ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

TREATMENT OF SEWAGE SLUDGE BY ANAEROBIC MEMBRANE BIOREACTOR TECHNOLOGY

M.Sc. THESIS

Muhammed Furkan ARAS

Department of Environmental Engineering

Environmental Science, Engineering and Management Programme

MAY 2023

ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

TREATMENT OF SEWAGE SLUDGE BY ANAEROBIC MEMBRANE BIOREACTOR TECHNOLOGY

M.Sc. THESIS

Muhammed Furkan ARAS
(501191722)

Department of Environmental Engineering

Environmental Science, Engineering and Management

Thesis Advisor: Assoc. Prof. Dr. Hale ÖZGÜN

MAY 2023

ISTANBUL TEKNİK ÜNİVERSİTESİ ★ LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ

EVSEL ATIKSU ARITMA TESİSİ ÇAMURLARININ ANAEROBİK MEMBRAN BİYOREAKTÖR TEKNOLOJİSİ İLE ARITILMASI

YÜKSEK LİSANS TEZİ

Muhammed Furkan ARAS
(501191722)

Çevre Mühendisliği Anabilim Dalı

Çevre Bilimleri, Mühendisliği ve Yönetimi Programı

Tez Danışmanı: Doç. Dr. Hale ÖZGÜN

MAYIS 2023

Muhammed Furkan ARAS, a M.Sc. student of ITU Graduate School student ID 501191722, successfully defended the thesis entitled “TREATMENT OF SEWAGE SLUDGE BY ANAEROBIC MEMBRANE BIOREACTOR TECHNOLOGY” which he prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

Thesis Advisor :	**Assoc. Prof. Dr. Hale ÖZGÜN** İstanbul Technical University	....
Jury Members :	**Assist. Prof. Dr. Türker TÜRKEN** Istanbul Technical University	....
	Assoc. Prof. Dr. Afşın Yusuf ÇETİNKAYA Yıldız Technical University	**…………..**

Date of Submission : 05.04.2023
Date of Defense : 03.05.2023

To my beloved wife and daughter,

FOREWORD

"The obstacle is the path", says a Zen proverb. This proverb encourages whoever comes across difficulties, I believe. Difficulties are the triggers, that allow us human beings to grow, as long as we're encouraged to take them. Having graduated from another university and starting an MSc. program at ITU was well enough to be stressed, to be honest. When I began my MSc. study at ITU, I had to be ready for the challenge of tough courses and long laboratory work. However, together with my passion for science and self-discipline, a well-coordinated lab group was the main source of my motivation with their dedicated and disciplined work style. Therefore, now I believe more in the fact that a team can do what one cannot.

From then to now, I have many people I owe appreciation to, who helped me throughout my path up to now. Assoc. Prof Hale ÖZGÜN, my supervisor, has been more than a supervisor with her very gentle and adviceful attitude. I have always thought of her self-discipline and motivation as a goal I want to reach. I appreciate your support and guidance throughout my MSc. journey. A special thanks to Assoc. Prof. Mustafa Evren ERŞAHİN, for his guidance and advice and for helping me gain a scientific perspective. I sincerely thank both of you especially because you set an example for me in my orientation toward academia.

Doubtlessly, I owe other special thanks to my laboratory partner Dr. Amr Mustafa ABDELRAHMAN. He introduced me to the anaerobic membrane bioreactor system and helped me learn a lot with his gentle and patient attitude. Without you, the laboratory work wouldn't have been so entertaining. You're such a passionate, inquisitive, and hardworking scientist, who the future ones must take as an example. I feel very fortunate to have worked under your guidance. The continued support you provided deserves more than thanks.

I would like to thank to Scientific Research Projects Department of Istanbul Technical University (Project No: MYL-2020-42701) for providing support. This research was partly funded by Istanbul Water and Sewerage Administration (ISKI) with the project titled as "Integration of High-rate Activated Sludge Process and Anaerobic Membrane Bioreactor Process for Energy Efficient Wastewater Treatment in Istanbul: Maximum Energy Recovery (MEGA2 Project)".

I would like to express my gratitude to Turkish Academy of Science (TÜBA) for the financial support during my thesis study. Very special thanks to Prof. İzzet ÖZTÜRK, who is an honorary member of TÜBA, for being an ongoing supporter along with my thesis with his deep knowledge on the area.It is not possible to forget my dear friends from En3Lab Research Group for converting the laboratory into a friendly environment and also for their continued physical and mental backup in the laboratory.

Final thanks go to my family for giving me the encouragement to pursue an academic career. I'm thankful to my dear father, mother, and brothers for their support throughout my whole educational life. It wouldn't be possible to plan my academic future without their support. Some words, even though they wouldn't summarize my

feelings, to my dear wife: since you came into my life, everything drastically changed from grayness to beauty and life became meaningful. Thank you for your patience in my busy times and you'll always be the main power in whatever I'll do.

May 2023

Muhammed Furkan ARAS
(Environmental Engineer)

TABLE OF CONTENTS

Page

ABBREVIATIONS

A-sludge	**:** Adsorption stage sludge
A-stage	**:** Adsorption stage
AD	**:** Anaerobic digestion
ADUF	**:** Anaerobic digestion ultrafiltration
AnMBR	**:** Anaerobic membrane bioreactor
AnDMBR	: Anaerobic dynamic membrane bioreactor
BOD	**:** Biochemical oxygen demand
BPC	: Biopolymer cluster
$CaCO_3$	**:** Calcium carbonate
CH_4	**:** Methane
CIP	**:** Clean-in-place
CLSM	**:** Confocal laser scanning microscopy
COD	**:** Chemical oxygen demand
COP	**:** Clean-out-of-place
CO_2	**:** Carbon dioxide
CST	**:** Capillary suction time
CSTR	**:** Completely stirred tank reactor
DM	**:** Dynamic membrane
DO	**:** Dissolved oxygen
DP	**:** Dissolved phosphorus
D50	**:** Median particle size
EC	**:** Evaporator condensate
EDTA	**:** Ethylenediaminetetraacetic acid
EPS	**:** Extracellular polymeric substances
ESEM	**:** Environmental scanning electron microscope
FID	**:** Flame ionization detector
FTIR	**:** Fourier transform infrared spectroscopy
FW	**:** Food waste
GC	**:** Gas chromatography
HCl	**:** Hydrochloric acid

HRAR : High-rate anaerobic reactor
HRAS : High-rate activated sludge
HRT : Hydraulic retention time
H_2S : Hydrogen sulfide
$K_2NH_4PO_4$: Potassium ammonium phosphate
LB-EPS : Loosely-bound EPS
MARS : Membrane anaerobic reactor system
MAS : Membrane anaerobic system
MBR : Membrane bioreactor
MF : Microfiltration
$MgNH_4PO_4$: Magnesium ammonium phosphate
MLSS : Mixed liquor suspended solids
MLVSS : Mixed liquor volatile suspended solids
MPN :Most probable nummber
MW : Molecular weight
NaClO : Sodium hypochlorite
NaOH : Sodium hydroxide
NH_3 : Ammonia
NH_4-N : Ammonium-nitrogen
OLR : Organic loading rate
ORP : Oxidation-reduction potential
Pd-Au : Palladium-gold
PE : Polyethylene
PES : Polyethersulfone
PLC : Programmable Logic Controller
PP : Polypropylene
PS : Primary sludge
PSD : Particle size distribution
PSF : Polysulfone
PVDF : Polyvinylidene fluoride
sCOD : Soluble chemical oxygen demand
SAnMBR : Submerged anaerobic membran bioreactor
SCADA : Supervisor control and data acquisition
SD : Standart deviation
SMA : Specific methanogenic activity

SMBR : Submerged membrane bioreactor
SMP : Soluble microbial products
SRT : Sludge retention time
SS : Suspended solids
TB-EPS : Tightly-bound EPS
TMP : Transmembrane pressure
TN : Total nitrogen
TP : Total phosphorus
TS : Total solids
TSS : Total suspended solids
TWAS : Thickened waste activated sludge
UF : Ultrafiltration
US : Ultrasonication
USEPA : United States Environmental Protection Agency
VFA : Volatile fatty acids
VS : Volatile solids
VSS : Volatile suspended solids
WAS : Waste activated sludge
WWTP : Wastewater treatment plant

SYMBOLS

H	**:** Henry’s law constant
N_w	**:** Number of water moles in 1 L solution
P	**:** Pressure
V_c	**:** Corrected volume of 1 mole of gas at 35 °C

LIST OF TABLES

Page

LIST OF FIGURES

Page

TREATMENT OF SEWAGE SLUDGE BY ANAEROBIC MEMBRANE BIOREACTOR TECHNOLOGY

SUMMARY

Wastewater treatment requires a substantial amount of energy to meet the discharge criteria. Energy can be recovered by anaerobic digestion of the produced sludge, which has a favorable impact on the energy balance. Conventional anaerobic digesters are constructed as completely mixed reactors operated at sludge retention times (SRTs) (< 30 days) to maximize solids conversion into biogas and sustain the methanogenic activity inside the reactor. In order to achieve a sufficient reduction of volatile solids (VS), anaerobic digesters are often constructed with huge volumes. Effective substitutes for conventional anaerobic digesters for the digestion of sludge are anaerobic membrane bioreactors (AnMBRs). Simply, AnMBR system is made up with the combination of a membrane and an anaerobic reactor. AnMBRs produce high-quality effluent, have a lower environmental impact, are resistant to toxic substrates, and have a high ability to transform carbonated organic molecules into biogas. AnMBRs can be operated at long SRT independent from hydraulic retention time (HRT), which allows the biomass to retain in the reactor for a longer time, thus results in higher digestion performance and biogas production.

As an alternative to primary clarifier, high-rate activated sludge (HRAS) system, referred as adsorption stage (A-stage), was used since more organic matter can be recovered by A-stage. The biogas produced during the digestion of each sludge type and methane content were measured. The permeate quality was assessed. The filtration performance of an ultrafiltration (UF) membrane was also observed. The membrane area was 0.012 m^2 and the flux was 5 $L/m^2.h$. Morphological analyses were conducted to make a broader evaluation of membrane fouling. This study makes a comparative evaluation of the biogas production, treatment performance, and filtration performance of primary sludge (PS) and adsorption stage sludge (A-sludge) treated by AnMBR under mesophilic conditions. Finally, a plant-wide chemical oxygen demand (COD) mass balance was conducted to evaluate the COD conversion of each sludge type.

Biogas production for PS was observed to be higher than for A-sludge, with average volumes of 5908 ± 352 and 5486 ± 238 mL/day, respectively. However, A-sludge contained a greater methane percentage (73%) in biogas than PS (62%). Similar COD removal efficiencies were obtained for each sludge type, approximately 96% for PS and 97% for A-sludge. Stable digester conditions in the digester were obtained, considering the optimum volatile fatty acids (VFA) to alkalinity ratio of nearly 0.08 found for each sludge type. Total nitrogen (TN) removal efficiency was 52.5% for PS, while nearly 19% was achieved for A-sludge. High total phosphorus (TP) removal efficiencies of 97% and 82% were acquired for PS and A-sludge, respectively. Total suspended solids (TSS) removal efficiency for each sludge was more than 99% thanks to the membrane, and almost no solids and fecal coliforms were found in the permeate. Extracellular polymeric substances (EPS) were found higher in AnMBR treating A-sludge. In correlation with this higher EPS, a higher capillary suction time (CST) value

(293 ± 11 sec) was observed for the anaerobic sludge fed with A-sludge. Average transmembrane pressure (TMP) was higher for A-sludge (223 ± 51 mbar) in comparison to PS (171 ± 53). Morphological analyses of membranes were conducted following the operation period. Environmental scanning electron microscopy (ESEM) analysis revealed that a denser cake layer was observed on the membrane of the system fed with A-sludge, which may be correlated with the higher EPS content in the sludge of the system fed with A-sludge. A plant-wide COD mass balance was conducted in the study and revealed that A-stage integration can convert 34.5% of COD in the wastewater into methane, while primary clarifier integrated with AnMBR can recover only 19.9% of COD into methane. Consequently, in terms of energy efficiency, integration of AnMBR with A-stage instead of primary clarifier can be applied and contributes to the energy efficiency of wastewater treatment plants (WWTPs).

.

EVSEL ATIKSU ARITMA TESİSİ ÇAMURLARININ ANAEROBİK MEMBRAN BİYOREAKTÖR TEKNOLOJİSİ İLE ARITILMASI

ÖZET

Deşarj kriterlerinin sağlanması için uygulanan atıksu arıtma teknolojileri önemli miktarda enerji gerektirir. Enerji, üretilen çamurun anaerobik çürütülmesi yoluyla geri kazanılabilir ve geri kazanılan enerji, enerji dengesi üzerinde olumlu bir etkiye sahip olur. Konvansiyonel anaerobik çürütücüler, tam karışımlı reaktörler olarak inşa edilirler ve uygun çamur bekletme sürelerinde (ÇBS) (<30 gün) işletilirler. Böylece organik içeriğin biyogaza dönüşümü gerçekleşir. Uçucu katı maddenin (UKM) yeterli seviyede giderimini sağlamak için anaerobik çürütücüler genellikle büyük hacimlerde inşa edilirler. Anaerobik membran biyoreaktörler (AnMBR) çamurun çürütülmesi için konvansiyonel anaerobik çürütücülere alternatif olarak kullanılan etkili bir teknolojidir. AnMBR sistemi, membran ve anaerobik reaktörün kombinasyonundan oluşur. AnMBR'ler yüksek kaliteli çıkış suyu üretme, düşük çevresel etkiye sahip olma, toksik akımlara karşı dayanıklı olma ve organik maddeyi biyogaza dönüştürme konusunda oldukça yüksek bir verime sahiptirler. AnMBR'ler, hidrolik bekletme süresinden (HBS) bağımsız olarak daha uzun ÇBS'lerde çalıştırılabilirler. Bu durum, çamurun reaktörde daha uzun süre tutulmasına, dolayısıyla daha yüksek çürütme verimi ve biyogaz oluşma potansiyeline neden olur.

Ön çökeltim sistemine alternatif olarak, A prosesi adı verilen yüksek hızlı aktif çamur sistemi (YHAÇ) kullanılabilir. A prosesi oldukça yüksek seviyelerde enerji geri kazanımı sağlamaktadır. Bu çalışmada, birincil ve A prosesi çamurlarının AnMBR'de çürütülmesi sonucu oluşan biyogaz miktarı karşılaştırmalı olarak değerlendirilmiştir. Ayrıca, süzüntü suyu kalitesi değerlendirilmiştir. Her iki durum için ultrafiltrasyon (UF) membranının filtrasyon performansı gözlenmiştir. UF membranı 0.012 m^2 membran alanı ile 5 L/m^2.sa akıda işletilmiştir. Membran tıkanmasının daha detaylı bir şekilde değerlendirmesini yapmak üzere morfolojik analizler yapılmıştır. Bu çalışmada, mezofilik koşullar altında AnMBR teknolojisi ile birincil ve A prosesi çamurlarının çürütülmesi sonucu elde edilen biyogaz üretimi, arıtma performansı ve filtrasyon performansı karşılaştırmalı olarak değerlendirilmiştir. Ayrıca bu çalışmada, her iki çamur türü için kimyasal oksijen ihtiyacı (KOİ) esaslı kütle dengesi gerçekleştirilmiştir.

Birincil ve A prosesi çamurları için ortalama biyogaz üretimi, sırasıyla 5908 ± 352 ve 5486 ± 238 mL/gün’dür. Bununla birlikte, A prosesi çamurundan elde edilen biyogazdaki metan içeriğinin (%73), birincil çamurdan elde edilen biyogazdaki metan içeriğine (%62) göre daha yüksek olduğu gözlenmiştir. Her iki çamur türü ile beslenen sistemde birincil çamur için yaklaşık %96 ve A prosesi çamuru için %97 olmak üzere benzer KOİ giderim verimleri elde edilmiştir. Her iki işletme koşulunda reaktörde elde edilen yaklaşık 0.08'lik uçucu yağ asitlerinin (UYA) alkaliniteye oranı dikkate alındığında, çürütücüde kararlı koşulların elde edildiği söylenebilir. Toplam azot (TN) giderim verimi birincil çamur için %52,5, A prosesi çamuru için yaklaşık %19 olarak bulunmuştur. Birincil ve A prosesi çamurları için sırasıyla %97 ve %82'lik oldukça yüksek toplam fosfor (TP) giderim verimleri elde edilmiştir. Membran kullanılması sonucu her iki çamur için toplam askıda katı madde (AKM) giderim verimi %99'un üzerinde olmakla birlikte, süzüntü akımında fekal koliform gözlenmemiştir. Hücredışı polimerik madde içeriği, A prosesi çamuru ile beslenen AnMBR’de daha yüksek bulunmuştur. Bununla bağlantılı olarak, anaerobik çamur için daha yüksek bir kapiler emme süresi (KES) değeri (293 ± 11 sn) gözlenmiştir. A prosesi ve birincil çamur ile beslenen sistemde ortalama transmembran basınç değerinin sırasıyla 223 ± 51 mbar ve 171 ± 53 mbar olduğu görülmüştür. Her iki çamur ile işletmeyi takiben membranlarda morfolojik analizler gerçekleştirilmiştir. Taramalı elektron mikroskobu (ESEM) ile elde edilen sonuçlara göre, A prosesi çamuru ile beslenen sistemde membran yüzeyinde daha yoğun bir kek tabakasının oluştuğu gözlenmiş, bu durum A prosesi çamuru ile beslenen reaktördeki çamurun daha yüksek hücredışı polimerik madde içeriği ile ilişkilendirilmiştir. Çalışmada kimyasal oksijen ihtiyacı (KOİ) esaslı kütle dengesi gerçekleştirilmiş ve A prosesi entegrasyonu sonucu AnMBR ile atıksudaki KOİ'nin %34,5'inin metana dönüştürülebildiği, ön çökeltim tankı entegrasyonu ile ise KOİ'nin yalnızca %19,9'unun metana dönüştürülebildiği hesaplanmıştır. Sonuç olarak, enerji verimliliği açısından, ön çökeltim sistemine alternatif olarak A prosesi ile entegre edilen AnMBR sistemlerinin teknik olarak uygulanabilir olduğu sonucuna varılmış ve atıksu arıtma tesislerinin (AAT) enerji verimliliğine önemli seviyede katkı sağlayacağı görülmüştür.

1. INTRODUCTION

1.1 Background

Sludge is formed as a residue in wastewater treatment plants (WWTPs) during wastewater treatment (Zhang et al., 2017). Due to the increase in population, water use has exponentially increased, resulting in an elevation in wastewater production. Sewage sludge may comprise several unwanted constituents such as organic matter, micropollutants, nutrients, and heavy metals (Abdelrahman et al., 2021). In the conventional WWTPs, mainly two types of sludge are produced, i.e. primary sludge (PS) from the primary clarifier and secondary sludge from the aerated activated sludge tank called waste activated sludge (WAS). To maintain environmental protection and human health, proper management of sludge is required. Efficient sludge management should cover both the reduction of sludge amount and the removal of these hazardous substances.

Sewage sludge contains a relatively high amount of energy, which can be recovered by anaerobic digestion (AD) process. The principal of AD process is the decomposition of organic matter into smaller compounds and finally into biogas, which mainly consists of methane (55-70%) and carbon dioxide (CO_2) (25-30%) (Singh et al., 2019). AD of sludge can reduce greenhouse gas emissions particularly due to the capture of methane in addition to the decrease in sludge volume. However, conventional anaerobic digesters have several handicaps such as limited organic loading rate (OLR), low hydrolysis rate, and thus low biogas production. Especially at low-temperature conditions (<20 °C), reaction rates restrict the overall process performance including digestion rates and biogas production rates. In addition, conventional anaerobic digesters require high reactor volumes that causes high capital costs (Ozgun et al., 2019). Overall, conventional anaerobic digesters still need modification in reducing footprints, increasing the process stabilization, optimizing pre-thickening to reduce reactor sizes, and increasing methane production (Abdelrahman et al., 2021).

Anaerobic membrane bioreactor (AnMBR) technology has been proposed to overcome the drawbacks of the conventional digesters. AnMBRs have long been applied for energy recovery to achieve energy-neutral or energy-positive WWTPs. Therefore, the amount of energy recovered from AnMBR systems has increasingly gained importance in recent decades, which lead to studies in which different sludge types have been examined in terms of their energy potential. WAS has been widely used in AnMBRs for digestion (Wen et al., 2008; Xu et al., 2010; Joshi and Parker, 2015; Yu et al., 2016). Co-digestion of PS and thickened waste activated sludge (TWAS) was also studied to investigate its biogas recovery (Pileggi and Parker, 2017). The coupled membrane allows an effective retention of biomass in the reactor for a longer time. This provides a higher digestibility of the substrate, higher solids destruction, and thus, higher biogas production as well as less sludge production (Yu et al., 2016). Due to its solid- and coliform-free permeate, AnMBRs can be used for irrigational purposes (Hafuka et al., 2019). Different membrane materials have been used so far, such as organic (polymeric), inorganic (ceramic), and metallic membranes. The most widely used membrane type was the polymeric membrane due to its relatively low cost (Santos and Judd, 2010). On the other hand, although ceramic membranes are more expensive, they are known to be more resistant to corrosion and have less irreversible fouling compared to other membrane types (Murić et al., 2014). The most widely used membrane types in AnMBR systems are ultrafiltration (UF) and microfiltration (MF) membranes configured as tubular, hollow fiber, flat sheet, or monolithic (Abdelrahman et al., 2021).

