PERFORMANCE EVALUATION OF LAB SCALE ANAEROBIC MEMBRANE BIOREACTOR FOR DOMESTIC WASTEWATER TREATMENT

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By

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Has been found satisfactory for partial fulfillment of the requirements of the degree of Masters of Science in Environmental Engineering

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Dedication:

This research work is dedicated to all the dreamers who dare to realize their destinities!

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ABSTRACT

The aim of this study was to evaluate the applicability of lab scale anaerobic membrane bioreactor (AnMBR) for domestic wastewater treatment. In initial stage, the hydraulic retention time (HRT) of lab scale continuously stirred tank reactor (CSTR) was optimized for maximum COD removal and biogas production. The CSTR was operated for 16 days and the COD removal observed at HRT of 24 hours and OLR of 0.5 kgCOD/m^3-day was 64.43% with the average biogas production of 2.51 L/day. In the attempt to reduce the HRT of the system, the CSTR was also operated at HRTs of 18, 12 and 6 hours in ‘test study’ for consecutive 3 days. Based on the results obtained, the HRT of the system was reduced to 12 hours resulting in OLR of 1.0 kgCOD/m^3-day for next phase of operation and the system was run for consecutive 42 days until steady state results were achieved. The COD removal obtained was 64.8% with the biogas yield of 5.15 L/day. In the next stage, the AnMBR was designed and established coupling CSTR with membrane tank while the membrane being in side stream submerged configuration. The performance of AnMBR was evaluated employing different sludge recirculation ratios at different fluxes. The system was fed with synthetic domestic wastewater of 500 mg/L COD at three different fluxes; 10.28 (R=1, phase I), 8.8 (R=2, phase II and R=3, phase III) and 6 LMH (R=2, phase IV and R=3, phase V) respectively. The operational cycle adopted was 8 min permeation and 2 min relaxation to reduce membrane fouling. The performance of the system was evaluated in terms of COD removal, VFAs and alkalinity accumulation and biogas yield. Sludge characteristics were measured in terms of mixed liquor suspended solids (MLSS), and mixed liquor volatile suspended solids (MLVSS). In comparison with the all operating protocols tested, optimum efficacy of the system was found at the net flux of 6 LMH and recirculation ratio (R) of 3 with the average COD removal of 96.7% and biogas yield of 0.44 L/gCOD$_{removed}$ while allowing the longest membrane run.

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List of Abbreviations

OLR	Organic loading rate
COD	Chemical oxygen demand
CSTR	Continuously stirred tank reactor
AnMBR	Anaerobic membrane bioreactor
HRT	Hydraulic retention time
LMH	Liters per meter square per hour
VFAs	Volatile fatty acids
MLSS	Mixed liquor suspended solids
MLVSS	Mixed liquor volatile suspended solids
PAC	Powdered activated carbon
GAC	Granular activated carbon
SRT	Solids retention time
UF	Ultrafiltration
IESE	Institute of environmental sciences and engineering
NUST	National university of sciences and technology
PVDF	Polyvinyl difluoride
TMP	Transmembrane pressure
ORP	Oxidation reduction potential

Chapter 1

INTRODUCTION

1.1 Background

The intolerable extraction of water resources and the unavoidable contamination of ground water and surface water has led to the scarcity of this natural resource which can further exacerbate the situation if continued. According to an estimation, almost 500 million people reside in areas where water demand exceeds the renewable water resources available locally by a factor of two. In most of the developing world, wastewater is directly disposed of into the water bodies leaving severe effects on human health, economic productivity and freshwater resources quality (Dinsdale et al., 1997).

The presence of many contaminants and compounds in ground and surface waters has been attributed to anthropogenic activities and excessive use of chemicals, fertilizers and insecticides that ultimately become part of surrounding water bodies. Water quality challenges need to be addressed in an integrated manner and by adopting pollution prevention strategies. Water pollution can be reduced by eliminating contaminants at source which is the most effective way to protect water quality.

The findings of various researches have indicated the deteriorating water quality in Pakistan resulting from the contamination and direct disposal of sewage which critically poses a threat to the growing population which is vulnerable to the negative impacts of both; water scarcity and energy crisis in the same time. The areas of dense population are majorly under threat where there is lack of water quality monitoring, management and provision of safe disposal options. Hence the wastewater mixed freely with surface water and groundwater thus opens a pool of problems including waterborne diseases, agricultural pollution and deteriorates ecosystems health.

The sustainable development goals (SDGs) put inevitable challenges for developing countries suffering from energy crisis, desertification and water scarcity issues. SDG 6.3 and 7 specifically focus on improving water quality by reducing pollution, halving the proportion of untreated wastewater and increasing recycling and safe reuse while ensuring affordable and sustainable energy for all. They highlight the increasing importance of water scarcity worldwide and the need for extensive integration and cooperation to ensure sustainable management of scarce water resources, both at international and local levels. Therefore, emphasis should be on the need for enhanced partnerships for sustainable developments, sharing of knowledge, expertise and technology for achieving Sustainable Development Goals (SDGs), particularly for developing countries like Pakistan.

On average, high-income countries treat about 70% of the generated municipal and industrial wastewater. That ratio drops to 38% in upper middle-income countries and further to 28% in lower middle-income countries. In low-income countries, only 8% is being treated by any means (Sato et al., 2013). These estimates conclude that globally, over 80% of all wastewater is discharged without treatment. Hence, the sustainable treatment of wastewater has been a failure owing to present energy crisis, lack of management and financial constraints which can compromise all other goals to achieve sustainable development if sustained.

Domestic wastewater is being seen as the potential resource than a problem. The conventional methods to treat the wastewater include activated sludge process, trickling filters and constructed wetlands which all contribute to enhanced cost being more energy intensive, vulnerable to more maintenance issues and while contributing to more capital cost. While the anaerobic systems eliminate aeration (often half of a WWTP's electricity consumption), reduce sludge wasting, and convert organic carbon to usable energy products (e.g., methane, hydrogen, electricity). So, the interests are being increased with time in anaerobic treatment which proves to be reliable, sustainable and cost effective at the same time.

One of the promising technologies in anaerobic domain is anaerobic digestion which converts waste COD to biogas, reserves useful nutrients, and requires less operational energy. One advancement is the up-gradation of this digestion system coupling with membrane tank which can provide better polishing of the effluent, retain complete biomass in the system and as a result, contribute to enhanced biogas yield. On one side, the net biomass production is low, up to ten times less than that of aerobic treatment. On the other side, the relatively poor settling properties of the biomass in conventional anaerobic biological treatment systems would result in the loss of biomass to effluent. This situation may lead to poor biomass retention in the conventional anaerobic biological system. A complete retention of all microorganisms in the bioreactor can be encompassed in membrane bioreactors (MBRs) by the use of microfiltration (MF) or Ultrafiltration (UF) modules (Lin et al., 2013). Several studies have used compact systems such as anaerobic membrane bioreactor (AnMBR) to meet the more stringent environment regulation and recover resources.

Research and development on AnMBRs have been extensive in the recent years, including reviewed studies which explored development and highlighted new research directions (Liao et al., 2006). However, a growing interest in the field of AnMBR is still on as depicted from the number of research studies in progress. One prime benefit of MBRs is they can be decouple solids retention time from hydraulic retention time providing complete retention of biomass in the system. Therefore, this became an attractive solution for the treatment of low (i.e., municipal wastewater) to high strength industrial wastewater with simultaneous energy recovery and less excess sludge production.

One major issue in the applications of MBRs is fouling which hinder its application at large scale. The complex biological process and the air tight reactor made observations on membrane fouling and the maintenance of a sustainable anaerobic process difficult. In light of this, recent research studies (Calderón et al., 2011; Huang et al., 2011) have considered investigating membrane fouling in AnMBRs. Hence, AnMBRs can yield more net energy while having environmental emissions in whole life cycle than activated sludge with anaerobic digestion process.

Different studies have been conducted on AnMBRs but the research has been limited to the performance evaluation of AnMBR consisting continuous stirred tank reactor coupled with immersed membrane system with recycling taking place into the anaerobic bioreactor. Moreover, the biogas produced from anaerobic bioreactors is mostly used for natural scouring of the membrane modules. However, un-stabilities and low production of biogas from the process may render biogas sparging ineffective for fouling control. So, there was a need to investigate some novel technique to minimize membrane fouling while ensuring constant flux filtration being more energy effective.

1.2 Objectives

The major advantage of this research was to conduct a comprehensive performance evaluation study of anaerobic MBR.

Therefore, the objectives of this study included:

1. Optimization of CSTR for the treatment of synthetic domestic wastewater of COD 500 mg/L

2. Performance Evaluation of anaerobic membrane bioreactor (AnMBR) in terms of COD removal and biogas production
3. Investigation of fouling mitigation potential of AnMBR at varying fluxes and sludge recirculation ratios

Chapter 2

LITERATURE REVIEW

This chapter outlines the relevant facts and figures from various researches and highlights the important clues stating the importance of anaerobic membrane bioreactors and the inquisitive studies being made into this domain particularly.

2.1 Available Wastewater Treatment Technologies

The term conventional wastewater treatment is used for physical, chemical, and biological mechanisms that remove solids, pathogens, organic matter, and nutrients. Biodegradation, sorption to excess sludge, and volatilization are the processes for the removal of contaminants present in wastewater. Biodegradability, mainly depends on the chemical structure of the molecules, physico-chemical properties, and the ability of microorganisms to degrade those molecules. Sorption is the removal mechanism for more hydrophobic compounds, accumulate onto primary and secondary sludge (Rogers, 1996). Biological treatment appears to be the best for wastewater treatment to attain revenue from Certified Emission Reduction (CER) credits, known as carbon credits for the production of methane gas from anaerobic digestion process which can be utilized as renewable energy. Almost all wastewaters with BOD/COD ratio of 0.5 or greater and having biodegradable components can be treated easily by biological means (Tchobanoglous et al., 2003a). It also incurs low treatment cost in comparison to other methods of wastewater treatment, with no secondary pollution (Sponza & Uluköy, 2005).

Aerobic and anaerobic treatments both can be used; the former involves the microorganisms (aerobes) which use free or dissolved oxygen in the biodegradation of organic wastes into biomass and CO_2 while in the latter complex organic matters are degraded in the absence of oxygen into methane, CO_2 and H_2O through three steps: hydrolysis, acidogenesis including acetogenesis and methanogenesis. Aerobic biological processes are used when high degree of treatment efficiency is required for the treatment of organic wastewaters while in anaerobic treatment, much progress has been made on the concept of resource recovery and utilization while achieving the primary objective of pollution control as reported by (Seghezzo et al., 1998; Yeoh, 1995).

2.2 Historical Background of Anaerobic Treatment

Anaerobic digestion (AD) is the most commonly used method for sludge stabilization to reduce odors, pathogens and volatile solids, where organic materials in sludge are converted to biogas mainly methane and CO_2 (Keating et al., 2016).

The rise of fossil fuels prices in early 1970s introduced application of anaerobic digestion to industrial wastewater and was also supported by the stringent pollution control regulations. The un-acceptability of the conventional digester for low strength industrial wastewater treatment and soluble organic contents, led to the idea of biological solids recycling and to the retention of biomass within the digester. Anaerobic digestion has been usedprimarily for the treatment of high-COD waste rather than as means of generating energy in the form of biogas. In western world (developed countries), anaerobic digestion has been used mainly for processing animal manure till the mid-1970s. The progressions in anaerobic digesters came with the introduction of anaerobic filter in 1967 (Abbasi et al., 2012).

