treatment has been mostly benefited from anaerobic technology owing to the development of anaerobic high-rate reactors such as the UASB reactors and the expanded granular sludge bed (EGSB) reactors (van Lier, 2008; Ersahin et al., 2011). Since the growth rate of the anaerobic microorganisms is much lower than that of aerobic ones, high biomass concentrations are needed inside the anaerobic reactors. High-rate anaerobic processes are characterized by an uncoupling of the SRT from the HRT. The increased SRT is a result of effective biomass retention, largely facilitated by (auto)immobilization of anaerobic bacteria in biofilms, flocs or granular sludge. When biomass immobilization cannot be guaranteed, alternatively membrane separation can be used to retain biomass. AnMBRs are of growing interest and have been researched for the treatment of different kinds of wastewater including municipal and industrial wastewaters (Liao et al., 2006; Dereli et al., 2012; Ozgun et al., 2013; Lin et al., 2013). AnMBRs combine the advantages of anaerobic processes with the production of solids free effluents. AnMBR technology has been considered as an appropriate alternative to provide a complete biomass retention enabling independent control of HRT and SRT (Jeison et al., 2008; Zhang et al., 2010).

Accumulation of solid particles such as microbial cells, extracellular organics, and inorganic precipitates on the membrane surface is a common phenomenon that occurs in (An)MBRs during filtration. The accumulated matter on the membrane surface becomes denser over time and forms a cake layer that governs fouling and flux limitation (Jeison and van Lier, 2008; Lin et al., 2009; Waeger et al., 2010). In fact, the cake layer is the most important barrier in AnMBR systems (Jeison and van Lier, 2008). The formation and the effective use of this cake layer on a support layer such as a mesh or woven filter cloth instead of a membrane presents a new concept, which is called DM filtration (Ersahin et al., 2012). Since the cake (DM) layer can easily be removed from the surface of the support material and can be re-established again in a short time, this layer is termed "dynamic membrane". DM layer can be used as a filter prior to the support material; thus, even the support material has a big pore size, the dense and compact DM layer provides an effective retention in AnDMBRs (Kiso et al., 2000; Jeison et al., 2008; Zhang et al., 2010). Therefore, cheap materials can be used as the support material, enabling AnMBR applications at much lower capital exploitation costs. In the proposed concept, the cake layer plays a crucial role. For effective DM layer formation and consolidation, the selection of appropriate type of support material regarding its structure, e.g. yarn type, pore size, and availability is an important issue (Ersahin et al., 2013). The most common support material types used in various studies, including both aerobic and anaerobic dynamic MBR applications, were mesh, woven and non-woven fabrics (Ersahin et al., 2012).

DM technology in AnMBRs was applied for the treatment of municipal wastewaters in various studies (Ho et al., 2007; An et al., 2009; Zhang et al., 2010; Zhang et al., 2011). Jeison et al. (2008) found that almost complete retention of solids could be achieved by AnDMBRs. However, they could not get a stable flux that had a range between 0.5-3 L/m^2.h under both thermophilic and mesophilic conditions. With non-woven fabric support layer, COD removal of 87% was achieved by an AnDMBR treating municipal wastewater (An et al., 2009). Zhang et al. (2011) located a DM module with a mesh support material at the top of a UASB reactor, thereby filtering the supernatant instead of the sludge. They found that high flux values, e.g. 65 L/m^2.h, are achievable in long-term operation. Although they had a stable COD removal of about 63.4%, this efficiency is significantly lower than those obtained by conventional AnMBRs. The research on DM technology has been mainly focused on the applications in aerobic MBRs rather than anaerobic ones (Ersahin et al., 2012). A few studies have been conducted for AnDMBRs, which focused mostly on the treatment of low strength wastewaters, e.g. municipal wastewaters. There is quite limited information about the potential and applicability of DM technology for treatment of high-strength/concentrated waste(water)s in AnMBRs. Therefore, the main goal of this chapter was to investigate the applicability of the DM technology in AnDMBRs treating high strength organic wastewaters. Within this concept, different SRTs were applied in a submerged AnDMBR in order to understand the effects of SRT on the removal efficiency and sludge filterability. For this purpose, COD removal, TSS retention capacity, biogas (methane) generation, evolution of TMP and specific resistance to filtration (SRF) change, PSD, and EPS/SMP formation in the bulk sludge were investigated.

4.2 Material and Methods

4.2.1 Experimental Set-up

A laboratory scale submerged AnDMBR set-up was used in this study (Figure 4.1). The AnDMBR system consisted of a completely mixed glass reactor that had an effective volume of 7.4 L and a submerged flat sheet (Figure 4.2) membrane module. The rectangular membrane module had two filtering sides with a total filtration area of 0.014 m^2. A mono-monofilament woven fabric, which was made of polypropylene material (Lampe BV, the Netherlands) with an average pore size of 10 µm, was used as the support material (Figure 4.3). Two peristaltic pumps (Watson Marlow 120U/DV) were separately used to feed substrate into the anaerobic reactor and to collect permeate from the membrane module. TMP was measured by a pressure sensor (AE Sensors, ATM -800/+600 mbar) placed on the permeate line. Produced biogas was recycled by a diaphragm pump (KNF, N86 KTDCB) via two diffusers to provide mixing inside the bioreactor and to scour the DM surface for fouling control. Mixing diffuser was located at the bottom of the bioreactor and the biogas sparging diffuser was placed under the membrane module (Figure 4.1). Biogas production was measured by a gas counter (Ritter, Milligas Counter MGC-1 PMMA). Two baffles were included inside the bioreactor in order to obtain even distributed mixing conditions. Temperature and pH inside the bioreactor were measured on-line by a probe combined with a transmitter (Elscolab, M300 ISM). The AnDMBR system (Figure 4.4) was connected to a computer equipped with a LabVIEW software (LabVIEW 10.0.1, National Instruments) for pumps control and data collection.

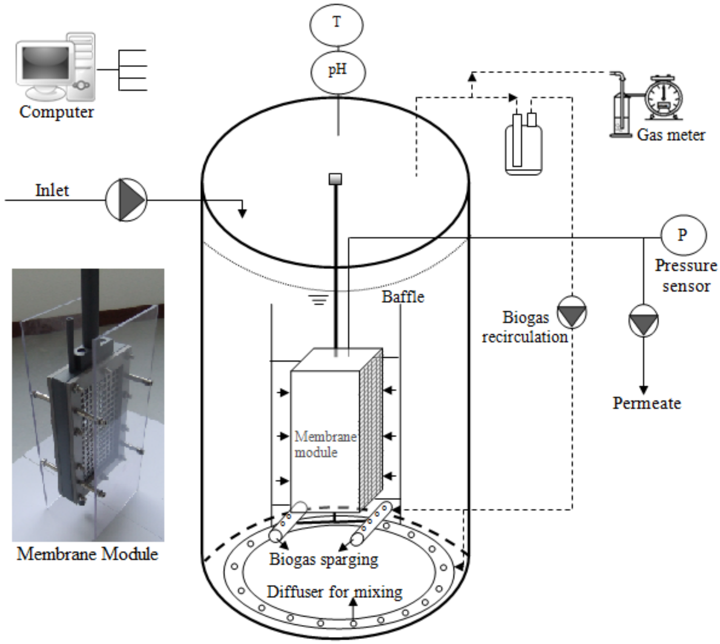


Figure 4.1. Schematic diagram of the submerged AnDMBR set-up.

4.2.2 Experimental Procedure

The AnDMBR was operated for 140 days at two different SRTs, i.e. 20 days and 40 days, respectively. Operational periods covering the SRT 20 of days and 40 days are referred to as R20 and R40, respectively. Average TSS concentration in the bioreactor increased from 5027±315 mg/L to 6450±480 mg/L at steady state conditions with the increase in SRT from 20 days to 40 days. AnDMBR operation was conducted sustainably at a flux of around 2.2 $L/m^2.h$ and no remarkable changes were observed at the applied different SRTs. A new support material was used for each SRT study. Organic loading rate (OLR) was kept at 2 kg $COD/m^3.d$ and HRT was set to 10 days during the entire study. The anaerobic bioreactor was operated at an average temperature of 35.7±0.1 ^oC. The average pH values were 7.87 ± 0.14 and 7.91 ± 0.05 in R20 and R40, respectively.

Figure 4.2. Submerged membrane module with two baffles.

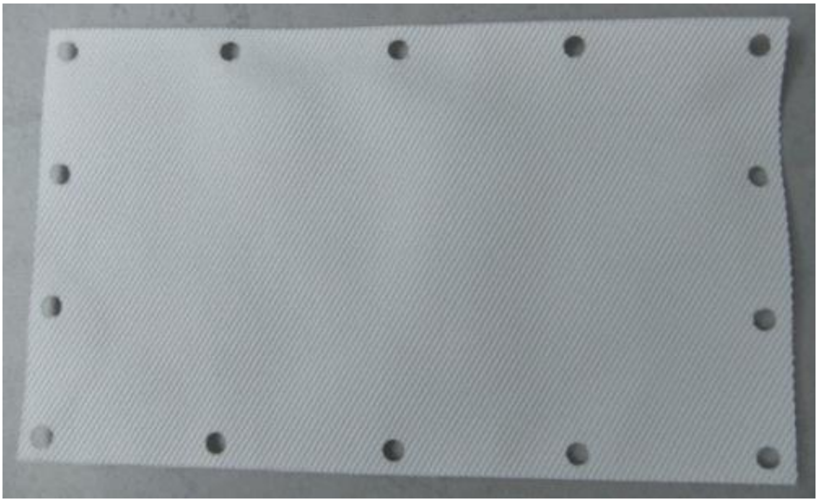
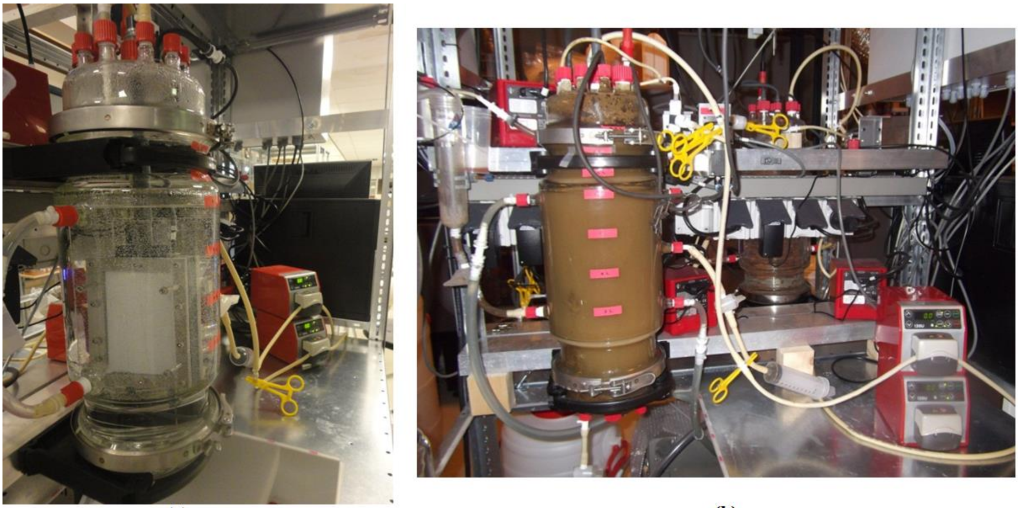


Figure 4.3. Mono-monofilament filter cloth.



(a) (b)

Figure 4.4. Submerged AnDMBR set-up: (a) without sludge, (b) with sludge during the operation.

To control both the dynamic cake layer thickness on the surface of the woven fabric and TMP, biogas sparging and backwashing were used. Biogas sparging rate, the recirculated biogas volume per cross-sectional area over the biogas sparging diffuser located under the membrane module, was 35 m/h. The DM unit was operated in cycles consisting of filtration and backwashing. The filtration phase was set to 190 seconds and backwashing phase was set to 35 seconds by reversing the direction of the permeate pump.

4.2.3 Wastewater Source and Seed Sludge

Synthetic concentrated wastewater was used as substrate. Macronutrient and micronutrient compositions were slightly modified from the ones given in Aiyuk and Verstraete (2004) and Martin et al. (2010), respectively. The composition and characterization of the synthetic concentrated wastewater are given in Table 4.1 and Table 4.2, respectively.

Table 4.1. Composition of the synthetic wastewater.

Macronutrients	Concentration (g/L)	Micronutrients	Concentration (g/L)
Urea	3	$FeCl_3 \cdot 4H_2O$	1
NH_4Cl	0.56	$CoCl_2 \cdot 6H_2O$	1
$NaCH_3COOH \cdot 3H_2O$	6.3	$MnCl_2 \cdot 4H_2O$	0.25
$MgSO_4 \cdot 7H_2O$	0.25	$CuCl_2 \cdot 2H_2O$	0.015
K_2HPO_4	2.2	$ZnCl_2$	0.025
$CaCl_2.2H_2O$	0.37	$NiCl_2 \cdot 6H_2O$	0.025
Ovoalbumin	0.84	$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$	0.045
Starch	5.9	$Na_2SeO_3 \cdot 5H_2O$	0.05
Milk Powder	5.6	Boric Acid	0.025
Yeast Extract	2.5	EDTA	0.5
Sunflower Oil	1.4 ml	HCl 36%	0.5 ml
Micronutrient	1 ml	Resazurin	0.25

Table 4.2. Characterization of the synthetic wastewater.

Parameter	Unit	Value
COD	mg/L	20100±310
Soluble COD	mg/L	11500±95
TSS	mg/L	7400±1100
NH_4-N	mg/L	195±5
Total Nitrogen (TN)	mg/L	2340±145
Total Phosphorus (TP)	mg/L	470±10
pH	-	7.3
Turbidity	NTU	3920±135

The AnDMBR was inoculated with sludge from a pilot-scale UASB reactor treating black water at 35 ^oC. The characterization of the seed sludge is given in Table 4.3. The bioreactor was filled with seed sludge up to effective volume for start-up. Acclimation period using the concentrated synthetic wastewater (Table 4.2) lasted 30 days before the SRT study.

Table 4.3. Characterization of the seed sludge.

Parameter	Unit	Value
TS	mg/L	22000±300
VS	mg/L	16900±235
TSS	mg/L	20200±75
VSS	mg/L	16900±225
COD	mg/L	27100±330
pH	-	7.88
Specific Methanogenic Activity (SMA)	g CH_4-COD/g VS.d	0.3±0.03

4.2.4 Analytical Methods

4.2.4.1 Analysis Techniques

COD, TSS, VSS, ammonium nitrogen, TN and TP parameters were determined following Standard Methods (APHA, 2005). Soluble COD samples were filtered through 0.45 μm disposable filters before analysis. Turbidity measurements were carried out with Hach 2100N turbidimeter. The PSD of the anaerobic sludge was determined by a Mastersizer 2000 (Malvern Instruments, Hydro 2000 MU), which has a detection range of 0.02-2000 μm. Laser diffraction technique was used in order to measure the size of the particles. The methane content in biogas was measured with a Varian 3800 gas chromatograph equipped with a flame ionization detector (Varian, Palo Alto, CA). The chromatograph was fitted with a Varian Hayesep Q (80-100 mesh) Ultimetal micropacked column (1.2 m × 1/16" × 1 mm). Helium was used as the carrier gas at flow rate of 0.2 ml/min. The temperature of the injector port and the detector was set 200 °C, and the temperature of the oven was 50 °C.

For assessing the SMP content of sludge samples, a volume of 5 ml sludge was sampled, diluted by phosphate buffered saline (PBS, pH=7.2) and centrifuged at 7000xg for 7 minutes at 4 °C. The supernatant was filtered by a 0.45 μm filter and the filtrate was collected for SMP determination. The pellet was rewashed with 10 ml PBS and then ultrasonication was carried out at 40 kHz (Cole-Parmer Ultrasonic, the Netherlands) for 3 minutes. A high speed centrifuge (17000xg for 20 minutes at 4 °C) was applied to collect the supernatant and supernatant was filtered by a 0.45 μm filter for EPS measurement. The washing-ultrasonication-centrifuge process was repeated in order to extract the EPS. The phenol-sulphuric acid method was used to quantify polysaccharides (Dubois et al., 1956). The concentration of protein was determined using Bradford method (Bradford, 1976).

4.2.4.2 Bio-tests

SMA was determined in triplicate by using an Automated Methane Potential Test System (AMPTS, Bioprocess Control, Sweden) (Li et al., 2011). The SMA test was carried out in 500 ml serum bottles (with a working volume of 400 ml), which were filled with sludge, sodium acetate (0.5 g/L as COD), distilled water, pH buffer, nutrients and trace elements. The bottles were sparged with nitrogen gas to remove oxygen from the headspace. Inoculum (based on VS) to substrate ratio of 2:1 was used in the test. SMA tests were performed at 35 °C. The nutrient stock solution consisted of (g/L): NH_4Cl (170), $CaCl_2{\cdot}2H_2O$ (8), $MgSO_4{\cdot}7H_2O$ (9) and the trace element stock solution contained (g/L): $FeCl_3{\cdot}4H_2O$ (2), $CoCl_2{\cdot}6H_2O$ (2), $MnCl_2{\cdot}4H_2O$(0.5), $CuCl_2{\cdot}2H_2O$ (30), $ZnCl_2$ (50), H_3BO_3 (50), $(NH_4)_6Mo_7O_2{\cdot}4H_2O$ (90), $Na_2SeO_3{\cdot}5H_2O$ (100), $NiCl_2{\cdot}6H_2O$ (50), EDTA (1), HCl 36% (1 ml/L), Resazurine (0.5). The pH buffer stock solution was composed of $K_2HPO_4{\cdot}3H_2O$ (45.65 g/L) and $NaH_2PO_4{\cdot}2H_2O$ (31.20 g/L).

4.2.4.3 Filtration Tests

Capillary suction time (CST) is defined as a quantitative measure of the release rate of water from sludge based on time unit. A CST device (Triton Electronics, Model 304M) was used to conduct CST experiments. TSS concentration is closely related with CST (Sawalha and Scholz, 2010). Therefore, normalized CST (CST_n) can be used in order to minimize the effect of TSS by dividing CST to TSS concentration (Khan et al., 2008). To determine SRF of the sludge samples, a dead end filtration experiment was performed using an unstirred filtration cell (Amicon, Model 8050). Flat sheet glass microfiber filter (Whatman GF/F 1825-047) was used in the cell. The cell was filled with 40 ml of sludge sample and a constant pressure of 0.5 bar was applied by pressurized air. The mass of permeate was recorded in 15 s intervals by an electronic balance connected to a computer. SRF (m/kg) was calculated (Wang et al., 2007) by the equation (4.1) below:

$$\alpha = \frac{2000.A^2.\Delta P}{\mu.C} . \frac{t/V}{V} \tag{4.1}$$

where A is the filtration area (m^2), ΔP is the applied pressure (kPa), μ is the dynamic viscosity of permeate (Pa.s), C is the TSS concentration (kg/m^3), t is the time of filtration (s), and V is the filtrate volume (m^3).

Total filtration resistance (R_T) was determined as a function of the TMP, which was measured by a pressure sensor located on the permeate line. Resistance was calculated with equation (3.1).

4.3 Results and Discussion

4.3.1 Treatment Performance

High and stable total COD removal efficiencies of 99.1% and 99.5% were achieved during R20 and R40, respectively (Figure 4.5). The acclimation period for the sludge was initiated before the operation period of SRT 20 days. Therefore, the first data in Figure 4.5 was obtained just after the acclimation period. High COD removal efficiency was obtained regardless of the operating SRT which indicated that the DM layer had the capability to compensate the differences in operating conditions enabling a stable and sustainable permeate quality. Average permeate COD concentrations of 115±20 mg/L and 95±12 mg/L were obtained in R20 and R40, respectively. The specific COD removal rates in R20 and R40 were calculated as 0.38 g COD/g VSS.d and 0.4 g COD/g VSS.d, respectively. Figure 4.5 shows a period of stabilization prior to reach stable COD removal efficiencies at both SRTs.

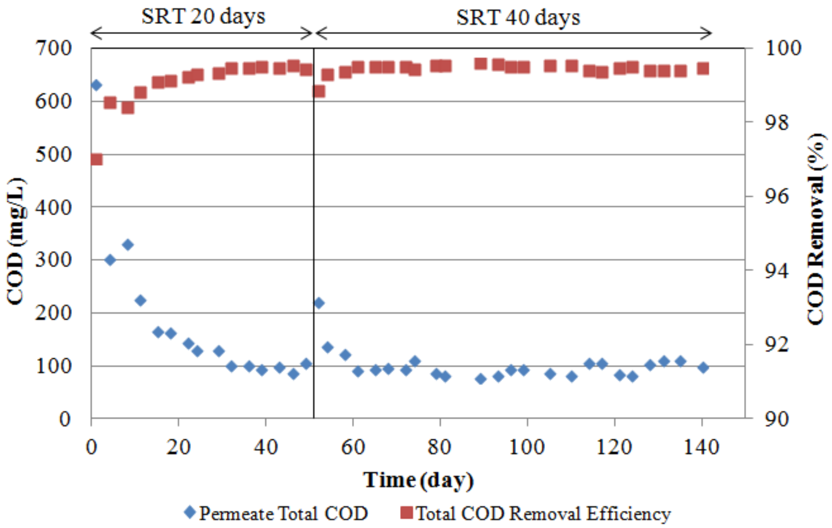


Figure 4.5. COD concentrations in the permeate and COD removal efficiency.

Average soluble COD concentrations in the bioreactor (excluding data of the first 10 days of the operation at both SRTs) were 360±50 mg/L and 240±30 mg/L in R20 and R40, respectively. Soluble COD removal efficiencies by the DM layer were 63.4% and 63.6% in R20 and R40, respectively. While the removal rates for the COD were high, the average elimination of TN and TP by the AnDMBR were 20% and 13%, respectively. Although a dynamic cake layer can form on the support layer only after a few minutes of filtration start-up (Seo et al., 2002; Park et al., 2004; Hu and Stuckey, 2006), an effective cake layer formation, by which a stable pollutant removal and permeate quality can be obtained, requires more time. In this study, an effective DM layer formation was reached in between 10-20 days (Figure 4.5 and Figure 4.7).

VSS/TSS ratio was calculated over 0.85 in the AnDMBR at both SRTs (Figure 4.6). F/M ratio, which represents the ratio between the COD loading fed into the bioreactor and the TSS concentration in the bioreactor, decreased from an average of 0.37 kg COD/kg TSS.d to 0.27 kg COD/kg TSS.d at steady state conditions when the SRT was shifted from 20 days to 40 days. However, there was no significant change observed in the permeate quality depending on the F/M ratio.

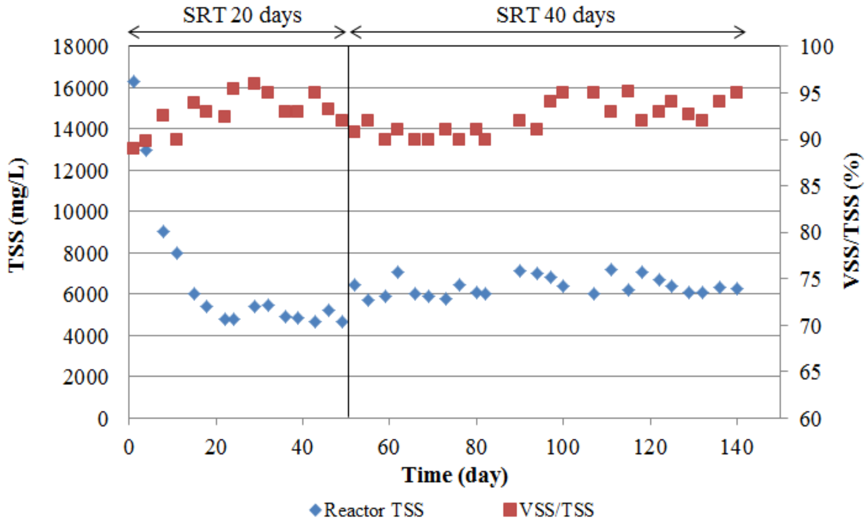


Figure 4.6. TSS concentration and VSS/TSS ratio in the AnDMBR.

After the formation of an effective DM layer, TSS concentration in the permeate was lower than 10 mg/L at both SRTs. This corresponds to TSS retention of >99% by the formed DM layer. Similar TSS concentrations ranged between 5-10 mg/L in the permeate were reported in aerobic dynamic MBR studies using a nylon mesh and/or a non-woven fabric filter (Kiso et al., 2000; Seo et al., 2002). Figure 4.7 shows the permeate turbidity at both SRTs. At the initial stage, permeate turbidity was 140 and 58 NTU in R20 and R40, respectively. The turbidity decreased during the formation of an effective DM layer and after 10 days, average turbidity of 11.4±2 NTU and 12.5±2.3 NTU were detected at SRT 20 days and 40 days, respectively. According to these results, a stable turbidity removal rate of >99% was obtained in the AnDMBR. In aerobic dynamic MBRs treating municipal wastewaters, it was also reported that a specific time period is needed to form a stable dynamic cake layer, after which a high turbidity removal rate can be obtained (Chu and Li, 2006; Ren et al., 2010).