1.2 Problem Statement

AnMBR technology has gained popularity in recent years mainly due to its energy recovery potential. Organic content of the sludge is related with the energy potential of anaerobic digestion processes. The use of a primary clarifier before biological reactors leads to a production of a higher organic-rich sludge that can be used to recover energy by AD. A typical primary clarifier with the hydraulic retention time (HRT) of 2-3 days recovers 40% of organic matter into the sludge stream. An alternative to primary clarification is adsorption stage (A-stage), that is a high-rate activated sludge (HRAS) system. A-stage can recover 66% of the organics of the stream. Therefore, more organics can be sent to the anaerobic digester compared to that of primary clarification, improving the energy balance of the WWTP (Wan et al., 2016). In the literature, no study has been conducted to observe the impact of A-stage integration instead of primary clarifier on sludge digestion in AnMBR.

1.3 Aim of Thesis

This thesis focused on comparing the treatment and filtration performances of AnMBR treating PS and adsorption stage sludge (A-sludge). AnMBR system was operated in the same conditions during each stage. The aim of this thesis was accomplished by the objectives below:

- Comparison of treatment performance of PS and A-sludge in terms of biogas production and organic matter removal efficiency, process stability and permeate quality,
- Determination of the filtration performance of UF membrane implemented for physical separation of anaerobic sludge,
- Assessment of morphological properties of the cake layer formation on the membrane at each stage to have a better understanding about the membrane fouling,
- Developing a chemical oxygen demand (COD) mass balance for AnMBRs digesting PS and A-sludge.

1.4 Outline of Thesis

The thesis consists of 5 chapters, in which all procedures, outcomes, and results were broadly given. The thesis content is briefly explained below:

Chapter 2 provides a brief literature review of sludge management and sludge treatment methods. Essentials of anaerobic digester and AnMBR processes are given. This chapter compares conventional anaerobic digester and AnMBR processes in terms of their treatment efficiency and applicability. Furthermore, an overview of AnMBR technology; including its historical development, different configurations, and substrate types are summarized. Additionally, parameters affecting the overall performance of AnMBR technology are given. Membrane characteristics and fouling control strategies are presented. Finally, treatment and filtration performance of AnMBR for sludge treatment are briefly reviewed.

Chapter 3 explains the experimental procedure used during the operation of the AnMBR. Characteristics of seed sludge and substrates are given in detail. A schematic diagram of the AnMBR setup, and supervisory control and data acquisition (SCADA) control system are illustrated. Membrane characteristics are given. Analytical techniques for the analysis of several parameters used in the study are explained in detail. Operational conditions of AnMBR are reported with the parameters such as HRT, sludge retention time (SRT), OLR, and temperature. The devices used for morphological analyses are illustrated and the methods used for each analysis are explained. Moreover, the calculations of the COD mass balance are given.

In chapter 4, the findings obtained in this study are thoroughly explained. Treatment and filtration performances of AnMBR for PS and A-sludge treatment are compared. The COD mass balance conducted for each sludge type are shown. Morphological analyses for cake layer formation on the membranes are reported.

Chapter 5 gives an overview of the study. It summarizes and debates the particular findings of the study. It also gives some recommendations for further research.

2. LITERATURE REVIEW

2.1 An Overview of Sludge Treatment

A massive amount of sludge is formed due to wastewater treatment worldwide, which needs to be disposed properly to overcome its harmful effect on the environment. Currently, the worldwide sewage sludge production rate is approximately 45 million dry tons of sludge per year, equivalent to circa 2 billion population equivalent (Zhang et al., 2017). The most populated countries such as China produce a huge amount of sludge to be treated. China yielded a large amount of sludge in 2013, only 25% of which was properly treated (Zhang et al., 2016). In 2016, China produced 30 million tons of wet sludge (with 80% moisture content) generated in 5300 WWTPs (Wang et al., 2017). In the United States, the energy consumption for water and wastewater treatment represents 3-4% of total energy consumption (Daw et al., 2012). However, this energy consumption can be reduced by capturing the energy inside the wastewater to generate electricity and heat.

Sludge management requires high capital and operational costs. In Australia and Europe, 150-350 USD /ton sludge and 165-550 USD /ton sludge are spent for the treatment of wastewater sludge, respectively (Batstone et al., 2011). Before 2013, 10 billion USD per year was an estimation for sludge handling costs throughout the world (Zhang et al., 2017). Therefore, studies have long focused on decreasing this high cost of sludge management by reusing sludge and producing less sludge amounts during treatment. According to Spinosa et al. (2011), almost 50% of the operational cost in WWTPs is related to sludge handling. On the other hand, sludge handling methods have increased recently. There are several methods to deal with the management of sludge, including AD, mechanical dewatering, thermal drying, and pyrolysis–gasification.

Wastewater treatment sludge generally contains organic matter, heavy metals, nutrients, and micropollutants (Abdelrahman et al., 2021). For proper handling of sludge, the characteristics of sludge must be known. Sludge is handled to reduce its volume, remove its contaminants, digest its organic matter, and stabilize its organic and inorganic matter for meeting the disposal regulations. In WWTPs, mainly two types of sludge form, i.e. PS and secondary sludge. PS occurs with the effect of gravitational force and contains high amount of organics. Secondary sludge often named activated sludge, occurs after the secondary treatment as sedimentation of biological solids produced in the aeration tank. Secondary sludge contains protozoa, rotifers, polysaccharides, protein-rich bacteria, and extracellular polymeric substances (EPS)-forming microorganisms (Markis et al., 2014).

AD is the mostly applied stabilization process used in WWTPs. AD has been well-known for centuries. Biogas was actively used in bathwater heating in the 16th century in Assyria and Persia. After Sir Humphry Davy explored the presence of methane in the produced gas in 1808, a few AD plants were established in South Asia and then spread to Europe (Uddin and Wright, 2022). Since then, the application of AD in WWTPs has increased exponentially to control odor, and decrease sludge volume. In anaerobic digesters, the organic matters are converted into biogas that is used as a renewable energy source thanks to its methane content.

Anaerobic process is carried out in the absence of oxygen and consists of four main steps: Hydrolysis, acidogenesis, acetogenesis, and methanogenesis. In the hydrolysis step, insoluble organic material and larger compounds such as polysaccharides, lipids, and proteins are hydrolyzed into soluble organic substances such as amino acids and fatty acids. The hydrolysis step is followed by acidogenesis, in which the soluble organic substances are converted to volatile fatty acids (VFA) by acidogenic bacteria together with ammonia (NH_3), CO_2, hydrogen sulfide (H_2S), and some other compounds. Acetogenesis is the next step in AD process, where higher organic acids are further digested by acetogenic bacteria to produce acetic acid, CO_2, and hydrogen. In the last step, methanogenesis, two groups of methanogenic archaea produce methane: the first group, acetoclastic methanogens, converts acetate into methane and carbon dioxide, and the second group, hydrogenotrophic methanogens, uses CO_2 as the electron acceptor and hydrogen as the electron donor to produce methane (Meegoda et al., 2018).

AD process is carried out at different temperatures. Mesophilic is the most widely used condition in AD systems with an optimum temperature of 35 °C. Other temperature conditions are psychrophilic and thermophilic with optimum temperatures of 20 °C and 55 °C, respectively. Besides the temperature, parameters affecting methane production can be sorted as pH, the biodegradability of the organic waste, and toxic compounds present in the substrate. Increase in VFA accumulation in anerobic reactor influences the methanogens and may result in decreased methane production. VFA accumulation is basically caused by shock loading, pH, temperature, and overloading or some inhibitory substances such as sulfur, ammonia, halogenated aliphatic, aromatic or phenolic compounds, or heavy metals (Borja, 2011).

2.2 AnMBR Technology

2.2.1 Historical development of AnMBR technology

AnMBRs have gained much attention in recent decades. Due to its high treatment efficiency and energy-efficient features, it is a highly promising and applicable process. The idea of utilizing membrane filtration in conjunction with anaerobic wastewater treatment seems to have been tested for the first time in 1978 by Grethlein (1978). Septic tank effluent was successfully treated by an external cross-flow membrane and reduced biochemical oxygen demand (BOD) by 85-95%, nitrate concentration by 72%, and orthophosphate concentration by 24-85% (Liao et al., 2006). The membrane anaerobic reactor system (MARS), the first commercially available AnMBR, was created by Dorr-Oliver in the early 1980s for the treatment of wastewater from high-strength whey production. The MARS system consisted of a fully mixed suspended growth anaerobic reactor for biodegradation and an external cross-flow membrane module for biomass separation. However, pilot-scale testing of the MARS process did not result in its full-scale implementation, probably because of the high cost of the membrane at that time (Strohwald and Ross, 1992). In the same time frame, the Japanese government launched the "Aqua-Renaissance '90" nationwide project, which sparked the creation of numerous AnMBR systems (Kimura, 1991; Minami et al., 1991). The majority of these commercially available AnMBR systems were set up using external configuration. For the treatment of industrial wastewater, a system known as anaerobic digestion ultrafiltration (ADUF) was created in South Africa in 1987 (Rossi et al., 1990). There are several full- and pilot-scale ADUF systems in use. The results of the pilot- and full-scale tests showed that COD removal efficiency was over 90% (Liao et al., 2006).

The first investigation on the use of AnMBR for digestion of sewage sludge was carried out towards the end of the 1980s and reported by Bindoff et al. (1988). Only a few researches focused on membrane-coupled anaerobic digesters for the digestion of sewage sludge (Kayawake et al., 1991; Pillay et al., 1994), primary sludge (Ghyoot and Verstraete, 1997), and WAS (Takashima et al., 1996) until the 2000s.

In the 2000s, studies began to concentrate on employing AnMBR for recovering VFAs from sludge. A photosynthetic reactor was operated to produce hydrogen gas from VFA recovered from WAS fermentation (Jeong et al., 2007). Since 2005, there have been an exponential increase in the number of scientific studies about AnMBRs. Approximately 93% of the studies focused on AnMBR for sludge treatment in the literature has been created in the last ten years (Abdelrahman et al., 2021).

AnMBR applications have gained a lot of attention since 2008, particularly for methane production from WAS digestion. To determine the optimum conditions and identify the technical constraints of the technology, different operational conditions of AnMBRs were tested (Meabe et al., 2013; Wandera et al., 2018; Hafuka et al., 2019). To increase the digestibility and/or filterability of sludge, sludge pretreatment and the addition of adsorbents were investigated (Yu et al., 2015; Joshi and Parker, 2015; Martin-Ryals et al., 2017). Additionally, some studies combined the use of ultrasound with AnMBR (Xu et al., 2011; Xu et al., 2013). In the studies of Qiao et al. (2013a) and Chen et al. (2019), sludge was also co-digested with certain other wastes such as coffee grounds and coffee processing effluent in AnMBR.

2.2.2 General information about AnMBR technology

AnMBR technology has been extensively used for sludge treatment in recent decades due to its several advantages over conventional anaerobic digesters. AnMBR system simply consists of a membrane and the conventional AD process. In sludge treatment, there has been an increasing interest in low-energy use, and by-product utilization, such as nitrogen and phosphorus. AnMBRs have lower footprint, are resistant to hazardous or inhibitory substrates, have high ability to convert carbonated organic molecules into biogas, and produce high-quality effluent (Abdelrahman et al., 2021). Sludge digesters in municipal WWTPs are typically low-loaded reactors with a typical OLRs of 1-3 kg COD/m^3.d. AnMBR technology provides a better digestion and higher biogas production due to its ability to decouple SRT and HRT compared to conventional anaerobic digesters, where SRT is equal to HRT. Decoupled SRT and HRT allows the slow-growing methanogenic bacteria to stay in the reactor longer, resulting in a higher methane production. Pillay et al. (1994) conducted a 20 years project life estimation, which indicated that a 27% and 12% decrease was observed in capital and total project costs of AnMBR, respectively. AnMBR technology has the potential to achieve energy efficiency in wastewater and sludge treatment processes resulting in an energy-positive and energy-neutral WWTPs. For that purpose, biogas produced can be used as the energy source for digestion and contribute to lower energy requirements in WWTPs. According to Yu et al. (2016), net energy demand can be reduced by 37% in AnMBR technology in comparison to conventional anaerobic digesters. Furthermore, a smaller reactor volume can be used by controlling HRT, resulting in a smaller footprint of the anaerobic digester. Smaller reactor volumes contribute to lower capital costs and heat losses. Thanks to the MF and UF membranes, pathogen- and solids-free permeate can be achieved. Moreover, no nutrient removal occurs in AD process; therefore, the nutrient-rich permeate can be used for irrigational purposes (Abdelrahman et al., 2021).

The performance of AnMBR systems is dependent on several factors. While treatment performance is highly affected by operational conditions such as temperature, SRT, HRT, OLR, adsorbent addition, pretreatment, and co-digestion, filtration performance depends on membrane characteristics, reactor configurations, and operational conditions.

Pretreatment of sludge can be performed before AD process to degrade biomass and make the organic matter more accessible to bacteria, hence accelerating the conversion of organic solids into methane. Before full-scale conventional anaerobic digesters, other disintegration methods, including mechanical, biological, and chemical processes, as well as mixtures of these processes, have been developed and are currently in use (Zhen et al., 2017; Abdelrahman et al., 2021).

Due to its ability for sludge disintegration and the transformation of organic matter into soluble molecules, ultrasonication (US) may improve sludge biodegradability (Abdelrahman et al., 2021). Xu et al. (2011) evaluated the performance of two simultaneous AnMBRs treating WAS with external membrane designs. Both system was operated in similar conditions and the first system was coupled with US equipment. It was observed that combining US and AnMBR processes somewhat improved AD performance, with a 0.6%-3.1% improvement in volatile solids (VS) removal efficiency because of an increase in the organic matter hydrolysis rate.

Sandino et al. (2005) reported that TWAS ultrasound conditioning before AD has been recently applied in a few treatment facilities in Europe and North America as a way to condition WAS for more complete digestion and to improve VS reduction. Increased VS reduction equates to more biogas output and less stabilized biosolids for disposal. Some of these experiences have also indicated an improvement in sludge dewaterability. Many of these installations showed that ultrasound conditioning resulted in a decrease (and, in some cases, complete elimination) in foaming. Carrère et al. (2010) mentioned that sonication before the AD process increased biogas production by 24-140% in batch systems and 10-45% in continuous or semi-continuous systems. However, not all research support increased VS destruction or biogas production. As a result of the sonication of WAS, just a minor increase in both VS destruction and mesophilic methane production was discovered (Sandino et al., 2005; Ariunbaatar et al., 2014).

2.2.3 Configurations

Two main strategies are available for membrane design and operation: Vacuum or pressure. If the membrane is separated from the bioreactor and a pump is needed to pump the sludge to the membrane unit, this configuration is referred as an external cross-flow membrane (Figure 2.1 (a)). For this configuration, the primary method to prevent cake formation on the membrane is the cross-flow velocity of the liquor across the membrane surface. When the membrane is submerged or immersed in the liquid, the configuration is referred as submerged or immersed (Figure 2.1 (b)). The permeate is forced through the membrane either by a pump or by gravity. Cake formation can be prevented by sparging gas across the membrane surface since the velocity of the liquid across the membrane cannot be regulated as easily. Two types of the vacuum-driven immersed membrane method are applied. The membrane can either be submerged in the bioreactor itself or submerged in a different chamber (Figure 2.1 (c)).

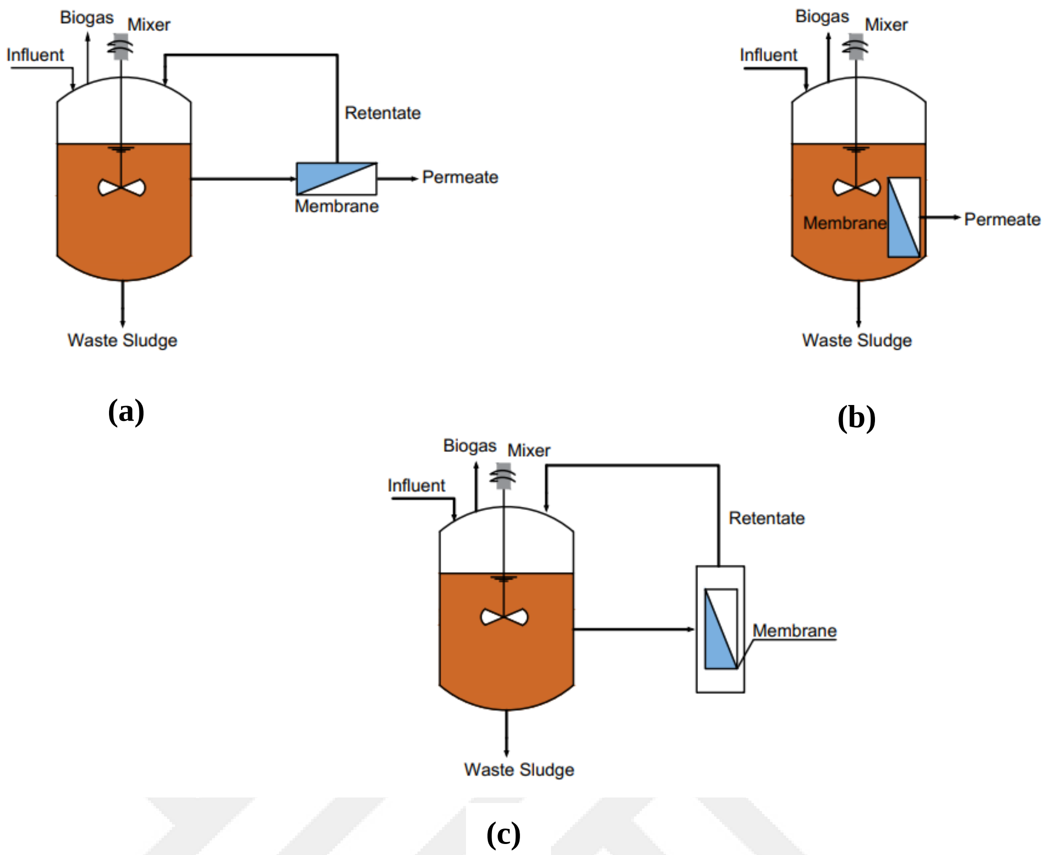


Figure 2.1 : Configurations of AnMBR: (a) External; (b) Submerged; (c) Externally submerged.

2.2.4 Membrane types

According to the literature review, the majority of the studies focusing on AnMBRs currently use MF or UF membranes. MF membranes typically have pores larger than 0.05 µm, whereas UF membranes have pores between 0.002 and 0.05 µm. Both membrane types retain particles, while UF retains more macromolecules and colloids. In order to reduce energy use and increase the flow, the membrane with the greatest pore size should be used (Liao et al., 2006). Xie et al. (2014) have evaluated a flat-sheet dynamic membrane (DM) in AnMBR for landfill leachate treatment. Unlike MF or UF membranes, the properties and performance of DM are primarily governed by the concentration, type, shape, and molecular weight (MW) of the solution being filtered, as well as the hydrodynamic conditions along the membrane.

Membrane materials are classified into three types: Polymeric, ceramic, and metallic. Polymeric membranes are less expensive than ceramic or metallic membranes. Thus, they are used for a wider range of applications, while ceramic or metallic membranes are employed for specific purposes. Polymeric membranes utilized in AnMBRs are commonly made of polyvinylidene difluoride (PVDF), polyethersulfone (PES), polyethylene (PE) (Vyrides and Stuckey, 2009), polypropylene (PP) (Jeong et al., 2010), or polysulfone (PSF) (Stuckey, 2012). However, compared to ceramic or metallic membranes, polymeric membranes have lower permeability and chemical cleaning stability (Dvořák et al., 2016).