The startup of anaerobic technology like upflow anaerobic sludge blanket (UASB) in 1980s (Lettinga et al., 1980), lead several countries to adopt anaerobic sewage treatment technology in the context of sewage treatment plants (STP). Anaerobic wastewater treatment, in various cases was equipped with units of aerobic systems serving as post treatment options. The favorable climate conditions and the continuous research efforts resulted in later frontrunner in the efficient use of UASB reactor systems for municipal wastewater treatment (Chernicharo et al., 2015).

With the introduction of anaerobic technology in Brazil, the inappropriate use of UASB systems damaged the credibility of this anaerobic technology within state legislative measures. However, the experience and operational upgradation of full-scale plants helped restoring the issue (Chernicharo, 2001).

The advancement was then followed by a number of more developments in this technology to treat a variety of biodegradable wastewaters. These developments in the process upgradation and in reactor designs have extensively supported the use of anaerobic digestion as a sustainable wastewater treatment process. Developed countries have already established wastewater treatment using anaerobic digestion which is being extensively followed all around the world (Abbasi et al., 2012).

2.3 Microbiology and Chemistry of Anaerobic Treatment

Process kinetics play an important role in developing anaerobic treatment systems. The thorough understanding of biochemistry of anaerobic process leads to a rational basis for anaerobic process monitoring, control, and design (Pavlostathis & Giraldo-Gomez, 1991).

The anaerobic degradation of particulate organic material is a multi-step process which continues in series and combinations of parallel reactions (Kaspar & Wuhrmann, 1978); (Bryant, 1979; Zehnder & Mitchell, 1978). First, complex polymers such as polysaccharides, proteins, and lipids are hydrolyzed by extracellular enzymes to small chunks to allow their transport across the cell membrane. These relatively simple compounds are then anaerobically oxidized forming short-chain fatty acids, alcohols, carbon dioxide, hydrogen, and ammonia. The short-chain fatty acids except acetate are transformed to acetate, hydrogen gas, and carbon dioxide. Lastly methanogenesis from carbon dioxide and acetate yields hydrogen (Pavlostathis & Giraldo-Gomez, 1991).

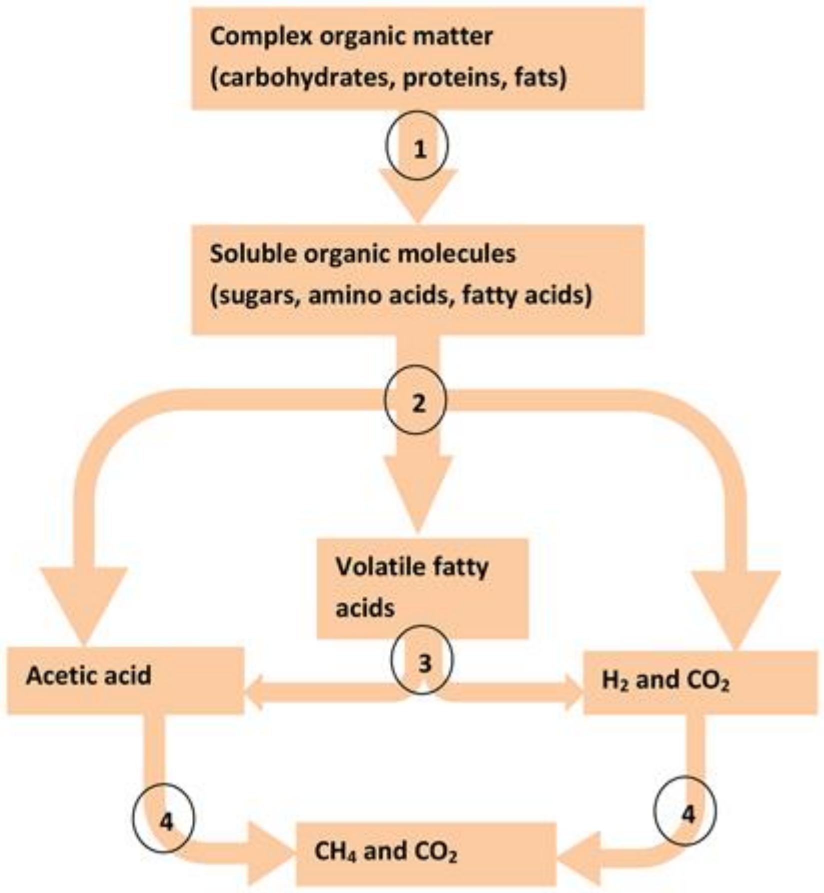


Figure 2.1. Bio-kinetics of anaerobic digestion process

In a compound multi-step process, the kinetics of the slowest step determine the overall kinetics of anaerobic process. The third process of methane fermentation is considered to be the slowest one (Andrews et al., 1964; McCarty, 1966). The complete process yields acetic and propionic acids which act as precursors to form approximately 85 percent of methane (Jeris & McCarty, 1965; McCarty et al., 1963). Hence, to successful design and analyze anaerobic systems, thorough knowledge of the process kinetics for the fermentation of methane from these acids is a basic element (Lawrence & McCarty, 1969). The major pathways for the conversion of organic matter in anaerobic treatment is shown in Figure 2.1.

2.4 Operational Parameters for Anaerobic Treatment

To prevent process upsets, proper system configuration and a rigorous control of the operational parameters are critical to maintain environmental variables within the optimal ranges (Labatut & Gooch, 2014). The most important process parameters to monitor in anaerobic systems are described below:

2.4.1 Volatile Fatty Acids (VFAs)

Volatile fatty acids (VFAs) concentration is considered to be the most sensitive to monitor. They can inhibit the digestion process ultimately leading to system failure. VFAs comprise of a group of compounds, i.e., acetic acid/acetate, propionic acid/propionate, butyric acid/butyrate, valeric acid/valerate, caproic acid/caproate, and enanthic acid/enanthate; acetate being the predominant (Labatut & Gooch, 2014).

A carefully designed and monitored system should have the concentration of total VFA less than 500 mg/L as acetic acid. However, smaller digesters can accumulate higher concentration of VFAs. Biogas yield can be hampered by VFAs concentrations over 1,500 – 2,000 mg/L. Moreover, the elevated concentrations of VFAs in the effluent can also be taken as indication of system upsets. Thus, VFAs are very important to monitor to have a successful running operation (Labatut & Gooch, 2014).

2.4.2 pH

An efficient and reliable anaerobic treatment requires the pH maintenance in a certain range. The generally optimal range lies between 6.5 and 7.5. The anaerobic digestion of organic substrates is carried out by the mutual working of several microorganisms, from which methanogens are more susceptible to pH change (Labatut & Gooch, 2014).

A system with less alkalinity being present and accumulation of acids can become "sour". Moreover, a well-maintained system can have a slight higher pH of the effluent as microbes produce alkalinity while consuming (protein-rich) organic matter (Labatut & Gooch, 2014).

2.4.3 Alkalinity (Alk)

The buffer capacity of an anaerobic reactor is determined by the amount of alkalinity present. The bicarbonate ion (HCO_3^-) plays the major role to maintain the system's pH in the range of 6.5 – 7.5. The amount of HCO_3^- in the system is related to the amount of carbon dioxide in the gas phase. In fact, cow manure can be used to increase the pH and buffering capacity of the influent mixture for the treatment of high strength and easily degradable industrial wastes (Labatut & Gooch, 2014).

2.4.4 Total Ammonia-Nitrogen (TAN)

Ammonia is generally produced from the digestion of protein-rich substrates. Likewise VFAs, ammonia can also inhibit the anaerobic process and hamper overall efficiency of the system. Ammonia-N concentrations over 1,500 mg/L can be toxic to a reliable anaerobic treatment process at higher pH (i.e., > 7.4) (Labatut & Gooch, 2014).

2.4.5 Temperature

The optimal temperature for anaerobic digestion system working in mesophilic conditions is considered 37°C (Van Lier et al., 1997). The digester temperature should be well maintained between 35°C and 40°C even keeping a safer margin level. The operation of system beyond the normal range might adversely affect the performance leading to lesser biogas production from less organic matter stabilization (Labatut & Gooch, 2014).

2.4.6 Biogas Production

The biogas yield is the most important performance parameter to be monitored in anaerobic systems. Biogas is composed of methane, carbon dioxide, and traces of ammonia nitrogen, hydrogen sulfide, and other gases (Labatut & Gooch, 2014).

Methane is the final end product of anaerobic treatment, and its yield is a reflection of digester's performance. The amount of methane production is directly co-related to the amount of stabilized organic matter (VS). A carefully monitored anaerobic process should yield consistent biogas. The drop in the biogas production is a close indicator of reactor's upset (Labatut & Gooch, 2014).

2.4.7 Methane Content

Biogas is mainly composed of two gases; methane (CH_4) and carbon dioxide (CO_2). The proportion of methane can be 58 – 65% in the treatment of dairy manure with the remaining gas mainly composed of carbon dioxide (Labatut & Gooch, 2014).

The co-digestion of manure with some additional substrate may yield more useful gas fractions. A stable content of methane content in the produced biogas is an indication of successful reactors operation while a steady drop of methane below the digester's average daily values is usually an indicator of a digester problem. However, the intermittent feeding of the substrate may also lead to un-steady biogas production (Labatut & Gooch, 2014).

2.4.8 Volatile Solids (VS)

The organic matter of the waste can be measured from the amount of total volatile solids in that waste. The amount of organic matter stabilized is associated with the system configuration and physicochemical characteristics of the substrate. The percent of stabilized VS in the digesters treating manure lies in the range of 30-42% (Gooch et al., 2011).The percent stabilization of the waste can be higher employing the co-substrates, but its magnitude depends on the types of co-substrates employed (Labatut & Gooch, 2014).

2.5 Anaerobic Treatment of Low Strength Domestic Wastewater

A two-stage anaerobic system was used for domestic wastewater treatment under temperatures from 21- 14º C. The (HRT) was varied from 5.7 to 2.8 h for the first stage (HUSB digester) and from 13.9 to 6.5 h for the second stage (UASB digester). The process gave a TCOD removal over 89%. The methane conversion of influent COD was 36.1%, and the plant's performance was observed to be directly influenced by raw wastewater concentration and the temperature the plant was operated (Alvarez et al., 2008).

An anaerobic filter reactor of 3.9-L was used to evaluate the treatment potential of low-strength wastewater over 32 months at temperature of 25.4ºC and 2 months at 15.5ºC, respectively. Different types of synthetic wastewaters were prepared and fed to the reactor with COD 325–403 mg/L, soluble COD (SCOD) 86–339 mg/L and total suspended solids (TSS) 65–156 mg/L at organic loading rates (OLR) ranging from 0.02 to 0.91 kgCOD m^3/d. A sugar-nutrient based supplement was used at OLR 0.27–0.91 kgCOD m^3/day which gave removal efficiencies in the range of 72–80% for COD and 80–92% for TSS. The optimum yield of biogas was found to be 63 mL/L at 1.0-d HRT with average organics to CH_4 conversion 0.067 m^3/kg COD removal (Manariotis & Grigoropoulos, 2003).