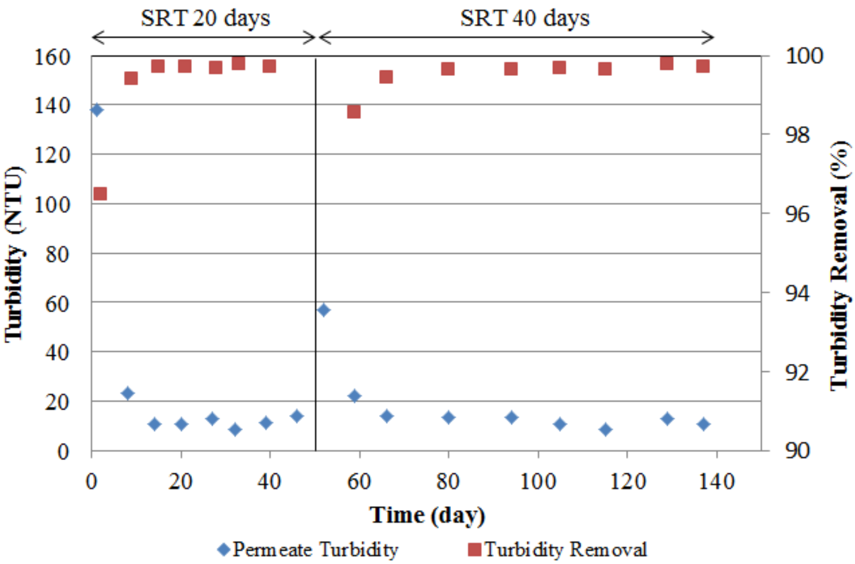


Figure 4.7. Turbidity removal in the AnDMBR.

Biogas and methane production measured in the study are depicted in Figure 4.8. After 20 days, the average biogas production in R20 and R40 were 3.20±0.13 L/day and 3.27±0.14 L/day, respectively. The methane content in the biogas was about 64% and 72% in R20 and R40, respectively. At SRT 20 days, an average methane yield of 0.31±0.02 L CH_4/g $COD_{removed}$ was obtained which represented 79% of the maximum theoretical value, 0.395 L CH_4/g $COD_{removed}$ at 35 °C. A slight increase in the methane yield to 0.34±0.04 L CH_4/g $COD_{removed}$, which was 86% of the maximum theoretical value, was observed at SRT 40 days. COD difference between influent, permeate and waste sludge of the AnDMBR was represented as the removed COD in the calculation of methane yield but apparently the actually degraded COD converted to methane was lower than the removed COD. Besides, some amount of the methane might be solubilized in the permeate. Smith et al. (2013) found that up to 40-50% of total methane generated in an AnMBR can be dissolved in the permeate. Methane yields below theoretical values are therefore commonly observed in AnMBR studies. Martinez-Sosa et al. (2011) and Huang et al. (2011) reported methane yields ranging from 0.124 to 0.27 L CH_4/g $COD_{removed}$ in AnMBR studies.

The data collected from SMA test showed that the sludge methanogenic capacity in R40 is higher compared to the R20 period (Table 4.4). Huang et al. (2011) also reported more methane production at longer SRTs compared to short SRTs in a submerged AnMBR. They attributed the increase in methane production to dominancy of acetoclastic methanogens at longer SRTs. Table 4.4 shows that SMAs of the bulk sludge were lower in comparison to seed sludge at both SRTs. A physical interruption of syntrophic associations might occur due to strong shear stress applied by the gas pump inside the AnDMBR.

Table 4.4. SMA of the different sludge types.

Sludge Sample	SMA (g CH_4-COD/g VS.d)
Seed Sludge	0.25±0.003
Bulk sludge (SRT 20 days)	0.12±0.003
Bulk sludge (SRT 40 days)	0.20±0.006

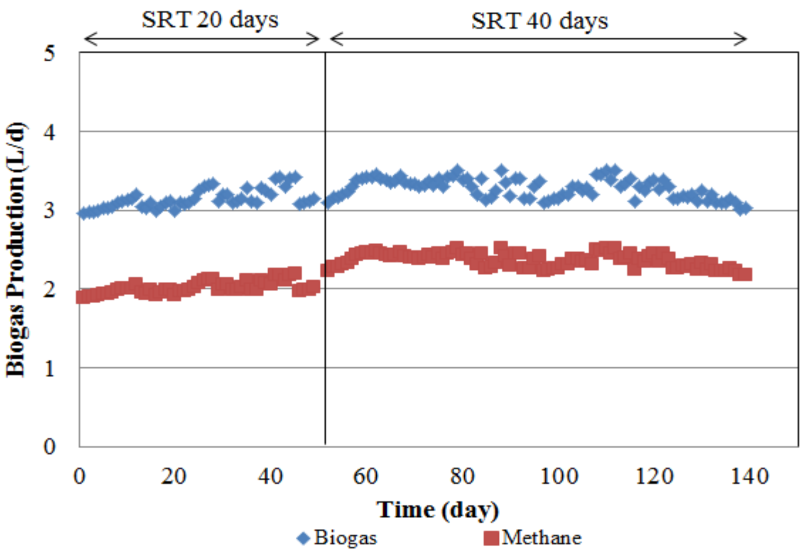


Figure 4.8. Biogas and methane production rates in the AnDMBR.

4.3.2 Filtration Performance

4.3.2.1 Flux

In the DM filtration concept, cake layer formation is the most important factor that determines the flux. Because of the prominent effect of the cake layer, other factors such as substrate type, temperature, biological operation conditions have less impact on the flux (Jeison and van Lier, 2007; Ersahin et al., 2012). To get an efficient retention and sustainable filtration with DM technology, it is vital to control the cake layer thickness on the support material surface since it provides the retention of particulate material inside the bioreactor but also causes filtration pressure increase (Ersahin et al., 2012). By controlling the cake layer, sudden changes in flux, TMP and permeate quality due to the possible unstable filtration can be prevented. In this study, biogas recirculation and backwashing were both used in order to control DM layer thickness and TMP. Biogas sparging rates in a range of 17.6-65 $m^3/m^2.h$ have been reported for pilot-scale AnMBR applications (Dereli et al., 2012). The biogas sparging rate of 35 m/h applied in this study is consistent with the previously reported data. Critical flux obtained with the mono-monofilament fabric used in this study was about 9.2 $L/m^2.h$ which is similar to critical fluxes obtained with conventional AnMBRs using polysulfone and/or polyolefin microfiltration membranes (Ersahin et al., 2013). The operational flux of 2.2 L/m^2.h obtained in this study is similar and/or higher than the values reported for aerobic submerged dynamic MBRs, e.g. 0.8-0.9 L/m^2.h (Satyawali and Balakrishnan, 2008), for submerged AnDMBRs, e.g. 0.5-3.0 L/m^2.h (Jeison et al., 2008) and for submerged AnMBRs, e.g. 2 L/m^2.h (Akram and Stuckey, 2008).

4.3.2.2 SMP and EPS

Various authors reported SMP and EPS as the main contributors to membrane fouling in MBRs (e.g. Ahmed et al., 2007; Satyawali and Balakrishnan, 2008; Meng et al., 2009; Huang et al., 2011; Zhang et al., 2011). EPS can be present in either the soluble or bound form. Soluble EPS can also be called SMP (Meng et al., 2009), which consists of the organic compounds that originate from substrate metabolism and/or biomass decay inside the bioreactor. Bound EPS is mainly composed of cell surface materials, e.g. proteins, polysaccharides, lipids, nucleic acids and humic acids (Meng et al., 2009). Bound EPS keeps the sludge flocs together on the membrane surface by surrounding them (Lin et al., 2011). Thus, the formation and consolidation of a DM layer may be significantly affected by these compounds.

SMP and EPS amounts in the bulk sludge decreased during the operation time at both SRTs (Table 4.5 and Table 4.6). Protein amounts in the SMP were 65 mg/g VSS and 120 mg/g VSS on the first day, which decreased to 32 mg/g VSS and 51 mg/g VSS at the end of operations in R20 and R40, respectively. A similar decreasing trend was also observed for polysaccharide amounts in the SMP and EPS compositions. Satyawali and Balakrishnan (2008) also observed a similar trend in an aerobic dynamic MBR treating distillery wastewaters. Reduction of SMP concentration in the bulk sludge may be attributed to the retention of these products by the DM layer. Besides, Drews et al. (2006) indicated that elimination of SMP can occur due to biodegradation in a MBR. Therefore, both accumulation on DM layer and biodegradation might be responsible for the SMP decrease. Lower SMP concentrations were determined in the bulk sludge in R20 than that in R40. Microorganisms have lower metabolism rates, less nutrition uptake and degradation due to the endogenous growth at longer SRTs. These conditions provide retention of higher SMP concentrations in the AnMBRs. More organic matters can be metabolized and less SMP is produced due to the higher activity rate of the microorganisms at low SRTs. Therefore, less SMP concentration in the system is reasonable at shorter SRTs (Shin and Kang, 2003; Ahmed et al., 2007; Huang et al., 2011). It means that more proteins and polysaccharides were introduced to the support layer surface at SRT 40 days. Su et al. (2011) also reported that carbohydrates and proteins in SMP increased as the SRT increased in a submerged MBR.

Table 4.5. SMP and EPS compositions in bulk sludge at SRT 20 days.

	SMP			EPS		
Day	**Protein (mg/g VSS)**	**Polysaccharide (mg/g VSS)**	**P/C**	**Protein (mg/g VSS)**	**Polysaccharide (mg/g VSS)**	**P/C**
1	65	20	3.2	9.5	5.6	1.7
15	58	17	3.4	3.3	3.5	0.9
30	50	13	3.7	2.0	3.4	0.6
45	34	16	2.1	1.5	2.9	0.5
50	32	13	2.5	1.8	3.0	0.6

Table 4.6. SMP and EPS compositions in the bulk sludge at SRT 40 days.

	SMP			EPS		
Day	**Protein (mg/g VSS)**	**Polysaccharide (mg/g VSS)**	**P/C**	**Protein (mg/g VSS)**	**Polysaccharide (mg/g VSS)**	**P/C**
1	120	47	2.6	4.0	2.1	1.9
15	62	39	1.6	1.8	1.7	1.1
30	58	35	1.7	1.9	1.7	1.1
45	50	29	1.7	1.6	1.8	0.9
70	51	27	1.9	1.5	1.6	0.9

EPS has a significant positive effect on particle flocculation and thus, particle size distribution in the bulk sludge. Finer particles may be present due to reduced flocculation at low EPS concentrations (Meng et al., 2006; Huang et al., 2011). Therefore, less EPS concentration in R40 in comparison to R20 may result in an increase in TMP and filtration resistance due to an increase in amount of small particles. A decrease in EPS concentration with an increase in SRT was also observed in aerobic MBRs due to the low formation rate of microbial substances at long SRTs (Lee et al., 2003; Masse et al., 2006; Ahmed et al., 2007).

It is obvious in Table 4.5 and Table 4.6 that protein is the major compound in SMP. Exo-enzymes in the sludge flocs, and cell lysis compounds might be responsible for the higher amount of proteins compared to polysaccharides (Neyens et al., 2004). In general, higher protein/carbohydrate (P/C) ratio in SMP was obtained in the AnDMBR at SRT of 20 days than that obtained at SRT of 40 days. P/C ratio in SMP ranged at 2.1-3.7 and 1.6-2.6 in R20 and R40, respectively. P/C ratio has been indicated as a factor that has a significant effect on the hydrophobicity and surface charge of the sludge and high P/C ratio results in a high hydrophobicity (Lee et al., 2003; Thuy and Visvanathan, 2006). This effect is mainly originated from proteins and the effect of polysaccharides may be negligible.

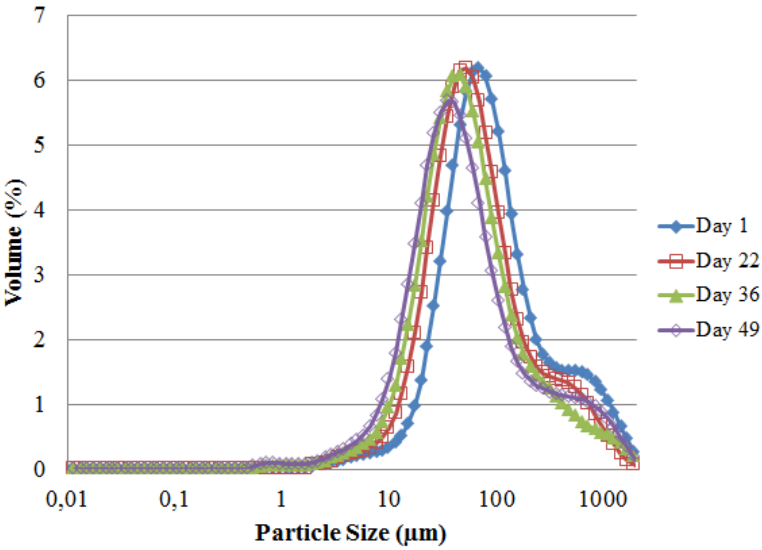
4.3.2.3 PSD

PSD analysis of bulk sludge in R20 and R40 are shown in Figure 4.9. At the first day of the operation, the median particle size by volume was 76.1 µm in R20. Along with the operation, the median particle size decreased to 55.3 µm, 45 µm, at day 22 and 36, respectively and then remained stable in R20. The median particle size of the particles was 41.1 µm at the initial stage and decreased to 37.1 µm after 18 days operation and then remained almost constant in

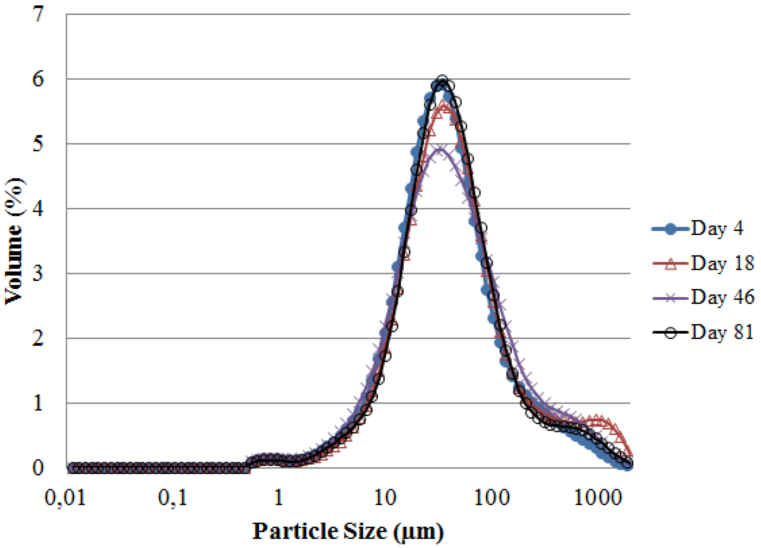
R40 during the study. A significant decrease in the particle size at the initial stage in R20 was possibly due to the effect of high biogas recirculation rate applied for mixing the reactor and sparging the surface of the support material. It was determined that the flocs in aerobic MBRs were finer than those in the conventional activated sludge systems due to the aeration turbulence inside the bioreactor and sparging of membrane surface (Gao et al., 2004; Durante et al., 2006). Moreover, 70% decrease in particle size of bulk sludge was reported in 102 days operation in a dynamic MBR equipped with a mesh filter (Satyawali and Balakrishnan, 2008). Small flocs may still provide appropriate conditions for mass and hydrogen transfer (Jeison and van Lier, 2007); however, they may increase the cake layer resistance due to the accumulation of small particles inside the cake layer leading to high cake compactness. As EPS amount affects particle flocculation, a decrease in particle size would be expected at low EPS amount in the bulk sludge. Since the EPS concentration was lower at SRT 40 days compared to SRT 20 days, it was reasonable to expect smaller particles in the bulk sludge in R40 compared to R20. This would result in an increased TMP and filtration resistance at SRT 40 days.

4.3.2.4 TMP and Filtration Resistances

Daily average TMP data obtained in R20 and R40 are given in Figure 4.10. TMP increased during the initial 10 days; thereafter, it showed a stable trend at both SRTs. The average TMP values during stable operation period were 530 mbar and 680 mbar at SRT 20 days and 40 days, respectively. The average total filtration resistances calculated as a function of TMP were 1.02×10^{17} m^{-1} and 1.30×10^{17} m^{-1} in R20 and R40, respectively. Higher TMP and total filtration resistance values determined in R40 are also in agreement with the results of EPS amounts and PSD in R20 and R40, which are explained above. Moreover, Lin et al. (2011) found that sludge with high P/C ratio in EPS resulted in a more sticky cake layer development with higher filtration resistances compared to sludge with a low P/C ratio in an AnMBR.



(a)



(b)

Figure 4.9. Particle size distribution expressed as % of total particle volume of the bulk sludge: (a) SRT 20 days, (b) SRT 40 days.

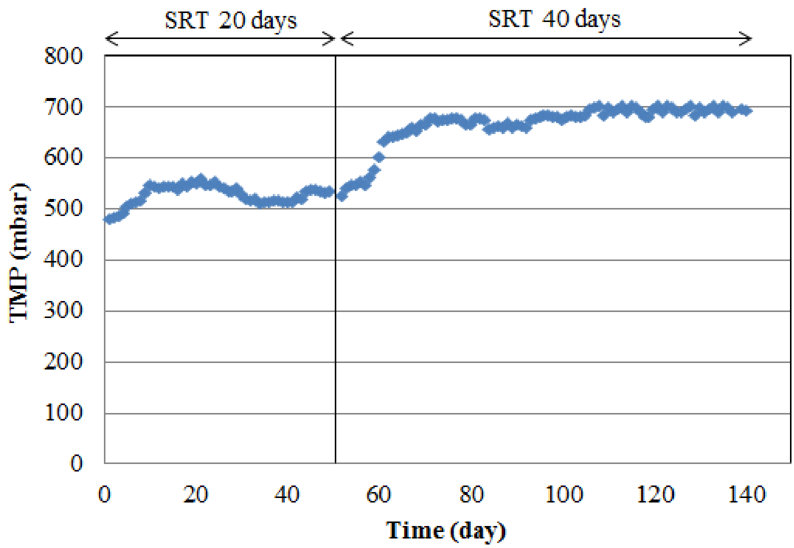


Figure 4.10. TMP profiles in the AnDMBR.

4.3.2.5 SRF and CST

SRF analyses were performed in order to assess the effect of SRT on sludge filterability in the AnDMBRs. The average SRFs were 7.70×10^{14} m/kg and 14.10×10^{14} m/kg in R20 and R40, respectively. The bulk sludge in R40 had an SRF value that was 1.8 times higher compared to that in R20. From a theoretical viewpoint, the SRF must be directly related to the PSD since small flocs can easily attach on the membrane surface and/or fill cavities in the DM layer and thus may contribute cake layer formation and compaction (Lin et al., 2011). The median particle size in the bulk sludge in R40 was lower than that in R20. Besides, TMP values were higher in comparison to SRT 20 days. These results indicated that the SRF is indeed closely related with the particle size of the flocs and EPS amount in AnDMBRs. These findings are in agreement with the previous studies performed by other authors (Xuan et al., 2010; Lin et al., 2011). SRF and PSD in the bulk sludge of an AnDMBR may be used as effective tools to characterize cake layer formation and compaction. The operation conditions (e.g. SRT, biogas sparging rate, etc.) affecting parameters such as EPS and PSD which are effective on cake layer formation, characteristics and compaction, can be adjusted to control cake layer characteristics and thus total filtration resistance using mitigating procedures.

Another parameter that can be used to estimate the filterability of the sludge is CST. SRF and CST can be used together to evaluate the filterability and dewaterability of the sludge. CST can be used as a supplementary data to assess the fouling potential of the sludge in AnMBRs. The average CST_n values of the bulk sludge were 14±3 s/(g/L) and 35±5 s/(g/L) in R20 and R40, respectively. CST results fully supported our above-described results. CST of the bulk sludge from R20 was almost half of that from R40, which is consistent to the SRF data. It can be inferred from the CST and SRF results that the bulk sludge in R20 had better filterability characteristics and lower potential for increasing cake compactness compared to the bulk sludge in R40.

4.3.3 Overall Discussion

The results showed that a stable operation was possible for a prolonged period of time. Combination of backwashing and biogas sparging enabled the control of the dynamic cake layer thickness, which is of pivotal importance for achieving stable operation and high quality permeate. Decreased EPS amounts in the bulk sludge, which was measured with prolonged SRT, resulted in an increase in both the amount of small particles and sludge SRF that caused higher TMP and higher filtration resistance. By finding optimum operation conditions, enabling an effective cake layer formation and consolidation for providing a stable and high quality permeate, together with reasonable filtration resistances, AnDMBR may be considered as a reliable and satisfactory alternative wastewater treatment technology.

4.4 Conclusions

The applicability of DM technology for the treatment of concentrated wastewaters was investigated in this study. The submerged AnDMBR achieved over 99% organic matter removal and TSS removal. As an alternative to microfiltration or ultrafiltration membranes, polypropylene mono-monofilament filter cloth was used as support material to form a DM (cake) layer and to provide high quality filtration by this self-forming layer. SRT was found to be an important factor, having a significant effect on SMP and EPS production, P/C ratio, bulk sludge PSD, DM layer formation and consolidation, as well as bulk sludge filterability. The DM filtration concept turns one of the most important disadvantages of MBRs, that is: membrane fouling, into an advantage. The use of low-cost support materials instead of membranes, combined with biogas production as an energy source, can make DM technology feasible for the anaerobic treatment of concentrated wastewaters.

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CHAPTER 5

CHARACTERISTICS AND ROLE OF DYNAMIC MEMBRANE LAYER IN ANAEROBIC MEMBRANE BIOREACTORS

Abstract
A laboratory scale submerged AnDMBR was operated for the treatment of concentrated wastewater. The DM or cake layer was characterized on its physicochemical and biological composition and the role of the DM layer in treatment and filtration performances was assessed. Pyrosequencing was employed to compare microbial communities between bulk sludge and DM layer. Total COD removal efficiencies of about 99% were achieved at a sludge retention time of 20 days. The results showed that the DM layer had an important role in organic matter removal. Morphological analyses indicated that both organic and inorganic materials, such as sludge particles, SMP, EPS, and Ca, N, P, Mg precipitations contributed to the DM layer formation. Furthermore, the results suggested that SMP and EPS contributed to the formation of a dense cake layer and thus, effective retention of very small particles by the DM layer was achieved. Pyrosequencing analyses showed that diversity and richness of the microbial communities in the DM layer were high and microbial population composition in the DM layer was different compared to the bulk sludge in the AnDMBR. Overall, this study provided a better understanding about the DM layer structure in AnDMBRs, which might lead to increased applicability of this promising technology for the treatment of concentrated wastewaters to obtain a stable and high quality permeate.

This chapter is based on:

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5 CHARACTERISTICS AND ROLE OF DYNAMIC MEMBRANE LAYER IN ANAEROBIC MEMBRANE BIOREACTORS

5.1 Introduction

MBRs have been used for many years as a promising and well known technology for the treatment of various kinds of wastewater (Judd, 2006). With growing application experiences from aerobic MBRs, AnMBRs have received much attention and become more attractive and feasible, due to their capability to achieve high permeate quality, energy production and complete biomass retention (Liao et al., 2006; Skouteris et al., 2012; Smith et al., 2012; Ozgun et al., 2013). However, MBR technology has still challenging problems that need to be solved. Membrane fouling is the most important obstacle that limits the practical applications of MBRs (Wang et al., 2008; Meng et al., 2009; Gao et al., 2010a). Moreover, fouling results in high TMP, high filtration resistance, low flux, and frequent membrane cleaning and/or replacement requirement, which increase the operational costs. Various types of foulants may be responsible for membrane fouling such as SMP, EPS, microorganisms, and/or inorganic precipitates (Meng et al., 2009; An et al., 2009).