2.2.5 Advantages and disadvantages

AnMBR technology provides operational stability, high treatment efficiency, and stable biogas output. In addition to the separation of suspended particulates, AnMBRs can remove bacteria and pathogens from wastewater while consuming little energy (Al-Hashimia et al., 2013). Depending on the required final water quality, AnMBR permeate can be reused for non-potable applications such as irrigation or process waters (Martinez-Sosa et al., 2011) since it contains nutrients such as nitrogen and phosphorus (Chan et al., 2009). AnMBRs provide significant operational advantages. For instance, since no oxygen is required for organic matter biotransformation, total energy consumption is minimized. In addition, the treatment of organic matter yields biogas as a useful end product. Biogas is often burned to generate power and heat. The heat is then employed to maintain proper temperatures in the reactor for AD processes. However, most studies so far have been conducted under mesophilic (35-37 °C) conditions. Therefore, there is a lack of studies conducted under thermophilic conditions, despite its improved filtration thanks to the enhanced sludge rheological properties and its ability to operate at higher OLRs under such conditions (Dvořák et al., 2016). AnMBR plants have much lower operational costs because no oxygen is required, and a large portion of the power and heating necessary to run the plant can be supplied by the biogas produced. The extent to which such costs are reimbursed will be dependent on the amount of biomass produced (Lin et al., 2011a). According to the study of Minami (1994), total AnMBR costs were considerably lower than those for aerobic treatment of Kraft mill effluent. As a result, the membrane and factors associated with membrane fouling account for the majority of both operational and capital costs during AnMBR operation (Lin et al., 2013). Membrane fouling, that reduces flow, is regarded as the primary obstacle to wider applications and faster commercialization of membrane technology in the field of wastewater treatment (Aquino et al., 2006).

While AnMBRs have some advantages over conventional systems, several issues persist. The most important disadvantage of AnMBRs, like aerobic MBRs, is membrane fouling. Membrane fouling reduces the hydraulic performance that limits the use of membranes more widely. Membrane fouling is a complex issue that is influenced by a variety of elements such as operational conditions, influent characteristics, membrane and biomass properties, and their mutual interaction. As a result, a wide spectrum of membrane fouling challenges has been extensively researched.

Membrane fouling occurs primarily due to the deposition and accumulation of microorganisms, solutes, colloids, and cell debris on or within the membrane (Dvořák et al., 2016). Inorganic compound precipitation, primarily struvite ($MgNH_4PO_4$; magnesium ammonium phosphate), has also been found as a significant component of irreversible fouling on membranes in AnMBRs (Choo and Lee, 1996). Other inorganic salts found in the fouling layer include potassium ammonium phosphate ($K_2NH_4PO_4$) and calcium carbonate ($CaCO_3$) (Meabe et al., 2013). Membrane characteristics and operating conditions can both influence the rate, at which inorganic chemicals precipitate. Meabe et al. (2013) revealed that struvite fouling increased at higher operating temperatures (55 °C vs. 35 °C) due to greater ammonia nitrogen concentrations.

The cost of the membrane is a significant barrier to the widespread use of AnMBR. Although membrane costs have decreased significantly in recent years, operational costs related to the filtration process continue to be a major drawback for membrane bioreactors (MBRs) in general. Based on the study of Pretel et al. (2014), up to 85-90% of AnMBR power needs were connected to the filtration process and membrane fouling control. More than 70% of the energy used by AnMBRs for overall operation is used for fouling control (Shin and Bae, 2018). Martin et al. (2011) conducted a study with both aerobic and anaerobic MBRs and found that the total specific energy consumption by aerobic MBR with full sludge retention was around 2 kWh/m^3, whereas the energy demand for AnMBR ranged from 0.03 to 5.7 kWh/m^3. Increased gas consumption for severe membrane fouling management resulted in the highest energy requirement.

2.2.6 Fouling control and cleaning methods

Membrane fouling is still a major impediment to the widespread use of AnMBR in wastewater treatment. Membrane fouling may limit system productivity, necessitate frequent cleaning, resulting in a shorter membrane lifespan and higher replacement costs, and increase the energy required for sludge recirculation or gas scouring (Lin et al., 2013).

Membrane fouling is further classified into two forms based on cleaning practice: Reversible fouling and irreversible fouling (Maaz et al., 2019). Cake formation, defined as a porous layer rejected on the membrane surface, correlates to reversible fouling. Physical techniques, such as relaxation or backwashing, are commonly used to eliminate reversible fouling (Calderón et al., 2011). Irreversible fouling cannot be reduced by physical cleaning procedures.

Long-term experiments are connected with irrecoverable fouling; when a membrane is fouled, the initial membrane permeability is never regained. This lingering resistance is known as "irrecoverable fouling," because it cannot be eliminated using standard chemical cleaning methods. Biofouling occurs when microorganisms accumulate and grow on membrane surfaces. One of the key factors in the biofouling process is the colonization of membrane surfaces with microorganisms. The deposition of EPS and soluble microbial products (SMP) on membrane and pore surfaces also contributes to biofouling. The deposition of macromolecular species (biopolymers) and organic components on membrane surfaces causes organic fouling (Huang et al., 2011).

Membrane fouling is unavoidable, however fouled membranes can be regenerated via physical, chemical, and biological methods. Membrane relaxing and membrane backwashing are the most common physical cleaning strategies for MBRs. US, a unique on-line physical cleaning approach, has been developed and intensively researched in MBRs, particularly in AnMBRs (Wen et al., 2008; Xu et al., 2011). Wen et al. (2008) proved that US can regulate cake formation on the membrane surface. The US mechanism for membrane fouling management was thought to be cavitation and acoustic streaming caused by ultrasonic waves, limiting cake formation and increasing membrane filtration rates. Meanwhile, ultrasonic irradiation has been shown to reduce anaerobic bacterial activity and cause membrane damage (Wen et al., 2008).

When physical cleaning approaches are ineffective in reducing fouling to an appropriate level, chemical cleaning of the membranes is required. Many chemical cleaning agents have been used for membrane cleaning in AnMBRs, including hydrochloric acid (HCl), citric acid, sodium hydroxide (NaOH), sodium hypochlorite (NaClO), nitric acid, and ethylenediaminetetraacetic acid (EDTA) (Zhang et al., 2007; Lin et al., 2011a; Mahendran et al., 2011). Proper chemical cleaning necessitates the use of cleaning chemicals that target the major components responsible for fouling while causing minimal harm to the membrane itself. To eliminate bacteria and organic foulants, oxidizing and alkaline chemicals such as NaClO and NaOH are commonly utilized (Lin et al., 2013). Metal-associated structures, such as metal organic foulant complexation and inorganic scales, are effectively broken down by acidic agents. Because of their exceptional binding affinity with metal ions, coordination agents such as EDTA and citric acid can also eliminate metallic foulants. A combination of cleaning agents, such as NaClO and NaOH, is clearly more efficient than single-agent techniques. A weekly clean-in-place (CIP) with 500 mg/L NaClO and 2000 mg/L citric acid, followed by a clean-out-of-place (COP) with 1000 mg/L NaClO and 2000 mg/L citric acid twice a year, is the common cleaning strategy employed in AnMBRs (Lin et al., 2011a).

2.2.7 Applications of AnMBR

A thorough examination reveals that, particularly in the last six years, researchers have devoted an increasing amount of attention and effort to AnMBR study. This scenario can be linked to two wastewater treatment trends. On one hand, the industrial sectors have been subjected to strict criteria for boosting water usage efficiency and closing industrial process water cycles, and this trend is expected to continue in the future. Meanwhile, extreme wastewater conditions are anticipated to grow increasingly widespread in the next years and beyond. On the other hand, although conventional technology costs are progressively growing due to personnel expenses and inflationary pressures, the costs of all membrane equipment have been continuously reducing over the previous decade (Lin et al., 2013). Furthermore, the benefits of biogas recovery associated with AnMBR treatment can greatly offset the operational costs. On a capital and operating cost basis, the possibility of AnMBR becoming a preferred alternative for any particular project grows over time.

AnMBR technology has been used for treating a broad range of wastewater types. Synthetic wastewaters are commonly used to test novel concepts like AnMBR. VFA, glucose, starch, cellulose, and yeast were among the substrates used. Industrial wastewaters have been recently treated by AnMBRs more than in previous years. Industrial wastewaters have industry specific features. Nevertheless, they generally have the potential for high organic strength and contain synthetic compounds that may be slowly degradable or non-biodegradable anaerobically, and/or hazardous (Liao et al., 2006). Toxicity to microorganisms is a major concern in the biological treatment of such wastewaters. However, hazardous chemicals in wastewater can still be anaerobically destroyed if proper safeguards are taken (Speece, 1983). Treatment of pulp and paper industry effluent by AnMBR has been documented several times in the literature (Liao et al., 2010; Lin et al., 2011b). Evaporator condensate (EC), one of the most important wastewaters produced by the pulp and paper industry, is distinguished by its high temperature, high organic strength (primarily due to methanol), low suspended solids (SS) (lower than 3 mg/L), and inhibitive materials such as turpene oils (Minami, 1994). A submerged anaerobic membrane bioreactor (SAnMBR) was operated for treating kraft EC at 37 ± 1 °C for 9 months. COD removal efficiency of 93-99% was reached for OLRs of 1-24 kg COD/m^3.d. Due to the fact that wastewater from the pulp and paper industry is typically high in temperature, operation at thermophilic conditions is of significant interest. Therefore, pre-cooling and post-heating employed in mesophilic treatment for subsequent reuse of treated effluent might be eliminated (Xie et al., 2010). Lin et al. (2009) evaluated two parallel SAnMBRs handling kraft EC that were run at mesophilic (37 °C) and thermophilic (55 °C) conditions, and found that a COD removal efficiency of 97-99% with the methane production rate of 0.35 ± 0.05 L methane (CH_4)/g COD removed was reached at a feed COD concentration of 10,000 mg/L at each SAnMBR. The results showed that both mesophilic and thermophilic SAnMBRs could be potentially beneficial technologies for kraft EC treatment in terms of COD removal and biogas production. However, thermophilic SAnMBRs faced severe membrane fouling due to higher temperature. More protein to polysaccharide ratio (PN/PS), SMP release, and greater portion of fine flocs (<15 µm) were observed on the membrane surface at thermophilic conditions, which resulted in more fouling of the membrane than at mesophilic conditions (Lin et al., 2009). Several lab-scale studies have been conducted to investigate the treatability of industrial wastewaters by AnMBRs. Abdurahman et al. (2011) operated a lab-scale membrane anaerobic system (MAS) for treating palm oil mill effluent at different HRTs ranging from 6.8 days to 600.4 days and achieved high fluxes up to 140 L/m^2.h. Furthermore, COD removal efficiencies from 96.6% to 98.4%, and methane yield from 0.25 to 0.57 L CH_4/g COD/d were achieved.

SRT, OLR, temperature, shear rate, and other parameters are expected to influence both treatability and filterability. Substrate composition and operational parameters have an indirect effect on fouling by altering sludge properties and membrane material. Shear rate has a direct effect on fouling by reducing membrane fouling caused by scouring the membrane surface, as well as an indirect effect on fouling by disturbing the bio-flocs and generating fine particles (Dereli, 2015).

The combination of membrane separation technology and an anaerobic bioreactor may enable sustainable municipal wastewater treatment with complete biomass retention, less sludge production, improved high quality effluent, net energy production, and without the additional costs for aeration associated with aerobic treatment processes. In the recent years, AnMBR technology has grown in popularity for municipal wastewater treatment (An et al., 2009). AnMBR technology has also been applied to low-strength municipal wastewaters. In an anaerobic bioreactor coupled with an external MF membrane for municipal wastewater treatment, at permeate flux of 80-450 L/m^2.h, Kocadagistan and Topcu (2007) found that COD, phosphorus, and SS removal efficiencies were 98%, 81%, and 99%, respectively.

2.2.8 Factors affecting AnMBR performance

2.2.8.1 Sludge characteristics

Characteristics of sludge to be treated in AnMBR directly influences the digestibility and membrane fouling in the system. The essential parameters regulating sludge cake formation and membrane fouling in AnMBR systems include sludge properties such as floc size, SMP, and bound EPS. The constant decrease of permeation flux caused by membrane fouling is still a significant barrier to the widespread deployment of MBRs. Hydrodynamic conditions, membrane materials and module design, as well as sludge characteristics (EPS, particle size, surface charge, hydrophobicity, etc.) have all been found as variables influencing membrane fouling (Lin et al., 2009; Meng et al., 2009). It has been found that biomass content and particle size distribution (PSD) are important factors governing membrane permeability (Bai and Leow, 2002). It was also reported that the deposition of inorganic foulants such as struvite ($MgNH_4PO_4$) along with the microbial cells adhering to the membrane surface played an important part in the creation of the tightly adherent cake layer that limited membrane permeability (Kim et al., 2007). Recent research has broadened the scope of foulant analysis. According to Pollice et al. (2005), fouling in subcritical flux operation was mostly caused by the accumulation of SMP and EPS in the pores and/or on the membrane surface. Tsuneda et al. (2003) found that increasing the EPS content improves sludge adherence through polymeric interactions. According to Lee et al. (2003), hydrophobicity and surface charge, that are related to the composition and characteristics of EPS, appeared to be critical parameters in microbial floc fouling. Furthermore, biopolymer cluster (BPC) was recently discovered to be an important foulant with a significant effect on membrane fouling (Wang and Li, 2008).

2.2.8.2 Membrane material

The long-term attainable flow was enhanced by decreasing the hydrophobicity of polypropylene membranes via graft polymerization with hydroxyethyl methacrylate (Sainbayar et al., 2001).

Negatively charged membranes showed a larger flow than noncharged and positively charged membranes due to the negative charge of sludge flocs. Ghyoot and Verstraete (1997) investigated the treatment of sewage sludge by AnMBR initially with a poly-ether sulfone microfiltration membrane with a membrane area of 0.3 m^2 and obtained a flux of 19 $L/m^2.h$ unlike a ceramic UF membrane with a membrane area of 0.05 m^2, by which 200-250 $L/m^2.h$ was reached in the same study.

Different membrane materials result in various fouling mechanisms. Inorganic membranes, for instance, were discovered to be fouled largely by $MgNH_4PO_4$, but organic membranes were fouled by both biomass and struvite (Liao et al., 2006).

2.2.8.3 Module type and configuration

The majority of membrane modules used in AnMBRs are made of MF or UF membranes in hollow fiber, flat sheet (plate or frame), or tubular configurations.

Hollow fiber membrane modules are the most commonly utilized in submerged membrane bioreactors (SMBRs) because of their high packing density and low cost. Nevertheless, flat sheet membrane modules remained popular, particularly within the research community, due to their advantages of good stability and simplicity of cleaning and replacing damaged membranes (Kim et al., 2007; Kocadagistan and Topcu, 2007; Lin et al., 2009). A tubular membrane module is made up of numerous tubes of tubular membranes. Low fouling, relatively easy cleaning, easy handling of suspended materials and viscous fluids, and the ability to repair or plug a damaged membrane are the key advantages, while the negatives include high capital cost, low packing density, high pumping costs, and large dead volume (Zhang et al., 2007; Herrera-Robledo et al., 2010; Torres et al., 2011). The majority of membranes used have pore sizes ranging from 0.03 to 1.0 μm, which is plainly smaller than the size of the majority of flocs or microorganisms in AnMBR, and so may almost entirely retain biomass (Lin et al., 2013).

2.2.8.4 Operational conditions

2.2.8.4.1 SRT

Due to the decoupling of SRT and HRT, AnMBR technology provides an advantage over conventional anaerobic sludge digesters. In comparison to conventional digesters, AnMBR technology thus achieves comparable or even superior digestion performances with much smaller (50%) reactor capacity (Dagnew et al., 2010). Increased retention of active biomass and particulates in the bioreactor is made possible by increase in SRT independent of HRT, which improves the hydrolysis of particulate organic matter.

Huang et al. (2011) conducted a study with SAnMBR treating low-strength synthetic wastewater to investigate the effect of SRT and HRT on treatment performance, biogas production, and membrane fouling. SRTs of 30 (R30), 60 (R60), and infinite (R∞) d at 12, 10, and 8 h of HRTs were set up in the study. It was concluded that a longer SRT achieved a better treatment performance for each HRT value and higher biomass concentration and biogas production were obtained at shorter HRT. Although COD removal efficiencies were similar (>97%) at all scenarios, a better methane yield rate of 1.290 ± 0.267 L CH_4/d was observed at R∞ followed by R60 (0.906 ± 0.357 L CH_4/d) and R30 (0.670 ± 0.203 L CH_4/d) at HRT of 12 h. Similarly, methane yield in terms of COD removal at all HRTs was also higher for R∞, for example (0.205 ± 0.049 L CH_4/g COD) compared to R60 (0.171 ± 0.039 L CH_4/g COD), and R30 (0.138 ± 0.031 L CH_4/g COD) at HRT of 12 h. Because of the more active metabolism of microorganisms at short SRT (30 d), more organic compounds were digested and less SMP was released, which reduced particle deposition, biofilm formation, and membrane fouling. Filtration performance is also affected by SRT. However, there is still more research to be conducted on the impact of high SRTs, particularly on membrane filtration performance. High SRTs could promote cell lysis, which would increase the release of soluble microbial metabolites and inert decay products, namely SMP. On the other hand, high SRTs result in high sludge concentrations, which causes a quick buildup of the cake layer, which serves as a barrier for blocking membrane pores. The drawback is that cake formation has become more prominent, which has caused flux to decline.

2.2.8.4.2 HRT

The capital cost of a reactor is strongly influenced by HRT. A greater OLR enables a shorter HRT and a smaller reactor for a given influent composition. For instance, Liao et al. (2006) mentioned that the use of a membrane could enhance the OLR of a totally mixed reactor from 4 to 12 kg COD/m^3.d. HRTs utilized with AnMBRs have typically been greater than those used with non-membrane high-rate anaerobic reactors (HRARs). For the treatment of soybean-processing wastewater and sewage, AnMBR with HRTs as low as 10 h have been applied, while HRARs normally have HRTs of 4–8 h. The projected decline in HRTs has not yet been brought on by the entire solid retention capabilities of AnMBRs (Liao et al., 2006).

2.2.8.4.3 OLR

AnMBRs for sewage sludge treatment were used at a variety of OLRs (Abdelrahman et al., 2021). However, the reported OLRs were much lower than the OLRs reported by research on AnMBRs treating industrial wastewater (Dereli et al., 2012), indicating that there may still be opportunity for process performance optimization (Abdelrahman et al., 2021). Qiao et al. (2013b) conducted a study co-digesting WAS and coffee ground with the OLRs of 2.2 – 33.7 kg COD/m^3.d and observed that biogas production rate increased from 0.53 m^3/m^3.d to 5.8 m^3/m^3.d, respectively. Correspondingly, COD removal efficiency also increased from 46.5% to 66.8%, respectively. It was seen that the system experienced no inhibition despite a 5-fold increase in OLR.

2.2.8.4.4 Temperature

Temperature considerably affects the biochemical reaction rates and efficiency of degradation of organic matter. Hydrolysis, at this point, is a rate limiting step in sludge digestion. In the digestion of sewage sludge, the hydrolysis of particulate matter is the rate-limiting stage. An increase in operating temperature accelerates the hydrolysis process and makes organic molecules more soluble. Thermophilic (55 °C) sludge digestion has some advantages over mesophilic (35 °C), including faster reaction rates, more organic load capacity, and greater pathogen destruction. However, as VFA accumulates as a result of the higher rate of acid generation, the process is more susceptible to instability and inhibition (Kim et al., 2002).

Meabe et al. (2013) examined the performance of an AnMBR treating sewage sludge under mesophilic and thermophilic conditions at 50 d of SRT and 7 d of HRT. Around 72% of the COD was converted to biogas, and digestion efficiency was equal under each circumstance. However, under thermophilic conditions, permeate quality declined as a result of the increased solubilization rate, which increased the concentration of soluble chemical oxygen demand (sCOD), VFA, and ammonia passing through the membrane.

2.2.8.4.5 Flux

Flux decline appears to be the most essential limitation for the applicability and viability of AnMBRs for sludge digestion, since it plays a significant role in determining the needed membrane area. Flux decrease is caused by a variety of reasons, such as membrane material, shear rate, operational conditions, and operation mode. As a result, studying membrane fouling behavior and processes necessitates an understanding of a variety of parameters such as membrane features, operational conditions, and sludge qualities. Due to the complexity of membrane foulants and the variability of operational conditions, membrane materials, configurations, and wastewaters in different investigations, membrane fouling in AnMBRs has not been thoroughly understood. Not only in full-scale plants, but also in controlled lab-systems, complex interactions appear between physical and biological variables (Ozgun et al., 2013). Hence, these parameters must be well evaluated in further studies.

Operational flux must be chosen carefully for proper management of fouling. Operating the system below critical flux has been proven to be a crucial method to avoid membrane fouling (Ozgun et al., 2013). Martinez-Sosa et al. (2011) demonstrated a stable operation with AnMBR treating municipal wastewater at a critical flux of 7 $L/m^2.h$ with a gas sparging velocity of 62 m/h. The study also proved that increasing the flux to 10 $L/m^2.h$ or 12 $L/m^2.h$ caused membrane fouling, thus an unstable operation, which could not be compensated even with high rate of gas sparging.