Another study was to develop the pilot plant model to find the optimum conditions for the treatment of municipal wastewater. The reactor was built having two anaerobic compartments (tanks) filled with fixed bed media plastic material making total volume of 0.85 m^3 and was operated on a flow of 5 m^3/day and total HRT of 69 hrs. The performance was observed operating the reactor on various organic loading rates (2.53, 1.95, 1.60 and 1.35 kgCOD/m^3-d) for 16 weeks. Based on the experimental results and model predictions, the removal efficiency fluctuated slightly while shifting the OLR from 1.60 to 1.35 kgCOD/m^3-d. Hence, the OLR of 1.60 kgCOD/m^3-d was proved to be the optimum one to have the better removal efficiency of the system (Shafi et al., 2009).

2.6 Limitations of Conventional Anaerobic Systems

Extensive modifications were made in last decade to UASB systems to widen their applicability to treat the various types of textile and industrial wastewaters. For many types of wastewaters, the conventional UASB systems showed limitations owing to problems related to mass transfer resistance and concentration gradients which usually build up inside the system. The result is usually the drop in the biogas production in the treatment of low strength wastewaters at cold temperatures, so mechanical mixing must be applied to have required mass transfer which could lead to better functioning of the systems (Rebac et al., 1999). Furthermore, the presence of concentration gradients can hinder the treatment of wastewaters containing protein (Rinzema et al., 1989), or biodegradable toxic substances, such as formaldehyde (Zoutberg & de Been, 1997).

The research brought the concept of fluidized bed (FB) reactor which can eliminate mass transfer limitations but the stability of biofilm in FB systems can be an issue, which is created as a result of biofilm segregation adjacent to inert packing material. Moreover, these systems may require high energy for biofilm to be kept in suspension.

2.7 Anaerobic Membrane Bioreactor and Its Developments

With upcoming research and application insights from MBRs (Santos et al., 2011), the advances in the research of anaerobic membrane bioreactor (AnMBR) technology have also started as alternative to conventional anaerobic technologies for municipal wastewater treatment (Martin et al., 2011).

In AnMBRs, membranes provide best suitable conditions for the degradation of organic matter without the reactor being carried over by suspended solids. Using membranes in anaerobic municipal wastewater treatment systems, superior quality of the effluent can be achieved in terms of COD, suspended solids and pathogens in comparison with conventional anaerobic processes, and a sustainable treatment performance can be ensured to meet stringent effluent discharge standards (Ho & Sung, 2010; Kocadagistan & Topcu, 2007).

It was supported from the literature that AnMBRs can provide an option for the agricultural use of the effluent and can effectively play a role in the region of water shortage (Martinez-Sosa et al., 2011b). But pathogen removal with macronutrients availability to a certain degree is demanded for the agricultural use of treated effluents. Since macronutrients such as nitrogen and phosphates cannot be completely removed by anaerobic processes while pathogens can reasonably be removed by the membrane unit (Ellouze et al., 2009; Saddoud et al., 2006).

2.7.1 Membrane configurations in AnMBR

Various researches conclude that treatment efficiencies obtained in AnMBRs are governed by the process configuration (Liao et al., 2006). The variations in system configuration of anaerobic bioreactors, including completely stirred tank reactors (CSTR), up-flow anaerobic sludge blanket (UASB), expanded granular sludge bed (EGSB), etc., have been studied and investigated configuring with various types of membranes. However, the most suitable process configuration, i.e. type of anaerobic bioreactor and the coupling of the bioreactor and membrane module yet need to be studied more considering all the parameters affecting performance (Smith et al., 2012).

2.7.2 Integration of Membrane with Different Reactors

Membranes are integrated to various anaerobic systems such as CSTRs, EGSB, and UASB reactors. The studies on AnMBRs with different reactor types have been conducted so far for the treatment of different types of wastewaters.

2.7.2.1 Completely stirred tank reactor (CSTR)

By far, CSTR is the most commonly used anaerobic process researches have been carried on in AnMBR systems (Kocadagistan & Topcu, 2007; Martinez-Sosa et al., 2011a). In CSTR, biomass can also be enhanced by using a secondary clarifier with recirculation, making it an anaerobic contact process. Simple flow through CSTRs without sludge separation generally have large reactor volumes because of low loading rates. However, in CSTRs coupled with membrane reactors, there is complete separation of solids and effluent in the reactor which decouples SRT and HRT leading to an increase in biomass concentration. The CSTRs are also coupled to external side stream membranes, which can create higher liquid turnover rates, and yet a well-mixed flow regime. Consequently, the prevailing high shear and rapid mixing can increase the methane production potential of the substrate from an AnMBR set-up (Liao et al., 2006).

On the other hand, membranes are exposed to bulk sludge in CSTRs, resulting in rapid membrane fouling which yield low fluxes (Liao et al., 2006). The influent with high solids loading when subjected to membrane separation in the reactor further enhances cake deposition in CSTR configuration. Moreover, there is a decrease in the mean particle size when sludge recirculation is applied through the feed pump, in side stream membrane configuration (Choo & Lee, 1998). The reduction in particle size can affect both way; on one hand, finer particles can positively affect hydrolysis, but conversely it may impact negatively on the juxtaposition of methanogens and acetogens, limiting the required specific methanogenic activity (SMA) (Brockmann & Seyfried, 1997).

A study on AnMBR system with an external ultrafiltration (UF) membrane coupled with CSTR for the treatment of municipal wastewater produced an effluent to be re-used for agricultural irrigation (Martinez-Sosa et al., 2011a). Another process, i.e. vibrating membranes integrated with CSTR, were proposed for municipal wastewater treatment. The removal efficiency was significant for total organic carbon (TOC) with an average removal of 92%. Subsequently, reverse osmosis (RO) as post-treatment was applied which concentrated the nutrients for further reuse for agricultural purposes (Grundestam & Hellström, 2007). Another study reported high removal efficiencies for COD for the treatment of synthetic municipal wastewater in membranes coupled to CSTR (Ho & Sung, 2010). Another study using hollow fiber membranes in submerged mode in an AnMBR system integrated to a CSTR achieved a COD removal efficiency of 90% at 10 L/m^2 h flux (Giménez et al., 2011).

2.7.2.2 High-rate anaerobic reactors

In high-rate anaerobic systems, biomass is evolved by the formation of thick flocculent sludge or it grows by attaching to a supporting material in case of sludge bed systems and anaerobic filters. They are able to take higher hydraulic loadings as the larger portion of influent SS is retained in the system resulting in significantly lower effluent SS concentration. For example, sludge bed systems are known to have total suspended solids (TSS) ranging between 20 and 40 kg/m^3 (van Lier et al., 2008). The biomass is not subjected to membrane filtration in these reactors and hence prone to less membrane fouling owing to lesser production of cake layer as compared to that in CSTRs integrated with membrane modules. Therefore, high rate anaerobic reactors prove to be better option to integrate with membranes for low SS concentration of effluent and biomass retention in the reactor or if the toxicity to municipal discharge systems is present, or to cater hydraulic overload events (Liao et al., 2006).

2.7.2.3 Upflow anaerobic sludge blanket reactor (UASB)

In UASB reactors integrated with membrane unit, sludge bed entrap particulate matter by adsorption and biodegradation and the influent concentration of suspended solids to membrane is reduced significantly (An et al., 2009b; Kataoka et al., 1992; Wu et al., 2009).

The biological processes occur inside the sludge bed in the lower part of UASB reactor. In fact, UASB reactors coupled to membrane prevent the membrane being excessively fouled as the high amount of suspended solids is retained in the UASB reactor which serves as the upfront as biofilter. In a study on UASB reactor, biomass concentration of 20–30 g/L was found while the suspended solids concentration was below 1 g/L in the effluent (Kleerebezem & Macarie, 2003). Another study reported the TSS concentrations of 11–32 g/L in a UASB reactor; while total solids (TS) concentration less than 50 mg/L was obtained in the effluent. In UASB reactors coupled to membrane, HRT and upflow velocity govern the efficiency of the system and membrane fouling propensity. UASB reactors also allow the application of long SRT and higher OLR without the effluent solids concentration being increased that has later to be subjected to membrane filtration. There are many researches determining the fouling trend of AnMBRs including UASB systems integrated with membranes.

The hydrolysis of the entrapped particulates for municipal wastewater treatment can become the rate-limiting step leading to the accumulation of solids in sludge which can result in activity loss under sub-mesophilic conditions (Lettinga et al., 2001). According to theresults, only the finer particles find out the operational flux of the membrane (Jeison et al., 2009a; Jeison et al., 2009b). Hence, the extent of small particles retention in sludge bed is of prime importance in declaring coupled membrane filtration as feasible option. In some studies, membrane filtration was used as polishing step after UASB systems without employing concentrate recirculation (Herrera-Robledo et al., 2011; Herrera-Robledo et al., 2010; Salazar-Peláez et al., 2011). These system configurations can be considered as tertiary filtration. This approach allows easier control of hydraulics in UASB system. However, the membrane can be exposed to higher concentration of suspended solids as it is concentrated in the concentrate collection tank (Ozgun et al., 2013).

2.7.2.4 Expanded granular sludge bed reactor

UASB reactors sometime have poor mixing regime operating at low temperature for the treatment of sewage which leads to a decrease in the efficiency for soluble COD removal. Various studies have proven that EGSB reactors can serve as best alternative, especially for low strength wastewater treatment at ambient temperatures, resulting from the enhanced contact of biomass and substrate resulting from high up-flow velocity (De Man, 1988; Kato et al., 1997; Lettinga et al., 1999). In addition to that, EGSB reactors can also work on higher OLRs as compared to UASB systems. However, suspended solids are not effectively separated from the wastewater which can lead to sludge washout in the system due to high up-flow velocity. Anaerobic treatment by EGSB systems is only limited to pre-settled sewage. According to a study, a membrane unit was proposed as alternative to pre-settling which can retain the suspended solids effectively inside the EGSB reactor. A high applied upflow velocity produced shear stress which helped in the significant reduction of membrane fouling submerging the membrane in the upper part of the EGSB reactor (Chu et al., 2005). Nonetheless, achievable flux from the hollow fiber membranes was determined based on cake layer resistance. The use of EGSB reactors based AnMBRs have proved as a vital technology for the treatment of municipal wastewater even at ambient temperature (Chu et al., 2005). However, proper membrane addition can eliminate the hydraulic pressure which is needed for granules formation, by eliminating the wash out of sludge having poor characteristics. Therefore, granulation is not observed in EGSB systems integrated to membrane unit, which decreases the settling ability of the biomass in long term operation.

2.8 Factors Affecting the Treatment Performance of AnMBR

The optimization of AnMBR processes relates with the improvement of biological efficiencies, or increasing the activity of the membrane separation process (Dereli et al., 2012; Visvanathan et al., 2000). Few researches have also been carried out in the combination of both processes (Chu et al., 2005). In subsequent paragraphs the effect of operational factors on the system, including temperature, HRT, OLR, and upflow velocity in both physical and biological contexts are further discussed.

2.8.1 Operational conditions

2.8.1.1 Temperature

The temperature puts a vital role in governing the efficiency of the biological treatment. Biological processes show significant decrease in efficiency if the temperature decreases, ultimately leading to decrease in COD removal efficiency. Temperature also has a strong effect on biogas solubility in the effluent, solubility of (in)organic compounds, and settlement of biological solids due to the change of water viscosity (Tchobanoglous et al., 2003b).