Membrane fouling occurs by cake layer and/or gel layer formation on the surface of the membrane and/or by pore clogging (Lee et al., 2001). Among these two causes, generally cake layer formation is the main contributor to the fouling in aerobic and anaerobic MBRs (Jeison and van Lier, 2008; Meng et al., 2009). However, a cake layer has the advantage of acting as a filter because it has a rejection capability. By this way, rejection properties are more dependent on the cake layer rather than the membrane itself and thus a cheap support material such as woven or non-woven filter cloth enabling the formation of a cake layer can be used instead of a membrane. Cake layer filtration is also referred as DM filtration in which selection of the appropriate support material, homogeneity of the cake layer on the support material surface, and maintenance of the cake (DM) layer of a certain thickness are essential to obtain a stable permeate quality (Ersahin et al., 2014). DM layer can be self-formed by the wastewater solid particles and by the prevailing microorganisms including their excretion products, such as EPS and SMPs. Since the filtration is accomplished by the DM layer, support materials with larger pore size are possible alternatives in DMBR technology instead of microfiltration or ultrafiltration membranes. DM layer can be removed easily by backwashing and/or air/biogas sparging without chemical cleaning (Chu et al., 2008; Ersahin et al., 2012). The possibility to use low-cost support materials would make wastewater treatment feasible by DM filtration in terms of operation and maintenance.

Stable flux and operation were obtained in aerobic DMBRs (Fan and Huang, 2002; Fuchs et al., 2005; Chu and Li, 2006; Ren et al., 2010; Xiong et al., 2014; Poostchi et al., 2015). Cake layer is mainly responsible from the retention in DMBRs. Thus, when the cake layer over the filter is completely removed, permeate quality deteriorates till the cake layer re-forms again. Appropriate monitoring system should be provided to take reasonable precautions in case of this kind of situation in DMBRs. Cake layer is the most important barrier in MBR systems causing fouling which results in low fluxes. Cake layer is used as an advantage in DMBR technology since it provides an effective retention. However, the flux values in DMBRs are generally lower in comparison to those obtained in conventional MBRs. Therefore, the primary benefit of the DMBR technology is to obtain a stable treatment performance and high permeate quality rather than to achieve high fluxes.

Generally lower fluxes were obtained in AnDMBRs compared to aerobic DMBRs (Ersahin et al., 2012). Different floc morphology and particle size distribution of anaerobic sludge might be the reason for the differences in performance between AnDMBRs and aerobic DMBRs. Biomass, SMP and EPS were indeed found to be the main contributors of DM formation in terms of organic components in AnDMBRs (Zhang et al., 2010; Gao et al., 2010a). Besides, a number of chemical elements including Mg, Al, Ca, Si, and Fe provide a rough and dense cake layer (An et al., 2009). Inorganic elements can also play a bridge function between biopolymers and cells, which increases the density and strength of the cake layer (Meng et al., 2007). Lin et al. (2011) found that colonization of bacterial clusters and small flocs enhanced cake formation on a membrane surface in a submerged AnMBR. They reported that microbial intensity and diversity inside the cake layer and bulk sludge were remarkably different, which contradicts the results of Zhang et al. (2011). Most of the studies about cake layer formation were conducted in conventional AnMBRs rather than AnDMBRs and there is limited research on AnDMBR technology. Therefore, further study is needed to understand the formation mechanisms of the DM layer and identify the optimum conditions for achieving an effective DM layer, by which a stable permeate quality and pollutant removal can be obtained. Moreover, cake layer characterization should be evaluated together with the operational results in an AnDMBR in order to determine the relationships between operational parameters and DM layer characterization. This approach would help to understand the cake layer formation that enables a stable operation in AnDMBRs.

The aims of this study were to characterize the DM layer and to investigate its role in the treatment of high strength organic wastewaters in AnDMBRs. The role of the DM layer in biological removal performance in terms of particulate and soluble organic matter removal was determined. Moreover, morphological and microbial characteristics of the DM layer were elucidated. The study focused on the structure of DM layer in order to obtain a detailed understanding of the cake layer formation. Pyrosequencing was used to compare the microbial community structures, including both archaeal and bacterial communities, of the bulk sludge and cake layer.

5.2 Material and Methods

5.2.1 Experimental Set-up

A submerged AnDMBR was used in the study (Figure 4.1). Details of the set-up are explained in Section 4.2.1.

5.2.2 Experimental Procedure

The AnDMBR was operated at a SRT of 20 days. OLR and HRT were set at 2 kg COD/m^3.d and 10 days, respectively. Average temperature inside the AnDMBR was controlled at 35.7±0.1 °C. AnDMBR operation was conducted at a flux of 2.2 L/m^2.h.

Biogas recirculation and backwashing procedures are explained in Section 4.2.2.

5.2.3 Wastewater Source and Seed Sludge

Details about the substrate, seed sludge and start-up period are explained in Section 4.2.3.

5.2.4 Methods

5.2.4.1 Analysis Techniques

Measurements of COD, TSS, VSS, TS and VS were performed following Standard Methods (APHA, 2005). Methods for the soluble COD, SMP, EPS, PSD and methane content analyses are explained in Section 4.2.4.1. Volatile fatty acids (VFAs) were measured using a Focus GC (Thermo Scientific) equipped with a flame ionization detector. A 30 m long column (Hewlett Packard HP INNOWAX) with an internal diameter of 0.25 mm and a film thickness of 0.25 µm were used to separate VFAs.

5.2.4.2 Environmental Scanning Electron Microscopy (ESEM) and Energy Dispersive X-ray (EDX) Analyzer

DM layer is formed by deposition of the bulk sludge (due to the adhesion and deposition of particles in the sludge) on the filter surface, and the DM layer can be easily removed physically. However, gel layer with a crystal structure is formed under the DM layer. The gel layer is adhered to the filter cloth very tightly and it is difficult to remove it physically.

DM (cake layer) specimens were freshly sampled over the filter surface and cut into small pieces (1 cm x 1 cm). The cake layer was physically removed in order to obtain the gel layer specimens. The cake layer was carefully scraped off by a plastic sheet and simultaneously flushed. The entire cake layer deposited over the filter surface was removed before taking the gel layer samples. Gel layer and cake layer specimens were firstly fixed by means of 2% (v/v) glutaraldehyde in 0.1 M phosphate buffer for 2 hours and then washed using phosphate buffer twice for 10 minutes each. All the samples were stored at -25 °C after air-drying. For ESEM analysis, samples were mounted on a 1 cm^2 metal support and kept in place with conductive tape and examined with an ESEM (Philips XL30). The ESEM photographs were taken at the end of the operation period. In order to identify the chemical components, elemental analysis was also performed on the samples. The EDX system by EDAX (EDAM 3 EDX system, SUTW 3.3 EDX window and 128.0 eV EDX resolution) was applied to determine the major elements of the DM layer.

5.2.4.3 Atomic Force Microscopy (AFM)

The surface morphology and roughness were determined by means of an AFM. The sample pretreatment for AFM analysis was the same as that applied for the ESEM analysis. The AFM analyses were carried out in tapping mode with the microscope P47-SPM-MDT (Russia, NT-MDT). AFM was equipped with silicon cantilevers having a tip radius less than 10 nm and 20 degree apex angle (NSC11, Estonia, Mikromasch) and conductive cantilevers (silicon coated with Ti-Pt) having a tip radius of 40 nm and 30 degree apex angle (CSC21, Estonia, Mikromasch).

5.2.4.4 Fourier Transform Infrared Spectroscopy (FTIR)

The DM specimens for FTIR analysis were air-dried before FTIR analysis (Perkin Elmer Spectrum 100). The FTIR spectra in absorption mode were recorded in the range of 400 to 4000 cm^{-1}.

5.2.4.5 Microbial Analysis

Many DNA sequences can be generated in a single run by pyrosequencing technology. Richness and diversity of species are determined with pyrosequencing (Sanapareddy, et al., 2009; Lim et al., 2012). Pyrosequencing was reported as a powerful molecular method to determine the complete structure of microbial communities in domestic wastewater treatment plants and also industrial wastewater treatment plants (Zhang et al., 2012; Hu et al., 2012; Zhu et al., 2013). In this study, pyrosequencing was used to compare the microbial community structures, including both archaeal and bacterial communities, of the bulk sludge and the cake layer.

Bulk sludge was sampled three times (day 1, 22, and 50) during the operation and DM samples were collected once at the end of the operating period (day 50). Fresh samples, i.e. 5 ml for bulk and seed sludge samples, and 2 cm x 2 cm for DM samples, were washed twice with PBS and then centrifuged at 10,000xg for 3 minutes. The supernatant was removed before storage. All samples were stored at -25 °C until DNA extraction.

DNA extraction was carried out using a MoBio UltraClean microbial DNA isolation kit (MoBIO Laboratories, Inc., CA, USA) following the manufacturer's protocol. A combination of heat, detergent, and mechanical force was used to increase the efficiency in DNA isolation process. A minor modification including twice bead-beating (5 minutes) and heating (65 °C, 5 minutes) was applied to the protocol in sequence in order to enhance the lysis efficiency of microbial cells. DNA isolation was confirmed by agarose gel electrophoresis and the concentration of DNA was measured using Nanodrop 1000 equipment (Thermo Scientific, Waltham, MA, USA).

The amplification of the 16S rRNA gene was carried out at Research and Testing Laboratory (Lubbock, TX, USA) with universal primers U515F (GTG CCA GCM GCC GCG GTA A) and U1071R (GAR CTG RCG RCR RCC ATG CA) (Wang and Qian, 2009). Pyrosequencing of the 16S rRNA gene was carried out by using a Roche 454 GS-FLX system (454 Life

Science, Branford, CT, USA) with titanium chemistry. By testing on Ribosomal Database Project (RDP, Maidak et al., 1997), forward and reverse primers target both bacterial and archaeal DNA. Pyrosequencing data were processed using the Quantitative Insights Into Microbial Ecology (QIIME, version 1.6.0) pipeline (Caporaso et al., 2010).

5.3 Results and Discussion

5.3.1 Treatment Performance

Soluble COD concentrations in the bulk sludge of the bioreactor and in the permeate were measured during the study. After a stable and effective DM layer was formed on the support layer, by which a high and stable removal efficiency and permeate quality could be obtained, the average soluble COD concentration in the permeate was 105±25 mg/L (Figure 5.1). Average methane production in the AnDMBR was 2.2 L/day with a methane content of 68%. Besides, total COD removal efficiency of the AnDMBR was over 99%. It is expected that the DM layer plays an important role in the removal of soluble organic matter in AnDMBRs. The importance of the cake layer with regard to soluble COD removal in conventional AnMBRs was reported in various studies (e.g. Jeison and van Lier, 2007; Lin et al., 2009; Ersahin et al., 2012). The positive effect of the cake layer on soluble COD removal may be attributed to the biodegradation in the DM layer. The other possible explanation for the difference between the soluble COD in the bulk sludge and permeate may be physical retention by DM layer of soluble organic matter with large molecular weight. The positive effect of cake layer on soluble COD removal was observed in AnMBRs using a microfiltration membrane (Hu and Stuckey, 2007; Xu et al., 2011; Smith et al., 2013). Moreover, similar results have been obtained for aerobic DMBRs (Fan and Huang, 2002; Wu et al., 2005). A soluble COD removal efficiency of 34% by the DM layer was reported by Fan and Huang (2002) in a submerged aerobic DMBR treating municipal wastewaters. Considering the above, we postulate that a combined effect of both biomass activity and physical retention in the DM layer might be responsible for the removal of soluble organics.

An effective DM layer was achieved in between 10-20 days in the study. It is important to take into consideration the time required to form an effective DM layer in AnDMBRs, since the support material, unlike a membrane, can provide only limited separation by itself. The required time may vary according to the characteristics of the support material, morphology and concentration of sludge in the bioreactor, substrate type and operating conditions. To keep the permeate quality high, the permeate flow can be returned to the bioreactor until the formation of an effective DM layer has been established. Furthermore, one of the most important challenges in AnDMBRs is to keep the thickness of the DM layer within an optimum range in order to achieve an effective treatment. This is a prerequisite to obtain a stable and high permeate quality and to avoid unexpected increases in TMP (Ersahin et al., 2012).

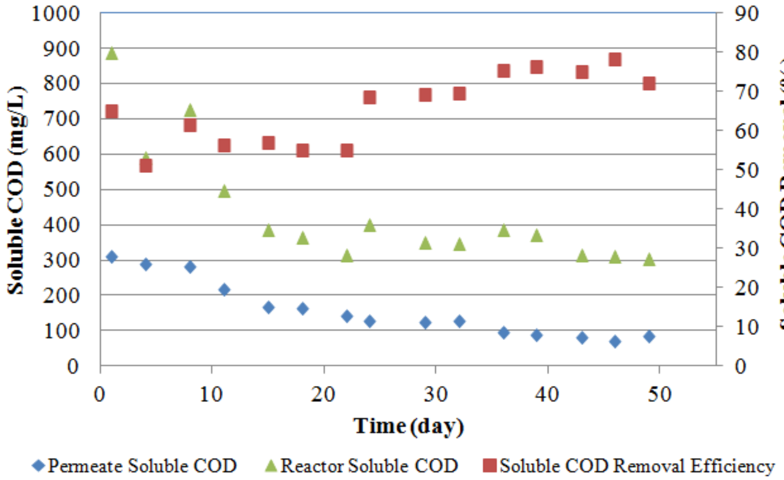

Figure 5.1. Soluble COD concentrations and soluble COD removal efficiency in the AnDMBR.

After starting up the operation with a new support material, initial TSS concentrations in the permeate were significantly high. TSS concentrations of up to 170 mg/L were measured in the permeate. A similar result was also observed by Kiso et al. (2000) and Seo et al. (2002) in DMBRs. With the formation of an effective DM layer, TSS concentrations decreased gradually to below 10 mg/L and became stable throughout the study period.

In order to determine the role of the DM layer in VFA removal, total VFA concentrations in the bulk sludge and permeate were measured. The highest concentration measured in the bulk sludge was that of acetic acid, which ranged between 20-40 mg/L. VFAs can pass through the pores of the support material (Martinez Sosa et al., 2011). Since the pore size of the support material was around 10 µm, it was expected to determine similar VFA concentrations in the permeate as in the bulk sludge. However, over 50% of the total VFA was removed by the DM layer. Total VFA concentration in the permeate was between 10-15 mg/L. This reduction could be attributed to microbial biodegradation in the DM layer. The role of the DM layer as a secondary membrane was mentioned by various studies (Jeison et al., 2008; Hu and Stuckey, 2007; Lin et al., 2009; Ersahin et al., 2013). Recently, the ability of the cake layer to remove VFA in submerged AnMBRs was also reported (Ho and Sung, 2010; Gao et al., 2010b; Martinez-Sosa et al., 2011).

5.3.2 Morphological, Chemical and Microbial Characteristics of DM Layer

5.3.2.1 ESEM-EDX Analyses

The surface of the virgin polypropylene mono-monofilament woven filter cloth is shown in Figure 5.2(a), which demonstrates a porous and smooth structure of the regularly oriented woven fibers. At the end of the operating period, a complex DM layer was quite obvious (Figure 5.2(b)). As can be seen in Figure 5.2(b)-(d), the DM layer was not formed from only one matter e.g. only biomass, but it also contained other accumulated materials such as EPS-like materials and various kinds of inorganic compounds. The support material was covered by a heterogeneous cake layer. Following the elemental analysis using the EDX analyzer, C, O, N, P, Mg, Ca, Na, Si, Al, Cl, and K were detected in the DM layer as the major elements. Some of the elements, more specifically Ca, Mg, Al, and Si have been reported to be important contributors for the formation of the cake layer in MBRs. These elements can play a bridge role, even at low concentrations, between the microbial cells and biopolymers. Moreover, microbial cells or biopolymers can catch the metal ions by charge neutralization and this result in the formation of enhanced DM layer (Seidel and Elimelech, 2002; Meng et al., 2007; Wang et al., 2008; Herrera-Robledo et al., 2010; Gao et al., 2011). Thus, a compact and less porous DM layer can form during filtration.

Some fluffy matters detected on the filter cloth (Figure 5.2(d)), seem to consist of EPS, following the elemental composition revealed by EDX. The main elements in this part of the cake layer were C, O, N, and P. Besides, precipitate-like materials in the DM layer were identified in Figure 5.2(c). The major elemental composition of this material was 44.1% O, 35.4% C, 8.5% N, 5.8% P, and 4.3% Mg. Choo and Lee (1996) reported that struvite, one of the main inorganic foulants in AnMBRs, plays an important role in the formation of cake layers. Moreover, since these elements mainly originated from the feed solution, the type of substrate is very important for the inorganic scaling in AnMBRs. Therefore, the concentration of inorganic compounds in the substrate should be considered, while dealing with control of the fouling and/or DM layer.

Different materials such as EPS, SMP and inorganic compounds accumulated in the DM layer. During long-term continuous operation, these materials will also cover the microbial cells. Therefore, it was difficult to identify the exact microbial morphology by using ESEM images. However, microbial analyses were carried out to obtain detailed information on the microbial composition in the DM layer and results are discussed in further sections.

A partial occurrence of a gel layer under the cake layer can be seen in Figure 5.2(e). The gel layer seems like mineral material (crystal structure), adhered to the surface of the support material and consisted of C, O, Cl, N, Na, Ca, P, Mg, and S. This gel layer adhered to the filter cloth very tightly and it was difficult to remove it physically. Gel layer formation was reported in dynamic MBRs previously (Fan and Huang, 2002; Satyawali and Balakrishnan, 2008). Besides, some porous and spherical structures were also identified under the cake layer (Figure 5.2(f)). The EDX analysis showed that the main elements detected in this part were 72.1% C, 10.4% Ca, and 8.4% O. Those spherical shaped deposits might be calcium carbonate (Al-Jaroidi et al., 2010). Ca was also reported to effect cake layer compactness and it has a bridging function in the cake layer in MBRs (Lin et al., 2009; Zhou et al., 2014).

Following the ESEM and EDX results, we confirmed accumulation of a mixture of mineral-like materials, inorganic deposits and EPS-like materials inside the DM. This accumulation provided a dense DM layer and provided a better retention of soluble COD.

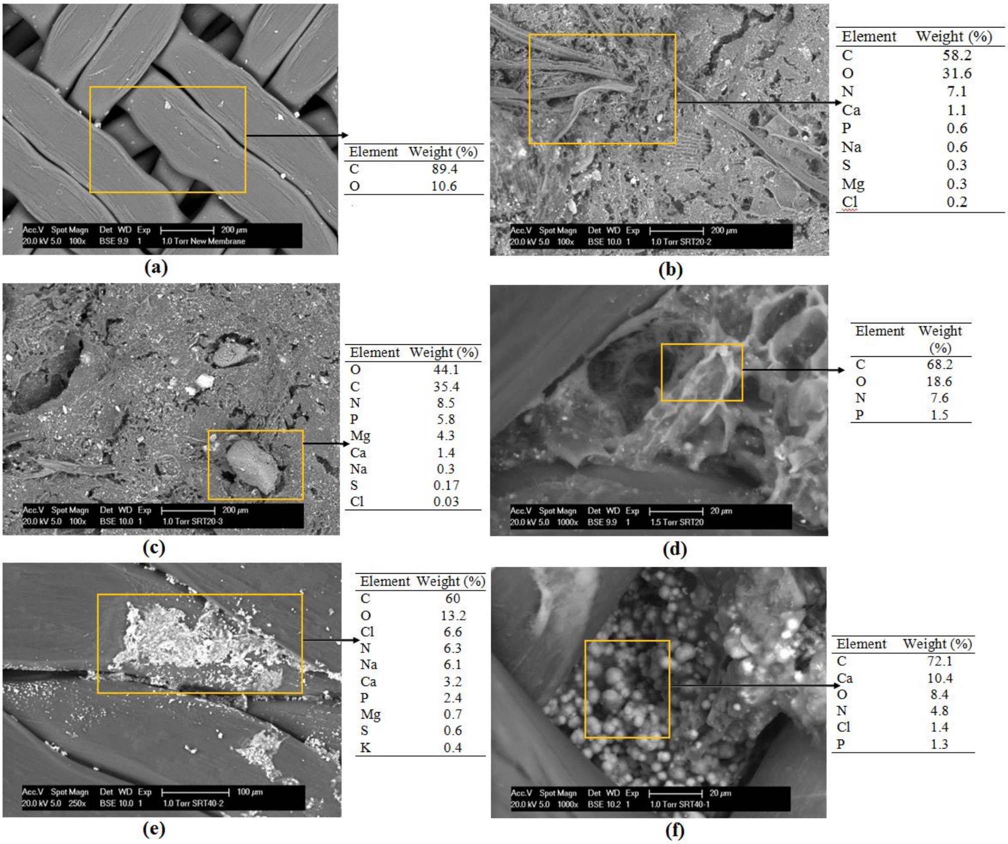

Figure 5.2. ESEM images: (a) virgin support material, (b)-(c) DM layer at the end of operation period, (d) EPS-like material, (e) partial gel layer under the DM layer, (f) spherical structures formed under the DM layer.

5.3.2.2 AFM Analyses

The surface roughness can be described as closely spaced irregularities, and it is quantified by the vertical spacing of a real surface from its ideal form (Thomas, 1999). The calculation of surface roughness by using AFM can be found in the study of Meng et al (2010). The surface roughness that can be observed by means of AFM analyses is generally used as an indicator of compactness for the DM layer and it also provides information about fouling in MBRs (Lin et al., 2009; Shen et al., 2010; Meng et al., 2010). A low roughness usually means a compact structure (Yu et al., 2006), thus, a DM layer with a high roughness may provide a better retention performance. AFM images of the cake layer structure are presented in Figure 5.3. The average roughness values were obtained based on a 30 µm x 30 µm scan area. Figure

5.3(a) shows that the virgin support material exhibited a smooth surface. The roughness of the virgin support material was 143 nm. Average roughness of the support layer after gel layer formation was 98 nm. These results show that the roughness of the support material decreased slightly after gel layer formation. This might be because the attachment of the gel layer on the surface of the support material resulted in a smoother surface in comparison to the virgin filter cloth. However, an increase in the roughness to 724 nm (Figure 5.3(b)) was measured after DM formation. This increase occurred possibly due to the deposition of different materials on the filter surface, and the uneven distribution of these materials. Moreover, the upper part of the DM layer was scraped off and the bottom part of the cake layer was also investigated by AFM based on 10 µm x 10 µm scan area (Figure 5.3(c)). It is possible to identify a few rod and berry shaped microbial cells in the bottom part of the DM layer. This showed the availability of biomass underneath the cake layer and the retained microorganisms might play a role in biodegradation of the organic matter during filtration through the DM layer.

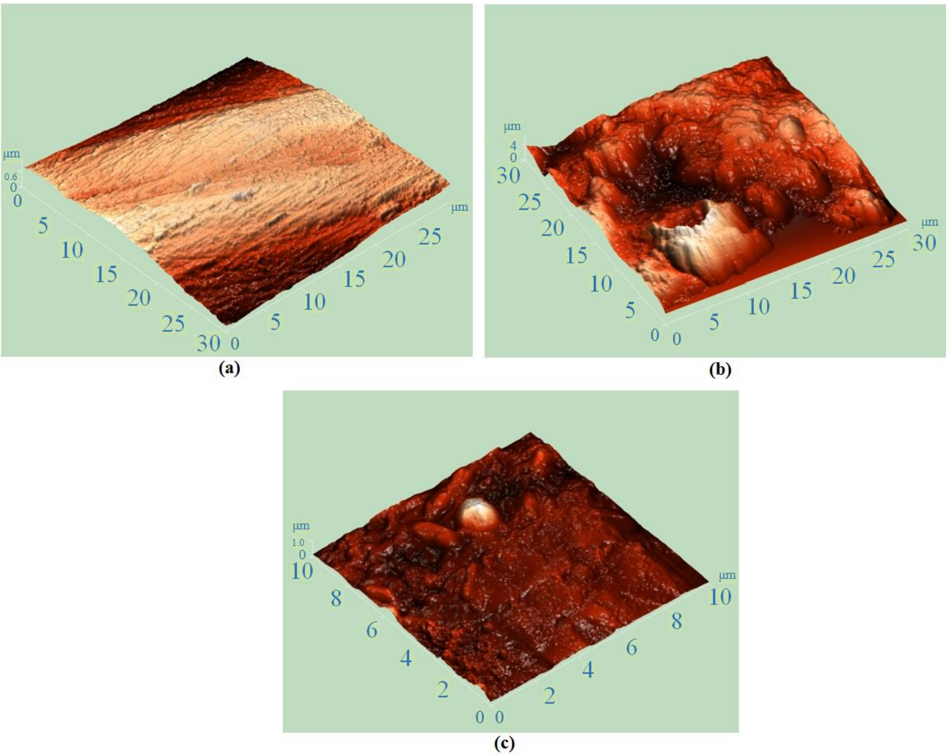

Figure 5.3. AFM images: (a) virgin support material, (b) DM layer, (c) bottom part of the DM layer.