2.2.8.4.6 Operation mode

Another factor affecting the AnMBR performance is the operation mode, which is a crucial aspect in achieving long-term and steady operation in AnMBRs. Together with the gas sparging and occasional chemical cleaning, frequent backwashing and/or relaxing can be employed to control membrane fouling. Continuous filtration and a filtration of 10 min and relaxation of 2 min cycle was applied by Yu et al. (2016) in anaerobic dynamic membrane bioreactor (AnDMBR) operation. When filtration/relaxation was used instead of continuous filtration, the length of operation without physical cleaning was nearly four times longer. Transmembrane pressure (TMP) was monitored by Dagnew et al. (2012) for WAS filtration at a flow of 30 $L/m^2.d$ in both continuous and intermittent filtration modes. The intermittent mode produced a lower TMP nearly 0.4 bar than the continuous mode, which produced an increasing TMP between 0.3-0.8 bar. In a study conducted by Chu et al. (2005), optimal operating modes were investigated and it was discovered that increasing the relaxation time increased permeability and improved permeate flux recovery. As a result of the relaxation, the cake layer was efficiently removed from the membrane surface.

2.3 AnMBR Application in Sludge Treatment

In recent years, AnMBRs have grown in popularity, and greater emphasis has been placed on the advancement of this technology for the treatment of high-concentration wastewater. However, an increasing attention has been paid on sewage sludge treatment nowadays. The treatment of sludge by AnMBR technology provides several benefits, including biogas recovery, nutrient recovery (phosphorus, ammonia etc.), high quality permeate, and stable operation under high OLRs. The methane content of the biogas recovered can be used for the energy requirement of the WWTPs, thus decreasing the energy costs of the plants. Verstraete and Vandevivere (1999) stated that the treatment of high-solids waste streams is typically carried out in completely stirred tank reactors (CSTRs) with low OLR of roughly between 1-3 g COD/L.d. Decoupling of HRT and SRT is of particular relevance in those systems, since a long SRT is crucial to deal with the poorer growth rate of anaerobic biomass and to efficiently remove VS. AnMBRs concentrate biomass inside the digester and physically keep particulate organic matter until it is susceptible to degrading, eliminating the volumetric load constraint seen in conventional digesters due to the poor hydrolysis rate. Furthermore, by running the system with a long SRT, the production of high-quality effluent is promoted, as is the generation of more stabilized and concentrated digested sludge. Finally, the biogas offsets the high energy requirements of cross-flow filtration that is a major disadvantage of membrane filtration-based systems (Meabe et al., 2013).

Table 2.1 : Treatment performance of AnMBRs for sludge treatment.

Reactor type/Membrane Configuration	Temperature (ºC)	Substrate	Influent Total Solids (TS) concentration (g/L)	HRT (d)	OLR (kg COD/m^3. d)	COD Removal (%)	Biogas Production rate (m^3/m^3. d)	CH_4 Content (%)	Reference
CSTR[a]/Submerged	37	WAS	11.6	15	0.66	95.7	NA[b]	43.4	Li et al. (2023)
CSTR[a]/Externally submerged	75	75% Synthetic FW + 25% sewage sludge	49.1	NA[b]	5.44[c]	NA[b]	NA[b]	89	Li et al. 2021b)
CSTR[a]/Externally submerged	75	Synthetic Food waste (FW)	50	NA[b]	5.44[c]	NA[b]	NA[b]	98	Li et al. (2021b)
Two-phased CSTR[a] /Submerged	37	PS	24.2-34	2, 16	1.86	52.4	NA[b]	71.2	Martin-Ryals et al. (2020)
CSTR[a]/-	35	WAS	10.8	20	0.55	98.3	0.094	68.3[d]	Niu et al. (2020)
CSTR[a]/Submerged	35	WAS	6	17	1.1-1.2[e]	NA[b]	0.15-0.18	60-70	Zhao et al. (2019)
CSTR[a]/Submerged	37	Thermally pretreated sewage sludge	4.5-4.9	5-20	1.39-5.72	79-96.3	0.43-1.3	70-78	Wandera et al. (2019)
CSTR[a]/External	35	WAS	5-10	5-6	0.15-0.55[e]	23-56	0.02-0.05	NA[b]	Hafuka et al. (2019)
CSTR[a]/External + coagulant + flocculant aid	35	WAS	48.2	18	NA[b]	NA[b]	NA[b]	NA[b]	Kooijman et al. (2017)
CSTR[a]/Externally submerged	35	WAS	NA[b]	5	NA[b]	NA[b]	0.15	72	Yu et al. (2016)
CSTR[a]/External	35	MBR excess sludge	15	34, 67	1.3-2.2[f]	98	0.1-1.3	54.5-68.5	Hafuka et al. (2016)
CSTR[a]/Submerged	35	WAS + pretreatment with 60 min US	7.06	3	NA[b]	63	NA[b]	NA[b]	Joshi and Parker (2015)

Table 2.1 (continued): Treatment performance of AnMBRs for sludge treatment.

Reactor Type/Membrane Configuration	Temperature (°C)	Substrate	Influent TS (g/L)	HRT (d)	OLR (kg COD/m³. d)	COD Removal (%)	Biogas Production Rate (m³/m³. d)	CH_4 Content (%)	Reference
CSTR[a]/Submerged	35	WAS + pretreatment with 20 min US	7.06	3	NA[b]	58	NA[b]	NA[b]	Joshi and Parker (2015)
CSTR[a]/Submerged	57	WAS + coffee grounds	100-150	7-70	2.2-33.7	46.5-66.8	0.53-5.8	51.5-6.1	Qiao et al. (2013b)
CSTR[a]/External	55	Sewage sludge	32.7	3-7	4.8-6.4	94	1.76	67.5	Meabe et al. (2013)
CSTR[a]/External	35	Sewage sludge	32.7	5-7	4.8	99	1.63	67.5	Meabe et al. (2013)
CSTR[a] /External	35	WAS	6.2-18.8	9	1.5-3.7[e]	NA[b]	NA[b]	NA[b]	Xu et al. (2011)
CSTR[a]/External	35	WAS + Thickened WAS	19.4	15	1.34	NA[b]	NA[b]	NA[b]	Dagnew et al. (2010)
CSTR[a]/External	NA[b]	Sewage sludge	NA[b]	7.8 – 943.4	0.1-10	96.5-98.8	NA	66.3-76.3	Liew Abdullah et al. (2005)
CSTR[a]/External + Alkaline heat post-treatment	35	WAS	20.7	30	NA[b]	NA[b]	NA[b]	71	Takashima et al. (1996)
CSTR[a]/External	35	WAS	20.7	30	NA[b]	NA[b]	NA[b]	57	Takashima et al. (1996)
CSTR[a]/External	NA[b]	Sewage sludge	NA[b]	14	NA[b]	NA[b]	NA[b]	NA[b]	Pillay et al. (1994)

[a]: Completely stirred reactor

[b]: Not available

[c]: At the 0[th] day of the operation only.

[d]: When the substrate was hydrolyzed at 125 °C.

[e]: kg VS/m³.d

[f]: kg COD/m³ (the system was fed twice a week)

Table 2.2 : Filtration performance of AnMBRs for sludge treatment.

Operation Mode/Module Configuration	Membrane configuration	Material	Membrane type	Pore Size (μm)	Cross-flow Velocity (m/s)	Operation Duration (d)	Filtration Area (L/m^2.h)	Flux (L/m^2.h)	TMP (bar)	Cleaning Type	Reference
CSTR[a]/Submerged	Flat sheet	PVDF	MF	0.4	NA[b]	NA[b]	NA[b]	8.6	0.033	NA[b]	Li et al. (2023)
CSTR[a]/Externally submerged	Hollow fiber	Polytetrafluoroethylene	MF	0.1	NA[b]	NA[b]	NA[b]	5	NA[b]	NA[b]	Li et al. (2021a)
Two-phased CSTR[a]/Submerged	Filter cartridges	NA[b]	NA[b]	10	NA[b]	130	0.22	NA[b]	NA[b]	NA[b]	Martin-Ryals et al. (2020)
CSTR[a]/Externally submerged	Flat sheet	PVDF	MF	0.4	NA[b]	115	NA[b]	10.8	0.047	Physical cleaning and chemical cleaning	Niu et al. (2020)
CSTR[a]/Submerged	Flat sheet	Cellulose triacetate	FO	NA[b]	NA[b]	105	0.005	0.3-1	NA[b]	Physical cleaning and chemical cleaning	Zhao et al. (2019)
CSTR[a]/Submerged	Flat sheet	NA[b]	MF	0.22	NA[b]	170	0.116	3.6-10.5	0.04-0.11	Water flushing and chemical cleaning	Wandera et al. (2019)
CSTR[a]/Submerged	Flat sheet	NA[b]	MF	0.2	NA[b]	155	0.116	4.4	0.06-0.18	NA[b]	Wandera et al. (2018)
CSTR[a]/External	Tubular	PVDF negatively charged	NA[b]	40	NA[b]	>730	0.4	NA[b]	NA[b]	Water flushing, abrasion and chemical cleaning	Pileggi and Parker (2017)
CSTR[a]/Externally submerged	Flat sheet	Dacron mesh	DM	39	NA[b]	200	0.38-0.46	15	0-30	Physical cleaning	Yu et al. 2016)
Two-phased CSTR[a]/External	Mesh screen	Naylon	NA[b]	100	1.47-3.92	190	NA[b]	10-15	1.47-3.92	Tap water rinsing and chemical cleaning	Joo et al. (2016)
CSTR[a]/Submerged	Flat sheet	Chlorinated polyethylene	MF	0.2	NA[b]	155	0.116	2-7.6	NA[b]	Chemical cleaning	Qiao et al. (2013b)
CSTR[a]/External + US	Hollow fiber	Polythene	NA[b]	0.4	1	54	0.012	3.5	NA[b]	NA[b]	Xu et al. (2013)

Table 2.2 (continued): Filtration performance of AnMBRs for sludge treatment.

Operation Mode/Module Configuration	Membrane configuration	Material	Membrane type	Pore Size (µm)	Cross-flow Velocity (m/s)	Operation Duration (d)	Filtration Area ($L/m^2.h$)	Flux ($L/m^2.h$)	TMP (bar)	Cleaning Type	Reference
CSTR[a]/External	Tubular	Neutral surface charged	UF	0.04	1	160	0.2	32.3	0.29	NA[b]	Dagnew et al. (2013)
CSTR[a]/External	Hollow fiber	Polythene	NA[b]	0.4	1	390	0.012	1.3-7	NA[b]	Water rinsing	Xu et al. (2011)
CSTR[a]/External	Hollow fiber	Polythene	NA[b]	0.4	1	77	0.05	NA[b]	NA[b]	Chemical cleaning	Xu et al. (2010)
CSTR[a]/External	Tubular	NA[b]	UF	120	1.2	180	0.2	40	28.2	Mechanical washing and chemical cleaning	Dagnew et al. (2010)
CSTR[a]/External	Vibrating configuration	Polymeric teflon	UF	0.05	3.45	56	1.6	66.7-83.3	3.45	Chemical cleaning (every 30 days)	Pierkiel and Lanting (2005)
CSTR[a]/External	Tubular	Titanium dioxide	UF	0.1	5	7	1.4	145.8	4.8-5.5	Chemical cleaning (every day)	Pierkiel and Lanting (2005)
Upflow anaerobic bioreactor/External	Tubular	Ceramic	MF	0.1	4.5	40	0.05	120-275	2	Tap water rinsing	Ghyoot and Verstraete (1997)
CSTR[a]/External	NA[b]	NA[b]	UF	30	NA[b]	124	0.0177	1-13	NA[b]	NA[b]	Takashima et al. (1996)
CSTR[a]/Submerged	Tubular	Ceramic	NA[b]	0.1	0.2-0.3	35-40	1.06	2.5-8.3	0.27	Nitrogen gas backwashing	Kayawake et al. (1991)

[a]: Completely stirred reactor

[b]: Not available

3. MATERIAL AND METHODS

3.1 Seed Sludge Characterization

The seed sludge was obtained from a full-scale advanced biological WWTP. The VS to total solids (TS) ratio was 41.3% and the average TS concentration was 49,795 mg/L. The seed sludge characteristics are given in Table 3.1.

Table 3.1 : Seed sludge characteristics.

Parameter	Unit	Value (Average ± Standard Deviation (SD))
TS	mg/L	49,795 ± 262
VS	mg/L	20,563 ± 244
Total Suspended Solids (TSS)	mg/L	48,600 ± 566
Volatile suspended solids (VSS)	mg/L	20,417 ± 212
COD	mg/L	41,268 ± 172
sCOD	mg/L	1,360 ± 11
Alkalinity	mg $CaCO_3$/L	8,188 ± 18
Ammonium nitrogen (NH_4-N)	mg/L	568 ± 11
Capillary suction time (CST)	sec	50.8 ± 1.0
Median particle size (D50)	µm	10.4 ± 0.6
VFA	mgCOD/L	3,942 ± 73
Specific methanogenic activity (SMA)	g CH_4-COD/g VS.d	0.12 ± 0.007

3.2 Substrate Characteristics

PS and A-sludge were used as substrates. The PS was obtained from a primary clarifier in a full-scale sewage treatment plant. A-sludge used in this study was obtained from waste sludge of a pilot-scale HRAS system (Figure 3.1). Dissolved oxygen (DO) concentration, HRT, and SRT of the pilot scale HRAS system were 0.5 mg/L, 75 d, and 0.5 d, respectively. Coarse particles were removed from the sludge by sieving it through a 2 mm mesh screen and the sieved sludge was stored at 4 °C. The characteristics of substrates are given in Table 3.2.

Figure 3.1 : HRAS system.

Table 3.2 : Characteristics of PS and A-sludge.

Parameter	Unit	Value (Average ± SD)	
		PS	A-sludge
TSS	mg/L	11,035 ± 714	9,555 ± 417
VSS	mg/L	5,198 ± 311	5,643 ± 231
VSS/TSS	%	47.1 ± 1.4	59.1 ± 0.8
TS	mg/L	12,553 ± 667	13,477 ± 405
VS	mg/L	5,853 ± 372	6,616 ± 184
COD	mg/L	10,100 ± 289	10,524 ± 380
sCOD	mg/L	1,953 ± 86	2,229 ± 66
Total nitrogen (TN)	mg/L	329 ± 11	609 ± 21
NH_4-N	mg/L	71 ± 4	216 ± 11
Total phosphorus (TP)	mg/L	53 ± 3	115 ± 5
Dissolved phosphorus (DP)	mg/L	1.66 ± 0.04	22.18 ± 1.73
pH	-	6.5 ± 0.2	6.7 ± 0.2
Conductivity	ms/cm	1.9 ± 0.1	6.5 ± 0.1
Fecal coliform	MPN*/g TS	$2x10^5 \pm 0.7x10^5$	$3.5x10^5 \pm 0.34x10^5$
Total coliform	MPN*/g TS	$3.7x10^5 \pm 0.35x10^5$	$6.5x10^5 \pm 0.32x10^5$
CST	sec	54 ± 3	117 ± 7
D50	µm	33 ± 1	206 ± 5

*MPN: Most probable number.

3.3 Experimental Setup

The schematic diagram of the lab-scale AnMBR system is shown in Figure 3.2 (a). AnMBR system consisted of a 7 L working volume cylindrical glass reactor with an external membrane configuration (Figure 3.2 (b)). The substrate was kept at 4 °C. A peristaltic pump (Watson Marlow 300 series, UK) was used to feed the substrate into the anaerobic digester, which was equipped with a water jacket for temperature control. Several sensors were used in the anaerobic digester, including pH, temperature, oxidation-reduction potential (ORP) and level sensors, and a gas meter to monitor the operational conditions inside the digester. Pressure transmitters, that are used to measure TMP, were fixed on the inlet, outlet, and permeate lines (Jumo Midas, Germany). Sludge circulation inside the membrane module to achieve a specific cross-flow velocity was obtained with a Mono^{TM} progressing cavity pump (Seepex BN 5-6L, Germany). Permeate from the membrane was obtained by using a vacuum pump.

To control the pumps and to record the data obtained from the sensors, a programmable logic controller (PLC) was used (Figure 3.2 (c)). SCADA software was used to connect the computer to the PLC.

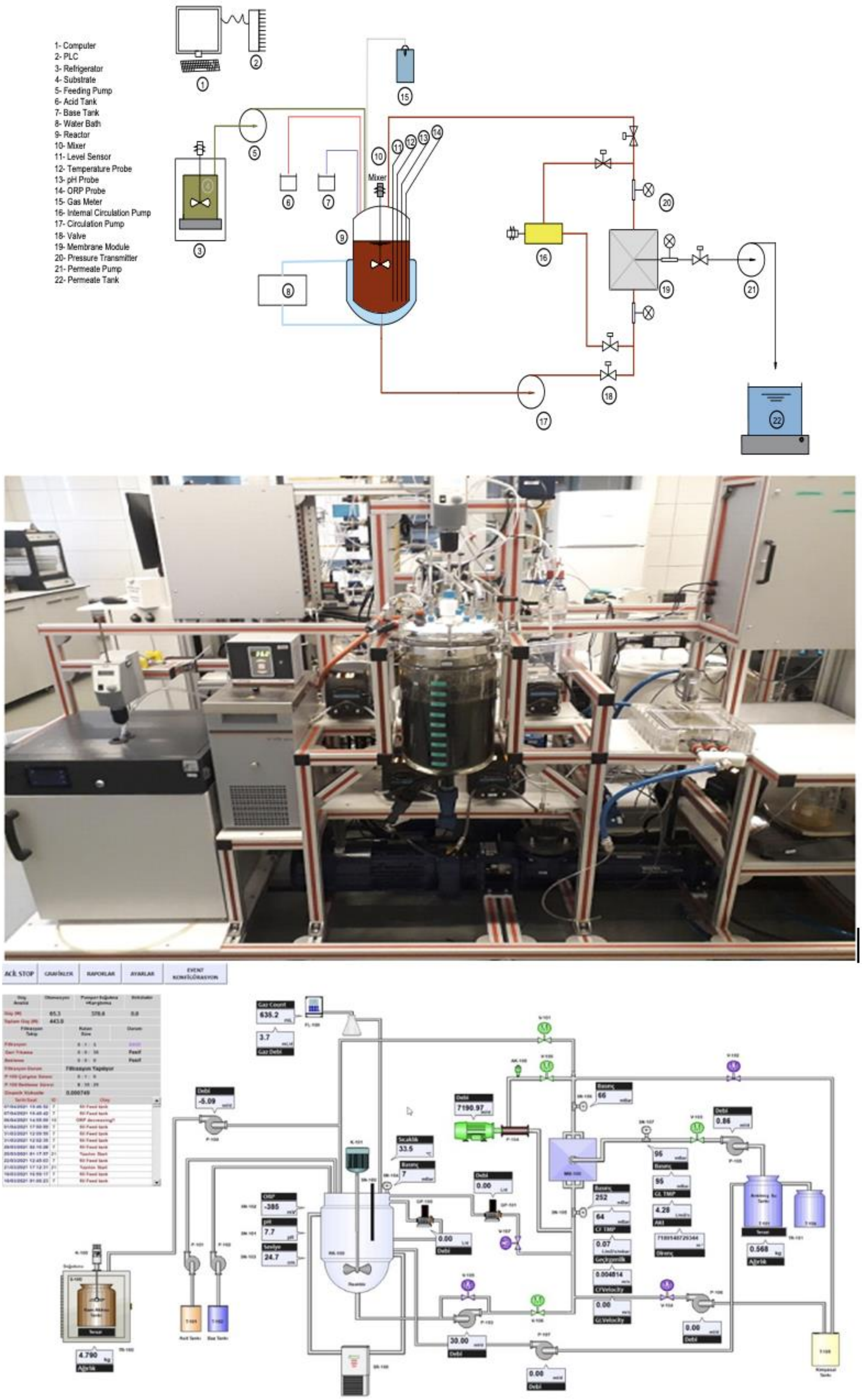


Figure 3.2 : Lab-scale AnMBR system: (a) Schematic diagram; (b) AnMBR setup; (c) SCADA software.

3.4 Membrane Characterization

In this study, a commercial UF membrane, made of PVDF, was used, which had a pore size of 0.02 μm and a membrane filtration area of 0.012 m^2. Characteristics of the membrane are shown in Table 3.3. The membrane module used for the AnMBR is given in Figure 3.3.

Table 3.3 : Characteristics of the membrane.