A study was conducted investigating the effect of temperature on the efficiency of EGSB reactor coupled to membrane unit. The results showed the rate of anaerobic conversion process is strongly dependent on the temperature and a decline in COD removal efficiency was observed when the temperature was reduced from 25ºC to 11ºC. However, the performance of the membrane for COD removal efficiency increased from 8.8% to 14.2% even at reduced temperatures (Chu et al., 2005).

This is in accordance with the findings where the effect of temperature was observed for municipal wastewater treatment by AnMBRs. Two similar AnMBRs were operated in parallel for 112 days at a temperature of 25 and 15ºC, respectively. They reported that the physical removal capacity of the membrane increased at 15ºC as higher rejection of soluble organics was observed but the biological removal rate and SMA decreased (Ho & Sung, 2010). Another study demonstrated the AnMBRs worked well at temperature variations between 12and 26ºC for municipal wastewater treatment. The COD removal at 12ºC was observed to be 88% (Wen et al., 1999). Another study investigating the effect of temperature on methane recovery in an anaerobic externally submerged MBR found COD removal efficiency close to 90% for the treatment of municipal wastewater. The temperature was maintained at 35 and 20ºC, respectively. A lower methane recovery was observed at 20ºC which can be due to higher solubility observed at lower temperatures while methane proportion of the biogas was observed to be higher at low temperatures. The observed trend can be due to the difference in the solubility of CO_2 and CH_4 at 20ºC compared to 35ºC. An increase from 80 to 88% in the gaseous methane was found as the major portion of CO_2 left the reactor being more soluble at 20ºC (Martinez-Sosa et al., 2011a).

2.8.1.2 Organic Loading Rate (OLR)

AnMBR processes can tolerate the changes in organic loading as they can tolerate the fluctuations in temperature. The OLRs in the range of 0.3 to 12.5 kgCOD/m^3-day have been applied in AnMBRs for municipal wastewater treatment. The excellent treatment efficiency of AnMBR was confirmed while operating at OLR between 0.5 and 12.5 kgCOD/m^{3-} day, unlike causing perturbations in conventional anaerobic reactors (Wen et al., 1999). Accordingly, another study confirmed the stability of the effluent COD in spite of the variations in influent COD. Moreover, the biogas yield observed to be increasing linearly with an increase in the systems organic loading (Lin et al., 2011).

2.8.1.3 Hydraulic Retention time (HRT)

HRT is considered important parameter as it governs capital costs, with the notion that shorter HRTs can allow reactors with small volumes (Salazar-Peláez et al., 2011). Therefore, various investigations have been made to check the effect of HRT on biological removal in AnMBRs for municipal wastewater treatment. A study on AnMBR operating at 35ºC concluded the increased COD concentrations both in the effluent and in the reactor with the decrease in HRT of the system and increase in organic loading (Hu & Stuckey, 2007). Another study was undertaken to see the treatment performance of an EGSB reactor coupled to membrane while operating at various temperatures and same HRT. It was observed that COD removal was not affected by HRT at temperatures higher than 15ºC (Chu et al., 2005). However, the COD removal observed to be increased with increasing the HRT at 11ºC, demonstrating the importance of HRT at lower temperatures (An et al., 2009b). Another study summarized that the TOC removal efficiency is increased of membrane coupled UASB reactor when the HRT was reduced from 10 h to 5.5 h. The result was due to improved wastewater distribution in the sludge bed and enhanced contact of biomass and substrate found at higher upflow velocity (Chu et al., 2005). Based on studies results, it can be summarized that the operational parameters like system hydraulics, wastewater characteristics and sludge properties determine the optimized HRT of each process in terms of sustainable filtration performance.

2.8.1.4 Sludge Characteristics

Biomass characteristics including the proportion of slow growing bacteria, and their nutrition are mainly determined by operating conditions of the bioreactors (Kataoka et al., 1992). This might be because of cell lysis at high shear or juxta-positioning disruption of hydrogenotrophic methanogens and hydrogen producing bacteria, which ultimately enlarge the hydrogen transfer distance between species. On the contrary, methanogenic and acetogenic activities were preserved in AnMBR system with crossflow mode having liquid velocities of 1– 1.5 m/s and gas upflow velocity of 0.1 m/s. SMAs of AnMBR sludge in which propionate was used as the substrate were observed to be better than that of a parallel operating UASB system (Jeison et al., 2009b). Some other studies have also focused on differences in the composition of microbes for the treatment of domestic wastewater (Gao et al., 2010; Ho & Sung, 2010). The SMA of the biomass attached to the membrane surface was found to be lowered compared to that of AnMBR sludge to treat municipal wastewater. Thus, no significant difference was found between attached sludge and suspended sludge for the biological efficiencies (Ho & Sung, 2010).

2.8.1.5 Addition of Adsorbents

The adsorbents like PAC and zeolites are used in AnMBRs to adsorb organic compounds. This technique has widely been used to reduce organic fouling, and results in enhanced membrane flux and affects biological treatment (Akram & Stuckey, 2008). Another study determined PAC and GAC effect on the performance of AnMBRs. The efficiency of PAC was much more significant than that of GAC in terms of COD removal. The trend might be ascribed to larger surface area per unit mass of PAC compared to less surface area to mass ratio of GAC, thus attachment of colloids and molecules to PAC is significant from the bulk solution. The absorption of VFAs on the activated carbon was found to be very limited. Interestingly, the SMA increased in the presence of activated carbon in the reactor and hence the sludge growing in the reactor with carbon addition gave highest performance. This enhanced SMA is due to the support surface which is obtained from activated carbon which helps the biomass protect from shear conditions (Hu & Stuckey, 2007). Besides, dissolved organic carbon (DOC) was also found to be decreasing with PAC addition resulting from adsorption of high molecular weight entities on its surface (Vyrides & Stuckey, 2009).

2.9 Membrane Fouling: Controlling and Cleaning

In AnMBR, the SMP proportion of mixed liquor is almost in a same range as in the case of aerobic MBR, but severe membrane fouling can be observed (Berube et al., 2006). Small floc sizes and different chemical properties of SMP can play a role to further exacerbate the cause. The sustainable flux of submerged membranes has been found out to be comparably less in case of AnMBRs as compared to that in aerobic MBR even when the same air bubbling rate is applied. The low flux can be a drawback of AnMBR because membrane flux is inversely related to surface area and membrane scouring energy. In a recent study (Dagnew et al., 2014), a stable flux of 6 to 10 LMH was obtained at 38.5°C for the treatment of refinery, petrochemical, or potato processing wastewaters using lab to pilot scale MBR systems equipped with submerged hollow fiber membranes (ZW500d®, GE). The wastewater source and tests purpose determined the operational MLSS and it was varied between 4.2 and 17.5 g/L.

Several efforts have been made to reduce membrane fouling using floating biocarriers directly contacted with membrane (Bae et al., 2013; Yoo et al., 2014; Yoo et al., 2012). The biocarriers are fluidized by two methods; recirculating produced gas through diffusers or up-flow velocity by recirculating the reactor contents through external loops in AnMBR. Biocarriers significantly reduce SMP concentrations by adsorbing them and provide enhanced contact with microorganisms. In a laboratory study, a tall fluidized AnMBR column having 50 cm height and 2.5 cm diameter was used for the treatment of municipal wastewater. The total surface area of the submerged hollow fibers was 0.0215 m^2, and 30 g of dimension 10 × 30 mesh granular activated carbon (GAC) was used as a biocarrier. The municipal wastewater was filtered by 2 mm screen and fed to the column, while the HRT was maintained at 2.3 h. The anaerobic broth through side-stream loop was recirculated at 0.75 L/min which generated cross-flow on the membrane surface and fluidized GAC particles. The filtration was observed to be sustainable at a flux of 9 LMH at temperature range of 10 to 25°C, which can be taken as the high end of AnMBR with submerged membranes (5–10 LMH). In the same experiment, the removal efficiencies for BOD and COD were found to be 94 and 89%, respectively while the sludge yield remained between 0.01 to 0.03 kg VSS/kg COD.

The common strategies to lessen membrane fouling rate and recover sustainable operation have been investigated: membrane surface modification (Bae et al., 2006), membrane relaxation (Wen et al., 1999), gas sparging (Huang et al., 2011), and control of reactor operational parameters such as SRT, HRT and the concentration of biomass (Liao et al., 2006), and addition adsorbents like carbon, coagulant or flocculent (Baêta et al., 2012).

One method of reducing fouling rate is operating the membrane below its critical flux. The concept of critical flux was presented over 15 years ago (Field et al., 1995) and is defined as the flux operating below which no fouling occurs. However, fouling cannot be avoided in complex systems because fouling occurs even when the membranes come in contact with the bulk sludge. Consequently, the critical flux is now defined as the flux above which there is non-linear relation between flux and TMP (Jeison, 2007). Several methods have been proposed to determine critical flux (Tiranuntakul et al., 2011) including the flux-step method that can easily be applied in process involving membrane (Le Clech et al., 2003). A certain flux for long term operation is only referred when it is below the critical flux with acceptable value of dTMP/dt, and gains tolerable fouling levels for the operation of MBRs for longer periods of time (Jeison & Van Lier, 2006). The stable operation of membranes can also be maintained by membrane relaxation (flux stoppage) that is generally applied to ensure diffusive back transport of fouling materials away from the membrane surface when concentration gradient is present. The back transport can also be enhanced by gas scouring of membrane surface and the resulting shear force.

Studies have been made to modify the suspension properties and testing them to control membrane fouling which could enhance membrane filterability. The addition of powdered/granular activated carbon (PAC/GAC), membrane flux enhancer, or zeolite makes better flocs of activated sludge, entraps the soluble compounds, and leads to the better formation of cake layer (Akram & Stuckey, 2008; Wu et al., 2009). The better flocs formation of sludge on the surface of PAC results in less accumulation of biomass over membrane surface leading to less fouling (Hu & Stuckey, 2007). It was also observed that the lower amount of PAC addition (1.67 g/L) can enhance membrane performance while the addition of higher dosage (3.4 g/L) makes the sludge more viscous and consequently leading to lower flux (Akram & Stuckey, 2008). The coagulants which are used in water treatment can also be applied into MBRs for the removal of colloids and soluble organics, and reduces fouling (Meng et al., 2009). The ultrasound technology has also proved to enhance membrane permeability in AnMBRs (Xu et al., 2011). However, the irradiations from ultrasonic technology can also affect negatively anaerobic bacteria (Sui et al., 2008). A number of new techniques to alleviate membrane fouling of AnMBRs are being investigated. For instance, sludge can also be removed from the membrane by the application of electric repulsive force, as the surface of the activated sludge is negatively charged (Akamatsu et al., 2010).