5.3.2.3 FTIR Analyses

The FTIR spectra of the virgin filter cloth, gel layer and DM layer are presented in Figure 5.4. The peaks appearing in the spectra of the virgin support material and the gel layer were close to each other. However, a significant difference was observed between the DM layer and virgin filter cloth. Two peaks at 1643 cm^{-1} and 1541 cm^{-1} in the spectrum of the DM layer indicated a protein secondary structure; amides I (stretching of C=O and C–N bonds) and amides II (deformation of N–H and C=N bonds), respectively (Maruyama et al., 2001; Gao et al., 2011). The peaks of 1446 cm^{-1} and 1249 cm^{-1} represented the existence of amides III (C–N stretching) (Lin et al., 2009). Moreover, there was a quite distinct peak at 1025 cm^{-1} which is typical for polysaccharides-like substances including C–O bonds (Kimura et al., 2005). The peak at 3286 cm^{-1} was also indicative for the stretching of the O–H bonds in polysaccharides and the peak at 2920 cm^{-1} corresponded to aliphatic C–H stretching (An et al., 2009; Gao et al., 2011). Furthermore, the peak at 1727 cm^{-1} was found to be representative for humic acids (stretching vibration of COO^-) (Kimura et al., 2005). The results of FTIR spectrum showed the existence of proteins- and polysaccharides-like substances in the DM layer. Therefore, it can be expected that the amount of EPS and SMP would be high in the DM layer since these materials would accumulate on the support material surface. Based on the ESEM-EDX and FTIR results, it was concluded that the DM layer was composed of both organic and inorganic matter that accumulated on the filter cloth surface.

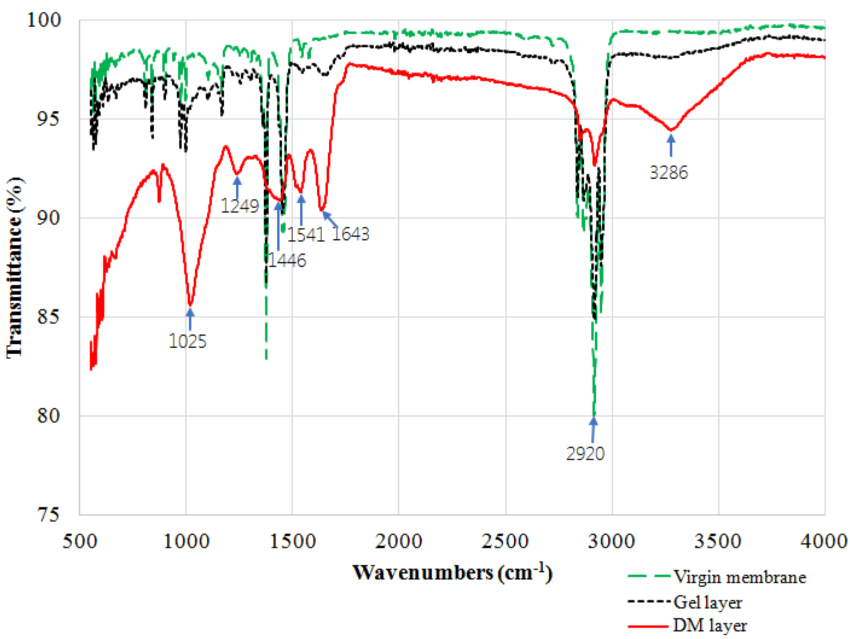

Figure 5.4. FTIR spectrum of the virgin and used support layer surfaces.

5.3.2.4 SMP-EPS Analyses

In order to determine whether there was a substantial accumulation in the DM layer, SMP and EPS contents were measured in both the DM layer and the bulk sludge at the end of the operational period (Table 5.1). The SMP and EPS contents in the bulk sludge were remarkably lower than those in the DM layer (Table 5.1). Average EPS and SMP contents of the DM layer were over 21.5 and 5.8 times higher than those of bulk sludge, respectively. A

P/C ratio of 1.9 was obtained for EPS in the DM layer, which was over 3 times higher than the ratio obtained for the bulk sludge. The results in Table 5.1 showed that SMP could be retained by the DM layer, which is also consistent with the difference in soluble COD concentrations between bulk sludge and permeate (Figure 5.1). High EPS accumulation enhances sludge adhesion by polymeric interactions and contributes to membrane fouling (Tsuneda et al., 2003; Meng et al., 2009; Gao et al., 2010a). Moreover, it was reported that the affinity between proteins and sludge particles was greater compared to that between polysaccharides and sludge particles (Masse et al., 2006; Lin et al., 2011). Therefore, the increase in P/C ratio with the accumulation of EPS provided a tight cake layer and thus, an efficient retention performance could be achieved by the DM layer. However, a high P/C ratio in the DM layer in AnDMBRs may result in a higher TMP and filtration resistance during operation compared to conventional MBRs that are operated without a DM.

Table 5.1. SMP and EPS compositions in the bulk sludge and DM layer.

	SMP			EPS		
Sample	**Protein (mg/g VSS)**	**Polysaccharide (mg/g VSS)**	**P/C**	**Protein (mg/g VSS)**	**Polysaccharide (mg/g VSS)**	**P/C**
Bulk Sludge	32	13	2.5	1.8	3	0.6
DM Layer	143	116	1.2	68	35	1.9

5.3.2.5 PSD and TS/VS Analyses

In AnMBRs, small particles have a tendency to accumulate in the cake layer rather than bigger particles (Lin et al., 2011). However, these particles can adher together in the presence of EPS-like material, in which bivalent cations may act as electrostatic bridges. Therefore, the particle size might increase on the surface of the support material due to tight adherence. The PSD of both the bulk sludge and the DM layer is shown in Figure 5.5. In the bulk sludge, the average particle size was 45 µm, whereas this size was 66 µm in the DM layer. Small particles attached on the support layer surface might stuck together with the help of polymeric substances, e.g. EPS, and some inorganic elements which have a bridging effect between the cells and polymers. This strong adhesion might result in an increase in the PSD of the DM layer.

TS and VS compositions in the DM layer were also analyzed. TS and VS mass were found as 28.4 mg TS/cm^2 and 22.9 mg VS/cm^2, respectively. The VS/TS ratio of 81% indicated that mainly the organic fraction contributed to the DM layer. These results are consistent with the findings obtained in AnMBRs treating municipal wastewater (Herrera-Robledo, 2010; Zhang et al., 2011). However, the inorganic fraction in the cake layer (almost 20%) should not be underestimated. ESEM-EDX, AFM and FTIR results showed the existence of organic substances, inorganic precipitates and cellular biomass in the DM layer (Figure 5.2-5.4).

Average soluble COD removal rates in the bulk sludge and DM layer were 0.19 g COD/g VS.d and 0.07 g COD/g VS.d, respectively. The soluble COD removal rate in the bulk sludge was over 2.5 times higher than that in the DM layer. VFA removal rate by the DM layer was about 0.02 g VFA-COD/g VS.d. Zhang et al. (2011) also reported that the activity in the cake layer was lower than the activity in the bulk sludge in an AnDMBR system. Substrate and nutrient transfer inside the DM layer might be difficult due to the high amount of solids accumulation on the support material.

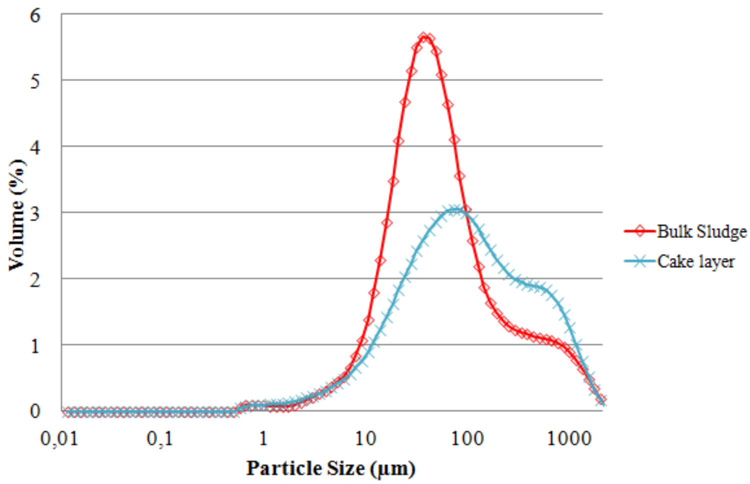

Figure 5.5. PSD of the bulk sludge and DM layer.

5.3.2.6 Microbial Community Analysis

Pyrosequencing of the five samples (seed sludge; bulk sludge samples taken on day 1, 22, 50, and DM layer sample taken on day 50) yielded 26318 sequences in total. All the bacterial and archaeal species detected in the seed sludge, bulk sludge and DM layer are presented in Table A1 and Table A2.

Firmicutes, *Bacteroidetes*, *Proteobacteria*, *Chloroflexi* and *Acidobacteria* were the five most predominant bacterial phyla in all samples (Figure 5.6). All the other phyla together only consisted about 2% of the total phyla. These five phyla contain several species that are known to participate in key anaerobic digestion processes such as hydrolysis, acidogenesis and syntrophic acetogenesis. *Firmicutes* was the most dominant phylum of bacteria in the AnDMBR and accounted for more than 40% of the total phyla in both bulk sludge and DM (Figure 5.6). *Firmicutes* were previously detected in cake layers or biofilms of AnMBRs (Yu et al., 2012; Calderon et al., 2011). *Bacteroidetes* were the second largest bacterial phylum in each sample (Figure 5.6). Many species belonging to this phylum have been reported to be capable of releasing high amounts of proteinaceous EPS in order to form biofilm (Gao et al., 2010a). The relative abundance of the phylum *Proteobacteria* increased from 11% in the seed sludge to 14% in the bulk sludge and 22% in the DM layer (Figure 5.6). The relative abundance of the phyla *Chloroflexi* and *Acidobacteria* were low but quite stable, varying in a range of 2~10% and 2~6%, respectively.

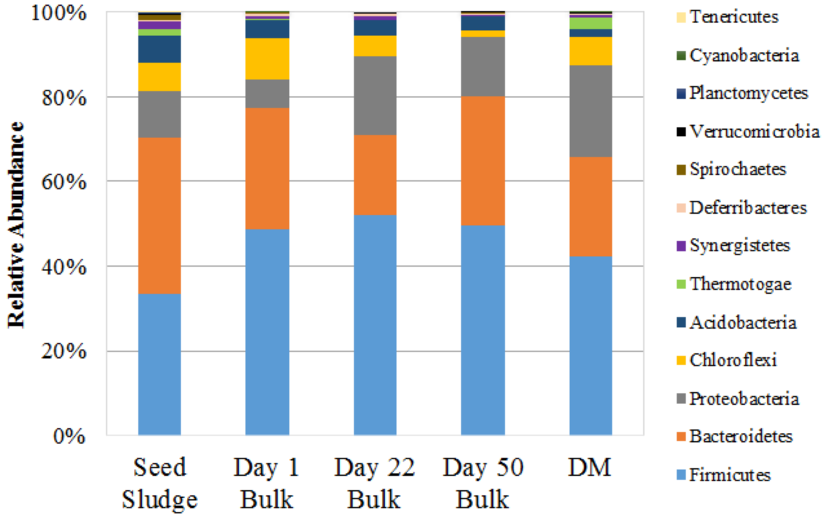

Figure 5.6. Classification at the phylum level of the bacterial communities in the seed sludge, bulk sludge and DM layer.

Remarkably, the abundance of genus *Syntrophus* in the bulk sludge decreased by 70% in comparison to the seed sludge, whereas it accumulated in the DM layer with an increase by 40% (Figure 5.7). It has been demonstrated that many species of genus *Syntrophus* are acetogens, which are highly dependent on the presence of hydrogenotrophic methanogens as syntrophic partners in anaerobic environments (Chen et al. 2005, Lykidis et al. 2011). In fact, a hydrogenotrophic methanogen, *Methanolinea mesophila* (Sakai et al. 2012), became the most dominant archaeal species in the DM layer (Figure 5.8), which is in accordance with the accumulation of *Syntrophus sp* in the DM layer, indicating a syntrophic interaction between those microorganisms. Previous studies have shown that the presence of hydrogenotrophic methanogens in cake layers was essential for interspecies electron/hydrogen transfer and improving the overall conversion of organic substances (Yu et al., 2012; Summers et al., 2010).

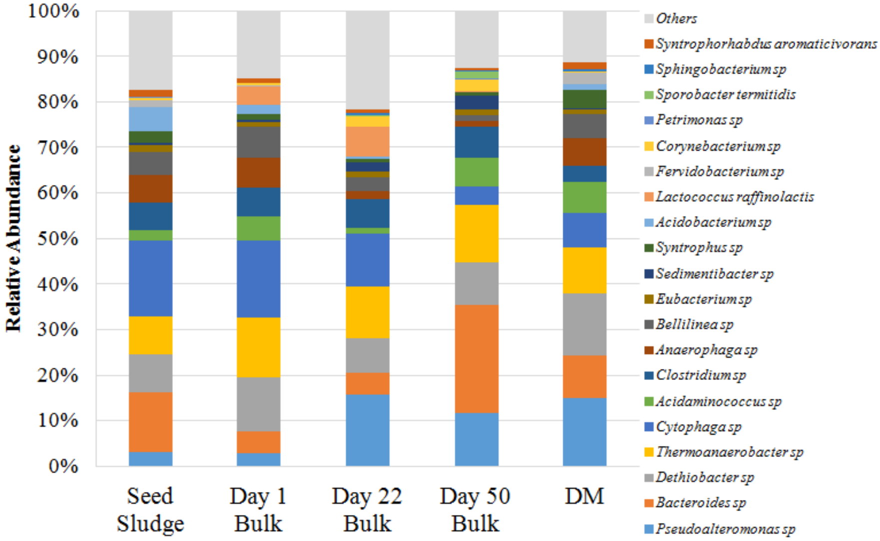

Figure 5.7. Major (top 20) bacterial species and their relative abundances in the bulk sludge and DM layer.

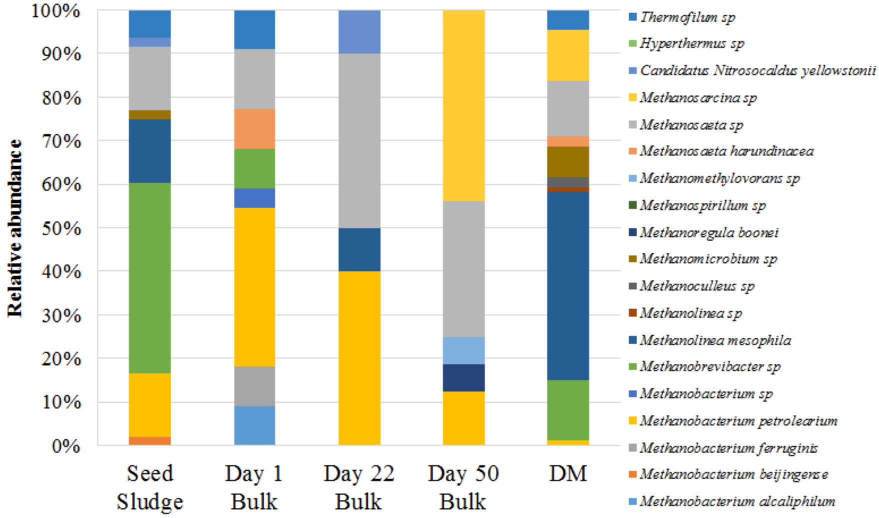

Figure 5.8. Archaeal species and their relative abundances in the bulk sludge and DM layer.

The genus *Pseudoalteromonas* was one of the most dominant bacterial genera in the DM layer (Figure 5.7). Some species of *Pseudoalteromonas* are commonly discovered on the surface of marine biotic/abiotic materials and they are associated with the production of biologically active extracellular agents (Holmstrom and Kjelleberg, 1999). Another study also demonstrated that some *Pseudoalteromonas* species can form biofilms and even produce extracellular protease (Xiong et al., 2007). Under laboratory conditions, *Pseudoalteromonas* species were proven to be able to form robust biofilms and produce extracellular proteases (Iijima et al., 2009). Above literature references indicate that the genus *Pseudoalteromonas* is highly involved in biofilm formation and maintenance. A dense and robust cake layer plays an important role in filtration by biomass activity and physical retention capacity in the AnDMBRs. In this study, the high abundance of *Pseudoalteromonas* bacteria in the cake layer might be linked to the high filtration performance of the AnDMBR.

Some other bacterial genera are also capable of adhering to the woven fibers, facilitating the development and the colonization of biofilms. EPS production is generally associated to carbohydrate degrading bacteria (Fukuzaki et al., 1995). Since EPS is a key material keeping sludge flocs together in biofilms, it is reasonable to link the high abundance (Figure 5.7) of carbohydrate degrading bacteria, such as *Bacteroides* (9%), *Thermoanaerobacter* (10%) and *Dethiobacter* (14%) to a well-functioning DM layer.

In addition to the hydrogenotrophic methanogen *Methanolinea mesophila*, the methanogens *Methanobrevibacter sp* (14%), *Methanosaeta sp* (13%) and *Methanosarcina sp* (12%) were also abundant in the cake layer (Figure 5.8). *Methanobrevibacter sp* was the most dominant (44%) archaeal species in the inoculum. However, it was not detected in the bulk sludge samples taken at days 22 and 50. This observation indicated that this genus was out-competed by other hydrogenotrophic mehanogens, but could survive in the DM layer. Previous studies proved that *Methanobrevibacter* had high abundance in biofilm-based anaerobic reactors (Araujo et al., 2004; Lu et al., 2012).

The microbial diversity results indicated obvious differences among samples. Both alpha diversity (Phylogenetic Diversity, Observed Species Number, Shannon-Wiener Index; Table 5.2) and beta diversity (Principal Co-ordinate Analysis plots; Figure 5.9) results were used in order to compare the microbial communities in the seed sludge, bulk sludge and DM layer. Alpha diversity and beta diversity can be used to characterize the microbial diversity over spatial scales (Peet, 1974). Alpha diversity refers to the diversity within a particular ecosystem (in our case, seed sludge, bulk sludge or DM layer samples), and is usually expressed by species richness and evenness. The richness was indicated by Phylogenetic Diversity Index and Observed Species Number, while the evenness was shown by Shannon-Wiener Index. Beta diversity is commonly used to examine the diversity variation among ecosystems. The seed sludge had the highest biodiversity and the DM had a slightly lower one. The bulk sludge community had much lower diversity compared to the DM one. Such difference in microbial diversity between bulk sludge and cake layer was also reported by PCR-DGGE analysis in a submerged AnMBR (Lin et al. 2011).

Although the alpha diversity level of the DM community was similar to that of the seed sludge, the beta diversity results showed that the microbial communities between the seed sludge and DM layer were remarkably different from each other (Figure 5.9), likely occurred because of niche differentiation. There are considerable differences in (micro)environments among the reactor where the seed sludge was taken, bulk sludge and the DM layer. Possible differences can be pH gradient, organic bioavailability, shear strength and hydrogen pressure. These differences, from an ecological perspective, could drive different microbial communities (Gao et al., 2012; Xia et al., 2012; Carbonero et al., 2012).

Table 5.2. Alpha biodiversity of seed sludge, bulk sludge and DM layer.

Biodiversity	Seed Sludge	Day 1 Bulk Sludge	Day 22 Bulk Sludge	Day 50 Bulk Sludge	DM
Phylogenetic Diversity	24.13±1.13	21.06±3.11	17.14±1.90	17.85±0.95	23.92±4.09
Observed Species Number	392.40±20.85	315.60±44.65	232.10±31.39	272.80±17.76	384.10±50.73
Shannon-Wiener Index	6.19±0.09	6.03±0.13	4.44±0.16	4.62±0.09	5.70±0.12

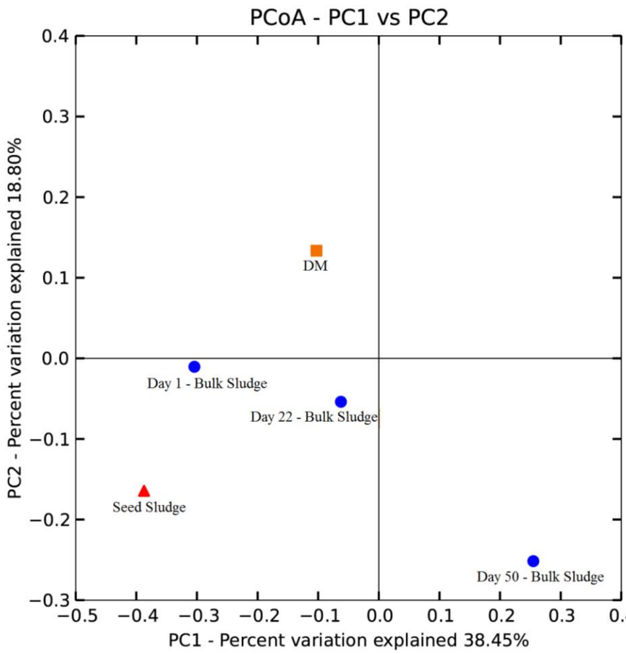

Figure 5.9. Principal co-ordinate analyses plots of bulk sludge and DM layer determined using the weighted UniFrac distance metric.

5.4 Conclusions

DM technology was applied in a submerged AnMBR in this study and structure of the DM layer was investigated in order to determine its role in filtration and treatment performance. The results showed that a stable and high organic matter removal efficiency was achieved with the AnDMBR. A removal efficiency of 99% for total COD, 65% for soluble COD and over 50% for VFA were obtained by the DM layer. The DM layer played a significant role in the removal of organic matter in the AnDMBR. The combined effect of biomass activity and physical retention capacity in the cake layer might be responsible for the removal by the DM layer. Pyrosequencing analyses demonstrated that diversity and richness of the microbial communities including bacteria and archaea in the DM layer were high and microbial population composition in the DM layer was different compared to the bulk sludge in the AnDMBR. According to the characterization analyses results, the DM layer was formed by both organic and inorganic materials. Besides, a partial gel layer formation under the cake layer, and accumulation of some mineral matter and sphere-like inorganic materials were detected. High accumulation of SMP and EPS in the DM layer contributed to the formation of a tight cake layer and effective retention of soluble COD. However, accumulation of the proteins and formation of a tight cake layer also caused an increase in the filtration resistance. Therefore, focusing on improvement of the current methodologies and/or development of new methods to control cake layer thickness and porosity, while minimizing energy input (e.g. required for biogas recirculation) and filtration resistance, and maximizing the flux, would be beneficial for the future applications of DM technology. Overall, this study provided a better understanding of the morphological and microbial characteristics of the DM layer in AnDMBRs.

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CHAPTER 6

IMPACT OF MEMBRANE CONFIGURATION ON TREATMENT AND FILTERABILITY PERFORMANCE OF ANAEROBIC DYNAMIC MEMBRANE BIOREACTORS

Abstract

Submerged and external AnDMBRs have been compared in terms of removal efficiency, filtration characteristics and microbial community structure. High COD removal efficiencies were obtained with both submerged and external AnDMBRs. To obtain an effective DM layer enabling high quality permeate, longer time was required in the external configuration compared to the submerged one. Suprisingly, a difference in microbial community structure was identified using pyrosequencing analyses between the submerged and external AnDMBRs. The number of archaeal types decreased in the bulk sludge of the external AnDMBR. External sludge recirculation might have had a negative effect on the archaeal community in the bulk sludge of the external AnDMBR. However, the sludge recirculation in the external configuration led to a filtration at lower total filtration resistance and TMP in comparison to the submerged one at the same gas sparging rate. Results showed that the submerged AnDMBR system can provide a shorter start-up period, slightly better permeate quality in terms of COD concentration and higher biogas production in comparison to the external one in gas-lift mode.

This chapter is based on:

Ersahin, M.E., Ozgun, H., Tao, Y., Gimenez, J.B., Spanjers, H., van Lier, J.B. Impact of membrane configuration on treatment and filterability performance of anaerobic dynamic membrane bioreactors. Under review.

6 IMPACT OF MEMBRANE CONFIGURATION ON TREATMENT AND FILTERABILITY PERFORMANCE OF ANAEROBIC DYNAMIC MEMBRANE BIOREACTORS

6.1 Introduction

Membrane integrated anaerobic bioreactor processes (AnMBRs) offer many advantages such as independent control possibility of SRT and HRT, small footprint, low sludge production, high effluent quality, and net energy production. Therefore, recently, a large number of scientific investigations have been performed from laboratory scale to full-scale applications for the treatment of various kinds of wastewater by AnMBRs (Liao et al., 2006; Stuckey, 2012; Skouteris et al., 2012; Ozgun et al., 2013; Lin et al., 2013). However, membrane fouling causing flux decrease and negative consequences in terms of operating costs is still an important problem that limits the widespread application of AnMBRs, especially at full-scale applications. Cake layer formation by organic and inorganic particles on the membrane surface is the major contributor of the fouling in AnMBRs (Jeison and van Lier, 2007; Xie et al., 2010).