Specifications	Unit	Value (Average ± SD)
Material	-	PVDF
Pore Size	μm	0.02
Module Type	-	Flat sheet
Permeability	$L/m^2.h.bar$	0.05 ± 0.01

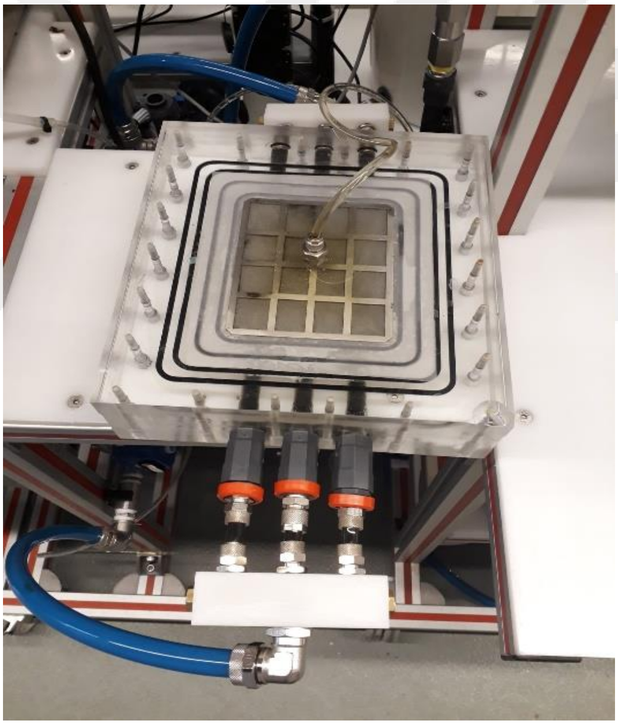


Figure 3.3 : Cross-flow membrane module.

3.5 Experimental Procedure

This study consisted of two stages. In the first stage, AnMBR system was fed with PS for 94 days. Afterward, AnMBR system was fed with A-sludge, and operated for 109 days. Operation under stable conditions lasted for 55 days for both stages. Stable conditions were maintained when the daily variation of biogas production was less than 10% for 10 days. The average values described in this study were calculated according to the stable process performance. The temperature was kept at 35 °C in the reactor to maintain mesophilic conditions. HRT and OLR values were 3.33 days and 3 kg COD/m^3.d during the operation, respectively. The membrane flux was set to 11 L/m^2.h by increasing it in three steps. The operational conditions of the AnMBR system are given in in Table 3.4.

The membrane in this study was operated in cycles of filtration and backwashing. The filtration period was 190 seconds, while the backwashing period was 35 seconds, which was performed by utilizing the permeate. The cross-flow velocity in the membrane was set around 0.1 m/sec by adjusting the recirculation rate at 50 L/min.

Table 3.4 : Operational conditions of AnMBR system.

Parameter	Unit	Value
HRT	days	3.33
OLR	kg COD/m^3.d	3
Temperature	°C	35
Flux	L/m^2.h.bar	11
Filtration mode	sec	190
Backwashing mode	sec	35

3.6 Experimental Analysis

3.6.1 Analytical techniques

TSS, VSS, TS, VS, COD, sCOD, TP, DP, TN, NH_4-N, alkalinity, fecal coliform, and total coliform were measured according to Standard Methods (APHA, 2017). The D50 values of the anaerobic sludge and the substrates were measured by a Mastersizer 2000 (Malvern Instruments, Hydro 2000 MU, UK) (Figure 3.4). VFA analyses were conducted by a gas chromatography (GC) equipped with flame ionization detector (FID) (Shimadzu, Japan). CST measurements for feed sludge and anaerobic sludge were determined by a CST analyzer (Triton Electronics, Type 304 M, UK). The methane content in biogas was detected via GC-FID (Agilent 7890 A, USA). The student’s t-test was performed with a significance level of probability (p-value) of 0.05.

SMA is another important measurement, which was in this study measured by an Automated Methane Potential Test System II (Bioprocess Control, Sweden). In analyzing SMA, samples and blanks were measured as triplicates in 500 mL bottles with a working volume of 400 mL. To maintain anaerobic conditions in the bottles, nitrogen gas was flushed into the bottles. As substrate, sodium acetate was used for samples. Macronutrients, phosphate buffer solution, and trace elements were added based on the study of Ozgun et al. (2015). VS concentration of sludge was kept as twice the substrate COD concentration.

Figure 3.4 : PSD analyzer.

SMP and EPS, including tightly-bound EPS (TB-EPS) and loosely bound EPS (LB-EPS), were measured in anaerobic sludge samples taken weekly. The samples were filtered through 0.45-μm filters to measure SMP (Kinyua et al., 2017). Carbohydrate and protein fractions of SMP, TB-EPS, and LB-EPS were detected with the procedures described by Dubois et al. (1956) and the modified Lowry method (Frølund et al., 1996), respectively.

3.6.2 Morphological analyses

3.6.2.1 Environmental scanning electron microscopy (ESEM)

Environmental scanning electron microscopy (ESEM) (Thermo Fisher Scientific Inc., FEI Quanta FEG 250 ESEM, UK) was used to visualize the membrane surface morphology (Figure 3.5). Membrane samples were located inside 4 °C fridge for drying before performing the morphological analyses. Before the analysis, the samples were coated with palladium and gold (Pd-Au) with a thickness of 3-4 nm via a vacuum evaporator (Quorum SC7620, UK) to increase the conductivity.

Figure 3.5 : ESEM

3.6.2.2 Fourier transform infrared spectroscopy (FTIR)

FTIR spectroscopy (Perkin-Elmer Inc., Spectrum 100 spectrometer, USA) was used to identify organic compounds on the membrane surfaces (Figure 3.6). Records for adsorption mode FTIR spectra ranged from 400 to 4000 cm^{-1}.

Figure 3.6 : FTIR

3.6.2.3 Confocal laser scanning microscopy (CLSM)

CLSM (Thermofisher Scientific, USA) was used to visualize the biofilms attached to the membrane (Figure 3.7). Membrane samples were stained with Live/Dead Baclight™ Bacterial viability kit. For the sample preparation, 1 µL SYTO 9 green-fluorescent nucleic acid stain (Component A) and 1 µL propidium iodide red-fluorescent nucleic acid stain (Component B) were mixed with 1 mL distilled water.

Figure 3.7 : CLSM

3.7 COD Mass Balance Calculations

The evaluation of the digestibility of the sludge in AnMBR was calculated by applying a mass balance for COD. The equation (Equation 3.1) used for the mass balance is as below:

$$Q_I x COD_I = Q_P x COD_P x Q_B x P_M x \frac{1}{0.35\ L\ methane/g\ COD} + Q_{WS} x COD_{WS} + Q_P + C_{DM}\ x \frac{1}{0.35\ L\ methane/g\ COD} \quad (3.1)$$

where Q_I (L/d), Q_P (L/d), Q_B (L/d), and Q_{WS} (L/d) are the flow rates of influent, permeate, biogas, and sludge wasting, respectively; COD_I(g/L), COD_P (g/L), and COD_{WS} (g/L) are the COD concentrations of influent, permeate, and sludge wasting, respectively; P_M (%) is the methane content in biogas; P is the pressure (atm); C_{DM} ($L_{Methane}$ / L_{Liquid}) is dissolved methane content in liquid, which was estimated based on Equation 3.2:

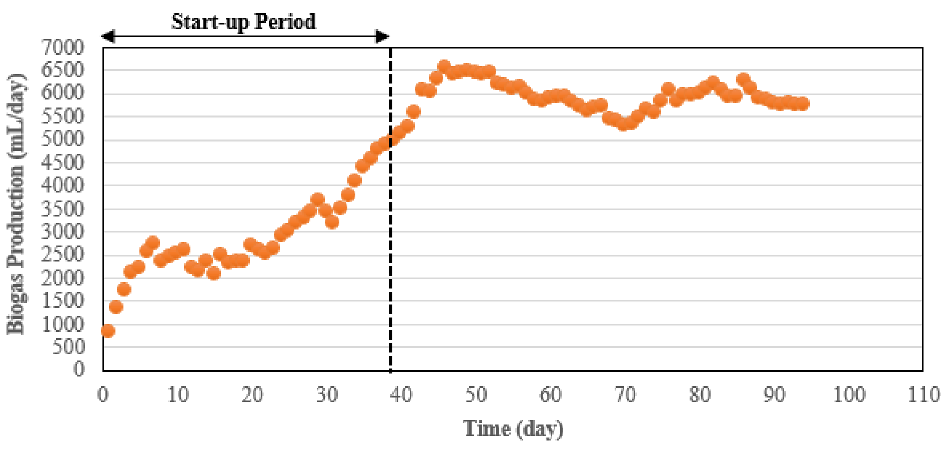
$$C_{DM} = \frac{P_M x P x V_c x N_w}{H} \quad (3.2)$$

where, P_M is the methane content in biogas; P is the pressure (1 atm); V_C is the corrected volume of 1 mol of gas at 35 °C (25.27 L/mole); H is Henry's law constant of methane at 35 °C (4.845 × 104 atm/mole fraction); N_W is the number of water moles contained in 1 L solution (55.6 mol/L).

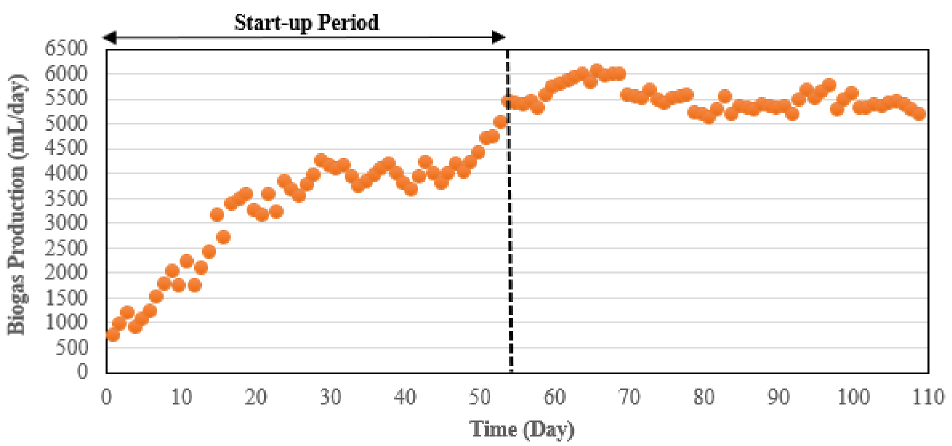
4. RESULTS AND DISCUSSIONS

4.1 Treatment Performance

Biogas production and methane content of the digested sludge is one of the most important indicators of AD process. During the digestion processes of both PS and A-sludge, biogas production rates and methane yields were monitored. The average biogas production rate of digestion of PS and A-sludge was 5908 ± 352 and 5486 ±238 mL/day, respectively (Figure 4.1). Although a higher biogas production was observed for the PS, A-sludge exhibited a higher methane production with a methane content of 73%, while 62% for PS. While the average methane yield of PS was 0.173 ± 0.012 mL CH_4/g COD_{fed}, A-sludge showed an average methane yield of 0.182 ± 0.009 mL CH_4/g COD_{fed}, respectively. The statistical t-test showed that the methane yields of both sludge types were significantly different (p-value=0.047). The reason for the higher methane yield of A-sludge may be attributed to the higher protein content of A-sludge, which can be seen from the TN concentration in A-sludge shown in Table 3.2. According to Hu et al. (2020), digestion of protein can result in higher methane content in biogas compared to carbohydrate digestion.



(a)



(b)

Figure 4.1 : Biogas production: (a) PS; (b) A-sludge.

Since methanogenesis is a rate-limiting step in AD processes, SMA test was carried out in this study to understand the methane-producing ability of the anaerobic organisms. The methanogenic population is crucial for achieving an effective sludge treatment. For the SMA tests, sodium acetate was used as substrate because acetoclastic methanogens can transform nearly 70% of COD to methane (Abdelrahman et al., 2022). In this study, SMA values of 0.13 ± 0.01 g CH_4-COD/gVS.d and 0.19 ± 0.01 CH_4-COD/g VS.d were obtained from anaerobic sludge fed with PS and A-sludge, respectively. The SMA value for PS was close to the SMA of the seed sludge, while the SMA of A-sludge showed an increase in acetoclastic methanogenesis activity.

The average COD concentrations of AnMBR were similar when fed with PS and A sludge (34,656 ± 2,637 and 33,276 ± 1,173 mg/L, respectively). A gradual decrease was observed in COD concentration of permeate during the start-up period, reaching average concentrations of 440 ± 151 and 281 ± 51 mg/L after the start-up period with removal efficiencies of 95.6 ± 1.5% and 97.3 ± 0.5% for PS and A-sludge, respectively (Figure 4.2). AnMBR has been proven to show a considerably high COD removal efficiency, which can be related to the complete retention of suspended solids by the membrane. In the literature, similar results were observed, as in the study of Cheng et al. (2021), which exhibited a 99.3% COD removal with the HRT of 15 d in AnMBR system treating sludge. Similarly, Hafuka et al. (2016) achieved COD removal efficiency of 98% in AnMBR treating excess sludge.

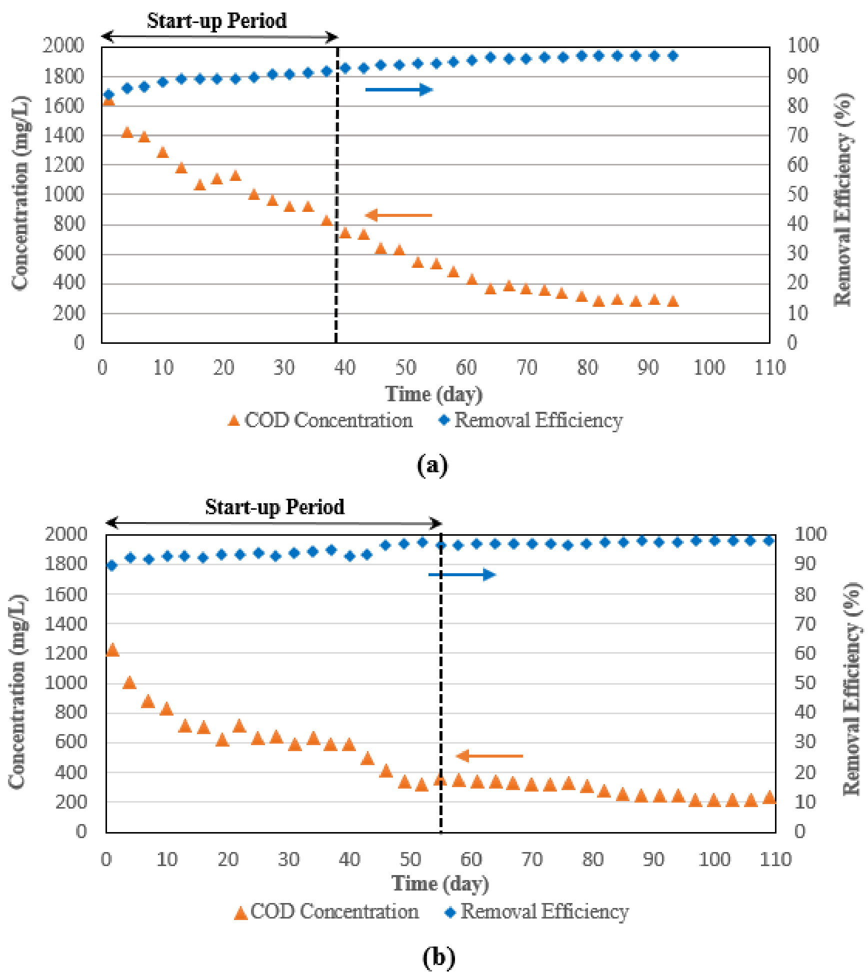
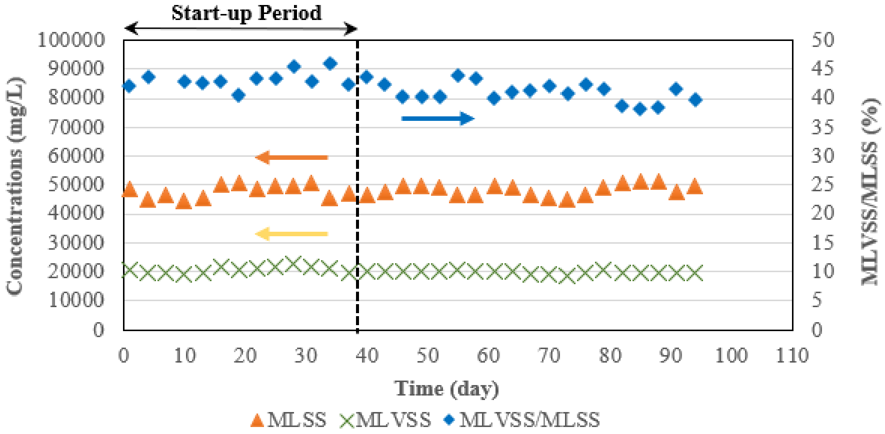


Figure 4.2 : Permeate COD concentrations and removal efficiencies: (a) PS; (b) A-sludge.

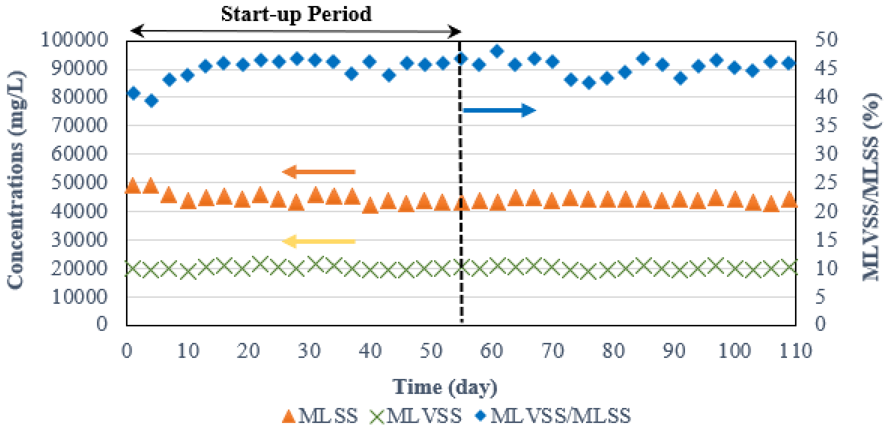
4.1.1 Process stability of the AnMBR

One of the advantages of AnMBR systems compared to conventional anaerobic digesters is the accumulation of high active biomass concentration in the anaerobic reactor. This high biomass concentration is obtained due to the decoupling of HRT and

SRT due to the solid-liquid separation by the membrane, which also results in a high suspended solids concentration in the reactor. Mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS) concentrations were fairly stable as a result of daily wasting of sludge to maintain SRT for 25 days. In the reactor fed with PS, MLSS and MLVSS concentrations were 48,329 ± 1,851 and 19,824 ± 502 mg/L, respectively, which were very similar to A-sludge with MLSS and MLVSS concentrations of 44,089 ± 633 and 19,964 ± 685 mg/L (Figure 4.3). The MLVSS/MLSS ratio was slightly lower for the AnMBR fed with PS (41 ± 2%) compared to A-sludge (45 ± 1%). This difference can be attributed to the lower VSS/TSS ratio of PS in comparison to A-sludge. Almost no solids were observed in the permeate of the AnMBR system. Therefore, very low concentration of TSS (<38 mg/L) and turbidity (<15 NTU) were found in the permeates of both sludge, which is undoubtedly due to the membrane separation.



(a)



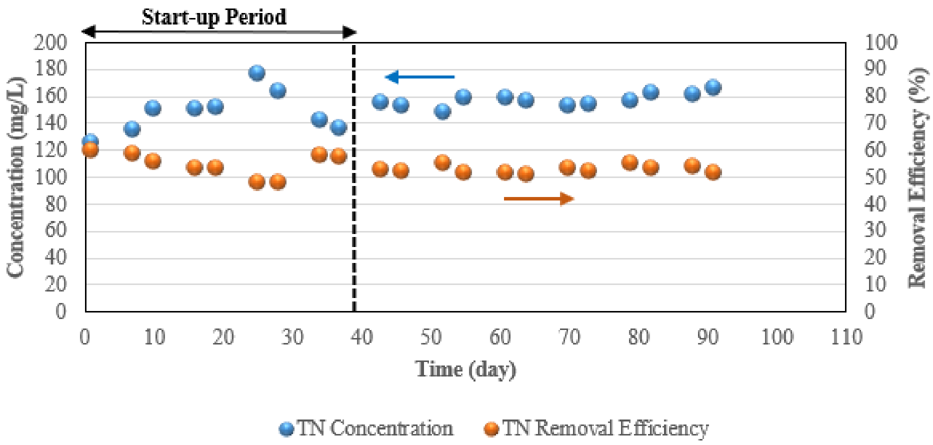
(b)

Figure 4.3 : MLSS, MLVSS concentrations, and MLVSS/MLSS ratios: (a) PS; (b) A-sludge

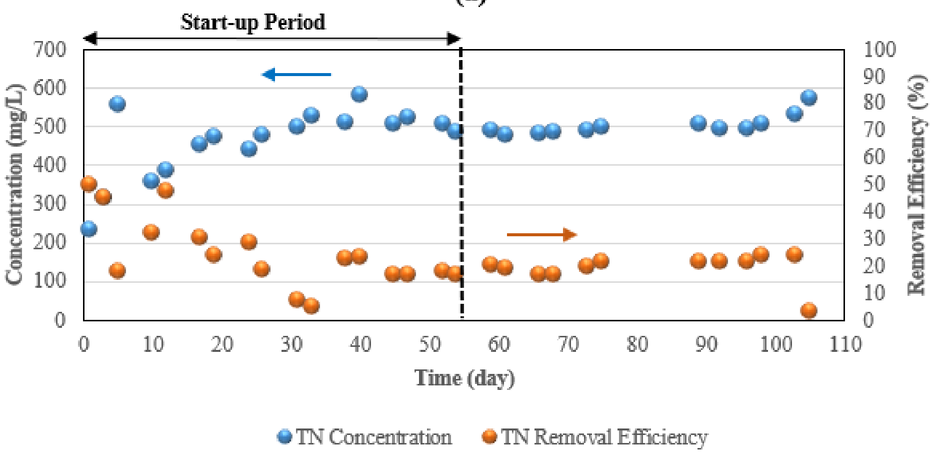
The stability of a digester is typically evaluated with VFA, alkalinity, and pH parameters. During feeding with PS and A-sludge, the average VFA concentrations in the anaerobic reactor were 426 ± 43 and 573 ± 117, respectively. For a stable digester operation, lower than 1500-3000 mg/L of VFA is recommended (Wu et al., 2019). Major VFAs were propionate and acetate in both AnMBRs. The acetate accounted for 67% of total VFAs in the reactor fed with PS. In the reactor fed with A-sludge, acetate was dominant with 83% of total VFAs. The average alkalinity in the AnMBR was 9792 ± 570 mg $CaCO_3$/L for PS and 7388 ± 154 mg $CaCO_3$/L for A-sludge. Cook et al. (2017) reported that the minimum alkalinity value of 2000 mg $CaCO_3$/L is required to have a stable operation for anaerobic digesters. Another crucial parameter indicating the stability of the digester and the risk of VFA accumulation in AnMBRs is VFA/Alkalinity ratio, which was lower than 0.08 for both sludges in this study. In the literature, it was reported that there is no risk of VFA accumulation, and the digester is considered stable if VFA/Alkalinity ratio is less than 0.3 (Liu et al., 2012). In this study, the average ORP value and pH were -462.5 ± 5.4 mV and 7.02 ± 0.04, respectively, in the reactor fed with PS, while ORP and pH were -482.3 ± 4.7 mV and 7.25, respectively, when fed with A-sludge. The pH value was stable around the neutrality value throughout the whole operation. The favorable ORP for methanogens is recommended as below -300 mV and the optimum pH is between 7.0 and 8.0 (Amani et al., 2010). Therefore, it can be concluded that the anaerobic system was favorably stable during the whole operation.