2.10 Application of AnMBR for Treatment of Different Wastewater Types

2.10.1 Municipal Wastewater Treatment

Municipal wastewater is considered as low-strength wastewater as the chemical oxygen demand (COD) ranges from 250–800 mg/L; for which aerobic activated sludge process is used (Liao et al., 2006). The large quantity of municipal wastewater generated puts the pressure for its high-rate treatment but at low temperatures and short HRTs (Smith et al., 2012). Aerobic MBRs have extensively been used for municipal wastewater treatment. The treatment of low-strength wastewater, in recent years, has been investigated with growing interest in AnMBRs as they have the potential to recover energy (biogas) (Berube et al., 2006; Smith et al., 2012). In addition to that, the presence of membrane prevents the sludge being washed out ensuring a long SRT. The sufficient concentration of biomass is maintained for effective treatment of low-strength wastewaters, even at cold climates when biomass growth is hindered. The studies on application of AnMBR for municipal wastewater treatment are summarized in Table 2.1. In order to reduce the heating cost for the operation of AnMBRs at mesophilic or thermophilic temperature, AnMBRs were operated at the ambient temperatures and the results showed COD removal of above 80% (An et al., 2009a; Martinez-Sosa et al., 2012). At higher OLR of 2.36 kgCOD/m^3-day, the COD removal was reasonable with the effluent COD concentration of 77.5 mg/L (An et al., 2009a). Moreover, another study operated the AnMBR and an aerobic MBR under same operating conditions and the results inferred similar COD removal for domestic wastewater treatment, although the acclimatization period of AnMBRs was longer compared to aerobic MBRs (Achilli et al., 2011; Baek & Pagilla, 2006). On the other hand, anaerobic biomass may not have the potential to acclimatize and grow under very low wastewater strength, and the presence of toxicity (Saddoud et al., 2009; Saddoud et al., 2006). AnMBRs offers the potential for energy recovery, but the drawback of methane being dissolved in the effluent while treating low-strength wastewater treatment is possible. A study found out almost 50-70% of the total methane yield exited remained in gas phase (Lefebvre et al., 2011; Smith et al., 2011). Several methods like air stripping, degassing of membrane, and hanging sponge reactor with down-flow have been attempted for the recovery of dissolved methane, but there has been limitation regarding their economical and practical feasibility (Bandara et al., 2011; Hatamoto et al., 2010; McCarty et al., 2011). Only a few studies on pilot-scale AnMBR have been reported for municipal wastewater treatment (Calderón et al., 2011; Dagnew et al., 2011; Giménez et al., 2011). These pilot-scale studies indicate the viability of AnMBRs for municipal wastewater treatment.

Table 2.1. Case studies on municipal wastewater treatment

Type of reactor	Configuration, Scale and volume	Characteristics of membrane	Flux (L/m^2h)	Temp (C^0)	HRT (d)	SRT (d)	OLR (kg COD/m^3 d)	MLSS (g/L)	Feed COD (mg/L)	Effluent COD (mg/L)	COD removal
CSTR/AnMBR	Submerged, P (1.3 m^3)	Hollow fiber UF membrane, pore size 0.05 µm	20	33	0.25-0.83	70		6-22	445±95	77	87%
CSTR	Submerged, P (350 L)	Flat sheet polyether sulfone UF membrane, pore size: 38 nm	7	35/28/20	0.8	680	0.5-1.1	15-21	630±82	80	<90%
CSTR	Submerged, P (630 L)	Hollow fiber ZeeWeedTM	17	22	0.35	80-100			224	<47	79%
UASB	P (849 L)	PVDF, tubular UF membrane, MWCO: 100 kDa	45-50	22	0.25	180			445±138	33	93%

2.10.2 Industrial wastewater treatment

Mostly anaerobic treatment is preferred for high strength wastewaters for which aerobic processes are not viable. Industrial wastewater generally has high organic strength (1–200 g COD/L), incurs variable temperatures, and different levels of salinity, suspended solids, turbidity, and heavy metals (Lin et al., 2012). Additionally, the presence of some elements in the industrial wastewaters may hinder anaerobic degradation. The harsh process conditions induced by these factors can limit the applicability of anaerobic processes, where AnMBRs suit best for the treatment of industrial wastewaters (Dereli et al., 2012).

The treatment performance of AnMBRs is illustrated in Table 2.2, 2.3, and 2.4. The wastewaters from food processing industries are high in organic strength and readily biodegradable. The full-scale applications illustrated the merits of the process for the treatment of high-strength wastewaters and the production of a solids-free effluent.

Table 2.2. Case studies on food processing wastewater treatment

Type of wastewater	**Type of reactor**	**Configuration, scale and volume**	**Characteristics of membrane**
Cheese whey	Two phase (acidogenic/methanogenic) CSTR+M	External, L (5 L/15 L)	Ceramic membrane, pore size: 0.2 µm
Brewery wastewater	CSTR	External, L (4.5 L)	Tubular membrane module, ceramic (pore size: 0.2 µm) and polymeric (pore size: 30 num) membrane
Food processing and washing wastewater	CSTR	External, P (0.4 m^3)	PES UF membranes, MWCO: 20,000-70,000 Da
Snacks factory wastewater	UASB	Submerged, P (0.76)	Hollow fiber, PVDF, pore size: 0.4 µm
Food processing wastewater	CSTR	Submerged, P (20 m^3)	Rotating membrane module

Table 2.3. Case studies on various kinds of wastewater treatment

Type of wastewater	Type of reactor	Configuration, scale and volume	Characteristics of membrane
Petrochemical effluent	CSTR	Submerged, L (50 L)	Flat sheet, pore size: 0.45 µm
Slaughterhouse wastewater	CSTR	External, L (50 L)	UF membrane, MWCO: 100 kDa
Slaughterhouse wastewater	Two phase (Acidogenic/methanogenic) CSTR+M	External, L (50 L/50 L)	UF membrane, MWCO: 100 kDa
Thin stillage wastewater	CSTR+M	Submerged, P (12 m^3)	Flat sheet UF membrane, PVDF, pore size: 0.08 µm
Animal waste	CMAD+AnMBR	External, P (100 L/100 L)	Tubular UF membrane, PVDF, pore size: 0.03 µm

Table 2.4. Case studies on leachate treatment

Type of wastewater	Type of reactor	Configuration, scale and volume	Characteristics of membrane
Landfill leachate	CSTR	External, L (50 L)	UF, MWCO= 100,000 Da
Diluted landfill leachate	CSTR	Submerged, L (29 L)	UF membrane, pore size: 0.1 µm

2.11 Challenge, Constraints and Future Potentials of AnMBR

The existing anaerobic treatment systems can be upgraded to AnMBRs for municipal wastewater treatment which can serve as robust systems, especially when fine effluent quality and/or treated effluents to be reused are considered. AnMBR effluents can be used for horticulture purpose and are pathogen free (Ellouze et al., 2009; Saddoud et al., 2006). However, AnMBRs application in domestic utilities is needed to investigate more the process operational viability which is hindered by membrane fouling issues (Chu et al., 2005). Therefore, AnMBR systems can find extensive applications once technologies to prevent fouling are developed.

The precipitates of struvite ($MgNH_4PO_4$-.$6H_2O$), $K_2NH_4PO_4$ and/or $CaCO_3$ cause inorganic fouling which further is of greater concern as it is associated with the release of ammonia and phosphate from organic nitrogen and phosphorus during anaerobic digestion while the changes in carbon dioxide partial pressure and increase in alkalinity generation leads to associated fluctuations in pH in AnMBRs (Liao et al., 2006). Another study estimated the undersaturation of struvite with lesser concentrations of $NH4^+$, $PO^{2-}{}_4$ and Mg^+ comparative to industrial wastewaters leading to probable occurrence of struvite. However, membrane properties also play a vital role in struvite precipitates formation (Kang et al., 2002). Therefore, further investigations should be made into the process causing irreversible fouling with long term studies in AnMBRs for municipal wastewater treatment.

The reuse of treated municipal wastewater in horticulture is concerned with the sufficient removal of endocrine disrupting substances. The sewer systems can be alarmingly polluted when industrial discharges are made into them which also causes serious toxicity and results into severe shock loads in the wastewater treatment plants. The treatability of the combined effluents from municipal sewers including industrial discharges have also been studied in AnMBR connected to a cross-flow UF module. The results concluded the treatment to be inefficient, as reflected by the unstable biogas yield and varying composition, mainly caused by high fluctuations in the industrial toxicants (Saddoud et al., 2006). The residual toxicity in the treated effluents with AnMBRs has been found in another study also due to toxic compounds found in industrial discharges. However, micro-toxicity analysis revealed the significantly less toxicity of AnMBR permeate than of aerated lagoon and activated sludge (Ellouze et al., 2009). The studies addressing the treatment of endocrine disrupting chemicals present in municipal wastewater have also been made in MBR systems (Boonyaroj et al., 2012; Cases et al., 2011). Aerobic MBRs have proved well to treat the phenolic compounds, phthalates and estrogens in comparison to conventional treatment systems (Cases et al., 2011) but very little knowledge exists on the fate and biodegradation of the same in AnMBRs (Ho et al., 2007).

The coastal residential areas mostly deal with the salinity problem in sewage resulting from the infiltration of sea water into sewer systems from improper infrastructure which allows the stated problem. The treatment of saline municipal wastewater has also been examined by submerged AnMBR where fluctuations were found in salinity. The DOC removal at 35 g NaCl/L was observed to be 99% while the removal efficiency remained very low (40–60% DOC) inside the reactor. The results summarized that membrane rejection led to the accumulation of SMPs and colloidal COD in the reactor. But fouling control should be focused more in order to make AnMBR a feasible treatment technology (Vyrides & Stuckey, 2009).

Chapter 3

METHODOLOGY

The details of materials and methods used during the course of this research have been explained in this chapter. The contents include detailed reactor design, installation and optimization including sludge acclimatization, feed solution preparation and sample analysis etc. The chapter also highlights the operational conditions, procedures, and detailed specifications of the equipments used.

3.1 Experimental Set-Up: Bench Scale Anaerobic Membrane Bioreactor (AnMBR)

A lab scale anaerobic membrane reactor was established in Water and Wastewater Lab at Institute of Environmental Engineering and Sciences (IESE) NUST, Pakistan consisting of two acrylic reactors; anaerobic digestion tank (CSTR) followed by membrane tank having effective volume of 5 L and 6.5 L respectively.

The system consisted of 70 L feed tank, 1.5 L wastewater level controller for the anaerobic digestion tank (CSTR), 2 gas collection bags and effluent collection tank. The anaerobic digestion tank was provided with a mixer (Cole Parmer, Stir- Pak, model no 50002-20, USA) to continuously mix the contents of the tank and to provide a better contact of the sludge and the influent wastewater. A photo of the lab scale set up has been shown in Figure 3.1.