The applicability of the cake layer formed on a support material as a filter for treatment of wastewaters has been researched in recent years (Jeison et al., 2008; An et al., 2009; Zhang et al., 2010; Ersahin et al., 2014). Different types of low cost materials can be used as support material enabling the formation of a cake layer, which is called a DM layer. Filtration is conducted by the DM layer instead of the filter itself in DM filtration technology. DM technology can be used in aerobic and/or anaerobic MBRs (Satyawali and Balakrishnan, 2008; An et al., 2009; Zhang et al., 2010; Ersahin et al., 2012). High organic and particulate matter removal/retention efficiency reaching 99%, was achieved by submerged AnDMBRs treating high strength wastewaters in long-term operation period (Ersahin et al., 2014). However, higher filtration resistances and lower fluxes may be obtained in AnDMBRs compared to conventional AnMBRs because the cake layer, which is manifested in AnDMBR systems, is the main contributor to total filtration resistance and fouling. Nonetheless, AnDMBR system may represent a cost effective alternative, owing to the use of low cost filter materials compared to more costly microfiltration or ultrafiltration membranes (Ersahin et al., 2013). Moreover, the DM layer can be removed when it is necessary by several physical methods without chemical cleaning, including backwashing, vibration, brushing and/or biogas sparging, and the DM layer can re-form on the support material. Development of cost-effective filter materials, using no chemical reagents for cleaning, and net energy production can make AnDMBRs feasible for the treatment of waste(water) treatment, including concentrated industrial or domestic (black water) wastewater and/or sludge.

The membrane unit can either be located inside or outside the bioreactor in AnMBR applications. In submerged AnMBR configurations, in which the membrane is located inside the bioreactor, the membrane is operated under a vacuum, brought about at the permeate site. When the membrane is located outside the bioreactor, that is: external AnMBR configuration, the membrane unit can be operated under a vacuum at the permeate site or pressure at the feed site (Liao et al., 2006). In the external AnMBR configurations, liquid can be delivered to the membrane unit by a liquid pump at a pre-determined cross-flow velocity, or biogas can be the driving force for the mixed liquor transfer from bioreactor to the membrane unit when applying a specified gas sparging velocity. Applications of liquid pumped (He et al., 2005; Saddoud et al., 2007; Abdurrahman et al., 2011) and gas-lift (Jeison and van Lier, 2007; Huang et al., 2011) external AnMBRs have been investigated previously.

Biogas sparging has been generally used to scour the membrane surface for fouling control in submerged AnMBRs. Dereli et al. (2012) reported that most of the full-scale AnMBRs treating industrial wastewaters are operated in submerged configuration with high COD removal efficiencies, that is: ≥95%. So far, most of the AnDMBR research has been conducted in submerged configuration (Ersahin et al., 2012). Jeison and van Lier (2008) reported that gas sparging energy and membrane cost of a submerged AnMBR was approximately three times lower than that of an external (side-stream) configuration, for a given flux. Similarly, it was indicated that the energy demand per produced permeate flow volume for submerged AnMBR configurations was much lower than that for pumped external AnMBRs (Martin-Garcia et al., 2011). However, a direct comparison of submerged and external AnDMBR configurations in terms of removal efficiency and DM filterability has not been reported yet. The purpose of this chapter was therefore to compare the removal efficiency and filtration characteristics of submerged and external AnDMBRs treating concentrated wastewater enabling to determine the impact of membrane configuration on treatment and filterability performance. Moreover, microbial community structure including bacterial and archaeal communities and the relative abundance of microbial species in the bulk sludge of submerged and external AnDMBRs were compared by using pyrosequencing.

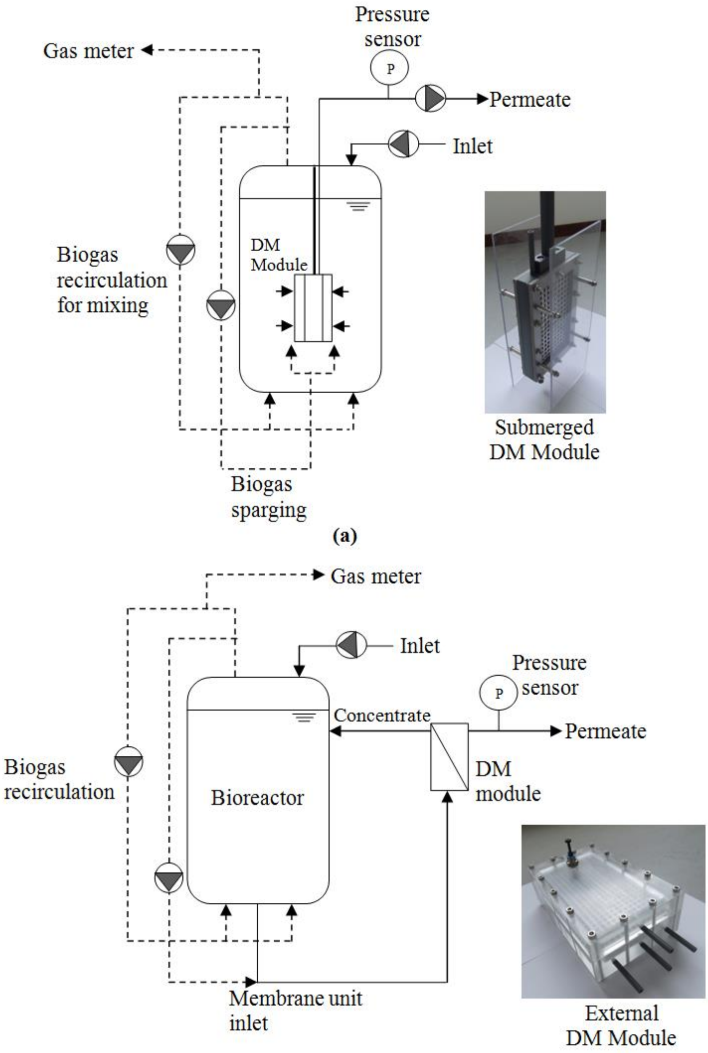
6.2 Material and Methods

6.2.1 Experimental Set-up

Laboratory scale submerged and external AnDMBR set-ups were used in this study (Figure 6.1). Submerged and external AnDMBRs are referred as RS and RE, respectively. Glass made completely mixed anaerobic reactors with an effective volume of 7.4 L were used in both set-ups. Flat sheet membrane modules with a total filtration area of 0.014 m^2 were used in the RS and RE. Filter material and equipments used in both set-ups were the same and are explained in Section 4.2.1. The external membrane module and AnDMBR set-up are shown in Figure 6.2 and Figure 6.3.

Both RS and RE were operated in gas-lift mode. Produced biogas was recycled by diaphragm pumps (KNF, N86 KTDCB) to provide mixing inside the bioreactors and to scour the DM surface for fouling control. Mixing diffuser was located at the bottom of the bioreactor and the biogas sparging diffuser was placed under the membrane module in RS (Figure 6.1).

Similarly, mixing was accomplished by a diffuser located at the bottom of the bioreactor in RE.



(b)

Figure 6.1. Laboratory scale set-ups: (a) submerged AnDMBR, (b) external AnDMBR.

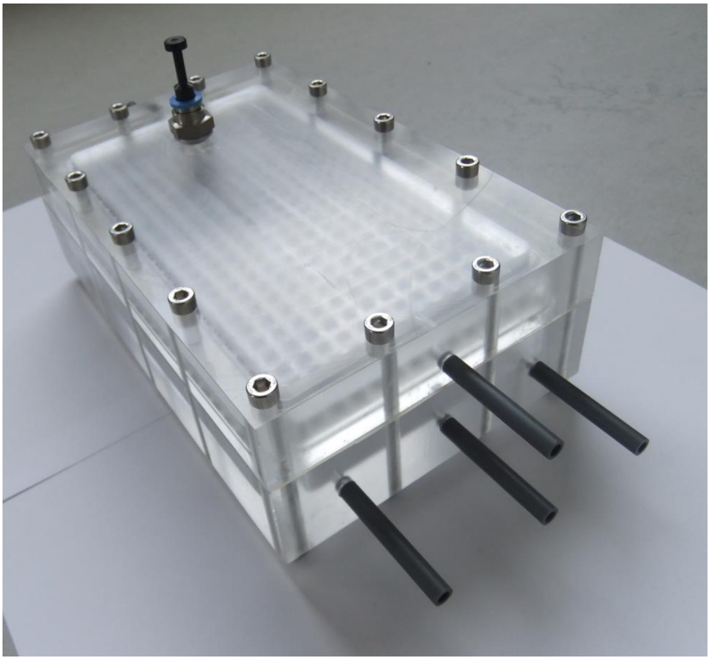


Figure 6.2. External membrane module.

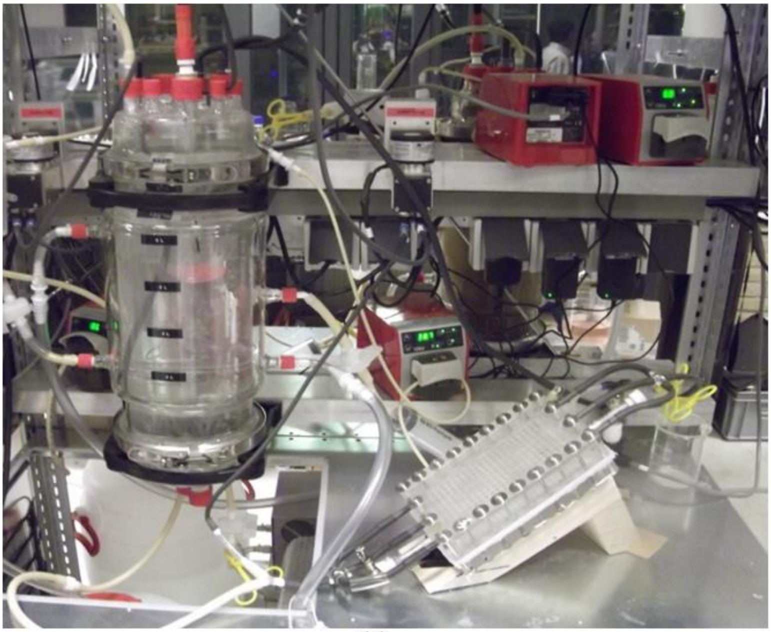
6.2.2 Experimental Procedure

RS and RE were operated at average temperatures of 35.7±0.1 °C and 35.50±0.4 °C, respectively. OLR of 2 kg COD/m^3.d was applied at a HRT of 10 days and a SRT of 40 days during the study. Average TSS concentrations inside the RS and RE were 6450±480 mg/L and 6400±470 mg/L, respectively. VSS/TSS ratio in the bioreactors was over 85% in both configurations. The AnDMBRs were operated at a flux of 2.2 L/m^2.h. F/M ratio, the ratio between the COD loading fed into the bioreactor and the MLSS concentration, was about 0.28 kg COD/kg MLSS.d in both RE and RS.

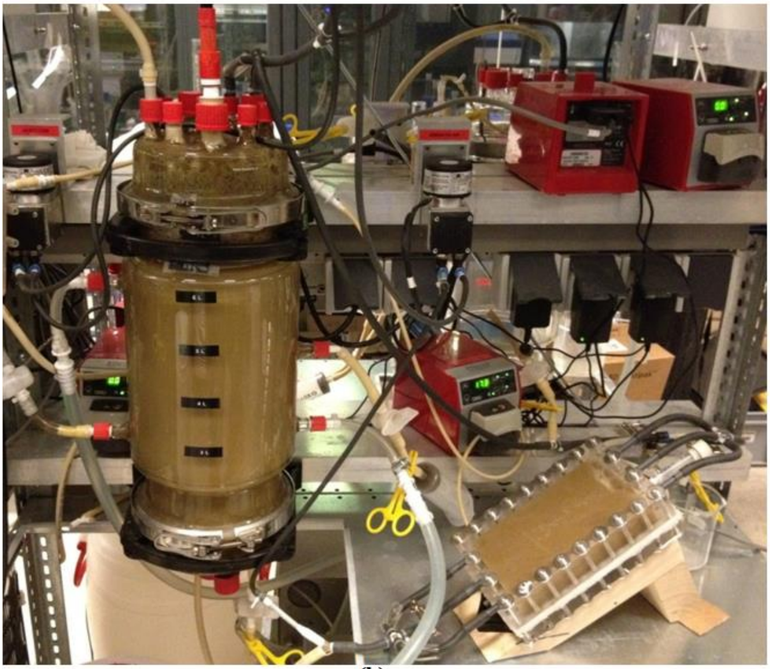
Biogas recirculation and backwashing procedures are explained in Section 4.2.2.

6.2.3 Wastewater Source and Seed Sludge

Details about the substrate are explained in Section 4.2.3. The AnDMBRs were inoculated with an anaerobic sludge from a submerged AnDMBR (Ersahin et al., 2014; Chapter 4) operated by feeding the same substrate under mesophilic conditions.



(a)



(b)

Figure 6.3. External AnDMBR set-up: (a) without sludge, (b) with sludge during the operation.

6.2.4 Methods

6.2.4.1 Analysis Techniques

Measurements of COD, TSS, VSS, TS and VS were performed following Standard Methods (APHA, 2005). Methods for the turbidity, soluble COD, VFA, PSD, methane content and SMA analyses are explained in Section 4.2.4. R_T was calculated as explained in Section 4.2.4.3.

6.2.4.2 Microbial Analysis

Bulk sludge samples were collected at the end of the operation periods in RS and RE. Details about the microbial analysis are explained in Section 5.2.4.5.

6.3 Results and Discussion

6.3.1 Treatment Performance

Permeate COD concentrations and total COD removal efficiencies of the RS and RE are given in Figure 6.4. After steady state was reached, similar and high total COD removal efficiencies (≥ 99%) were obtained in both AnDMBRs irrespective of the DM configuration. However, the permeate average total COD concentrations were 100±10 mg/L and 180±30 mg/L (after effective cake layer formation) in RS and RE, respectively, which showed that the performance of the RS was slightly higher than that of the RE. Formation of an effective cake layer is necessary in order to get a high and stable permeate quality by DM technology. As can be seen in Figure 6.4, an effective DM layer formed after 10 days in RS. However, it took 20 days in RE, two times longer than what was required in the submerged one. Therefore, permeate total COD concentrations were higher in RE than those in RS during the initial stage. Considering these results, the DM module configuration was effective with respect to the required time to achieve a high and stable removal of total COD by DM layer. Submerged AnDMBR was more appropriate to form a DM layer in terms of required time. Apart from mixing turbulence, there was no liquid flow across the support material inside the RS, meaning that sludge could easily attach on the surface of the filter cloth and the DM layer became denser. However, in the RE configuration, the mixed liquor inside the bioreactor was transferred by the gas pump to the DM module and passed through the filter cloth surface. The prevailing shear force across the filter cloth apparently limited particles to settle and retain, hampering the rapid formation of a thick DM layer on the filter cloth. As a result, a slightly less effective separation was obtained by the DM layer and more time was required to form an effective DM layer in the external AnDMBR configuration compared to the submerged one.

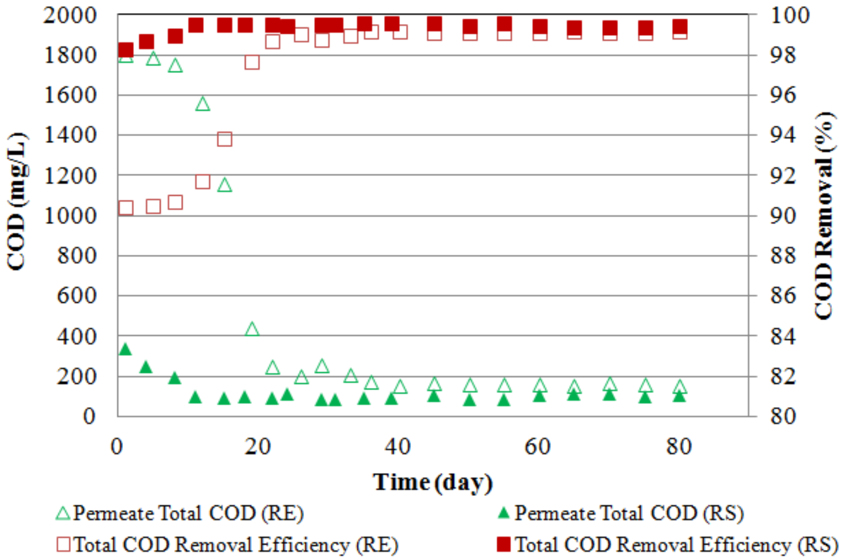


Figure 6.4. Permeate total COD concentrations and COD removal efficiencies in the submerged and external AnDMBRs.

After a stable removal efficiency was obtained, the average soluble COD concentrations in the permeate were 85±10 mg/L and 115±12 mg/L in RS and RE, respectively (Figure 6.5). Soluble COD concentrations inside the bioreactor were 240±30 mg/L and 760±50 mg/L in RS and RE, respectively. Kim et al. (2001) showed that soluble COD increased with the recirculation of the sludge due to the floc breakage in a cross-flow MBR. Although soluble COD concentration was 3.1 times higher in the bioreactor in RE, soluble COD concentration in the permeate was only 1.4 times higher in RE compared to RS. Obtained results showed that the DM layer was able to retain a large part of the soluble COD fraction, which was analyzed by using a filter of 0.45 µm. We postulate that physical retention and possibly some bio-conversion had contributed to this removal. Similarly to the results obtained from total COD trends, more time was required for an effective DM layer formation enabling to achieve a stable and low soluble COD concentration in the permeate of RE, namely 20 days, compared to RS. Considering these results, it can be concluded that the start-up period for submerged AnDMBRs would be shorter than that for external AnDMBRs. This is important, especially at large-scale applications, because permeate obtained at the start-up period and after intensive filter cloth cleaning, should be recycled to the bioreactor until the desired permeate quality is reached. Average soluble COD/total COD ratios were 0.85 and 0.64 in the permeate of RS and RE, respectively. This result indicated that more particulate COD could pass through DM layer in RE compared to RS possibly due to the less effective DM formation in RE caused by the difference in the membrane configuration.

Total VFA concentration in the bulk sludge was higher in RE compared to RS. Total VFA concentration in RS was between 30 mg/L and 55 mg/L after steady state condition was reached. However, it was 950 mg/L at the 10th day of the operation and decreased to 100 mg/L under the steady state conditions. The major VFA concentration difference between RS and RE was originated from C_2 (acetic acid) and C_3 (propionic acid) accumulation inside the RE. Over 50% VFA removal efficiency was achieved by the DM layer in both submerged and external AnDMBRs.

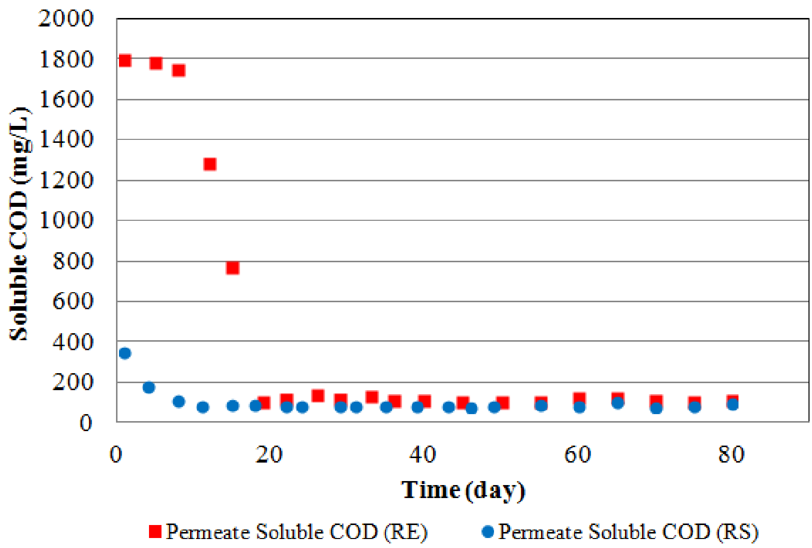


Figure 6.5. Soluble COD concentrations and removal efficiencies in the submerged and external AnDMBRs.

Although a stable and low turbidity in the permeate was reached after 10 days in RS, it required more than 20 days in RE (Figure 6.6). The average permeate turbidity values were 12.5±2.3 and 30±2 NTU in RS and RE, respectively after stable turbidity removal efficiency was obtained. The turbidity removal efficiency was ≥ 99% in both configurations; however, permeate turbidity was more than two times higher in RE compared to that in RS. Turbidity results were also consistent with the results of COD in terms of the difference in time needed for the effective DM layer formation between submerged and external AnDMBRs.

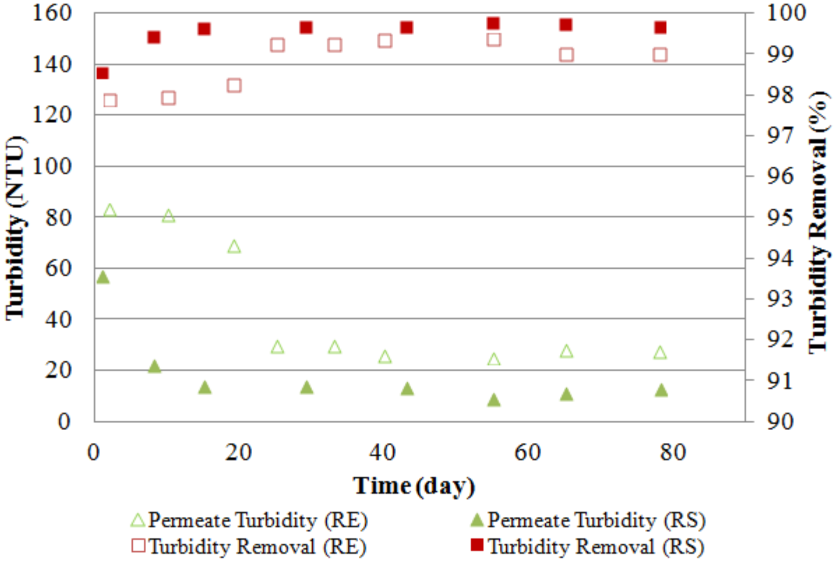


Figure 6.6. Permeate turbidity and turbidity removal efficiencies in the submerged and external AnDMBRs.

After stable biogas production was obtained, the average produced methane flows were 2.4±0.1 L/day and 1.9±0.1 L/day in RS and RE, respectively. Methane compositions of the biogas were 70% and 63% in RS and RE, respectively. Daily biogas production was lower in RE compared to RS. In addition, SMAs of the bulk sludge in RS and RE were measured to determine the impact of DM configuration on the methanogenic activity. SMAs were 0.20±0.01 g CH_4-COD/g VS.d and 0.15±0.01 g CH_4-COD/g VS.d in RS and RE, respectively. SMA results indicated 25% difference in the methanogenic activity in the external AnDMBR configuration compared to the submerged one. This result is consistent with the difference in the methane production between RS and RE. The obtained results indicate that the microbial activity in the external AnDMBR was slightly affected by the imposed process conditions. Sludge circulation required in the operation of side-stream membrane configurations has been reported as a negative factor that resulted in a decrease in microbial activity due to a possible disruption of the syntrophic relationship, that is: syntrophic hydrogen transfer between different groups of microorganism (Liao et al., 2006). Recirculation of the mixed liquor through the membrane unit by a pump resulted in a deteriorated microbial activity in aerobic and anaerobic MBRs (Brockman and Seyfried, 1996; Ghyoot and Verstraete, 1997; Kim et al., 2001). The presence of a balanced microbial ecosystem is of particular importance when feeding the reactor with complex substrates, such as used in this study. Therefore, the decrease in the methane production and also in the soluble COD removal efficiency inside the RE (Figure 6.5) might be attributed to the difference in DM configuration.

6.3.2 Filtration Performance

As depicted in Figure 6.7, higher TMP values were obtained in RS compared to RE. After stable TMP was reached, the average TMPs were 680 and 380 mbar in the submerged and external AnDMBRs, respectively. The average filtration resistances were 1.02×10^{17} m^{-1} and 7.4×10^{16} m^{-1} in the RS and RE, respectively at an operational flux of 2.2 $L/m^2.h$. About 28% lower total filtration resistance was obtained in RE compared to RS and the higher total filtration resistance obtained in the submerged AnDMBR is likely caused by the thicker DM layer. These results showed that the DM configuration affected the operational pressure and the total filtration resistance. External AnDMBR configuration allowed a decrease in the operational pressure by the sludge recirculation through the DM module. On the contrary, the DM layer thickness in RS was not controlled or decreased to the same level as in RE by bottom biogas sparging, albeit the same biogas flow was applied in RS and RE. Most likely, gas bubbles applied in RS could not sparge the DM layer as effectively as in RE because the gas diffusers were placed inside the mixed liquor and the mixed liquor had also a resistance against the gas sparging force in RS. Notwithstanding the above mentioned differences, comparable COD removal efficiencies were obtained with both configurations. Since the permeate turbidity and COD concentrations in RE were only slightly higher than those in RS, likely the development and/or density of the DM layer over a certain level had a minor effect on the COD and solids removal. In other words, DM layer formation achieved in both submerged and external AnDMBR apparently was sufficient for attaining a stable and high treatment efficiency.