4.1.2 Permeate quality

The average TN concentrations in the permeate of AnMBR fed with PS and A-sludge were 156.4 ± 4.8 mg/L (removal efficiency of 52.5%) and 494.9 ± 12.3 mg/L (removal efficiency of 18.8%), respectively (Figure 4.5). Relatively low TN removal efficiency was observed, which can be corresponded to the higher TN (protein) concentration in A-sludge. The nitrogenous compounds (proteins) release NH_4-N during digestion, and NH_4-N can pass through the membrane because NH_4-N is soluble. The fraction of NH_4-N in the permeate of the AnMBR fed with PS and A-sludge was 86.3% and 84.8% of TN, respectively. For the removal of NH_4-N, partial nitritation-Anammox technology can be used due to the high COD/nitrogen ratio, which is 2.8 ± 1.0 and 0.55 ± 0.10 for PS and A-sludge. This COD/nitrogen ratio is considered applicable for partial nitritation-Anammox technology (Molinuevo et al., 2009).



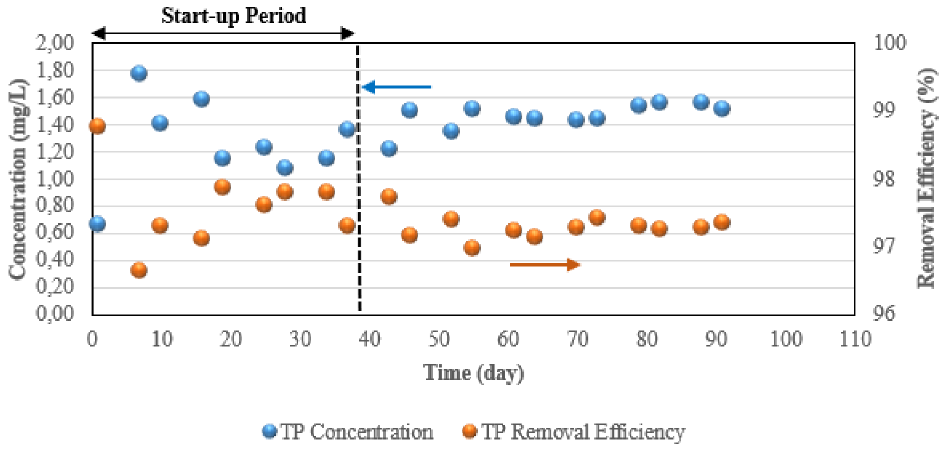
(a)



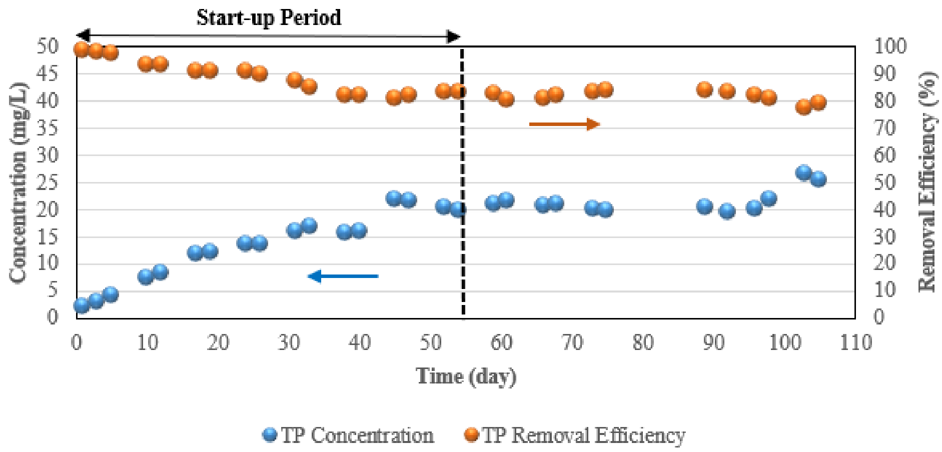
(b)

Figure 4.4 : Permeate TN concentrations and removal efficiencies: (a) PS; (b) A-sludge.

Considerably high TP removal efficiency was observed for both sludges. TP removal efficiency of AnMBR fed with PS was 97.3%, while 82.2% removal efficiency was reached when fed with A-sludge (Figure 4.6). The reason for a lower removal efficiency in the case of A-sludge was attributed to the relatively high DP concentration of A-sludge in comparison with PS. Thanks to the membrane, no total and fecal coliforms were observed in the permeates. Dagnew et al. (2010) operated a pilot-scale AnMBR and found no fecal coliform in the permeate, which is consistent with our study. Based on the fecal coliform results in this study, the permeate can be used for irrigational purposes according to the guidelines of the United States Environmental Protection Agency (USEPA), which reported that reclaimed water free of fecal can be used for irrigational purposes (USEPA, 2012).



(a)



(b)

Figure 4.5 : Permeate TP concentrations and removal efficiencies: (a) PS; (b) A-sludge

4.2 Filtration Performance

Table 4.1 illustrates the SMP and EPS concentrations in the AnMBR. Organic membrane fouling increases in line with SMP and bound EPS present in the reactor (Lin et al., 2010). The SMP highly contributes to irreversible fouling because of pore blocking (Meng et al., 2009; Shi et al., 2018). Carbohydrate concentration of SMP was higher in the AnMBR fed with A-sludge, while protein was dominant in SMP in the AnMBR fed with PS. Lin et al. (2010) also indicated that retained SMP and EPS may act as glue, resulting in the formation of a slime layer. In EPS, on the other hand, protein concentrations were found to be higher than carbohydrates in both sludge types. The higher protein concentration can correspond to a high amount of exoenzymes in sludge (Frølund et al., 1995). The higher total bound EPS was observed in A-sludge, due to the increase in the concentration of LB-EPS. LB-EPS has been proven to be greatly associated with increased membrane fouling and membrane resistance rather than TB-EPS (Wang et al., 2009). In this study, the protein/carbohydrate ratio was observed to be higher in LB-EPS in comparison to TB-EPS. In addition, TB-EPS concentrations were highly similar in both sludges.

Table 4.1 : SMP and EPS contents of the AnMBR system fed with PS and A-sludge.

Parameter	Unit	Average ± SD	
		PS	A-sludge
SMP			
Protein	mg/g VSS	15.9 ± 3.1	8.9 ± 2.4
Carbohydrates	mg/g VSS	3.7 ± 0.7	4.6 ± 1.0
Protein/Carbohydrates ratio	-	4.3 ± 0.2	1.9 ± 0.1
LB-EPS			
Protein	mg/g VSS	8.7 ± 1.0	12.9 ± 2.8
Carbohydrates	mg/g VSS	1.8 ± 0.3	3.6 ± 0.1
Protein/Carbohydrates ratio	-	4.9 ± 1.0	3.6 ± 0.7
TB-EPS			
Protein	mg/g VSS	16.2 ± 1.9	16.4 ± 1.5
Carbohydrates	mg/g VSS	4.2 ± 0.2	4.9 ± 0.04
Protein/Carbohydrates ratio	-	3.8 ± 0.6	3.4 ± 0.3
Total EPS			
Protein	mg/g VSS	24.9 ± 2.5	29.4 ± 4.3
Carbohydrates	mg/g VSS	6.1 ± 0.4	8.4 ± 0.03
Protein/Carbohydrates ratio	-	4.2 ± 0.7	3.5 ± 0.5

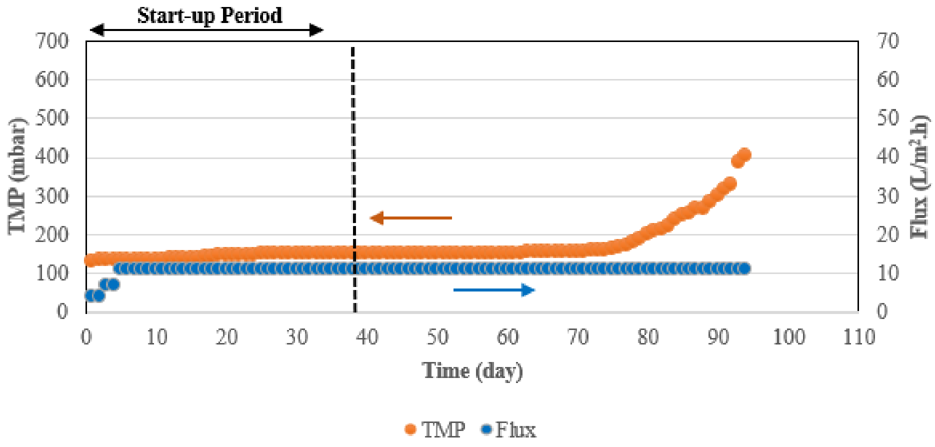
4.2.1 PSD and CST

In membrane processes, membrane fouling is greatly affected by the nature of biomass, one of which is PSD. Because smaller flocs contribute more to membrane fouling, PSD analysis is a must in membrane processes. The D50 values of the anaerobic sludge for PS and A-sludge were observed to be 7.75 ± 0.53 and 10.99 ± 0.42 µm, respectively. During the digestion of A-sludge, a higher D50 value was observed in the anaerobic sludge, which can be related to the higher D50 of A-sludge and higher EPS, particularly LB-EPS. It was previously reported that particle size decreased in parallel with the decrease in EPS concentration (Ersahin et al., 2016). CST is also a crucial parameter for determining the dewaterability and filterability of the sludge. Additionally, it was suggested that membrane fouling can be correlated to the dewaterability of the sludge (Huang et al., 2013). A higher CST value was observed for the anaerobic reactor fed with A-sludge (293 ± 11 sec) compared to the one with PS (87 ± 4 sec). The higher CST in the case of A-sludge may be related to the higher

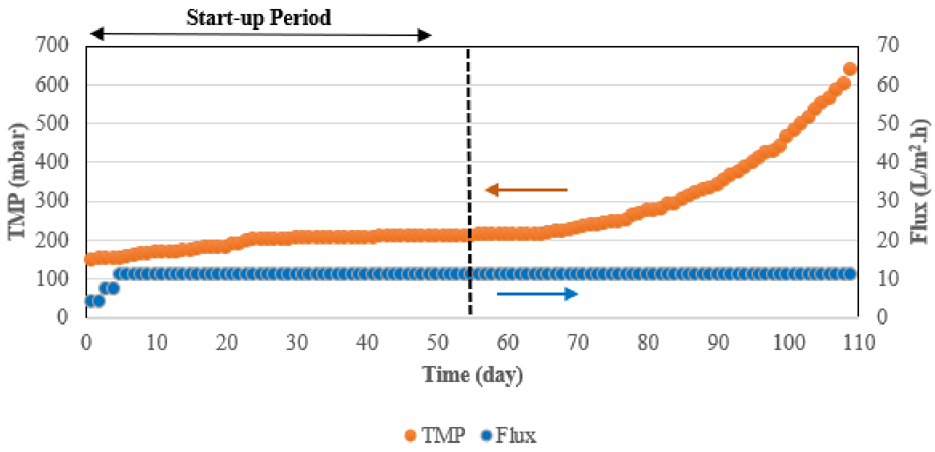
EPS available in A-sludge. This correlation is consistent with the study of Sahinkaya et al. (2018), in which a higher CST value was attributed to a higher EPS concentration.

4.2.2 TMP and filtration resistance

AnMBRs treating sludge for long-term operation are sensitive to the stability of flux and TMP values during the operational period since these values affect the applicability and economic feasibility of the AnMBR systems (Abdelrahman et al., 2021). The flux value in this study was chosen as 11 $L/m^2.h$ by raising the flux gradually in two steps to prevent any rapid fouling and sudden TMP increase (Figure 4.7). Membrane systems often encounter fouling problems and require appropriate cleaning that can be classified as physical and chemical cleaning. In this study, during the operation of the AnMBR system, neither physical nor chemical cleaning was applied for membrane cleaning, thanks to the filtration and backwashing cycles and also cross-flow shear force. TMP values for AnMBRs fed with PS and A-sludge were 171 ± 53 mbar and 223 ± 51 mbar, respectively. The average TMP increase rates were 3.0 and 4.6 mbar/d for the AnMBR fed with PS and A-sludge, respectively. Due to the higher EPS concentration and longer CST in the reactor fed with A-sludge, the membrane clogged earlier. Membrane permeability may be linked to several issues, such as higher hydrophobic protein concentration in EPS, which causes EPS accumulation on the membrane (Arabi and Nakhla, 2008).



(a)



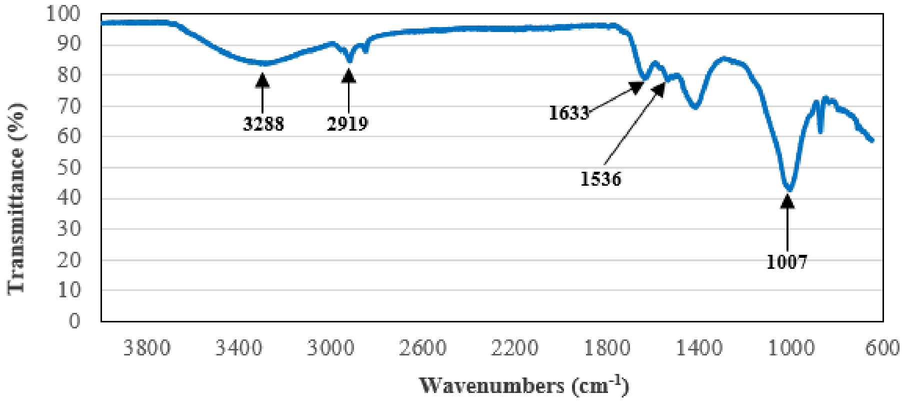
(b)

Figure 4.6 : TMP profile in the AnMBR: (a) PS; (b) A-sludge.

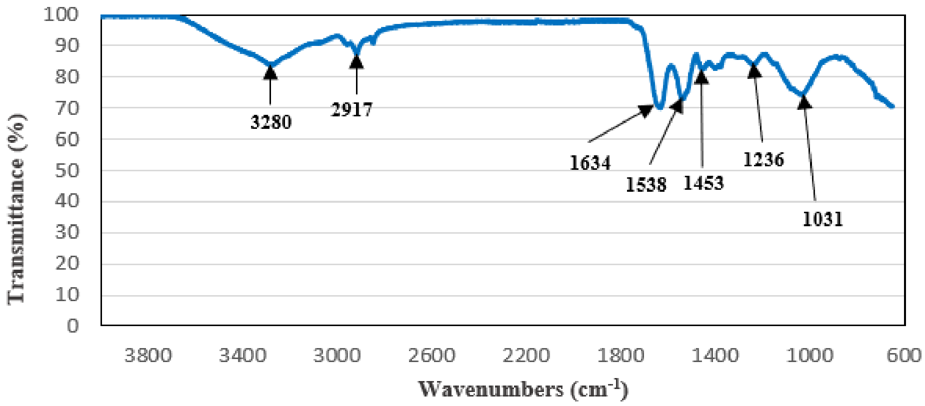
4.3 Morphological Analyses

4.3.1 FTIR

Similar peaks in FTIR spectra curves were observed for both sludges indicating that the cake layers had similar functional groups (Figure 4.8). The peaks observed at 3288 and 3280 cm^{-1} in the spectrum indicated the stretching of O-H bonds in the polysaccharide structure (Isik et al., 2019). The peaks at 2919 and 2917 cm^{-1} corresponded to the aliphatic C-H stretches of the polysaccharide (Gao et al., 2011). Peaks at 1633 and 1634 cm^{-1} showed amides I, namely stretching of C=O and C-N bonds while amides II (deformation of N-H bonds and C=N bonds) were correlated with peaks at 1536 and 1538 cm^{-1}. The presence of these amide groups indicates the secondary structure of protein (Isik et al., 2020). Similarly, amides III (C-N stretching) were also observed with the presence of peaks at 1416, 1453, and 1236 cm^{-1} (Ersahin et al., 2016; Wang et al., 2009). The peaks observed at 1007 and 1031 cm $^{-1}$ indicated symmetrical and asymmetrical C=O stretches (at 1000–1200 cm^{-1}) belonging to polysaccharides or polysaccharide-like substances (Ersahin et al., 2016). The peak <1000 cm^{-1} (fingerprint area) may correspond with the functional groups of nucleic acids, such as phosphate and sulfate (Gao et al., 2011). These results indicated the presence of polysaccharide-like and protein-like substances in the cake layer, which was expected due to the accumulation of SMP and EPS on the surface of the membrane.



(a)

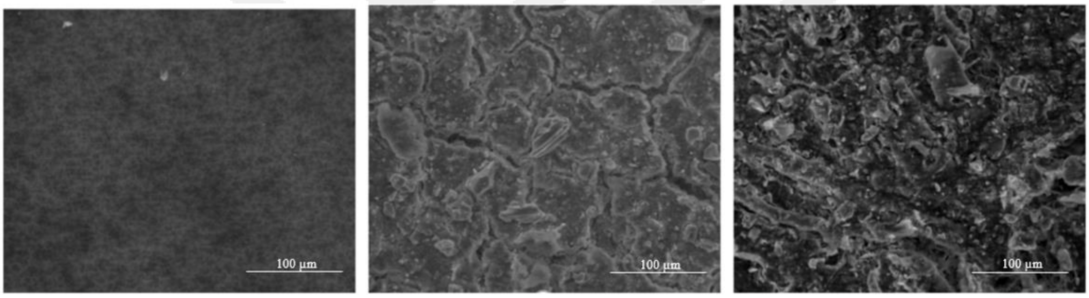


(b)

Figure 4.7 : FTIR spectrum of cake layer in AnMBR: (a) PS, (b) A-sludge

4.3.2 ESEM

ESEM analysis imaged the surface of the virgin membrane and cake layer after the operational period (Figure 4.9). Inorganic materials indicated by crystal-like materials can be observed on cake layers. Various studies suggested that mineral scales (i.e. inorganic particles) can accumulate on the membrane surface, resulting in roughness on the cake layer (Guo et al., 2012). Membrane fouling inorganic substances were found to be carbonate, calcium, magnesium, iron, silica, and sulfate (Potts et al., 1981). In this study, A-sludge digestion caused more accumulation on the membrane surface. This can be related to the comparatively higher EPS content of A-sludge, increasing the TMP. The formation of a compact fouling layer and increased membrane filtration resistance may be induced by intact microbial cells binding with colonizing bacterial-EPS clusters attached to inorganic particles, leading to filled spaces within the biopolymer.