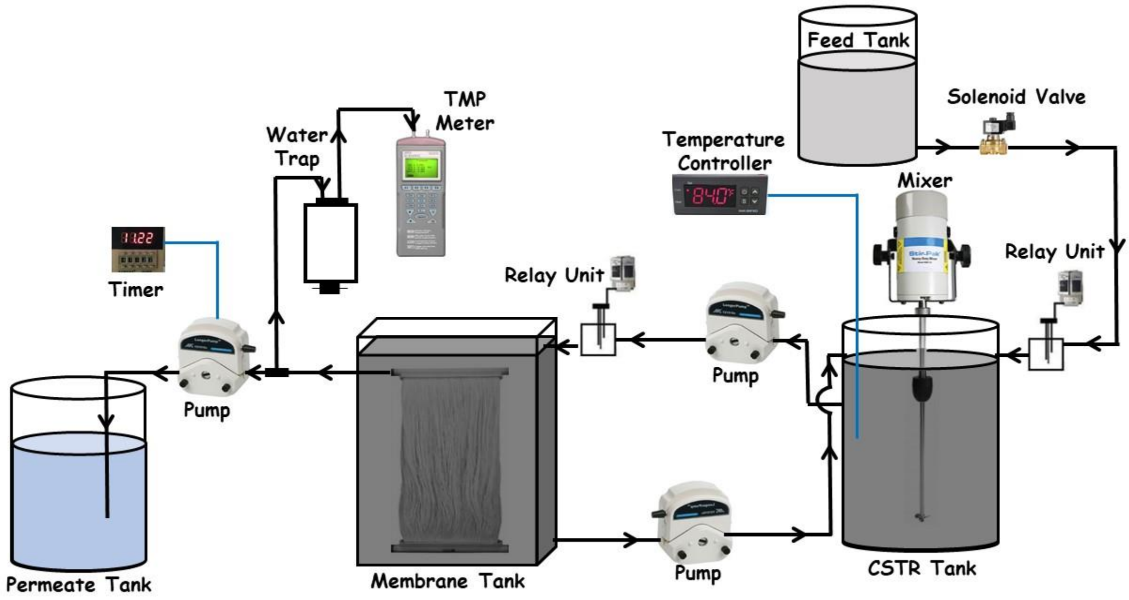


Figure 3.1. Schematic diagram of the lab scale AnMBR reactor

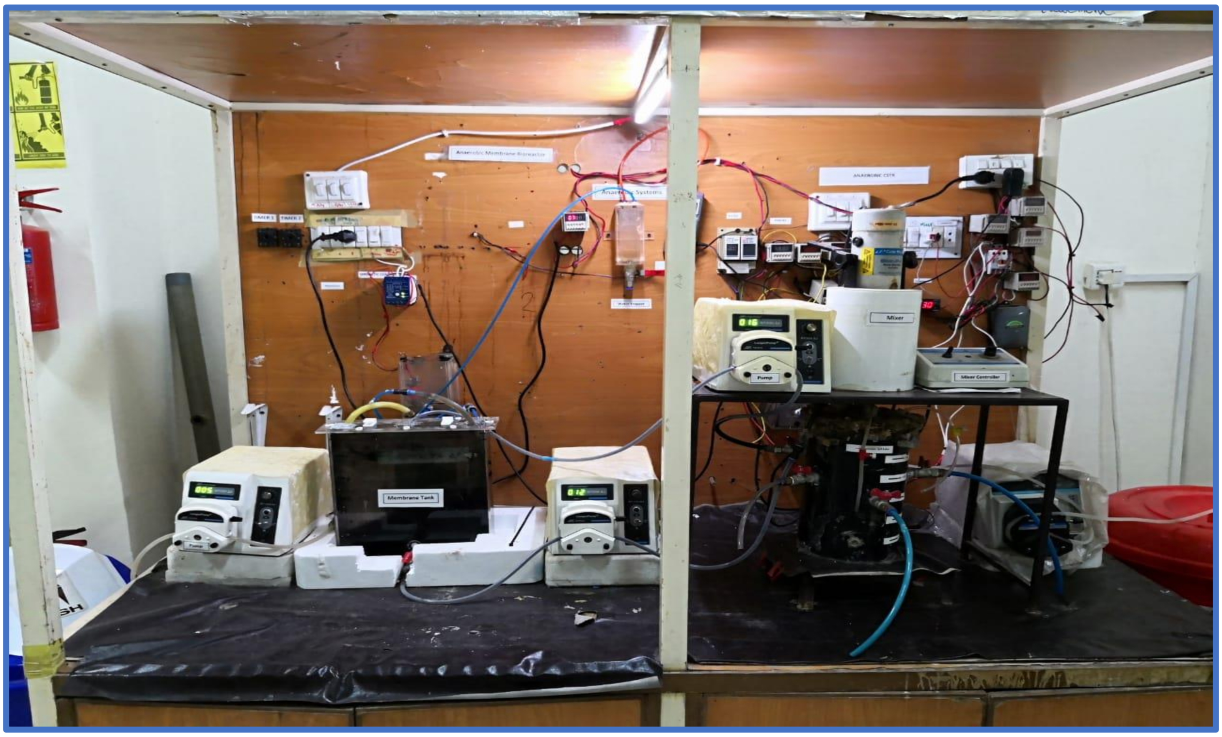


Figure 3.2. Lab Scale AnMBR Reactor

A hollow fiber PVDF membrane (Mitsubishi Chemical Aqua Solutions Co., Ltd., 50S0070SA, Japan) having filtration area of 0.073 m^2 with a pore size of 0.4 µm was used. Peristaltic pumps (Longer Precision Pump Co., Ltd, BT300-2J/YZ1515X, China) were used to continuously feed and draw the synthetic domestic wastewater from the system. Table 3.1 shows the detailed characteristics of the pumps used. The membrane tank had a pump working in operation cycle of 8 mins permeation and 2 mins relaxation for the membrane. The system was operated at different operating parameters in different phases as summarized in Table 3.2. Phase I to V were performed at 10.3, 8.8, 8.8, 6 and 6 LMH respectively with periodic maintenance cleaning required when the TMP in respective runs reached closer or equal to 300 mbar.

Table 3.1. Peristaltic pumps specifications

SPECIFICATION	VALUE
Speed	1 to 300 rpm, reversible
Speed resolution	1 rpm
Display	3-digit LED displays current speed
Power supply	AC 90 - 260V 50/60 Hz
Power consumption	< 48 W
Operating condition	Temperature 0 to 40℃ Relative humidity < 80%
Dimensions (L×W×H)	285×207×180 (mm)
Drive weight	3.6 kg

Table 3.2. Operating protocol used during AnMBR operation

OPERATING PROTOCOL	
PHASE	**CONDITIONS**
I	Flux= 10.3 LMH, R=1
II	Flux= 8.8 LMH, R=2
III	Flux= 8.8 LMH, R=3
IV	Flux= 6 LMH, R=2
V	Flux= 6LMH, R=3

Trans-membrane pressure was recorded using data logging manometer (Sper Scientific, 840099 15 PSI, USA) to note the membrane fouling tendency. The detailed characteristics of the manometer are specified in Table 3.3.

Table 3.3. Specifications of datalogging manometer

SPECIFICATION	VALUE
Accuracy	±0.3% full scale
Min pressure	0
Max pressure	15
Unit of measurement	psi, " WC, kPa, ft WC, " Hg, cm WC, mbar, bar, mm Hg, oz/in², kg/cm²
Battery type	Four AAA (included)
Media compatibility	Noncorrosive gases and liquids
Resolution	0.01 psi

3.2 Wastewater Composition

Synthetic wastewater was prepared using fresh tap water and adding into it major organic (macro) nutrients and trace (micro) nutrients. The synthetic wastewater was prepared as low strength domestic wastewater having concentrations of Dextrose 500 mg/L, Ammonium Chloride (NH4Cl) 191 mg/L, Potassium di-Hydrogen Phosphate (KH_2PO_4) 23.85 mg/L, Calcium Chloride ($CaCl_2$) 4.87mg/L, Magnesium Sulfate (MgSO4.7H2O) 4.87mg/L, Ferric Chloride (FeCl3) 0.5mg/L and Cobalt Chloride (CoCl), ZinC Chloride (ZnCl) and Nickel Chloride (NiCl) each 0.5 mg/L. The pH of the synthetic wastewater was maintained at 6.8-7.2 using Sodium Hydrogen Carbonate ($NaHCO_3$) 100 mg/L.

Seed activated sludge was collected from the full scale MBR plant (Capacity 50 m^3/day) at NUST Sector H-12 and was separately acclimatized to anaerobic conditions adding 500 mg/L Dextrose and 100 mg/L Sodium Hydrogen Carbonate ($NaHCO_3$)

respectively. The organic loading rate of 1 kgCOD/m^3-day was maintained in the anaerobic membrane bioreactor.

Table 3.4. Synthetic wastewater composition

Chemicals	**Unit**	**Concentration**
Dextrose (glucose)	mg/L	500
Ammonium chloride	mg/L	191
Potassium di hydrogen phosphate	mg/L	23.85
Calcium chloride	mg/L	4.87
Magnesium sulfate	mg/L	4.87
Ferric chloride	mg/L	0.5
Sodium hydrogen carbonate	mg/L	100
Cobalt chloride	mg/L	0.05
Zinc chloride	mg/L	0.05
Nickle chloride	mg/L	0.05

3.3 Experimental Conditions

The experimental conditions maintained in all the phases of the study have been elaborated below. The temperature of the system was maintained at 30°C throughout. During operation of reactor, the operating pH was continuously monitored and controlled using Sodium Hydrogen Carbonate ($NaHCO_3$).

Nitrogen purging was done frequently to bubble out the oxygen gas that went into the solution and also, it gave an opportunity to anaerobic microorganisms to reproduce faster. For purging, an inlet pipe from the nitrogen gas cylinder was inserted into the reactor (anaerobic digestion tank) and the gas valve was opened to let the gas flow at a moderate pressure. The gas was allowed to be moved outside opening a hole in the lid of anaerobic digestion tank for de-gassing. The purging was done for 5-7 minutes which ensured the complete anaerobic environment inside the reactor.

3.3.1 Phase I (Batch Study)

In the first phase of the study, the anaerobic digestion tank (CSTR) was optimized for the hydraulic retention time (HRT). The operating conditions and protocol are presented in Table 3.5 and Table 3.6. The digestion tank was operated for continuous 16 days at HRT of 1 day. The complete analysis of the samples was carried out for performance evaluation. The contents of the digestion tank were kept well mixed using mixer (Cole Parmer, Stir- Pak, model no 50002-20, USA).

Table 3.5. Working conditions for batch study

Working Conditions	
Operation Mode	Batch
Total Volume	6.5 L
Working Volume	5.5 L
COD	500 mg/L
HRT	24 Hours
SRT	Infinite
Volumetric Loading	0.5 kg/m^3-day
Temperature	32°C
pH	6.8-7.4

Table 3.6. Operating protocol for batch study

Operating Protocol	
Stage	Time
Feed	7.5 Minutes
React	23 Hours
Settle	45 Minutes
Decant	7.5 Minutes

3.3.2 Phase 2 (Batch Study)

The samples from digestion tank (CSTR) were taken at HRT of 6, 12, 18 and 24 hrs. Based on the laboratory experimental analysis, the HRT was reduced to 12 hrs. The digestion tank was then continuously operated for 24 days at optimized HRT of 12 hrs followed by laboratory experimental analysis. The operating conditions and operating protocol was kept same as in phase I of the study except volumetric loading (1 kgCOD/m^3-day) and react time (11 hours).

3.3.3 Phase 3 (Continuous Study)

The AnMBR was operated for 62 days consecutively at increasing HRT and decreasing flux while different sludge recirculation ratios were employed during each phase. Mode I to V were performed at 10.3, 8.8, 8.8, 6 and 6 LMH (litres/m^2 h) respectively with periodic maintenance cleaning required when the TMP in respective runs reached closer or equal to 300 mbar. The operational cycle adopted was 8 min permeation and 2 min relaxation to reduce membrane fouling. The net flux was calculated incorporating the periodic relaxation time of the membrane from the total filtration time of the membrane in each run. Sludge wasting was not performed in the whole operation cycle of AnMBR to maintain the infinite SRT of the system and to allow the growth and accumulation of biomass in the reactor.