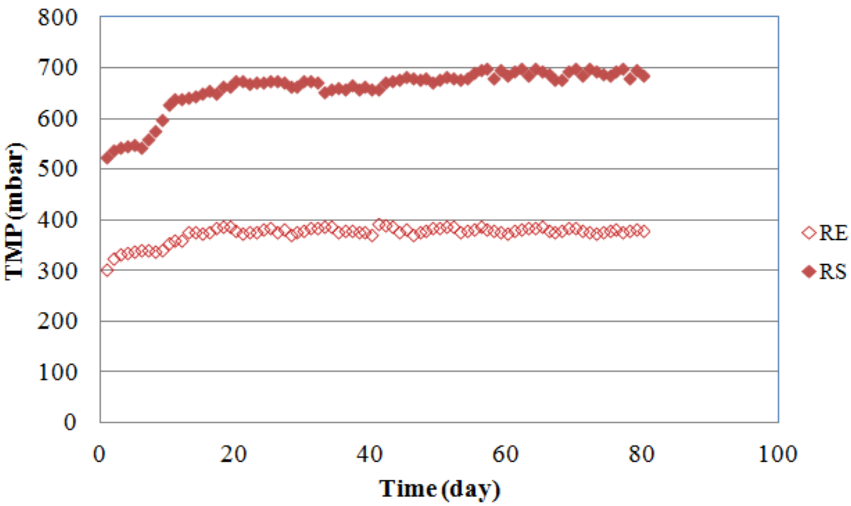


Figure 6.7. TMP profiles in the submerged and external AnDMBRs.

PSD of the bulk sludge in RS and RE are shown in Figure 6.8. The median particle sizes were 37.1 µm and 29.2 µm in RS and RE, respectively. The somewhat smaller median PSD of the bulk sludge in RE compared to that in RS might be caused by the applied higher shear forces in RE, resulting in floc erosion and/or poor flocculation (Choo and Lee, 1998; Stricot et al., 2010; Martin-Garcia et al., 2011). Shear forces applied by the sludge recirculation in the external configuration might be more detrimental for methanogens and/or acetogens and their syntrophic associations (McMahon et al., 2001; Speece et al., 2006) than for acidogenic bacteria. This is likely the explanation for the lower methane production observed in RE compared to RS. Jeison et al. (2009) reported that single cell acidogenic bacteria leads to a decrease in PSD of sludge in an AnMBR. Therefore, smaller average particle size might be attributed to a higher amount of single cell acidogenic bacteria in RE in comparison to RS.

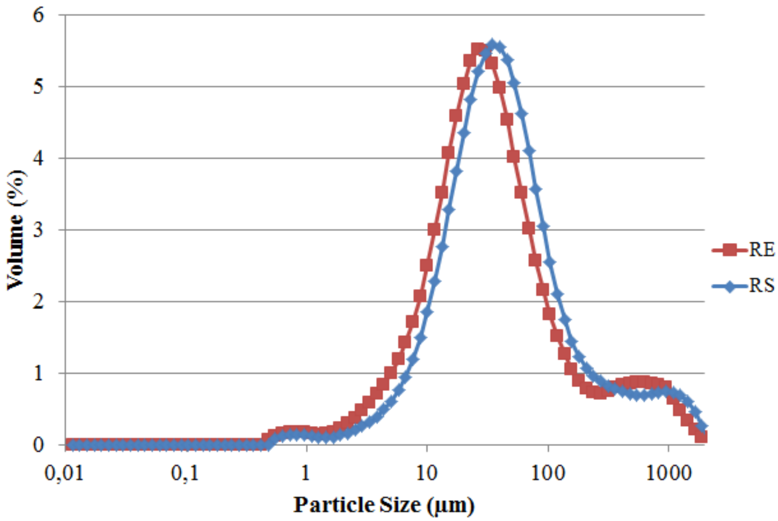


Figure 6.8. Particle size distribution of the bulk sludge in the submerged and external AnDMBRs.

6.3.3 Microbial Community Analysis

A total of 16124 reads were recruited by the 454 pyrosequencing analyses of the biomass samples from the seed sludge, RS and RE. All the bacterial and archaeal species detected in the seed sludge and bulk sludge are presented in Table B1 and Table B2. There were three dominant phyla in the seed sludge, namely *Bacteroidetes* (37%), *Firmicutes* (34%) and *Proteobacteria* (11%) (Figure 6.9). The abundances of *Bacteroidetes* increased to 53% in the submerged bioreactor and to 74% in the external bioreactor at the end of each operation period. The abundance of *Firmicutes* decreased by half in the RS and further decreased to 15% in the external one. The phylum *Proteobacteria* was largely eliminated from the RS and RE with a reduction of 76% and 95% in the relative abundance, respectively. The phylum *Bacteroidetes* have been proven to be dominant in many anaerobic reactors (Tang et al., 2007; Gao et al., 2010; Qiu et al., 2013; Ziganshin et al., 2013) with a function to degrade complex organic matters, such as starch and other polysaccharides (Bauer et al., 2006; Xie et al., 2007; Hanreich et al., 2013). Firmicutes were also reported the dominant group of bacteria in AnMBRs (Calderon et al., 2011). Similar to the findings of this study, an increasing abundance of *Bacteroidetes* and a decreasing abundance of *Firmicutes* were observed in an anaerobic batch reactor degrading straw and hay, which is consistent with the observation that the *Bacteroidetes* phylum is able to express a higher number of sugar converters than the *Firmicutes* phylum. Hence, *Bacteroidetes* phylum has higher potential to metabolize various glycans efficiently (Hanreich et al., 2013). In our study, complex carbohydrates were fed to the AnDMBRs, including starch, milk powder, yeast extract and ovoalbumin, which contributed to over 50% (in COD weight) in the total organic sources. This can be the reason that *Bacteroidetes* became dominant rather than other phyla in the AnDMBRs. Among the phylum *Bacteroidetes*, the genera of *Cytophaga*, *Bacteroides* and *Anaerophaga* were the top three dominant genera in the seed sludge, but the genus of *Bacteroides* became the pre-dominant genus in both RS and RE (Figure 6.10). The members of *Bacteroides* are capable of degrading protein and/or carbohydrates and were previously found in great abundance in anaerobic systems (Li et al., 2013; Panichnumsin et al., 2012; Yu et al., 2012). It is noteworthy that the members of *Clostridium*, *Aminobacterium* and *OP9* were also sub-dominant in the RS but their abundance decreased by 44%, 43% and 89%, respectively, in the RE.

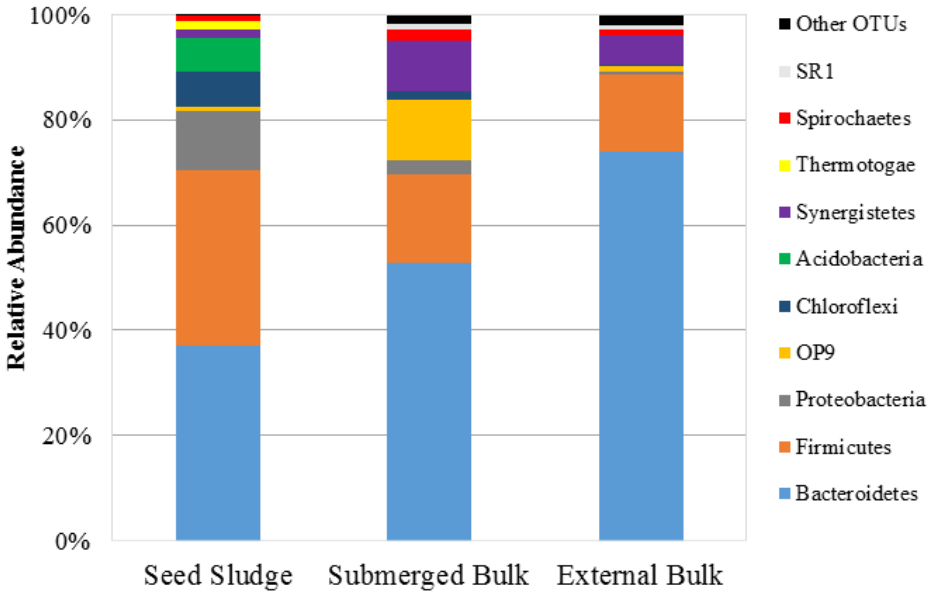


Figure 6.9. Classification at the phylum level of the bacterial communities in the seed sludge and bulk sludge of the submerged and external AnDMBRs.

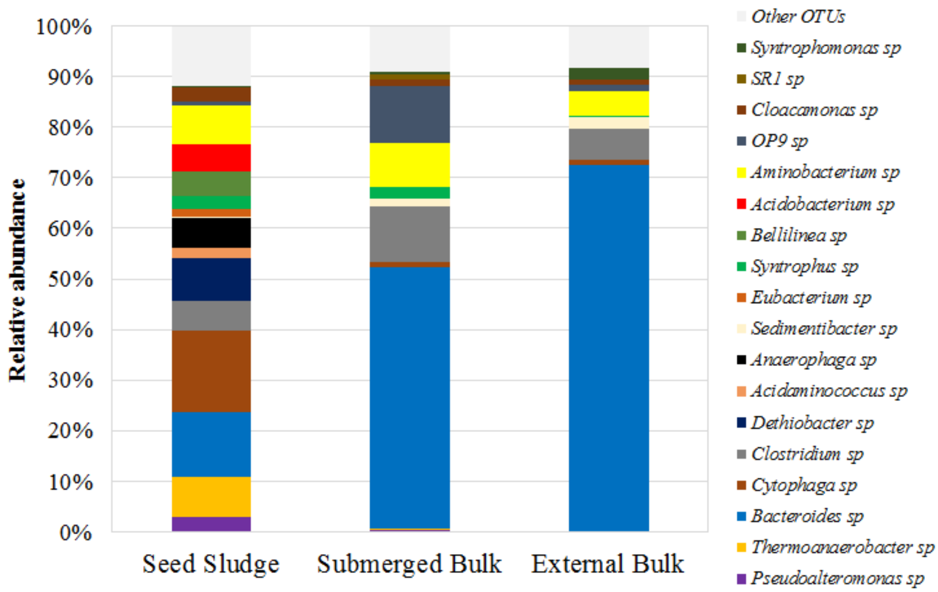


Figure 6.10. Major bacterial species and their relative abundances in the seed sludge and bulk sludge of the submerged and external AnDMBRs.

There was a substantial shift in the archaeal populations from the seed sludge to the submerged bioreactor community and eventually to the external one. A total of nine archaeal species were detected in the seed sludge with seven methanogenic archaea contributing to 92% of the total archaeal reads (Figure 6.11). The predominant methanogen group was genus *Methanobrevibacter* with abundance over 42%, followed by *Methanosaeta sp*, *Methanobacterium petrolearium* and *Methanolinea mesophila*, each of which accounted for about 14-15% of the total archaeal species. The methanogenic community turned to be more uniform in the submerged bioreactor compared to the seed sludge with the same dominant species. However, the number of archaeal types decreased to three species in the RE, with *Methanosaeta sp*, *Methanobacterium petrolearium* and *Methanosarcina sp*. Meanwhile, the SMA decreased by 25% in the external bioreactor in comparison to the submerged one. The combined information of archaeal community and SMA indicated a decrease in the methanogenic activity in the RE. The alpha-diversity calculated based on both bacterial and archaeal species also showed a clear decreasing trend from the seed sludge to the bulk sludge in the RS and RE (Figure 6.12). This implies a more stressful condition in the RE than the RS, probably brought about by the imposed high shear forces and the external sludge flow between the bioreactor and the membrane module in RE.

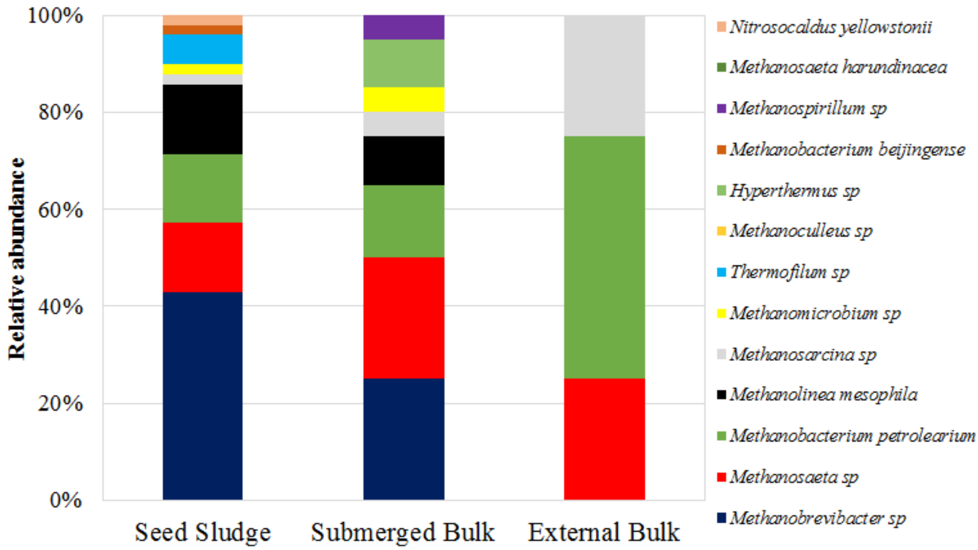


Figure 6.11. Archaeal species and their relative abundances in the seed sludge and bulk sludge of the submerged and external AnDMBRs.

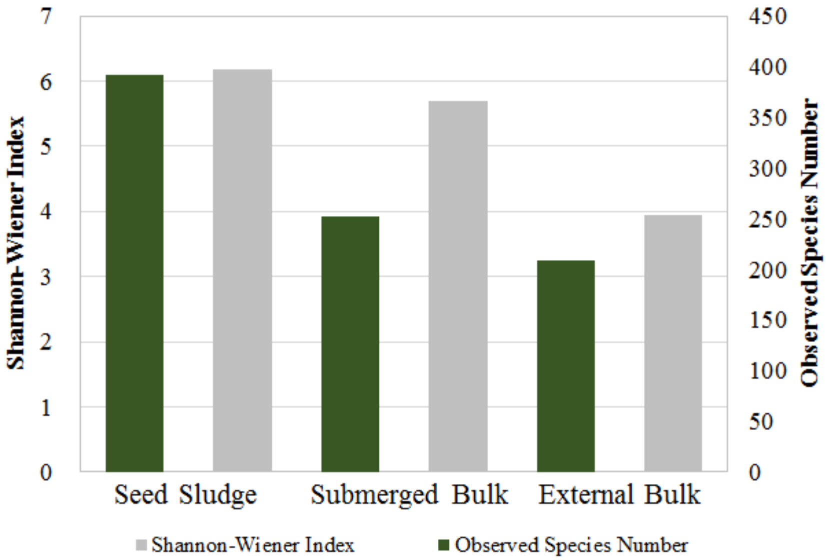


Figure 6.12. Shannon-Wiener index and observed species number of the seed sludge and bulk sludge from the submerged and external AnDMBRs.

It has been reported that the biomass encounters a more stressful environment in an external MBR compared to a submerged one, where the applied side-stream pumping and resulting shear may have a negative effect on the methanogenic activity (Stuckey, 2012). It also has been reported that the sole recirculation of biomass may cause as high as 90% loss of biomass (acidogenic and methanogenic microorganisms) activity (Brockmann and Seyfried, 1996). A further study proved that a high shear stress causes obvious changes in the sludge properties in a side-stream MBR, whereas almost no effect is observed at a low recirculation rate (Stricot et al., 2010). Based on above results, it is reasonable to attribute the observed lower methanogenic activity and community structure characteristics in the external AnDMBR compared to the submerged one, to the more stressful environment, resulting from external sludge recirculation.

6.4 Conclusions

Treatment and filtration performances of the submerged and external AnDMBRs treating high strength wastewater have been evaluated and compared to each other. Although slightly better permeate quality in terms of COD concentration was obtained by RS, over 99% COD removal efficiency was achieved in both configurations. Longer time was needed in RE compared to RS in order to form an effective DM layer enabling to achieve a stable removal efficiency and low soluble COD concentrations in the permeate. Therefore, submerged AnDMBR configuration appears more suitable when short start-up period is necessary or when periodic thorough filter cloth cleaning is considered. Higher methane production rates obtained in RS compared to RE reflected the negative impact of the imposed higher shear force and sludge recirculation in the external AnDMBR. The results obtained from pyrosequencing analyses revealed that diversity and richness of the microbial communities including bacteria and archaea in the seed sludge was high and microbial community structure in the bulk sludge of submerged AnDMBR was different compared to that of external AnDMBR. Archaeal community was apparently negatively affected by the sludge recirculation through the external DM module in the RE, most probably due to the environmental stress. At the same gas sparging rate, 28% lower total filtration resistance was obtained in the RE compared to RS, which was mainly caused by less compact DM layer in the RE in comparison to that in the RS. The results indicated that sludge recirculation in the external configuration was more effective in decreasing DM thickness/compactness, thus TMP, than the bottom biogas sparging in the submerged configuration. DM formation could offset the possible removal efficiency decrease due to the deterioration of the biomass activity caused by sludge recirculation in the RE.

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CHAPTER 7

GAS-LIFT ANAEROBIC DYNAMIC MEMBRANE BIOREACTOR FOR HIGH STRENGTH WASTEWATER TREATMENT: EFFECT OF BIOGAS SPARGING VELOCITY AND HRT ON THE TREATMENT AND FILTRATION PERFORMANCES

Abstract

A laboratory scale external AnDMBR treating high strength wastewater (influent COD ≈ 20 g/L) was operated to assess the effect of gas sparging velocity (GSV) and hydraulic retention time on removal efficiency and DM filtration characteristics. An increase in GSV resulted in a decrease in DM filtration resistance. DM or cake layer was identified as the main contributor to the total filtration resistance. The external AnDMBR achieved over 99% COD removal efficiency irrespective of the GSV. The results showed that the DM formation process proceeded until a stable cake layer was reached Reducing of HRT resulted in an increase in protein/carbohydrate ratio in SMP and an increase in biomass concentration in the bioreactor. Therefore, HRT affected TMP and total filtration resistance in the AnDMBR. A high permeate quality was obtained by an effective DM layer at OLRs between 2-3.6 kg COD/m^3.d. Based on the fluxes observed in this research, the filter cloth costs would be in the range of 0.17 €/m^3 of treated wastewater. The investment and operational costs of the AnDMBRs are expected to be substantially lower than that of conventional membrane filtration.

This chapter is based on:

Ersahin, M.E., Gimenez, J.B., Ozgun, H., Tao, Y., Spanjers, H., van Lier, J.B. Gas-lift anaerobic dynamic membrane bioreactors for high strength wastewater treatment: Effect of biogas sparging velocity and HRT on treatment performance. Under review.

7 GAS-LIFT ANAEROBIC DYNAMIC MEMBRANE BIOREACTOR FOR HIGH STRENGTH WASTEWATER TREATMENT: EFFECT OF BIOGAS SPARGING VELOCITY AND HRT ON THE TREATMENT AND FILTRATION PERFORMANCES

7.1 Introduction

The combination of an anaerobic treatment process and membrane technology, better known as AnMBR, is of growing interest based on expected high treatment efficiency, small footprint, and net energy production. AnMBRs have been successfully studied, generally at laboratory scale, for the treatment of both high and low strength wastewaters from industries and municipalities. Particularly the effective biomass retention and high quality effluents are conceived advantageous by the various authors (Liao et al., 2006; Jeison and van Lier, 2007; Huang et al., 2011; Robles et al., 2012; Dereli et al., 2012; Ozgun et al., 2013). SRT and HRT can be independently controlled in AnMBRs. Therefore, low strength wastewaters can be efficiently treated by AnMBRs in a feasible way, provided appropriate membrane fluxes can be achieved (Lew et al., 2009; Baek et al., 2010; Lin et al., 2013). HRT and SRT are two major factors that affect treatment performance and biomass characteristics, and thus affect membrane fouling in AnMBRs (Liao et al., 2006). AnMBRs are of particular interest when nutrient removal is not critical. There are several examples of AnMBR applications for the treatment of municipal wastewater, which achieved over 80% COD removal and 99% TSS removal (An et al., 2009; Herrera-Robledo et al., 2010; Lin et al., 2011; Gimenez et al., 2011). However, AnMBR technology was found to be more appropriate for high strength particulate wastewater treatment because of the relatively low membrane flux required leading to a reduction in capital costs. Moreover, the prevailing long SRTs may result in the degradation of slowly degradable particulate compounds in comparison to conventional anaerobic processes (Liao et al., 2006). For example, 98% COD removal efficiency was achieved in a completely stirred tank reactor (CSTR) coupled to a membrane separation device treating high strength petrochemical wastewaters (Van Zyl et al., 2008). Evaporator condensate wastewater originated from pulp and paper industry was treated by a submerged AnMBR with a COD removal efficiency over 93% at an OLR up to 24 kg COD/m^3.d (Xie et al., 2010).

DM technology in AnMBRs is becoming an interesting concept in which a low-cost filter material, e.g. woven or non-woven cloth, can be used instead of a conventional membrane (Ersahin et al., 2012). Suspended solid particles, e.g. microbial cells and flocs, in the bulk solution can accumulate and form a dynamic cake layer on an underlying support material. This phenomenon is similar to cake formation on a microfiltration or ultrafiltration membrane. Since the retention of solid particles is provided by the DM layer, the support filter material is protected against fouling, whereas the filter itself is not a critical factor in the filtration. Support material properties, including material type and filament structure are critical for the formation of a cake (DM) layer over the filter surface in DM filtration technology. Ersahin et al. (2013) concluded that mono-monofilament filter cloth is much more appropriate for DM technology in comparison to staple filter cloths. Results showed that the structure of staple filter cloth provides retention not only on the filter cloth surface but also inside the filter cloth pores, which makes it more appropriate for depth filtration. However, the structure of mono-monofilament filter cloth is appropriate for surface filtration and thus for DM filtration.

So far, applicability of the DM technology, including aerobic and anaerobic dynamic MBRs, has been investigated mainly for treatment of low/medium strength wastewaters, such as municipal wastewaters (Fan and Huang, 2002; Zhang et al., 2010; Ersahin et al., 2012). Although in principle higher permeate fluxes can be obtained due to the larger pore size of the filters in DM technology, the actual COD removal efficiency by solely the filter cloth would be lower compared to traditional membrane filtration. The build-up of an effective cake layer is crucial for enabling a stable and high quality permeate. In order to provide a feasible and stable operation, the DM layer thickness and DM porosity should be controlled. Biogas sparging over the support material surface can provide the required shear force to detach excess DM layer. In addition, periodic backwash may stabilize the DM porosity. Considering the need for proper DM consolidation, DM filtration technology would be much more appropriate for the treatment of high strength particulate wastewaters than the treatment of diluted wastewaters. With low-strength wastewater, a significant compromise has to be made on COD removal efficiency when large fluxes need to be achieved. Lin et al. (2013) reported that a high flux, i.e. 65 $L/m^2.h$, with an AnDMBR treating municipal wastewaters was obtained using a mesh with a pore size of 61 μm. However, a COD removal efficiency of only 57% was achieved due to the ineffective DM layer formation. Research done with AnDMBRs for treatment of high strength wastewaters is quite limited in the literature.

In the present chapter, long-term operation of an external AnDMBR for the treatment of high strength wastewater under mesophilic conditions was evaluated. The effect of GSV and HRT on the removal efficiency and filtration characteristics were investigated. Moreover, cost estimation in terms of support material acquisition was also presented.

7.2 Material and Methods

7.2.1 Experimental Set-up

Laboratory scale gas-lift external AnDMBR set-up was used in this study (Figure 6.1b). Design information of the external AnDMBR is given in Section 6.2.1. Filter material and equipments used in both set-ups are explained in Section 4.2.1.

7.2.2 Experimental Procedure

The AnDMBR was operated for 200 days at an average temperature of 35.5±0.2 °C and an SRT of 40 days. The average pH in the reactor was 7.9±0.1 during the study. During the first part of the study (first 140 days), an OLR of 2 kg $COD/m^3.d$ was applied at an HRT of 10 days, and GSVs of 17, 35 and 52 m/h were tested. GSV is defined as the recirculated biogas volume per cross-sectional area of the membrane module per hour. During the second part of the study, which lasted 60 days, a constant GSV of 35 m/h was applied and the HRT was decreased to 7 and 5.5 days with an increase in OLR to 3 and 3.6 kg $COD/m^3.d$, respectively. Before collecting data for each HRT period, the AnDMBR was operated for duration of 3 times the HRT to obtain steady state conditions.