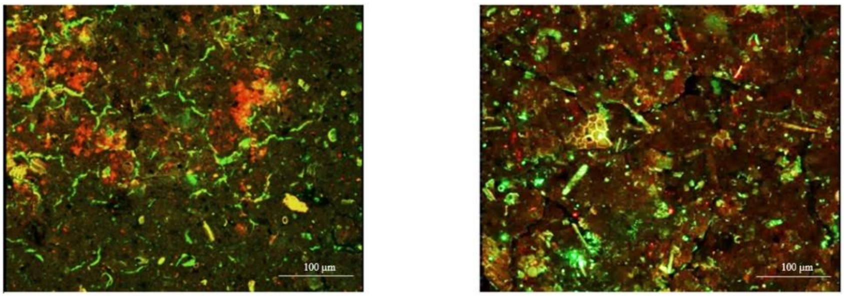


(a) **(b)** **(c)**

Figure 4.8 : ESEM images: (a) Virgin membrane; (b) Cake layer-PS; (c) Cake layer-A-sludge.

4.3.3 CLSM

Accumulation of live and dead bacteria on membrane surfaces was visualized by CLSM, which was applied for both cake layers (Figure 4.10). The cake layer of the AnMBR fed with A-sludge was observed to contain more dead cells, compared to that of PS. This can be correlated with the presence of more aerobic biomass in A-sludge, which accumulated on the membrane surface.



(a) (b)

Figure 4.9 : CLSM images of cake layer in AnMBR: (a) PS; (b) A-sludge (Green color represents live cells and red color represents dead cells).

4.4 COD Mass Balance

Based on the COD mass balance, A-stage integration to AnMBR can achieve more than 34.5% of COD recovery from wastewater into methane gas, while 19.9% of COD in the wastewater can be converted into methane gas when AnMBR is integrated with primary sludge. More methane production in the case of A-stage integration is attributed to the higher COD recovery of A-stage (>50%) (Guthi et al., 2022) in comparison to primary clarifier (40%) (Wan et al., 2016). With primary clarifier and A-stage integration, dissolved methane contributed 0.2% and 0.4% of the wastewater COD, respectively. Innovative methods with little energy requirements, including membrane contactors, can recover the dissolved methane (Velasco et al., 2018; Kalakech et al., 2022). Recovery of methane in membrane contactors can be more efficient with lower hydraulic flow, since the higher retention time of a liquid in the membrane module provides a longer time period for methane transfer (Li et al., 2021a). Consequently, given that the sludge is generated at low flow rates, it will be feasible to recover dissolved methane from the permeate.

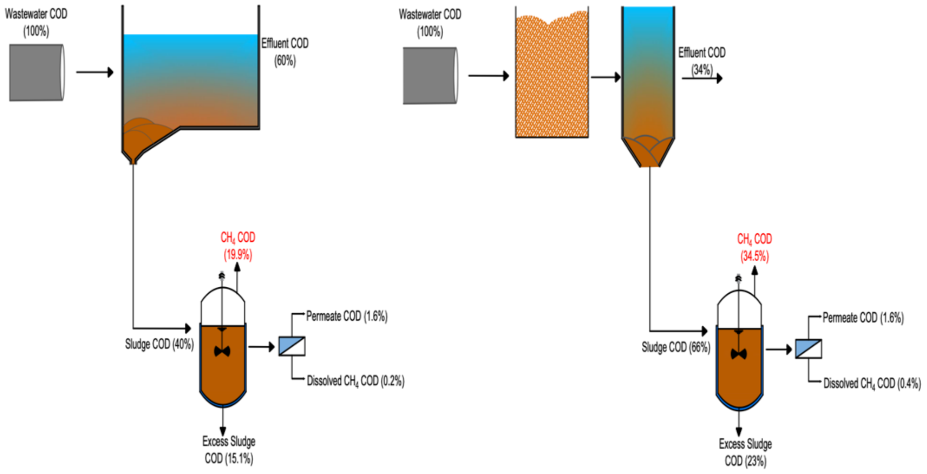


Figure 4.10 : COD mass balance for AnMBR: (a) PS; (b) A- sludge.

5. CONCLUSIONS AND FUTURE PERPECTIVES

5.1 Conclusions

This thesis focused on comparing the treatment and filtration performances of AnMBR treating PS and A-sludge in two separate stages. In this context, a lab-scale mesophilic AnMBR was operated for the digestion of both sludge types. For filtration, an external configuration with a commercial flat sheet UF membrane was used. The membrane area was 0.012 m^2 with a pore size of 0.02 µm. The AnMBR system was operated with PS for 94 days followed by the operational period with A-sludge for 109 days. Methane content and removal efficiencies of several parameters were observed. Moreover, morphological analyses of the membrane were also investigated. Consequently, a COD mass balance over both AnMBRs was conducted.

Following results were obtained as a result of the operation of the lab-scale AnMBRs:

- Although a higher biogas production was obtained with the digestion of PS, methane content in biogas was higher for A-sludge (73%) in comparison to PS (62%). More methane content obtained during the digestion of A-sludge can be related to its higher protein content. The average SMA of anaerobic sludge fed with PS and A-sludge was 0.13 ± 0.01 g CH_4-COD/g VS·d and 0.19 ± 0.01 g CH_4-COD/g VS·d, respectively.
- Similar COD removal efficiencies were obtained for the PS and A-sludge (95.6% and 97.3%, respectively).
- TSS removal efficiency for PS and A-sludge was more than 99% due to the membrane filtration, and almost no solids and fecal coliforms were observed in permeates. Therefore, the permeates have the potential to be used directly for irrigational purposes.
- Stable digester conditions were achieved in AnMBR during the digestion of PS and A-sludge, with the average VFA concentrations of 426 mg/L and 573 mg/L, respectively. The VFA/alkalinity ratio was lower than 0.08 for both

sludges, which was lower than the optimum ratio of 0.3 as reported in the literature.

- TN removal efficiencies for PS and A-sludge were 52.5% and 18.8%. The lower removal efficiency for A-sludge was due to a higher TN concentration in A-sludge, resulting in a high NH_4-N concentration due to protein hydrolysis. Since NH_4-N can pass through the membrane, more TN concentration was observed in the permeate.
- High TP removal efficiencies with an average of 97.3% and 82.2% were obtained for PS and A-sludge, respectively. Higher DP concentration in A-sludge led to the lower removal efficiency.
- A higher TMP was observed during the digestion of A-sludge. This can be linked to the higher EPS concentration and longer CST of A-sludge.
- Based on the morphological analyses, more accumulation was observed with A-sludge digestion, which might be related with its higher EPS concentration. Moreover, based on CLSM images, higher dead bacterial cells accumulation was observed on the cake layer of AnMBR treating A-sludge.
- COD mass balance indicated that integration of A-stage and PS can recover wastewater COD into methane gas with the percentage of 34.5% and 19.9%, respectively.

5.2 Future Perspectives

Based on the results obtained in this study, integration of A-stage with AnMBR instead of primary clarifier resulted in an increase in methane yield and methanogenic activity due to its potential for higher COD recovery. Thus, A-stage integration to AnMBR can be a solution for high energy consumption in WWTPs. Furthermore, membrane fouling when digesting A-sludge can be decreased by using different technologies to mitigate EPS accumulation on the membrane surface. However, few studies were conducted for the integration of A-stage with AnMBR; therefore, further investigation can be held in order to increase the methane yield of the system. Additionally, high TN concentration in permeate due to high ammonia concentration can be removed by partial nitritation-Anammox technology.

REFERENCES

Abdelrahman, A. M., Aras, M. F., Cicekalan, B., Fakioglu, M., Cingoz, S., Basa, S., Guven, H., Ozgun H., Ozturk, I., Koyuncu, I., van Lier, J.B., Volcke, E.I.P., and Ersahin, M. E. (2022). Primary and A-sludge treatment by anaerobic membrane bioreactors in view of energy-positive wastewater treatment plants. *Bioresource Technology, 351,* 126965.

Abdelrahman, A. M., Ozgun, H., Dereli, R. K., Isik, O., Ozcan, O. Y., van Lier, J. B., Ozturk, I., and Ersahin, M. E. (2021). Anaerobic membrane bioreactors for sludge digestion: Current status and future perspectives. *Critical Reviews in Environmental Science and Technology, 51(18),* 2119–2157.

Abdurahman, N. H., Rosli, Y. M., and Azhari, N. H. (2011). Development of a membrane anaerobic system (MAS) for palm oil mill effluent (POME) treatment. *Desalination*, *266*(1–3), 208–212.

Al-Hashimia, M. A. I., Abbas, T. R., and Jasema, Y. I. (2013). Performance of sequencing anoxic/anaerobic membrane bioreactor (SAM) system in hospital wastewater treatment and reuse. *European Scientific Journal*, *9*(15).

Amani, T., Nosrati, M., and Sreekrishnan, T. R. (2010). Anaerobic digestion from the viewpoint of microbiological, chemical, and operational aspects - a review. *Environmental Reviews*, *18*(NA), 255-278.

An, Y., Wang, Z., Wu, Z., Yang, D., and Zhou, Q. (2009). Characterization of membrane foulants in an anaerobic non-woven fabric membrane bioreactor for municipal wastewater treatment. *Chemical Engineering Journal*, *155*(3), 709–715.

APHA (2017). Standard Methods for Examination of Water and Wastewater, 23rd ed. *American Public 307 Health Association/American Water Works Association/Water Environment Federation*, 308 Washington DC, USA.

Aquino, S. F., Hu, A. Y., Akram, A., and Stuckey, D. C. (2006). Characterization of dissolved compounds in submerged anaerobic membrane bioreactors (SAMBRs). *Journal of Chemical Technology and Biotechnology*, *81*(12), 1894–1904.

Arabi, S., and Nakhla, G. (2008). Impact of protein/carbohydrate ratio in the feed wastewater on the membrane fouling in membrane bioreactors. *Journal of Membrane Science*, *324*(1–2), 142–150.

Ariunbaatar, J., Panico, A., Esposito, G., Pirozzi, F., and Lens, P. N. (2014). Pretreatment methods to enhance anaerobic digestion of organic solid waste. *Applied energy*, *123*, 143-156.

Bai, R., and Leow, H. F. (2002). Microfiltration of activated sludge wastewater-the effect of system operation parameters. *Separation and Purification Technology 29*.

Batstone, D. J., Jensen, P. D., and Ge, H. (2011). Biochemical treatment of biosolids-emerging technologies. *Water*, *5*, 90-93.

Bindoff, A. M., Treffry-Goatley, K., Fortmann, N. E., Hunt, J. W., and Buckley, C. A. (1988). The application of cross-flow microfiltration technology to the concentration of sewage works sludge streams. *Water and Environment Journal*, *2*(5), 513-522.

Borja, R. (2011). 2.55-Biogas Production. Comprehensive *Biotechnology (Second Edition)*, 785-798.

Calderón, K., Rodelas, B., Cabirol, N., González-López, J., and Noyola, A. (2011). Analysis of microbial communities developed on the fouling layers of a membrane-coupled anaerobic bioreactor applied to wastewater treatment. *Bioresource Technology*, *102*(7), 4618–4627.

Carrère, H., Dumas, C., Battimelli, A., Batstone, D. J., Delgenes, J. P., Steyer, J. P., and Ferrer, I. (2010). Pretreatment methods to improve sludge anaerobic degradability: a review. *Journal of Hazardous Materials*, *183*(1-3), 1-15.

Chan, Y. J., Chong, M. F., Law, C. L., and Hassell, D. G. (2009). A review on anaerobic–aerobic treatment of industrial and municipal wastewater. *Chemical Engineering Journal*, *155*(1-2), 1-18.

Chen, R., Wen, W., Jiang, H., Lei, Z., Li, M., and Li, Y. Y. (2019). Energy recovery potential of thermophilic high-solids co-digestion of coffee processing wastewater and waste activated sludge by anaerobic membrane bioreactor. *Bioresource Technology*, *274*, 127-133.

Cheng, H., Li, Y., Hu, Y., Guo, G., Cong, M., Xiao, B., and Li, Y. Y. (2021). Bioenergy recovery from methanogenic co-digestion of food waste and sewage sludge by a high-solid anaerobic membrane bioreactor (AnMBR): mass balance and energy potential. *Bioresource Technology*, *326*, 124754.

Choo, K. H., and Lee, C. H. (1996). Membrane fouling mechanisms in the membrane-coupled anaerobic bioreactor. *Water Research*, *30*(8), 1771-1780.

Chu, L. B., Yang, F. L., and Zhang, X. W. (2005). Anaerobic treatment of domestic wastewater in a membrane-coupled expanded granular sludge bed (EGSB) reactor under moderate to low temperature. *Process Biochemistry*, *40*(3–4), 1063–1070.

Cook, S. M., Skerlos, S. J., Raskin, L., and Love, N. G. (2017). A stability assessment tool for anaerobic codigestion. *Water Research*, *112*, 19-28.

Dagnew, M., Parker, W., and Seto, P. (2012). Anaerobic membrane bioreactors for treating waste activated sludge: Short term membrane fouling characterization and control tests. Journal of Membrane Science, 421, 103-110.

Dagnew, M., Parker, W. J., and Seto, P. (2010). A pilot study of anaerobic membrane digesters for concurrent thickening and digestion of waste activated sludge (WAS). *Water Science and Technology, 61*(6), 1451–1458.

Dagnew, M., Pickel, J., Parker, W., and Seto, P. (2013). Anaerobic membrane bio-reactors for waste activated sludge digestion: Tubular versus hollow fiber membrane configurations. *Environmental Progress and Sustainable Energy, 32*(3), 598–604.

Daw, J., Hallett, K., DeWolfe, J., and Venner, I. (2012). Energy efficiency strategies for municipal wastewater treatment facilities (No. NREL/TP-7A20-53341). National Renewable Energy Lab. (NREL), Golden, CO (United States).

Dereli, R. (2015). Treatment of industrial wastewaters by anaerobic membrane bioreactors: Implications of substrate characteristics (Doctoral Dissertation). Retrieved from https://repository.tudelft.nl/islandora/object/uuid%3A4ad3a7cb-cf9f-45e4-8b50-f6d48f669b50

Dereli, R. K., Ersahin, M. E., Ozgun, H., Ozturk, I., Jeison, D., van der Zee, F., and van Lier, J. B. (2012). Potentials of anaerobic membrane bioreactors to overcome treatment limitations induced by industrial wastewaters. *Bioresource Technology*, *122*, 160–170.

Dubois, M., Gilles, K. A., Hamilton, J. K., Rebers, P. T., and Smith, F. (1956). Colorimetric method for determination of sugars and related substances. *Analytical Chemistry*, *28*(3), 350-356.

Dvořák, L., Gómez, M., Dolina, J., and Černín, A. (2016). Anaerobic membrane bioreactors—a mini-review with emphasis on industrial wastewater treatment: applications, limitations, and perspectives. *Desalination and Water Treatment*, *57*(41), 19062–19076.

Ersahin, M. E., Tao, Y., Ozgun, H., Spanjers, H., and van Lier, J. B. (2016). Characteristics and role of dynamic membrane layer in anaerobic membrane bioreactors; Characteristics and role of dynamic membrane layer in anaerobic membrane bioreactors. *Biotechnology and Bioengineering, 113*, 761–771.

Frølund, B., Griebe, T., and Nielsen, P. H. (1995). Enzymatic activity in the activated-sludge floc matrix. *Applied Microbiology and Biotechnology*, *43*, 755-761.

Frølund, B., Palmgren, R., Keiding, K., and Nielsen, P. H. (1996). Extraction of extracellular polymers from activated sludge using a cation exchange resin. *Water Research*, *30*(8), 1749-1758.

Gao, W. J., Lin, H. J., Leung, K. T., Schraft, H., and Liao, B. Q. (2011). Structure of cake layer in a submerged anaerobic membrane bioreactor. *Journal of Membrane Science*, *374*(1–2), 110–120.

Ghyoot, W. R., and Verstraete, W. H. (1997). Coupling membrane filtration to anaerobic primary sludge digestion. *Environmental Technology (United Kingdom)*, *18*(6), 569–580.

Grethlein, H. E. (1978). Anaerobic digestion and membrane separation of domestic wastewater. *Journal of Water Pollution Control Federation*, 754-763.

Guo, W., Ngo, H. H., and Li, J. (2012). A mini-review on membrane fouling. *Bioresource Technology*, *122*, 27–34.

Guthi, R. S., Tondera, K., Gillot, S., Buffière, P., Boillot, M., and Chazarenc, F. (2022). A-Stage process–challenges and drawbacks from lab to full scale studies: a review. *Water Research*, 119044.

Hafuka, A., Mashiko, R., Odashima, R., Yamamura, H., Satoh, H., and Watanabe, Y. (2019). Digestion performance and contributions of organic and inorganic fouling in an anaerobic membrane bioreactor treating waste activated sludge. *Bioresource Technology*, *272*, 63–69.

Hafuka, A., Mimura, K., Ding, Q., Yamamura, H., Satoh, H., and Watanabe, Y. (2016). Performance of anaerobic membrane bioreactor during digestion and thickening of aerobic membrane bioreactor excess sludge. *Bioresource Technology*, *218*, 476–479.

Herrera-Robledo, M., Morgan-Sagastume, J. M., and Noyola, A. (2010). Biofouling and pollutant removal during long-term operation of an anaerobic membrane bioreactor treating municipal wastewater. *Biofouling*, *26*(1), 23–30.

Hu, Y., Cheng, H., Ji, J., and Li, Y. Y. (2020). A review of anaerobic membrane bioreactors for municipal wastewater treatment with a focus on multicomponent biogas and membrane fouling control. Environmental Science: *Water Research and Technology*, *6*(10), 2641-2663.

Huang, Z., Ong, S. L., and Ng, H. Y. (2011). Submerged anaerobic membrane bioreactor for low-strength wastewater treatment: Effect of HRT and SRT on treatment performance and membrane fouling. *Water Research*, *45*(2), 705–713.

Huang, Z., Ong, S. L., and Ng, H. Y. (2013). Performance of submerged anaerobic membrane bioreactor at different SRTs for domestic wastewater treatment. *Journal of Biotechnology*, *164*(1), 82–90.

Isik, O., Abdelrahman, A. M., Ozgun, H., Ersahin, M. E., Demir, I., and Koyuncu, I. (2019). Comparative evaluation of ultrafiltration and dynamic membranes in an aerobic membrane bioreactor for municipal wastewater treatment. *Environmental Science and Pollution Research*, *26*(32), 32723–32733.

Isik, O., Hudayarizka, R., Abdelrahman, A. M., Ozgun, H., Ersahin, M. E., Demir, I., and Koyuncu, I. (2020). Impact of support material type on performance of dynamic membrane bioreactors treating municipal wastewater. *Journal of Chemical Technology and Biotechnology*, *95*(9), 2437–2446.

Jeong, E., Kim, H. W., Nam, J. Y., Ahn, Y. T., and Shin, H. S. (2010). Effects of the hydraulic retention time on the fouling characteristics of an anaerobic membrane bioreactor for treating acidified wastewater. *Desalination and Water Treatment*, *18*(1–3), 251–256.

Jeong, T. Y., Cha, G. C., Yoo, I. K., and Kim, D. J. (2007). Hydrogen production from waste activated sludge by using separation membrane acid fermentation reactor and photosynthetic reactor. *International Journal of Hydrogen Energy*, *32*(5), 525-530.

Joo, J. Y., Park, C. H., and Han, G. B. (2016). Optimization of two-phased anaerobic sludge digestion using the pressurized ultra filtration membrane with a mesh screen (MS-PUFM). *Chemical Engineering Journal*, *300*, 20–28.

Joshi, P., and Parker, W. (2015). Effect of pretreatment using ultrasound and hydrogen peroxide on digestion of waste activated sludge in an anaerobic membrane bioreactor. *Environmental Progress and Sustainable Energy*, *34*(6), 1724–1730.

Kalakech, C., Sohaib, Q., Lesage, G., and Mericq, J. P. (2022). Progress and challenges in recovering dissolved methane from anaerobic bioreactor permeate using membrane contactors: A comprehensive review. *Journal of Water Process Engineering*, *50*, 103218.

Kayawake, E., Narukami, Y., and Yamagata, M. (1991). Anaerobic digestion by a ceramic membrane-enclosed reactor. *Journal of Fermentation and Bioengineering*, *71*(2), 122-125.

Kim, J., Lee, C. H., and Choo, K. H. (2007). Control of struvite precipitation by selective removal of NH_4^+ with dialyzer/zeolite in an anaerobic membrane bioreactor. *Applied Microbiology and Biotechnology*, *75*(1), 187–193.

Kim, M., Ahn, Y. H., and Speece, R. E. (2002). Comparative process stability and efficiency of anaerobic digestion; mesophilic vs. thermophilic. *Water Research*, *36*(17), 4369-4385.

Kimura, S. (1991). Japan's aqua renaissance'90 project. *Water Science and Technology*, *23*(7-9), 1573-1582.

Kinyua, M. N., Elliott, M., Wett, B., Murthy, S., Chandran, K., and Bott, C. B. (2017). The role of extracellular polymeric substances on carbon capture in a high rate activated sludge A-stage system. *Chemical Engineering Journal*, *322*, 428–434.