The resistance test was performed before and after the start of each run to check the intrinsic resistance and cake layer and pore resistance respectively. The following equation from Darcy's law was used to measure the resistance:

$R_t = \Delta P / J.\mu.f_t$

Where:

$R_t = R_c + R_p + R_m$

Where R_t (1/m) is the total hydraulic resistance, R_c, R_p and R_m are cake layer resistance, pore blockage resistance and intrinsic resistance respectively. The cake layer resistance is mainly offered due to deposition of sludge flocs and colloids while the pore resistance is due to blockage of membrane pores by colloids and dissolved matter. J is the permeate flux, ΔP is trans-membrane pressure, μ is the dynamic viscosity of water, while $f_t = e^{-0.0239(T-20)}$ is the correction factor for temperature. The data obtained for TMP over filtration time was used to calculate the full resistance of the membrane (R_t). The value of R_p was calculated removing the sludge cake from the membrane surface and measuring the resistance filtering distilled water while R_m was calculated after chemical cleaning of the membrane filtering distilled water before the start of each run.

3.4 Sampling and Preservation

The samples were taken into the sampling bottles into the sampling bottles. Some parameters are continuously monitored during reactor operation to maintain the stability of the process like ph, temperature, volatile fatty acids (VFA) and alkalinity.

For other parameters, the samples were preserved labelling the sampling bottles in fridge at 40 °F or below so as not to degrade the samples.

The COD, total phosphates, nitrates, and nitrites tests were performed filtering the samples into beakers using conical funnel and Wattsman filter paper no. 1. The filtrate was then used for the analysis which helped uniform and accurate measurement among all the samples taken.

3.5 Analytical Methods

The effluent quality parameters analyzed include COD, phosphates, and biogas production while the stability parameters analyzed include volatile fatty acids (VFA), alkalinity, pH and temperature. The sludge was characterized measuring mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS). The reference of the methods used has been given in Table 3.7.

Table 3.7. Analytical tests and used methods

Parameter	**Method**	**Reference**
Chemical oxygen demand (COD)	Close Reflux Titrimetric Method	APHA et al. (2012)
VFAs and Alkalinity	Titration Method	APHA et al. (2012)
Biogas Production	Water displacement method	Patil et al. (2011)
MLSS/MLVSS	Filtration-Evaporation	APHA et al. (2012)
pH	pH meter	Cybesrscan 500

3.5.1 Chemical Oxygen Demand (COD)

COD determination for the effluent was carried out using closed reflux titrimetric method. A volume of 2.5 mL of sample, 1.5 mL of digestion reagent (K_2Cr_2O7) and 3.5 mL of sulfuric acid (H_2SO_4) reagent was added in COD vials, capped and placed in preheated ($150^{\circ}C$) COD digester for 2 hours.

The vials were then cooled at room temperature and the mixture was titrated with ferrous ammonium sulfate (FAS) using ferroin as an indicator. Following equation was used for the COD calculation:

COD (mg/L)= (A-B)*1000*8/Sample Volume (mL)

Where:

A= Volume of FAS used to titrate the sample (mL)

B= Volume of FAS used to titrate the blank (mL)

3.5.2 Volatile Fatty Acids (VFA) and Alkalinity

The samples from digestion tank (CSTR) were collected and the pH was measured by pH meter (Cyberscan 500). If the pH was above 6.5 then it was titrated with 0.1 N H_2SO_4 to bring its pH value to 4.3. The volume for acid consumption was measure and used for alkalinity calculations.

Following was used for alkalinity calculations:

Alkalinity (mg/L)= Volume of acid consumed*Normality of acid used*5000/Sample Volume (mL)

For VFA measurement, the same titrated sample was used. The beaker containing sample was then placed on heating plate and temperature was allowed to reach up to 70 to 80°C. It was then cooled down at room temperature. After that sample was titrated against 0.1 N NaOH until the pH of the sample reached to 6.5. The volume of NaOH consumed was measured and used for VFA calculations.

Following equation was used for VFA calculations:

VFA (mg/L)= Volume of alkali consumed*Normality of alkali used*5000/Sample Volume (mL)

3.5.3 MLSS/MLVSS:

Whattman filter paper was firstly dried in oven at 105°C for 30 minutes to remove moisture from filter paper if there was any. The filter paper was then placed in desiccator till it cooled down to room temperature and initial weight of the filter paper was measured.

10 mL sludge sample (collected during mixing phase of the digestion tank) was then filtered placing the weighed filter paper in filtration assembly.

When the water was completely removed from filter paper and a sludge cake was developed, the filtration assembly was switched off. Filter paper was then placed in china dish and was put in oven at 105°C for 1 hour. The china dish was taken out, filter paper allowed to cool down to room temperature and the weight was measured again. The MLSS was measured using the formula below:

MLSS (mg/L)= (A-B)*1000/Sample volume (mL)

Where:

A = Weight of filter paper + residues after drying at 105°C

B = Initial weight of filter paper

The measurement for MLVSS was carried out using the same filter paper. The oven dried filter paper containing residues was then placed in muffle furnace for ignition at 550°C for 30 minutes. The china dish was allowed to cool down to room temperature and the filter paper was then weighed. MLVSS in sludge sample was measured using following formula:

MLVSS (mg/L)= (A-B)*1000/Sample Volume (mL)

Where:

A = Weight of filter paper + residues after drying at 105°C

B = Weight of filter paper + residues after ignition at 550°C

Chapter 4

RESULTS AND DISCUSSION

4.1 Phase A: Anaerobic Digestion Tank (CSTR)

4.1.1 Effect of Hydraulic Retention Time (HRT) on COD Removal

The figure 4.1 (a) illustrates the COD removal of anaerobic digestion tank (CSTR) in the initial stages of reactor operation. The reactor was operated at 24 h HRT for 16 days and the constant organic loading rate of 0.5 kgCOD/m^3-day was maintained throughout. In the start, COD removal was low and then gradually increased with time as the microbes adapted to new conditions and a fed with constant loading.

At OLR of 0.5 kgCOD/m^3-day, the average COD removed was 64.43% while the effluent contained COD of 177.84 mg/L.

An experimental evaluation was conducted taking the samples at HRT of 6, 12, 18 hrs and it was observed that the COD removal showed a negative trend with the increase of organic loading rate of the system which was also observed in anaerobic co-digestion of food waste and domestic wastewater (Chan et al., 2017). The overall COD removal didn't affect much at HRT of 12 hrs due the action of methanogenic and syntrophic anaerobic microbial species as is depicted from the Figure 4.1 (b). Hence, the HRT was decreased to 12 hrs (optimized) leading to OLR of 1 kgCOD/m^3-day. This decrease of HRT was also supported from the other researches to prevent the possibility of washout and to ensure the sufficient number of microorganisms in the system (Bal AS, 2001; Smith et al., 2011).

In the second phase of reactor operation, the run was carried for consecutive 42 days until the effluent quality got steady state. The Figure 4.1 (c) represents the COD removal over the course of operation with an average of 64.78%. The removal of COD in this phase was also supported from other researches at a little greater HRT i.e 14.7 hrs for the treatment of olive mill wastewater and municipal wastewater (Gizgis et al., 2006). The decrease in HRT didn't affect the removal efficiency much and also, the trend of COD removal got better as is depicted from the figure 4.1 (a).

Overall the quality of anaerobic effluent at OLR of 0.5 and 1 kgCOD/m^3-day was 177.84 and 176.12 mg/L respectively. Therefore, the optimum HRT for anaerobic system in treating domestic wastewater is inferred as 12 hrs for maximum COD removal efficiency. Moreover, the effluent concentration of COD at optimized HRT reflect its need to be further treated to meet the NEQS i.e COD of 150 mg/L.

The overall effect of variation of reactors HRT in each phase was reduction in the operational performance of the system owing to the fact that it takes necessary acclimatization period for microbes to acclimatize to changing environmental conditions (Gangagni Rao et al., 2005).

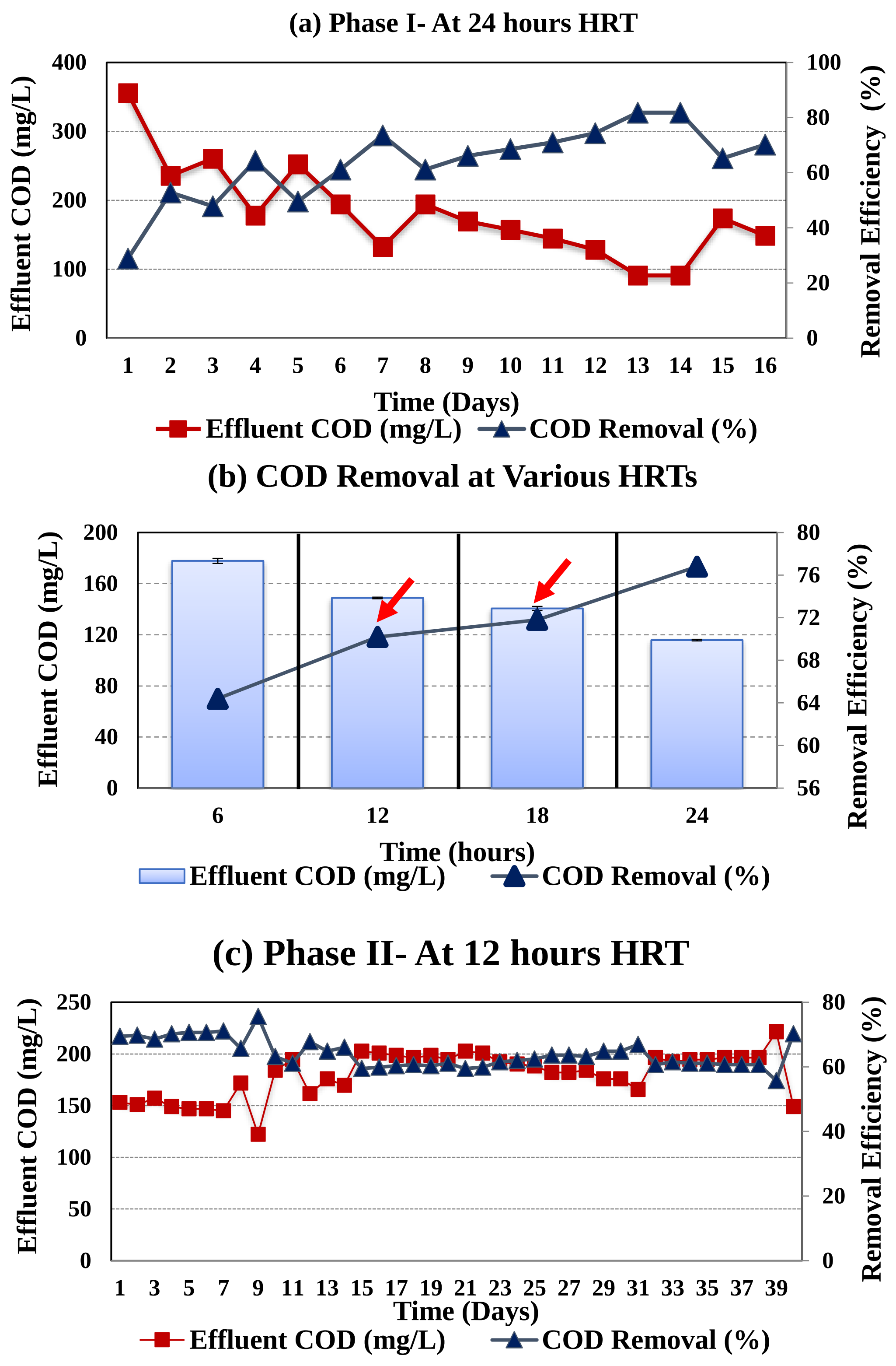


Figure 4.1. COD removal (a) at 24 hours HRT (b) at various HRTs (c) at 12 hours HRT

4.1.2 Effect of HRT on Stability Indicators and Biogas Production

In an anaerobic digester, a bicarbonate alkalinity of about 1000-3000 mg/L is required for a stable operation. In the first phase of study, at HRT of 24 hrs and OLR of 0.5 kgCOD/m^3-day, the presence of volatile fatty acids were higher (110-1040 mg/L) and to cater those relatively less concentration of alkalinity (1,250-2,500 mg/L) was present and hence the overall VFA/alkalinity ranged between 0.05-0.46. The same results having VFA/alkalinity ratio less than 0.3 were observed for the treatment of domestic wastewater by anaerobic up flow fluidized bed reactor (Moharram et al., 2016). The stability of reactor operation was kept supplemented y the addition of $NaHCO_3$ and adjusting the pH of the system. The result was improved stability of the process and hence the ratio VFA/alkalinity remained 0.15 and less. Moreover, fluctuating VFA/alkalinity ratio didn't affect the systems performance because of the better adaptation of microbial population involved in treatment performance.