Backwashing procedure is explained in Section 4.2.2.

7.2.3 Wastewater Source and Seed Sludge

Details about the substrate are explained in Section 4.2.3. The AnDMBRs were inoculated with an anaerobic sludge from an AnDMBR (Ersahin et al., 2014) operated by feeding the same substrate under mesophilic conditions.

7.2.4 Methods

7.2.4.1 Analysis Techniques

Measurements of COD, TN, TP, TSS and VSS were performed following Standard Methods (APHA, 2005). Methods for the turbidity, soluble COD, VFA, SMP, methane content and SMA analyses are explained in Section 4.2.4.

Filtration resistances were calculated based on the permeation data. Total filtration resistance (R_T) was calculated as explained in Section 4.2.4.3. Cake resistance was calculated using equation (7.1) following the method described by Fan and Huang (2002) and Zhang et al. (2010):

$$R_T = R_m + R_c + R_p \qquad (7.1)$$

where R_m is the intrinsic resistance of the filter cloth (m^{-1}), R_c is the cake layer (DM) resistance (m^{-1}), and R_p is the pore-clogging resistance (m^{-1}). After physical cleaning by tap water at the end of each operation period (each GSV and each HRT study), filtration resistance was measured. R_m, which was calculated from tap water filtration with a virgin filter cloth, was subtracted from the total filtration resistance (obtained after physical cleaning) to obtain R_p. R_c was calculated by subtracting the sum of R_m and R_p from R_T that was measured during the operation.

7.3 Results and Discussion

7.3.1 Effect of GSV

DM formation immediately started after starting the permeate pump in agreement with other studies (Park et al., 2004; Hu and Stuckey, 2006) and became effective in terms of COD removal in several days. DM formation duration depends on the substrate type, sludge concentration in the bioreactor and membrane module configuration. Effective DM layer formation was reached in 15-20 days at each GSV in this study. During the experimental study, average influent COD concentration was 20100 mg/L. Figure 7.1 shows the performance of the AnDMBR with respect to the removal of total COD at different GSVs. After the effective DM formation, a stable COD removal efficiency over 99% was achieved irrespective of GSV within the tested range, reaching a permeate COD concentration below 200 mg/L. There was no significant difference between COD removal efficiencies obtained from the operation periods conducted at different GSVs. The average elimination of TN and TP by the AnDMBR were 19% and 16%, respectively. TSS concentration of the permeate was less than 10 mg/L corresponding to an average TSS removal efficiency of 99% during the entire study. The average concentration for the mixed liquor TSS concentration in the bioreactor was 6410±455 mg/L. The percentage of VSS was 85% of the TSS.

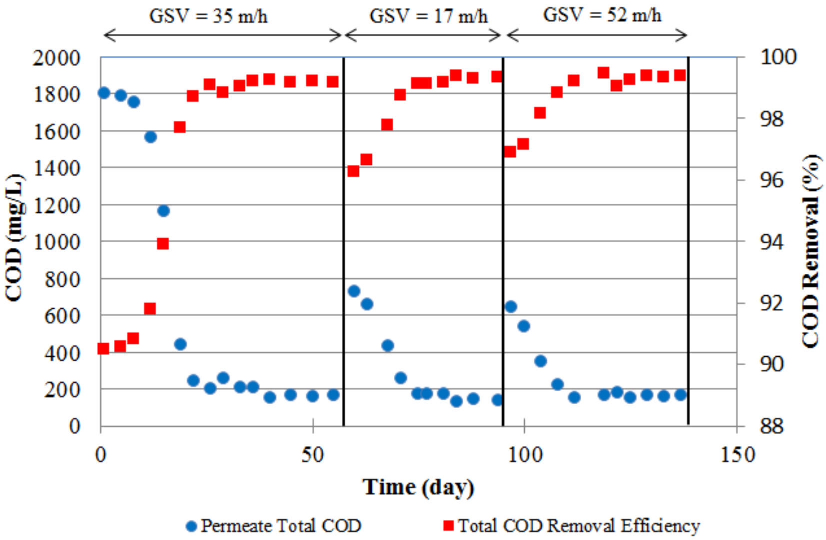

Figure 7.1. Permeate total COD concentration and total COD removal at different GSVs (HRT: 10 days).

High soluble organic matter removal was obtained by the AnDMBR at all GSVs tested in this study (Figure 7.2). After an effective DM layer was established, average soluble COD concentration in the permeate was 115±15 mg/L at all GSVs. This corresponded to a soluble COD removal efficiency of 99% by the AnDMBR. Figure 7.2 shows a similar trend as the total COD removal. Besides, soluble COD removal across the DM was achieved and consistent differences have been determined between the bulk sludge and permeate soluble COD concentrations. The average soluble COD removal by the DM layer was over 70%. This level of soluble COD removal was higher than some of the reported soluble COD removal efficiencies by membranes in AnMBRs in the literature (Lin et al., 2010; Smith et al., 2013). Reasons for the removal of soluble organics by DM might be microbial activity in the cake layer and/or physical retention capacity of the cake layer itself. Turbidity removal trend was also similar to the permeate COD results. The average permeate turbidity was 24.5±9.3 NTU resulting in a turbidity removal efficiency over 99% independently of GSV.

The biogas production was between 2.65-2.85 L/day and methane content of the biogas was in the range of 60-65%. The average methane yield ranged between 0.28 and 0.31 L CH_4/g $COD_{removed}$ irrespective of GSV. A similar observation of low methane yields in AnMBRs was also reported by other researchers, and was attributed to the solubility of methane in the permeate (Huang et al., 2011; Smith et al., 2013). Smith et al. (2013) indicated that methane oversaturation due to the pressure differential across the membrane may be responsible for yielding low methane in AnMBRs. In addition, methane production by the methanogens existed in the DM layer near the filter surface may result in methane oversaturation in the permeate. Methanogenic activity through the cake layer is expected in AnDMBRs, therefore, most likely, the combination of the pressure differential and methane production near the filter surface was responsible for low methane yields in this study. Any noticeable effect of GSV was observed neither on the biogas production in the AnDMBR nor in the specific methanogenic activity. SMA was 0.15±0.01 g CH_4-COD/g VS.d at a GSV of 35 m/h and changed ±5% throughout the GSV study.

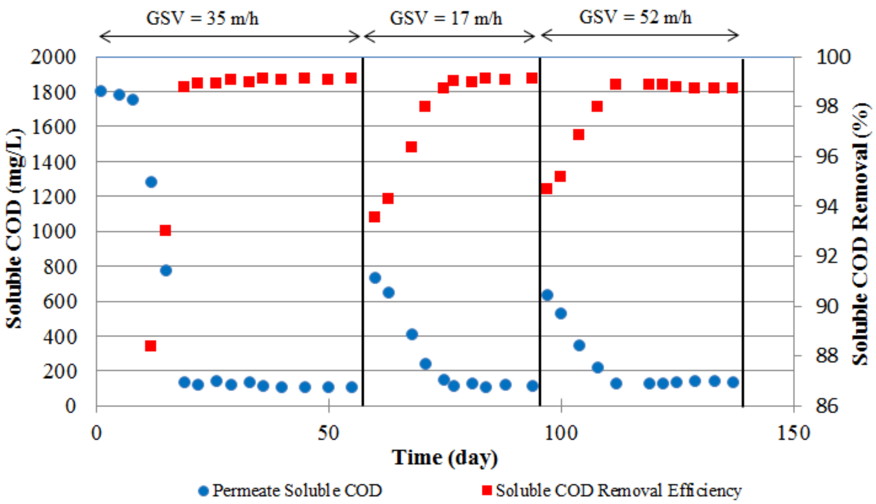

Figure 7.2. Permeate soluble COD concentration and soluble COD removal of the AnDMBR at different GSVs.

The applied flux was approximately 2.2 L/m^2.h during the experiments. As shown in Figure 7.3, TMP increased with operation time during the first 15-20 days until a stable value was achieved. Stabilized TMP values were on average 415, 380 and 360 mbar at GSV of 17, 35 and 52 m/h, respectively. These results indicate that low GSV yielded a more compact and/or thicker DM layer in comparison to higher GSV. Most likely, at higher GSVs, more abrasion occurs on the support material. However, although the GSV was increased by a factor 1.5, the effect of GSV on TMP was not in the same order of magnitude, suggesting the existence of an energetic optimum.

The average total filtration resistances (R_T) were $7.96x10^{16}$ m^{-1}, $7.40x10^{16}$ m^{-1} and $7.11x10^{16}$ m^{-1} at GSVs of 17 m/h, 35 m/h and 52 m/h, respectively. Cake (DM) layer resistance is expected to be the main contributor of the filtration resistance since the retention is caused by the DM layer in AnDMBRs. The resistance analysis showed that cake layer resistance contributed over 99% to the total filtration resistance independently of GSV. Nonetheless, the cake resistance was 35% higher at GSV of 17 m/h compared to the GSV of 52 m/h. Considering the filtration resistances, it can be concluded that biogas sparging can be used to reduce the excess cake layer thickness and/or cake layer compactness in AnDMBRs. Although the biogas sparging could remove excess cake layer formed on the support material surface and prevent TMP increase, an effective DM layer could be attained. Consequently, high COD removal efficiencies were obtained throughout the entire study. Therefore, the optimum GSV providing a high COD removal should be selected considering the energy consumption to obtain a feasible treatment. Increasing GSV is resulted in an increase in the energy consumption of the gas recirculation pump. In addition to GSV related energy requirement, also the TMP should be evaluated together with the permeate quality to identify the optimum GSV for the AnDMBR operation. Optimum GSV can be determined by observing the relationship between TMP and permeate quality at different GSVs. Permeate quality was high, quite stable and similar at all GSVs (Figure 7.1 and Figure 7.2). Energy consumption for biogas sparging would be lower at lower GSVs. In this regard, our results showed that GSVs of 17 m/h and 35 m/h gave better results compared to the GSV of 52 m/h, when permeate quality, TMP evolution and energy consumption were considered together.

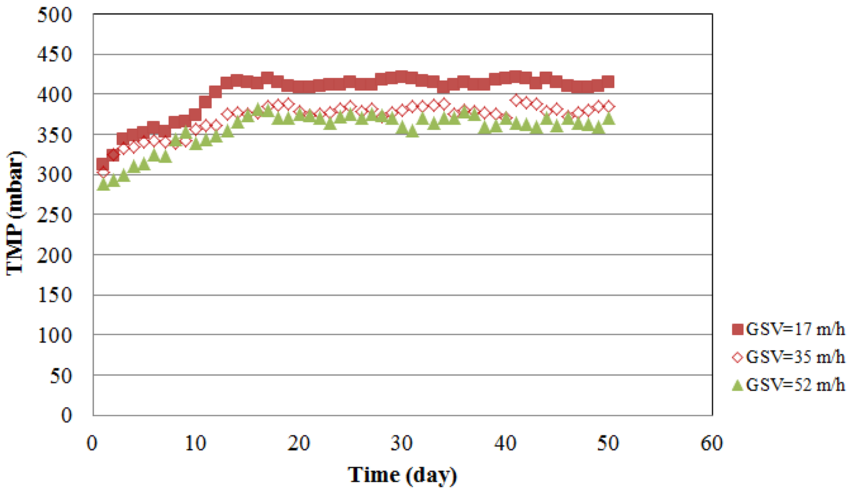

Figure 7.3. TMP at different GSVs.

7.3.2 Effect of HRT

HRTs of 7 and 5.5 days were applied in the AnDMBR at a GSV of 35 m/h. After the effective DM layer formed, very high total COD removal efficiencies and high quality permeate with non-detectable solids were achieved. COD removal efficiency of the system was over 99% at both HRTs (Figure 7.4), which was similar to the removal efficiency obtained at HRT of 10 days (Figure 7.1). TSS and turbidity removal efficiencies of 99% were achieved at all HRTs. An increase of 15% was observed in the average soluble COD concentration inside the bioreactor when HRT decreased. However, the DM could offset the rise in COD concentration and the permeate COD remained stable below 200 mg/L at both HRTs. Similar to COD concentration, average VFA concentration increased from 28 mg/L to 52 mg/L in the bulk sludge with the decrease in HRT from 10 days to 5.5 days. Total VFA in the bulk sludge was mainly composed of acetate which constituted 70-80% of the total VFA. Propionate contributed about 10% in concentration. The rest of the total VFA was comprised of isobutyrate, butyrate, isovalerate, valerate, and formate. The permeate total VFA concentration was stable and maintained between 10-15 mg/L at all HRTs. Apparently, the DM layer was responsible for a VFA removal of 55-65% based on the difference in VFA concentration between the bioreactor bulk and the permeate. Higher TSS and VSS concentrations and more methane production were observed with the reduction of HRT from 10 days to 5.5 days. TSS concentration in the bioreactor was increased to 8100 ± 240 mg/L with a VSS/TSS ratio of 77%. Average biogas production increased from 2.75 to 4.6 L/day when the OLR increased from 2 to 3.6 kg COD/m^3.d and HRT decreased from 10 to 5.5 days. Increasing of OLR by reducing HRT resulted in more biomass multiplication and higher conversion of organic matters to methane gas in an AnMBR (Huang et al., 2011).

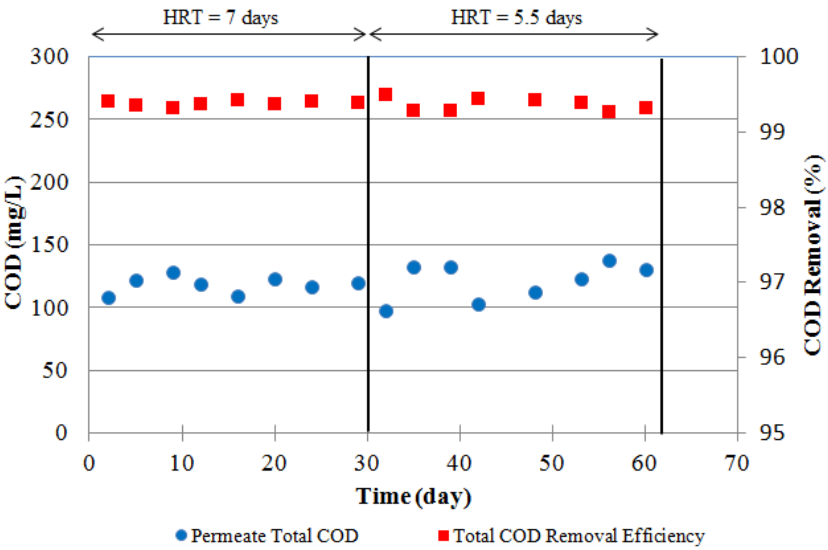

Figure 7.4. Permeate total COD concentration and total COD removal at different HRTs.

Daily averaged TMP obtained at HRTs of 10 days, 7 days and 5.5 days is depicted in Figure 7.5. An increase was observed in total filtration resistances at HRTs of 7 and 5.5 days compared to that at HRT of 10 days. This means that when HRT was decreased, AnDMBR could be operated in shorter duration without severe fouling and/or cleaning of cake layer on the filter surface in comparison to longer HRTs. The average filtration resistance increased from 7.33×10^{16} m^{-1} to 8.66×10^{16} m^{-1} which corresponded to an 18% increase compared to HRT of 10 days. However, cake resistance contributed over 99% to the total resistance independently of HRT similar to our observations in the above mentioned GSV study. SMP is one of the main contributors to fouling in aerobic and anaerobic MBRs (Satyawali and Balakrishnan, 2008; Meng et al., 2009; Zhang et al., 2011). Indeed, formation and consolidation of a DM layer in AnDMBRs are significantly affected by SMP (Ersahin et al., 2014). When HRT is reduced by increasing the OLR, the concentrations of SMP and undegraded substrates can increase in the bulk sludge (Huang et al., 2011). P/C ratio of SMP is an important parameter to assess fouling potential in AnMBRs. P/C ratio affects hydrophobicity and surface charge of the sludge in MBRs (Lee et al., 2003; Thuy and Visvanathan, 2006). P/C ratio in SMP increased from 2.8 to 5.4 with decreasing HRT from 10 days to 5.5 days in this study. It means that more proteins were introduced to the support filter surface at reduced HRTs. Protein is the major compound in SMP and increase of the protein amount in the bioreactor results in an increase in total filtration resistance due to the enhancement of the DM layer and reduction in the DM layer porosity. Another reason causing an increase in total filtration resistance might be the increase of TSS concentration in the bulk sludge at shorter HRTs due to the contribution of TSS to consolidation of the DM layer.

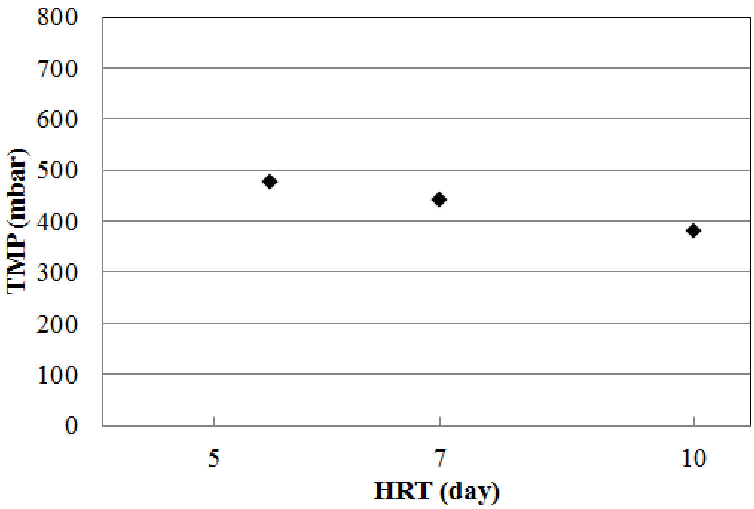

Figure 7.5. TMP profile at different HRTs.

7.3.3 Economic Feasibility

Membrane cost and biogas scouring energy needed to control the fouling were identified as the most important costs for AnMBRs (Jeison and van Lier, 2008). Costs are very sensitive to either applicable flux or membrane prices, and membrane cost represents a much more important economic factor than energy cost (Jeison and van Lier, 2007). Different kinds of simple low-cost materials can be used as the support material to form a DM layer in AnDMBRs. Thus, the most important benefit of DM technology in terms of total treatment cost is the replacement of the membrane for a low cost support material that carries the DM. Based on the flux applied in this study, support material cost would be close to 0.17 € per m^3 of permeate, assuming a filter cloth lifetime of 4 years and a mono-monofilament filter cloth price of 13 €/m^2. The support material cost was calculated using the equation given by Jeison and van Lier (2007). Chemical cost for membrane cleaning is another factor that makes AnDMBRs more advantageous in comparison to AnMBRs since chemical cleaning is not necessary for DM filtration.

Figure 7.6 shows the costs of the filter material for DM technology in function of the applicable flux. The support material cost can be decreased by using cheaper support material alternatives that are appropriate for DM formation. Alternatively, the permeate flux should increase, which restricts the potential application of AnDMBRs. Obviously, at low fluxes, the support material costs are higher in comparison to the cost at high fluxes. For example, filter cloth cost is 0.37 € per m^3 of permeate at a flux of 1 L/m^2.h; however the cost decreases to 0.04 € per m^3 of permeate at a flux of 10 L/m^2.h.

Other costs, such as those for maintenance, construction, etc. have not been considered because they are likely to be similar for AnMBRs and AnDMBRs. Since the increase in GSV had no remarkable effect on the pollutant removal efficiency of the AnDMBR in this study, low GSVs can be applied depending on TMP in order to decrease the energy cost required for biogas sparging.

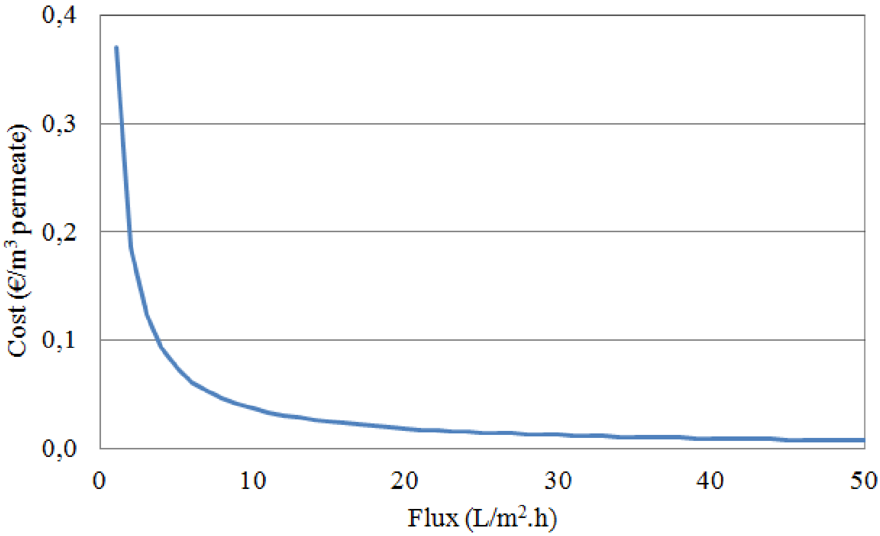

Figure 7.6. Mono-monofilament filter cloth cost for AnDMBRs (the cost only include membrane acquisition/replacement).

7.4 Conclusions

The external AnDMBR process for high strength wastewater treatment achieved over 99% COD removal irrespective of the GSV used, even though the total filtration resistance increased with GSV decrease. Total filtration resistance was mainly caused by the DM layer that provided effective and stable COD removal. Cake layer formation can be controlled effectively by applying a sufficient surface shear by increasing GSV. A decrease in TMP was observed with the increase in GSV within the tested range. Therefore, energy consumption for biogas sparging, TMP, and permeate quality must be evaluated concurrently to determine the optimum GSV by maximizing treatment performance and minimizing energy consumption for gas sparging. Biogas amount increased at shorter HRTs due to increase in OLR and biomass concentration in the bioreactor. Soluble COD concentration and VFA concentration increased in the bulk sludge at shorter HRTs. Higher total filtration resistance was obtained at HRT of 5.5 days due to the increases in P/C ratio in SMP and biomass concentration in comparison to HRT of 10 days. The AnDMBR achieved high COD removal efficiency at an HRT of 5.5 days and an OLR of 3.6 kg COD/m^3.d. Considering the lab-scale data obtained in this study, support material cost was calculated at about 0.17 €/m^3 of treated wastewater. Therefore, research should focus on development of new filter materials and/or membrane modules for AnDMBR applications enabling high operational flux and high permeate quality by keeping TMP at reasonable levels. Low capital costs of support material, and energy generation can make AnDMBRs feasible for those situations in which a high flux is not necessary such as in the treatment of sludge and slurry, black water or highly concentrated industrial wastewaters.

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CHAPTER 8

OVERALL CONCLUSIONS, PROBLEMS ENCOUNTERED AND FUTURE PERSPECTIVES

8 OVERALL CONCLUSIONS, PROBLEMS ENCOUNTERED AND FUTURE PERSPECTIVES

8.1 Conclusions

This thesis investigated the applicability of DM technology for the treatment of concentrated wastewaters in AnMBRs. A mono-monofilament filter cloth instead of a conventional membrane was used as a support layer to form the DM layer. The biological removal capacity and the filtration performance at low fluxes were investigated using two AnDMBR configurations: submerged and external. Besides, morphological and microbial characteristics of the DM layer were investigated.

An optimum support material was identified to form an effective DM layer enabling a stable and high permeate quality. The effects of different reactor operational conditions including SRT, HRT, F/M ratio, and GSV on the biological removal efficiency and filtration characteristics of DM were determined. The characteristics of the DM layer were investigated, the bulk sludge and cake layer characteristics were compared in order to understand the role and formation mechanism of DM layer. The impacts of membrane configuration on the treatment and filtration performances were evaluated by testing submerged and external AnDMBR configurations.

Following the achievements summarized above, the main conclusions that can be drawn from this thesis are given below:

- DM technology is applicable in AnMBRs.
- The DM filtration concept can turn one of the most important disadvantages of MBRs, membrane fouling, into an advantage.
- High removal efficiencies (i.e. 99% total COD removal) comparable to conventional AnMBR systems are obtained with AnDMBR technology. This can be accomplished by formation of a porous and compressible cake layer on the support material surface.
- As an alternative to MF or UF membranes, polypropylene mono-monofilament filter cloth can be used to provide high quality filtration by self-forming DM layer.
- Support material properties, such as yarn type, are critical for the formation of an effective cake layer over the filter surface in DM filtration technology.
- Staple filter cloth is more suitable for depth filtration, whereas, mono-monofilament filter is more suitable for cake filtration. Therefore, mono-monofilament filter is considered more appropriate for DM filtration systems.
- A stable operation for the treatment of concentrated wastewaters with AnDMBRs is possible for a prolonged period of time.
- Combination of backwashing and biogas sparging enables the control of the DM layer thickness, which is of pivotal importance for achieving stable operation and high quality permeate.