Kocadagistan, E., and Topcu, N. (2007). Treatment investigation of the Erzurum City municipal wastewaters with anaerobic membrane bioreactors. *Desalination*, *216*(1–3), 367–376.

Kooijman, G., Lopes, W., Zhou, Z., Guo, H., de Kreuk, M., Spanjers, H., and van Lier, J. (2017). Impact of coagulant and flocculant addition to an anaerobic dynamic membrane bioreactor (AnDMBR) treating waste activated sludge. *Membranes*, *7*(2).

Lee, W., Kang, S., and Shin, H. (2003). Sludge characteristics and their contribution to microfiltration in submerged membrane bioreactors. *Journal of Membrane Science*, *216*(1–2), 217–227.

Li, W., Cai, T., Lu, X., Han, Y., Kudisi, D., Chang, G., Dong, K., and Zhen, G. (2023). Two-Phase improves Bio-hydrogen and Bio-methane production of anaerobic membrane bioreactor from waste activated sludge with digestate recirculation. *Chemical Engineering Journal*, *452*, 139547.

Li, X., Lee, H. S., Wang, Z., and Lee, J. (2021a). State-of-the-art management technologies of dissolved methane in anaerobically-treated low-strength wastewaters: A review. *Water Research*, *200*, 117269.

Li, Y., Ni, J., Cheng, H., Zhu, A., Guo, G., Qin, Y., and Li, Y. Y. (2021b). Methanogenic performance and microbial community during thermophilic digestion of food waste and sewage sludge in a high-solid anaerobic membrane bioreactor. *Bioresource Technology*, *342*.

Liao, B. Q., Kraemer, J. T., and Bagley, D. M. (2006). Anaerobic membrane bioreactors: applications and research directions. *Critical Reviews in Environmental Science and Technology*, *36*(6), 489-530.

Liao, B. Q., Xie, K., Lin, H. J., and Bertoldo, D. (2010). Treatment of kraft evaporator condensate using a thermophilic submerged anaerobic membrane bioreactor. *Water Science and Technology*, *61*(9), 2177–2183.

Liew Abdullah, A. G., Idris, A., Ahmadun, F. R., Baharin, B. S., Emby, F., Megat Mohd Noor, M. J., and Nour, A. H. (2005). A kinetic study of a membrane anaerobic reactor (MAR) for treatment of sewage sludge. *Desalination*, *183*(1–3), 439–445.

Lin, H., Chen, J., Wang, F., Ding, L., and Hong, H. (2011a). Feasibility evaluation of submerged anaerobic membrane bioreactor for municipal secondary wastewater treatment. *Desalination*, *280*(1–3), 120–126.

Lin, H. J., Xie, K., Mahendran, B., Bagley, D. M., Leung, K. T., Liss, S. N., and Liao, B. Q. (2009). Sludge properties and their effects on membrane fouling in submerged anaerobic membrane bioreactors (SAnMBRs). *Water Research*, *43*(15), 3827–3837.

Lin, H. J., Xie, K., Mahendran, B., Bagley, D. M., Leung, K. T., Liss, S. N., and Liao, B. Q. (2010). Factors affecting sludge cake formation in a submerged anaerobic membrane bioreactor. *Journal of Membrane Science*, *361*(1–2), 126–134.

Lin, H., Liao, B. Q., Chen, J., Gao, W., Wang, L., Wang, F., and Lu, X. (2011b). New insights into membrane fouling in a submerged anaerobic membrane bioreactor based on characterization of cake sludge and bulk sludge. *Bioresource Technology*, *102*(3), 2373–2379.

Lin, H., Peng, W., Zhang, M., Chen, J., Hong, H., and Zhang, Y. (2013). A review on anaerobic membrane bioreactors: applications, membrane fouling and future perspectives. *Desalination*, *314*, 169-188.

Liu, X., Gao, X., Wang, W., Zheng, L., Zhou, Y., and Sun, Y. (2012). Pilot-scale anaerobic co-digestion of municipal biomass waste: Focusing on biogas production and GHG reduction. *Renewable Energy*, *44*, 463-468.

Maaz, M., Yasin, M., Aslam, M., Kumar, G., Atabani, A. E., Idrees, M., Anjum, F., Jamil, F., Ahmad, R., Khan, A.L., Lesage, G., Heran, M., and Kim, J. (2019). Anaerobic membrane bioreactors for wastewater treatment: Novel configurations, fouling control and energy considerations. *Bioresource Technology*, *283*, 358-372.

Mahendran, B., Lin, H., Liao, B., and Liss, S. N. (2011). Surface Properties of Biofouled Membranes from a Submerged Anaerobic Membrane Bioreactor after Cleaning. *Journal of Environmental Engineering*, *137*(6), 504–513.

Markis, F., Baudez, J. C., Parthasarathy, R., Slatter, P., and Eshtiaghi, N. (2014). Rheological characterization of primary and secondary sludge: Impact of solids concentration. *Chemical Engineering Journal*, *253*, 526–537.

Martin, I., Pidou, M., Soares, A., Judd, S., and Jefferson, B. (2011). Modelling the energy demands of aerobic and anaerobic membrane bioreactors for wastewater treatment. *Environmental Technology*, *32*(9), 921-932.

Martin-Ryals, A. D., Schideman, L. C., & Guy, K. (2017). Utilizing ion-exchange resin to improve recovery from organic shock-loading in an AnMBR treating sewage sludge. *Water Research*, *126*, 285-298.

Martin-Ryals, A. D., Schideman, L. C., and Ong, M. (2020). Utilizing bioaugmentation to improve performance of a two-phase AnMBR treating sewage sludge. *Environmental Technology (United Kingdom)*, *41*(10), 1322–1336.

Martinez-Sosa, D., Helmreich, B., Netter, T., Paris, S., Bischof, F., and Horn, H. (2011). Anaerobic submerged membrane bioreactor (AnSMBR) for municipal wastewater treatment under mesophilic and psychrophilic temperature conditions. *Bioresource Technology*, *102*(22), 10377–10385.

Meabe, E., Déléris, S., Soroa, S., and Sancho, L. (2013). Performance of anaerobic membrane bioreactor for sewage sludge treatment: Mesophilic and thermophilic processes. *Journal of Membrane Science*, *446*, 26–33.

Meegoda, J. N., Li, B., Patel, K., and Wang, L. B. (2018). A review of the processes, parameters, and optimization of anaerobic digestion. *International Journal of Environmental Research and Public Health*, *15*(10), 2224.

Meng, F., Chae, S. R., Drews, A., Kraume, M., Shin, H. S., and Yang, F. (2009). Recent advances in membrane bioreactors (MBRs): membrane fouling and membrane material. *Water Research*, *43*(6), 1489-1512.

Minami, K. (1994). A trial of high-performance anaerobic treatment on wastewater from a kraft pulp mill. *Desalination*, *98*(1-3), 273-283.

Minami, K., Okamura, K., Ogawa, S., and Naritomi, T. (1991). Continuous anaerobic treatment of wastewater from a kraft pulp mill. *Journal of Fermentation and Bioengineering*, *71*(4), 270-274.

Molinuevo, B., García, M. C., Karakashev, D., and Angelidaki, I. (2009). Anammox for ammonia removal from pig manure effluents: Effect of organic matter content on process performance. *Bioresource Technology*, *100*(7), 2171–2175.

Murić, A., Petrinić, I., and Christensen, M. L. (2014). Comparison of ceramic and polymeric ultrafiltration membranes for treating wastewater from metalworking industry. *Chemical Engineering Journal*, *255*, 403–410.

Niu, C., Pan, Y., Lu, X., Wang, S., Zhang, Z., Zheng, C., Tan, Y., Zhen, G., Zhao, Y., and Li, Y. Y. (2020). Mesophilic anaerobic digestion of thermally hydrolyzed sludge in anaerobic membrane bioreactor: Long-term performance, microbial community dynamics and membrane fouling mitigation. *Journal of Membrane Science*, *612*.

Ozgun, H., Ersahin, M. E., Zhou, Z., Tao, Y., Spanjers, H., and van Lier, J. B. (2019). Comparative evaluation of the sludge characteristics along the height of upflow anaerobic sludge blanket coupled ultrafiltration systems. *Biomass and Bioenergy*, *125*, 114–122.

Ozgun, H., Dereli, R. K., Ersahin, M. E., Kinaci, C., Spanjers, H., and van Lier, J. B. (2013). A review of anaerobic membrane bioreactors for municipal wastewater treatment: Integration options, limitations and expectations. *Separation and Purification Technology*, *118*, 89-104.

Ozgun, H., Tao, Y., Ersahin, M. E., Zhou, Z., Gimenez, J. B., Spanjers, H., and van Lier, J. B. (2015). Impact of temperature on feed-flow characteristics and filtration performance of an upflow anaerobic sludge blanket coupled ultrafiltration membrane treating municipal wastewater. *Water Research*, *83*, 71-83.

Pierkiel, A., and Lanting, J. (2005). Membrane-coupled anaerobic digestion of municipal sewage sludge. *Water Science and Technology*, *52*(1-2), 253-258.

Pileggi, V., and Parker, W. J. (2017). AnMBR digestion of mixed WRRF sludges: Impact of digester loading and temperature. *Journal of Water Process Engineering*, *19*, 74–80.

Pillay, V. L., Townsend, B., and Buckley, C. A. (1994). Improving the performance of anaerobic digesters at wastewater treatment works: the coupled cross-flow microfiltration/digester process. *Water Science and Technology*, *30*(12), 329.

Pollice, A., Brookes, A., Jefferson, B., and Judd, S. (2005). Sub-critical flux fouling in membrane bioreactors—a review of recent literature. *Desalination*, *174*(3), 221-230.

Potts, D. E., Ahlert, R. C., and Wang, S. S. (1981). A critical review of fouling of reverse osmosis membranes. *Desalination*, *36*(3), 235–264.

Pretel, R., Robles, A., Ruano, M. v., Seco, A., and Ferrer, J. (2014). The operating cost of an anaerobic membrane bioreactor (AnMBR) treating sulphate-rich urban wastewater. *Separation and Purification Technology*, *126*, 30–38.

Qiao, W., Takayanagi, K., Niu, Q., Shofie, M., and Li, Y. Y. (2013a). Long-term stability of thermophilic co-digestion submerged anaerobic membrane reactor encountering high organic loading rate, persistent propionate and detectable hydrogen in biogas. *Bioresource Technology*, *149*, 92–102.

Qiao, W., Takayanagi, K., Shofie, M., Niu, Q., Yu, H. Q., and Li, Y. Y. (2013b). Thermophilic anaerobic digestion of coffee grounds with and without waste activated sludge as co-substrate using a submerged AnMBR: System amendments and membrane performance. *Bioresource Technology*, *150*, 249–258.

Rossi, WR, Barnard, JP, Ie Roux, J. and de Villiers, H. (1990). Application of ultrafiltration membranes for solids liquid separation in anaerobic digestion systems: the ADUF process. *Water SA.*, *16*(2), 85-91.

Sahinkaya, E., Yurtsever, A., Isler, E., Coban, I., and Aktaş, Ö. (2018). Sulfate reduction and filtration performances of an anaerobic membrane bioreactor (AnMBR). *Chemical Engineering Journal*, *349*, 47–55.

Sainbayar, A., Kim, J. S., Jung, W. J., Lee, Y. S., and Lee, C. H. (2001). Application of surface modified polypropylene membranes to an anaerobic membrane bioreactor. *Environmental Technology (United Kingdom)*, *22*(9), 1035–1042.

Sandino, J., Santha, H., Rogowski, S., Anderson, W., Sung, S., and Isik, F. (2005). Applicability of ultrasound pre-conditioning of WAS to reduce foaming potential in mesophilic digesters, *Residuals and Biosolids Conference 2005* (pp. 819-835). Water Environment Federation.

Santos, A., and Judd, S. (2010). The commercial status of membrane bioreactors for municipal wastewater. *Separation Science and Technology*, *45*(7), 850–857.

Shi, Y., Huang, J., Zeng, G., Gu, Y., Hu, Y., Tang, B., Zhou, J., Yang, Y., and Shi, L. (2018). Evaluation of soluble microbial products (SMP) on membrane fouling in membrane bioreactors (MBRs) at the fractional and overall level: a review. *Reviews in Environmental Science and Biotechnology*, *17*, 71-85.

Shin, C., and Bae, J. (2018). Current status of the pilot-scale anaerobic membrane bioreactor treatments of domestic wastewaters: A critical review. *Bioresource Technology*, *247*, 1038-1046.

Singh, B., Szamosi, Z., and Siménfalvi, Z. (2019). State of the art on mixing in an anaerobic digester: A review. *Renewable Energy*, *141*, 922-936.

Speece, R. E. (1983). Anaerobic biotechnology for industrial wastewater treatment. *Environmental Science and Technology*, *17*(9), 416A-427A.

Spinosa, L., Ayol, A., Baudez, J. C., Canziani, R., Jenicek, P., Leonard, A., Rulkens, W., Xu, G., and van Dijk, L. (2011). Sustainable and innovative solutions for sewage sludge management. *Water (Switzerland)*, *3*(2), 702–717.

Strohwald, N. K. H., & Ross, W. R. (1992). Application of the ADUFR process to brewery effluent on a laboratory scale. *Water Science and Technology*, *25*(10), 95-105.

Stuckey, D. C. (2012). Recent developments in anaerobic membrane reactors. *Bioresource Technology*, *122*, 137-148.

Takashima, M., Kudoh, Y., and Tabata, N. (1996). Complete anaerobic digestion of activated sludge by combining membrane separation and alkaline heat post-treatment. *Water Science and Technology*, *34*(5-6), 477-481.

Torres, A., Hemmelmann, A., Vergara, C., and Jeison, D. (2011). Application of two-phase slug-flow regime to control flux reduction on anaerobic membrane bioreactors treating wastewaters with high suspended solids concentration. *Separation and Purification Technology*, *79*(1), 20–25.

Tsuneda, S., Aikawa, H., Hayashi, H., Yuasa, A., and Hirata, A. (2003). Extracellular polymeric substances responsible for bacterial adhesion onto solid surface. *FEMS Microbiology Letters*, *223*(2), 287–292.

Uddin, M. M., and Wright, M. M. (2022). Anaerobic digestion fundamentals, challenges, and technological advances. *Physical Sciences Reviews*.

USEPA (2012). Guidelines for Water Reuse. EPA/600/R-12/618. U.S. Environmental Protection Agency, Washington D.C. (2012). *2012 Guidelines for Water Reuse*.

Velasco, P., Jegatheesan, V., and Othman, M. (2018). Recovery of dissolved methane from anaerobic membrane bioreactor using degassing membrane contactors. *Frontiers in Environmental Science*, *6*, 151.

Verstraete, W., and Vandevivere, P. (1999). New and broader applications of anaerobic digestion. In *Critical Reviews in Environmental Science and Technology* (Vol. 29, Issue 2, pp. 151–173). CRC Press LLC.

Vyrides, I., and Stuckey, D. C. (2009). Saline sewage treatment using a submerged anaerobic membrane reactor (SAMBR): Effects of activated carbon addition and biogas-sparging time. *Water Research*, *43*(4), 933–942.

Wan, J., Gu, J., Zhao, Q., and Liu, Y. (2016). COD capture: a feasible option towards energy self-sufficient domestic wastewater treatment. *Scientific Reports*, *6*(1), 1-9.

Wandera, S. M., Qiao, W., Jiang, M., Gapani, D. E., Bi, S., and Dong, R. (2018). AnMBR as alternative to conventional CSTR to achieve efficient methane production from thermal hydrolyzed sludge at short HRTs. *Energy*, *159*, 588–598.

Wandera, S. M., Qiao, W., Jiang, M., Mahdy, A., Yin, D., and Dong, R. (2019). Enhanced methanization of sewage sludge using an anaerobic membrane bioreactor integrated with hyperthermophilic biological hydrolysis. *Energy Conversion and Management*, *196*, 846–855.

Wang, K., Zheng, Y., Zhu, X., Brewer, C. E., and Brown, R. C. (2017). Ex-situ catalytic pyrolysis of wastewater sewage sludge – A micro-pyrolysis study. *Bioresource Technology*, *232*, 229–234.

Wang, X. M., and Li, X. Y. (2008). Accumulation of biopolymer clusters in a submerged membrane bioreactor and its effect on membrane fouling. *Water Research*, *42*(4–5), 855–862.

Wang, Z., Wu, Z., and Tang, S. (2009). Extracellular polymeric substances (EPS) properties and their effects on membrane fouling in a submerged membrane bioreactor. *Water Research*, *43*(9), 2504–2512.

Wen, X., Sui, P., and Huang, X. (2008). Exerting ultrasound to control the membrane fouling in filtration of anaerobic activated sludge - Mechanism and membrane damage. *Water Science and Technology*, *57*(5), 773–779.

Wu, D., Li, L., Zhao, X., Peng, Y., Yang, P., and Peng, X. (2019). Anaerobic digestion: A review on process monitoring. *Renewable and Sustainable Energy Reviews*, *103*, 1-12.

Xie, K., Lin, H. J., Mahendran, B., Bagley, D. M., Leung, K. T., Liss, S. N., and Liao, B. Q. (2010). Performance and fouling characteristics of a submerged anaerobic membrane bioreactor for kraft evaporator condensate treatment. *Environmental Technology*, *31*(5), 511–521.

Xie, Z., Wang, Z., Wang, Q., Zhu, C., and Wu, Z. (2014). An anaerobic dynamic membrane bioreactor (AnDMBR) for landfill leachate treatment: performance and microbial community identification. *Bioresource Technology, 161*, 29-39.

Xu, M., Wen, X., Huang, X., and Li, Y. (2010). Membrane fouling control in an anaerobic membrane bioreactor coupled with online ultrasound equipment for digestion of waste activated sludge. *Separation Science and Technology*, *45*(7), 941–947.

Xu, M., Wen, X., Huang, X., Yu, Z., and Zhu, M. (2013). Mechanisms of membrane fouling controlled by online ultrasound in an anaerobic membrane bioreactor for digestion of waste activated sludge. *Journal of Membrane Science*, *445*, 119–126.

Xu, M., Wen, X., Yu, Z., Li, Y., and Huang, X. (2011). A hybrid anaerobic membrane bioreactor coupled with online ultrasonic equipment for digestion of waste activated sludge. *Bioresource Technology*, *102*(10), 5617–5625.

Yu, H., Wang, Z., Wu, Z., and Zhu, C. (2016). Enhanced waste activated sludge digestion using a submerged anaerobic dynamic membrane bioreactor: performance, sludge characteristics and microbial community. *Scientific Reports*, *6*(1), 20111.

Yu, Z., Song, Z., Wen, X., and Huang, X. (2015). Using polyaluminum chloride and polyacrylamide to control membrane fouling in a cross-flow anaerobic membrane bioreactor. *Journal of Membrane Science*, *479*, 20-27.

Zhang, J., Padmasiri, S. I., Fitch, M., Norddahl, B., Raskin, L., and Morgenroth, E. (2007). Influence of cleaning frequency and membrane history on fouling in an anaerobic membrane bioreactor. *Desalination*, *207*(1–3), 153–166.

Zhang, Q., Hu, J., Lee, D. J., Chang, Y., and Lee, Y. J. (2017). Sludge treatment: Current research trends. *Bioresource Technology*, *243*, 1159-1172.

Zhang, Q. H., Yang, W. N., Ngo, H. H., Guo, W. S., Jin, P. K., Dzakpasu, M., Yang, S. J., Wang, Q., Wang, X. C., and Ao, D. (2016). Current status of urban wastewater treatment plants in China. *Environment International*, *92–93*, 11–22.

Zhao, J., Li, Y., Pan, S., Tu, Q., and Zhu, H. (2019). Performance of a forward osmotic membrane bioreactor for anaerobic digestion of waste sludge with increasing solid concentration. *Journal of Environmental Management*, *246*, 239–246.

Zhen, G., Lu, X., Kato, H., Zhao, Y., and Li, Y. Y. (2017). Overview of pretreatment strategies for enhancing sewage sludge disintegration and subsequent anaerobic digestion: Current advances, full-scale application and future perspectives. *Renewable and Sustainable Energy Reviews*, *69*, 559-577.

CURRICULUM VITAE

Name Surname **:** Muhammed Furkan ARAS

EDUCATION

- **B.Sc.** **:**2018, Kahramanmaras Sutcu Imam University, Faculty of Engineering and Architecture, Department of Environmental Engineering

PUBLICATIONS, PRESENTATIONS AND PATENTS ON THE THESIS:

- **Abdelrahman, A. M., Aras, M. F., Cicekalan, B., Fakioglu, M., Cingoz, S., Basa, S., Guven, H., Ozgun H., Ozturk, I., Koyuncu, I., van Lier, J.B., Volcke, E.I.P., and Ersahin, M. E.** (2022). Primary and A-sludge treatment by anaerobic membrane bioreactors in view of energy-positive wastewater treatment plants. *Bioresource Technology, 351*, 126965.