- SRT was found to be an important factor, having a significant effect on SMP and EPS production, P/C ratio, sludge PSD, DM layer formation and consolidation, as well as bulk sludge filterability.
- Decreased EPS concentrations in the bulk sludge, which was measured with prolonged SRT, resulted in an increase in both the amount of small particles and sludge SRF that caused higher TMP and higher filtration resistance.
- A DM layer consisted of both organic and inorganic materials and the DM layer played a significant role in the removal of organic matters in the AnDMBR.
- The combined effect of biomass activity and physical retention in the cake layer is responsible for the removal by the DM layer.
- High accumulation of SMP and EPS in the DM layer likely led to the formation of a tight cake layer and effective retention of soluble COD. However, accumulation of the proteins and formation of a tight cake layer also cause an increase in the filtration resistance.
- Diversity and richness of the microbial communities including bacteria and archaea in the DM layer were high and microbial population composition in the DM layer was different compared to the bulk sludge in the AnDMBR.
- A longer time was needed in the external AnDMBR compared to the submerged one to form an effective DM layer, enabling the achievement of a stable removal efficiency and low soluble COD concentration in the permeate.
- Submerged AnDMBR configuration appears more suitable when a short start-up period is necessary or when frequent filter cleaning is considered.
- Higher methane production rate was obtained in the submerged AnDMBR compared to external AnDMBR, reflecting the negative impact of sludge recirculation in the external AnDMBR configuration on microbial community structure.
- Sludge recirculation in the external configuration is more effective in decreasing DM thickness/compactness, thus TMP, than the bottom biogas sparging in the submerged configuration.
- An overall 99% COD removal efficiency was achieved, irrespective of the GSV used in the AnDMBR, even though the total filtration resistance increased with GSV decrease.
- Total filtration resistance was by far mainly caused by the DM layer.
- A slight decrease in TMP was observed with the increase in GSV. Therefore, energy consumption for biogas sparging, and permeate quality must be evaluated concurrently to determine the optimum GSV.
- Low capital costs of support material, and energy generation can make AnDMBRs feasible for those situations in which a high flux is not necessary such as in sludge and slurry treatment or highly concentrated industrial wastewater treatment.

8.2 Problems Encountered and Future Perspectives

In general, the cake or DM layer governs membrane resistance both in aerobic and anaerobic MBRs. In fact, the same is true for DM filtration systems with the cake layer as the separating functional unit. In most of the studies reported for DMBRs, the DM layer could be easily scoured off with air when the TMP or the water head reached to a certain level.

The formation of SFDM is a complex process including many physicochemical and microbiological mechanisms, such as gel layer formation and cake formation. So far, the formation mechanism and structure of a DM in MBRs have not been completely understood (Liu et al., 2009). There is still limited information on the characteristics of the cake layer formed on the supporting layers, such as cloth or mesh.

The formation conditions applied to generate pre-coated DMs are dependent on the purpose of the studies. In many studies, the effects of individual formation parameters such as formation pressure, cross-flow velocity, concentration of DM layer forming material, and pH on separation performance and DM layer characteristics have been investigated (Ersahin et al., 2012). Because these parameters were investigated individually at different operating conditions, it is difficult to determine the most critical parameters controlling DM formation.

Research on the impact of shear stress by air or biogas sparging for DM formation, control, and process performance yields contradictory results. Some researchers report that a more intensive aeration leads to higher effluent turbidity in mesh filtration (Kiso et al., 2000), whereas others observed that aeration intensity has no significant effect on the effluent SS and turbidity (Alavi Moghaddam et al., 2002).

Thus far, the DM concept has been generally researched for application in aerobic MBRs, treating municipal sewage and low to medium strength synthetic wastewaters under lab-scale conditions. With low-strength wastewater, a significant compromise has to be made on the COD removal efficiency when large fluxes need to be achieved. Lin et al. (2013) reported that a high flux, i.e. 65 $L/m^2.h$, with an AnDMBR treating municipal wastewaters was obtained, however a COD removal efficiency of only 57% was achieved due to the ineffective DM layer formation.

Research done with AnDMBRs for the treatment of high strength wastewaters is quite limited in the literature (this thesis; Ersahin et al., 2014). The implementation of DM approach in AnDMBRs requires optimum conditions that allow satisfactory DM layer formation and effective cake layer control. Cake layer thickness/compactness can be controlled by the mixed liquor characteristics and/or the shear stress at the filtration surface, preventing excessive filtration resistance build up. By finding optimum operating conditions, enabling an effective DM layer formation and consolidation for providing a stable and high quality permeate at reasonable filtration resistances, AnDMBRs can be considered as a reliable and satisfactory alternative treatment technology. However, long-term reliability and operability of the DM applications needs further research at large-scale applications, likely in conjunction with the effect of fluid dynamics and sludge properties for full-scale applications.

AnDMBRs may be feasible for treatment of concentrated waste(water)s especially for those situations in which a high flux is not necessary such as in sludge and slurry treatment or highly concentrated industrial wastewater treatment. The use of low-cost support materials instead of membranes, combined with biogas production as an energy source could make DM technology feasible for the anaerobic treatment of concentrated wastewaters. Dynamic membrane filtration of wastewaters and/or sludge slurries may require less energy and lower capital costs compared to MBRs. Thus, DM filtration can be used in several processes of municipal wastewater treatment plants. Application of DMs in sewage sludge digestion, i.e. separating HRT from SRT, may result in higher SRTs and thus higher sludge concentrations, retaining slowly growing biomass and slowly degradable organic matter in the bioreactor. Furthermore, DM filtration can also be used as an alternative to primary settlers to remove the particulate organic matter with a high efficiency in municipal wastewater treatment plants. Especially for capacity extension of existing wastewater treatment plants with limited available area, system compactness is of high interest.

Focusing on improvement of the current methodologies and/or development of new methods to control cake layer thickness and porosity, while minimizing energy input, e.g. required for biogas recirculation, and filtration resistance, and maximizing the flux, would be beneficial for the future applications of DM technology. An economic feasibility study also would be necessary in order to decide the right membrane module configuration, i.e. submerged or external, to be used for any application of AnDMBRs. Thus, a stable filterability can be obtained with high fluxes in AnDMBRs, which can propose DM filtration as a reliable and promising treatment technology even for large-scale applications.

8.3 References

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APPENDIX

Table A.1. Bacterial species (Chapter 5).

Species Name	Seed Sludge	Day 1 Bulk Sludge	Day 22 Bulk Sludge	Day 50 Bulk Sludge	DM Layer
Cytophaga sp	16.6%	17.0%	11.7%	3.9%	7.6%
Bacteroides sp	13.2%	4.8%	4.7%	23.7%	9.3%
Thermoanaerobacter sp	8.2%	12.9%	11.3%	12.6%	10.3%
Dethiobacter sp	8.5%	11.8%	7.7%	9.3%	13.5%
Pseudoalteromonas sp	3.0%	2.9%	15.8%	11.7%	15.1%
Clostridium sp	6.2%	6.3%	6.3%	6.6%	3.7%
Bellilinea sp	5.1%	7.0%	3.2%	1.2%	5.5%
Acidaminococcus sp	2.3%	5.4%	1.1%	6.5%	6.6%
Anaerophaga sp	6.0%	6.4%	1.7%	1.3%	5.9%
Lactococcus raffinolactis	0.0%	4.1%	6.6%	0.2%	0.0%
Syntrophus sp	2.6%	1.4%	0.7%	0.8%	4.1%
Acidobacterium sp	5.4%	2.0%	0.6%	0.1%	1.3%
Sedimentibacter sp	0.3%	0.5%	2.1%	2.9%	0.3%
Eubacterium sp	1.6%	0.8%	1.1%	1.5%	0.8%
Corynebacterium sp	0.4%	0.5%	2.0%	2.6%	0.2%
Syntrophorhabdus aromaticivorans	1.3%	0.8%	0.9%	0.5%	1.5%
Fervidobacterium sp	1.5%	0.2%	0.0%	0.0%	2.5%
Bacillus sp	0.5%	0.8%	0.3%	0.4%	1.2%
Synergistes sp	1.4%	0.5%	0.5%	0.1%	0.5%
Clostridium acetireducens	0.5%	0.3%	0.6%	0.7%	0.8%
Trigonala elaeagnus	0.6%	0.5%	0.8%	0.0%	0.6%
Desulfotomaculum sp	0.1%	0.1%	0.3%	1.0%	0.9%
Streptomyces sp	0.1%	1.2%	0.5%	0.3%	0.2%
Deferribacter sp	0.3%	0.4%	0.8%	0.4%	0.2%
Chloroflexus sp	0.5%	0.8%	0.4%	0.2%	0.2%
Bdellovibrio sp	0.7%	0.6%	0.3%	0.2%	0.2%
Caloramator sp	0.1%	0.8%	0.6%	0.1%	0.2%
Enterococcus aquimarinus	0.0%	0.2%	1.7%	0.0%	0.1%
Sporobacter termitidis	0.0%	0.0%	0.3%	1.5%	0.1%
Lutispora thermophila	0.2%	0.3%	0.4%	0.2%	0.7%
Erysipelothrix inopinata	0.0%	0.4%	1.0%	0.2%	0.0%
Erysipelothrix sp	0.1%	0.0%	1.3%	0.2%	0.1%
Syntrophomonas sp	0.4%	0.5%	0.5%	0.0%	0.2%
Longilinea sp	0.2%	0.4%	0.8%	0.1%	0.2%
Vagococcus sp	0.0%	0.5%	1.0%	0.0%	0.1%
Proteocatella sphenisci	0.0%	0.2%	1.3%	0.1%	0.0%

Leptolinea tardivitalis	0.3%	0.8%	0.3%	0.0%	0.2%
Pelotomaculum isophthalicicum	0.4%	0.0%	0.5%	0.3%	0.2%
Leptospira sp	0.9%	0.2%	0.1%	0.1%	0.0%
Aminobacterium sp	0.3%	0.2%	0.4%	0.1%	0.2%
Rubrobacter sp	0.4%	0.5%	0.2%	0.1%	0.1%
Smithella propionica	0.4%	0.1%	0.3%	0.1%	0.4%
Sphingobacterium sp	0.0%	0.1%	0.4%	0.3%	0.4%
Anaerolinea sp	0.2%	0.4%	0.1%	0.1%	0.3%
Erysipelothrix muris	0.1%	0.0%	1.0%	0.0%	0.1%
Clostridium aminobutyricum	0.0%	0.3%	0.4%	0.3%	0.1%
Tissierella sp	0.2%	0.2%	0.5%	0.2%	0.0%
Enterococcus sp	0.1%	0.0%	0.5%	0.4%	0.0%
Clostridium viride	0.0%	0.0%	0.0%	1.1%	0.0%
Nitrosovibrio sp	0.6%	0.2%	0.1%	0.1%	0.1%
Levilinea saccharolytica	0.4%	0.4%	0.1%	0.0%	0.2%
Geobacter sp	0.6%	0.3%	0.1%	0.1%	0.0%
Enterococcus inusitatus	0.0%	0.1%	0.8%	0.0%	0.0%
Papillibacter cinnamivorans	0.0%	0.0%	0.1%	0.7%	0.0%
Moorella sp	0.4%	0.0%	0.3%	0.1%	0.0%
Ruminococcus sp	0.7%	0.0%	0.0%	0.0%	0.0%
Parabacteroides goldsteinii	0.0%	0.0%	0.1%	0.6%	0.0%
Carboxydibrachium sp	0.2%	0.5%	0.1%	0.0%	0.0%
Verrucomicrobium sp	0.4%	0.0%	0.2%	0.1%	0.0%
Clostridium pascui	0.0%	0.0%	0.3%	0.3%	0.1%
Bacteroides salanitronis	0.6%	0.0%	0.0%	0.1%	0.0%
Desulfocella halophila	0.3%	0.1%	0.2%	0.1%	0.0%
Spirochaeta sp	0.3%	0.1%	0.0%	0.1%	0.2%
Desulfitobacterium sp	0.1%	0.2%	0.0%	0.2%	0.2%
Parabacteroides distasonis	0.3%	0.1%	0.1%	0.1%	0.0%
Petrimonas sp	0.3%	0.0%	0.1%	0.1%	0.1%
Pseudomonas sp	0.4%	0.0%	0.1%	0.1%	0.0%
Actinomyces marimammalium	0.2%	0.2%	0.1%	0.0%	0.1%
Ruminofilibacter xylanolyticum	0.2%	0.3%	0.0%	0.0%	0.1%
Tissierella creatinini	0.4%	0.1%	0.0%	0.0%	0.0%
Clostridium bartlettii	0.2%	0.2%	0.1%	0.1%	0.0%
Anaeromyxobacter sp	0.2%	0.1%	0.1%	0.0%	0.1%
Clostridium thermocellum	0.0%	0.1%	0.1%	0.2%	0.0%
Lactococcus sp	0.0%	0.0%	0.4%	0.0%	0.0%
Syntrophobacter sp	0.3%	0.1%	0.0%	0.0%	0.0%

Pelobacter sp	0.2%	0.1%	0.0%	0.0%	0.1%
Caldilinea sp	0.1%	0.1%	0.1%	0.1%	0.0%
Alkaliflexus sp	0.0%	0.0%	0.0%	0.3%	0.0%
Planctomyces sp	0.2%	0.0%	0.0%	0.0%	0.1%
Bacillus chagannorensis	0.1%	0.1%	0.1%	0.0%	0.1%
Ralstonia sp	0.3%	0.0%	0.0%	0.0%	0.0%
Tissierella praeacuta	0.0%	0.0%	0.1%	0.2%	0.0%
Gracilibacter thermotolerans	0.1%	0.1%	0.0%	0.1%	0.0%
Solobacterium moorei	0.0%	0.2%	0.0%	0.0%	0.1%
Peptococcus sp	0.0%	0.0%	0.1%	0.2%	0.0%
Dehalobacter sp	0.1%	0.0%	0.1%	0.0%	0.0%
Byssovorax sp	0.0%	0.0%	0.1%	0.1%	0.0%
Catabacter hongkongensis	0.0%	0.0%	0.1%	0.1%	0.0%
Clostridium sporogenes	0.1%	0.0%	0.0%	0.0%	0.1%
Azoarcus sp	0.0%	0.0%	0.0%	0.1%	0.1%
Syntrophomonas zehnderi	0.0%	0.0%	0.0%	0.2%	0.0%
Thermobifida sp	0.0%	0.0%	0.0%	0.2%	0.0%
Clostridium propionicum	0.0%	0.0%	0.0%	0.1%	0.0%
Longilinea arvoryzae	0.0%	0.0%	0.1%	0.0%	0.1%
Aminomonas paucivorans	0.0%	0.0%	0.0%	0.1%	0.0%
Syntrophomonas wolfei	0.0%	0.0%	0.0%	0.2%	0.0%
Desulfosporosinus sp	0.0%	0.0%	0.1%	0.0%	0.1%
Isobaculum melis	0.0%	0.0%	0.0%	0.1%	0.0%
Parvimonas micra	0.0%	0.0%	0.0%	0.1%	0.0%
Desulfomicrobium sp	0.0%	0.1%	0.0%	0.0%	0.0%
Aquimarina sp	0.0%	0.0%	0.0%	0.1%	0.0%
Anaerovorax odorimutans	0.0%	0.0%	0.1%	0.0%	0.0%
Prevotella sp	0.0%	0.0%	0.0%	0.1%	0.0%
Guggenheimella bovis	0.0%	0.0%	0.0%	0.1%	0.0%
Eubacterium aggregans	0.0%	0.0%	0.0%	0.1%	0.0%
Enterococcus faecium	0.0%	0.0%	0.1%	0.1%	0.0%
Citrobacter sp	0.0%	0.0%	0.0%	0.0%	0.1%
Sporobacter sp	0.0%	0.0%	0.0%	0.1%	0.0%
Flavobacterium sp	0.0%	0.0%	0.0%	0.1%	0.0%
Desulfobulbus elongatus	0.0%	0.0%	0.1%	0.0%	0.0%
Desulfovibrio paquesii	0.0%	0.0%	0.0%	0.1%	0.0%
Desulfobulbus propionicus	0.0%	0.0%	0.1%	0.0%	0.0%
Finegoldia magna	0.0%	0.0%	0.0%	0.1%	0.0%
Sedimentibacter saalensis	0.0%	0.0%	0.0%	0.1%	0.0%

Table A.2. Archaeal species (Chapter 5).

Species Name	**Seed Sludge**	**Day 1 Bulk Sludge**	**Day 22 Bulk Sludge**	**Day 50 Bulk Sludge**	**DM Layer**
Methanobacterium alcaliphilum	0.0%	9.1%	0.0%	0.0%	0.0%
Methanobacterium beijingense	2.1%	0.0%	0.0%	0.0%	0.0%
Methanobacterium ferruginis	0.0%	9.1%	0.0%	0.0%	0.0%
Methanobacterium petrolearium	14.6%	36.4%	40.0%	12.5%	1.2%
Methanobacterium sp	0.0%	4.5%	0.0%	0.0%	0.0%
Methanobrevibacter sp	43.8%	9.1%	0.0%	0.0%	14.0%
Methanolinea mesophila	14.6%	0.0%	10.0%	0.0%	43.0%
Methanolinea sp	0.0%	0.0%	0.0%	0.0%	1.2%
Methanoculleus sp	0.0%	0.0%	0.0%	0.0%	2.3%
Methanomicrobium sp	2.1%	0.0%	0.0%	0.0%	7.0%
Methanoregula boonei	0.0%	0.0%	0.0%	6.3%	0.0%
Methanosaeta harundinacea	0.0%	9.1%	0.0%	0.0%	2.3%
Methanosaeta sp	14.6%	13.6%	40.0%	31.3%	12.8%
Methanosarcina sp	0.0%	0.0%	0.0%	43.8%	11.6%
Candidatus Nitrosocaldus yellowstonii	2.1%	0.0%	10.0%	0.0%	0.0%
Thermofilum sp	6.3%	9.1%	0.0%	0.0%	4.7%

Table B.1. Bacterial species (Chapter 6).

Species Name	**Seed Sludge**	**Submerged Bulk Sludge**	**External Bulk Sludge**
Pseudoalteromonas sp	3.0%	0.4%	0.0%
Thermoanaerobacter sp	8.2%	0.2%	0.0%
Bacteroides sp	13.2%	51.8%	72.6%
Cytophaga sp	16.6%	1.2%	0.9%
Clostridium sp	6.2%	10.9%	6.1%
Dethiobacter sp	8.5%	0.0%	0.0%
Acidaminococcus sp	2.3%	0.0%	0.0%
Anaerophaga sp	6.0%	0.0%	0.0%
Sedimentibacter sp	0.3%	1.7%	2.4%
Eubacterium sp	1.6%	0.0%	0.0%
Syntrophus sp	2.6%	2.3%	0.2%
Bellilinea sp	5.1%	0.0%	0.0%
Acidobacterium sp	5.4%	0.0%	0.0%
Aminobacterium sp	7.9%	8.8%	5.0%
OP9 sp	0.9%	11.1%	1.2%
Cloacamonas sp	2.8%	1.3%	0.9%
SR1 sp	0.1%	1.1%	0.0%
Syntrophomonas sp	0.1%	0.4%	2.4%
Petrimonas sp	0.1%	0.1%	0.0%
Corynebacterium sp	0.2%	0.4%	0.2%
Syntrophomonas wolfei	0.0%	0.4%	0.9%
Syntrophorhabdus aromaticivorans	0.3%	0.2%	0.2%
Synergistes sp	0.2%	0.5%	0.2%
Clostridium acetireducens	0.2%	0.5%	0.6%
Desulfotomaculum sp	0.1%	0.5%	0.4%
Clostridium viride	0.0%	0.4%	0.1%
Azoarcus sp	0.0%	0.4%	0.9%
Trigonala elaeagnus	0.2%	0.5%	0.4%
Aminobacterium sp	0.3%	0.3%	0.3%
Fervidobacterium sp	0.1%	0.0%	0.1%
Papillibacter cinnamivorans	0.0%	0.3%	0.1%
Thermobifida sp	0.0%	0.2%	0.0%
Clostridium aminobutyricum	0.0%	0.2%	0.2%
Pelotomaculum isophthalicicum	0.4%	0.3%	0.0%
Parabacteroides distasonis	0.3%	0.4%	0.1%
Bdellovibrio sp	0.3%	0.3%	0.0%

Leptospira sp	0.2%	0.0%	0.1%
Lutispora thermophile	0.2%	0.2%	0.2%
Syntrophomonas zehnderi	0.0%	0.0%	0.6%
Peptococcus sp	0.0%	0.0%	0.2%
Spirochaeta sp	0.1%	0.1%	0.1%
Nitrosovibrio sp	0.2%	0.1%	0.1%
Verrucomicrobium sp	0.4%	0.0%	0.1%
Chloroflexus sp	0.1%	0.2%	0.0%
Deferribacter sp	0.3%	0.2%	0.0%
Smithella propionica	0.4%	0.2%	0.0%
Geobacter sp	0.2%	0.0%	0.1%
Gracilibacter thermotolerans	0.1%	0.2%	0.2%
Caloramator sp	0.1%	0.2%	0.1%
Bacteroides salanitronis	0.2%	0.1%	0.0%
Ruminococcus sp	0.3%	0.0%	0.0%
Desulfobulbus propionicus	0.0%	0.1%	0.1%
Actinomyces marimammalium	0.2%	0.2%	0.0%
Parabacteroides goldsteinii	0.0%	0.1%	0.0%
Desulfovibrio paquesii	0.0%	0.1%	0.1%
Pseudomonas sp	0.4%	0.0%	0.1%
Flavobacterium sp	0.0%	0.2%	0.1%
Rubrobacter sp	0.4%	0.0%	0.0%
Tissierella sp	0.2%	0.1%	0.1%
Ralstonia sp	0.3%	0.0%	0.2%
Tissierella creatinine	0.2%	0.0%	0.1%
Lactococcus raffinolactis	0.1%	0.0%	0.1%
Levilinea saccharolytica	0.4%	0.0%	0.0%
Desulfocella halophile	0.3%	0.0%	0.1%
Pelotomaculum sp	0.0%	0.1%	0.1%
Desulfobulbus elongates	0.0%	0.0%	0.1%
Syntrophobacter sp	0.3%	0.0%	0.1%
Leptolinea tardivitalis	0.3%	0.1%	0.0%
Acholeplasma morum	0.0%	0.0%	0.1%
Streptomyces sp	0.1%	0.1%	0.0%
Longilinea sp	0.2%	0.0%	0.1%
Pelobacter sp	0.2%	0.1%	0.1%
Desulfitobacterium sp	0.1%	0.0%	0.1%
Dysgonomonas sp	0.0%	0.2%	0.1%

Table B.2. Archaeal species (Chapter 6).

Species Name	Seed Sludge	Submerged Bulk Sludge	External Bulk Sludge
Methanobrevibacter sp	*42.9%*	*25.0%*	*0.0%*
Methanosaeta sp	*14.3%*	*25.0%*	*25.0%*
Methanobacterium petrolearium	*14.3%*	*15.0%*	*50.0%*
Methanolinea mesophila	*14.3%*	*10.0%*	*0.0%*
Methanosarcina sp	*2.0%*	*5.0%*	*25.0%*
Methanomicrobium sp	*2.0%*	*5.0%*	*0.0%*
Thermofilum sp	*6.1%*	*0.0%*	*0.0%*
Hyperthermus sp	*0.0%*	*10.0%*	*0.0%*
Methanobacterium beijingense	*2.0%*	*0.0%*	*0.0%*
Methanospirillum sp	*0.0%*	*5.0%*	*0.0%*
Nitrosocaldus yellowstonii	*2.0%*	*0.0%*	*0.0%*

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Mustafa Evren Erşahin graduated as an environmental engineer from the Environmental Engineering Department of Trakya University in Turkey holding the first rank both in the department and faculty in 2003. He received his MSc degree from Department of Environmental Engineering at Istanbul Technical University. He has been working as a research assistant in the same department since 2005. After graduation he was involved in many research and development projects in the field of biological wastewater treatment, biomethanization of solid wastes, biosystem modelling, anaerobic biotechnology, energy efficient treatment processes, and membrane processes. He also assisted different courses including water and wastewater treatment, design of water and wastewater treatment plants, probability and statistics. In 2010, he has joined the Sanitary Engineering Section of the Watermanagement Department at TU Delft as a PhD researcher. He got the PhD fellowship award provided by HUYGENS Scholarship Programme in 2011. His PhD study focused on application of dynamic membranes in anaerobic membrane bioreactor systems under the supervision of Jules B. van Lier, İzzet Öztürk and Henri Spanjers.

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