In the first phase of the study, the biogas production was higher and showed a uniform trend in the range of 2.4 L/day to 2.7 L/day. The production of biogas has a close relation with the pH of the system as is illustrated in the Figure 4.3. A close and careful monitoring of the systems pH led to reliable and consistent performance in terms of biogas yield.

In the second phase of study, as the HRT decreased to 12 hrs and OLR increased to 1 kgCOD/m^3-day, the presence of VFAs were in the range of 128.6-290 mg/L while the concentration of alkalinity ranged between 525 and 592.5 mg/L. A consistent trend of VFA/Alkalinity ratio was observed which remained 0.22-0.55 and the operation was continuously monitored in the whole period.

Methanogenesis in particular is known to be hampered as a result of VFA/alkalinity ratio above 0.3 (Lefebvre et al., 2006) and hence leads to low production of biogas but in this case, even the microbes at higher VFA/Alkalinity ratio gave high biogas yield. The biogas yield increased to 5.15 L/day (average) and was found to be consistent at optimum HRT of 12 hrs (OLR 1 kgCOD/m^3-day) as the further increase in organic loading leads to increase of VFA and inhibits the methanogenic bacteria which in turn leads to decline in the biogas yield (Elango et al., 2007).

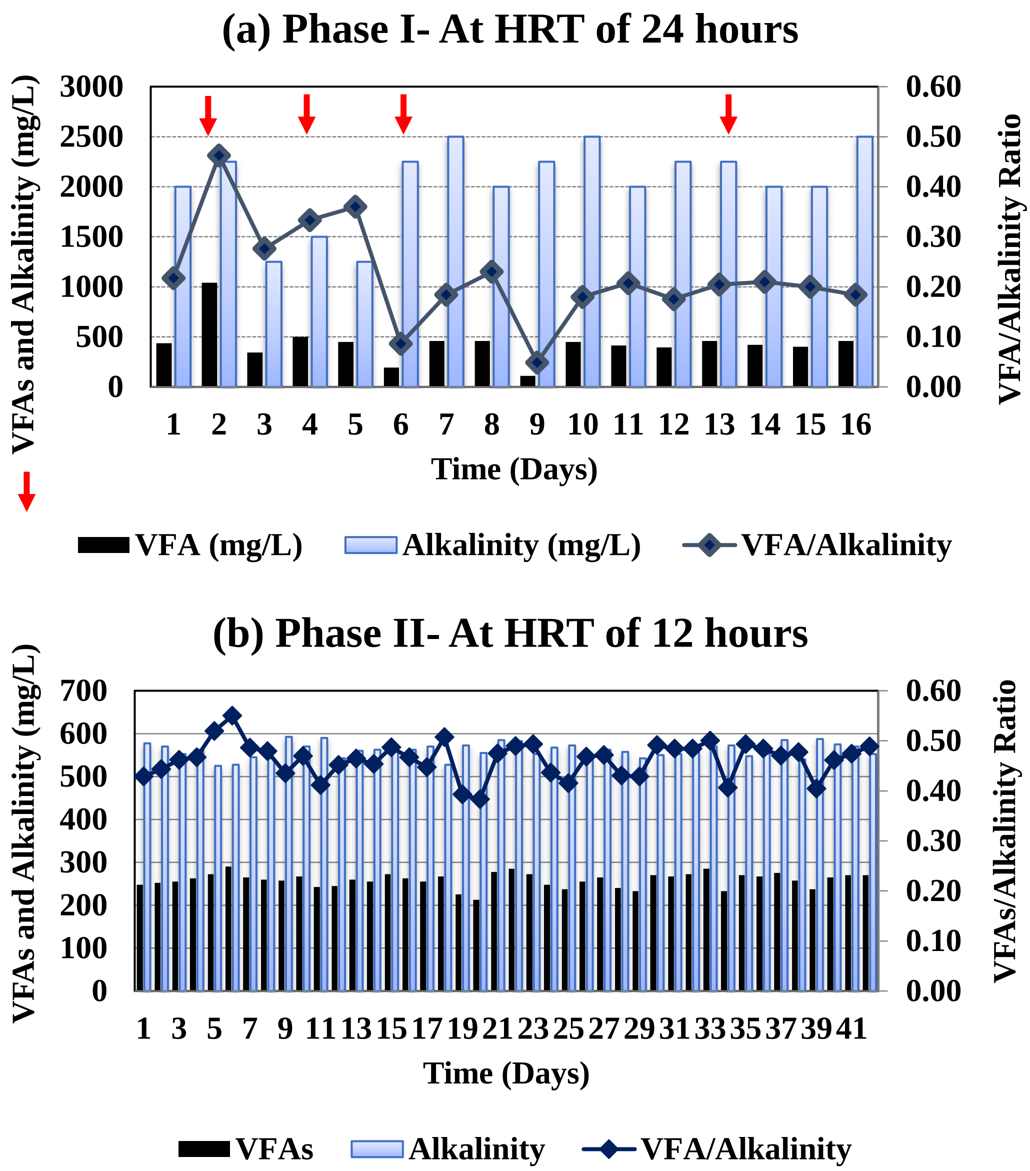
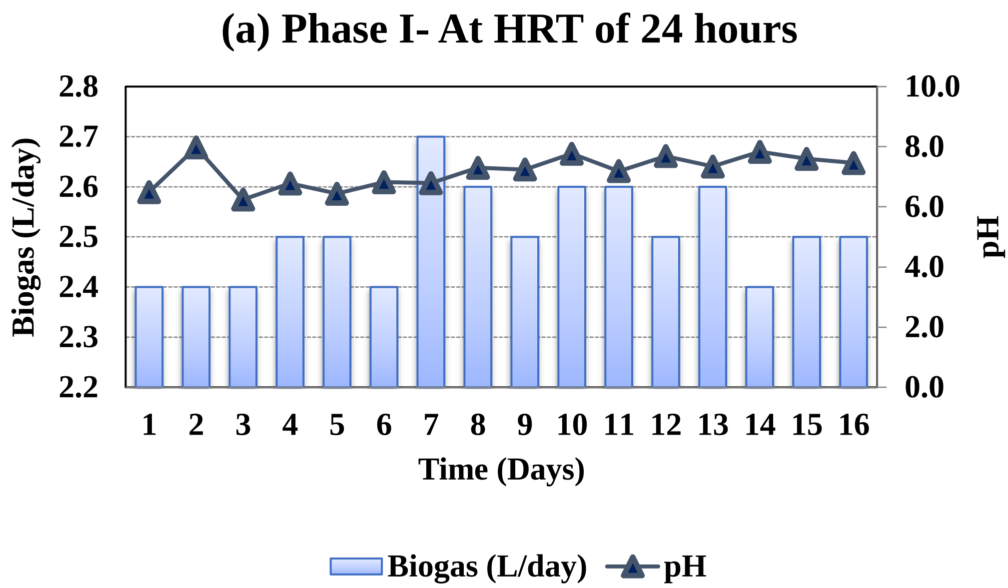


Figure 4.2. VFAs and Alkalinity (a) at 24 hours HRT (b) at 12 hours HRT



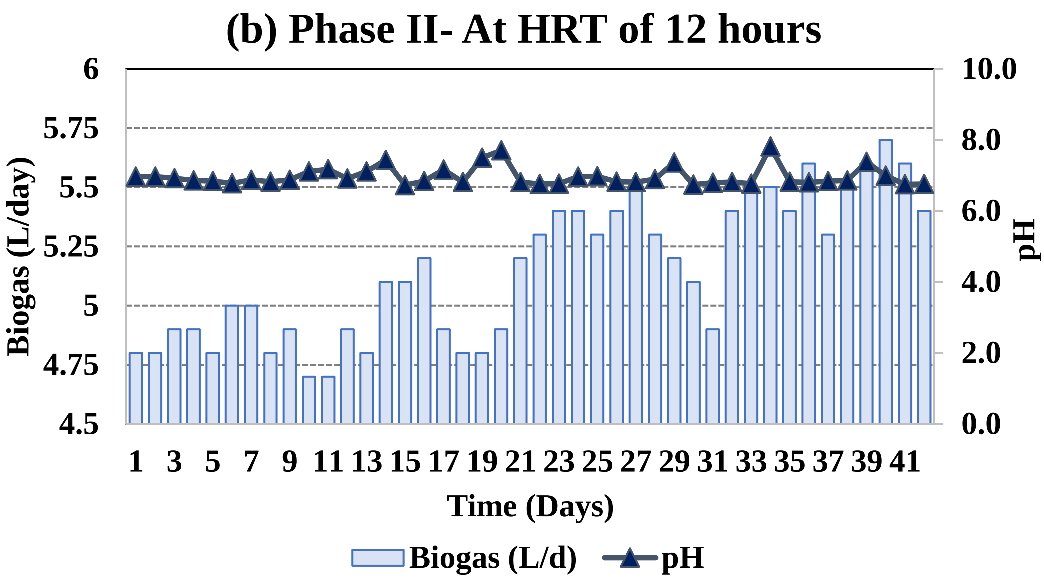


Figure 4.3. Biogas production (a) at 24 hours HRT (b) at 12 hours HRT

4.1.3 Effect of HRT on MLSS and MLVSS of the System

In case of biomass, MLSS and MLVSS concentrations were 4,500 mg/L and 2,750 mg/L respectively which kept increasing over time and observed to be 4,550 mg/L and 3,500 mg/L respectively in the last part of the run. The MLVSS/MLSS ratio ranged between 0.57 and 0.8 which was a good indicator of the stability of the reactor. The variation of MLSS and MLVSS with overall ratio of MLVSS/MLSS has been shown in Figure 4.4 (a).

No wasting of sludge was done in the whole study period and the microorganisms grew with the constant loading of the system. The analysis for MLSS and MLVSS was done every 7 days in the second phase of the study to ensure the presence of sufficient microorganisms in the system. The observations showed MLSS and MLVSS in the range of 4,800-4,900 mg/L and 3,200-3,400 mg/L with overall MLVSS/MLSS ratio of 0.65-0.71. The results confirmed a good growth of microorganisms with the higher organic load i.e 1 kgCOD/m^3-day as compared to the phase I (OLR= 0.5 kgCOD/m^3-day). The elaborative trend has been flashed in the Figure 4.4 (b).

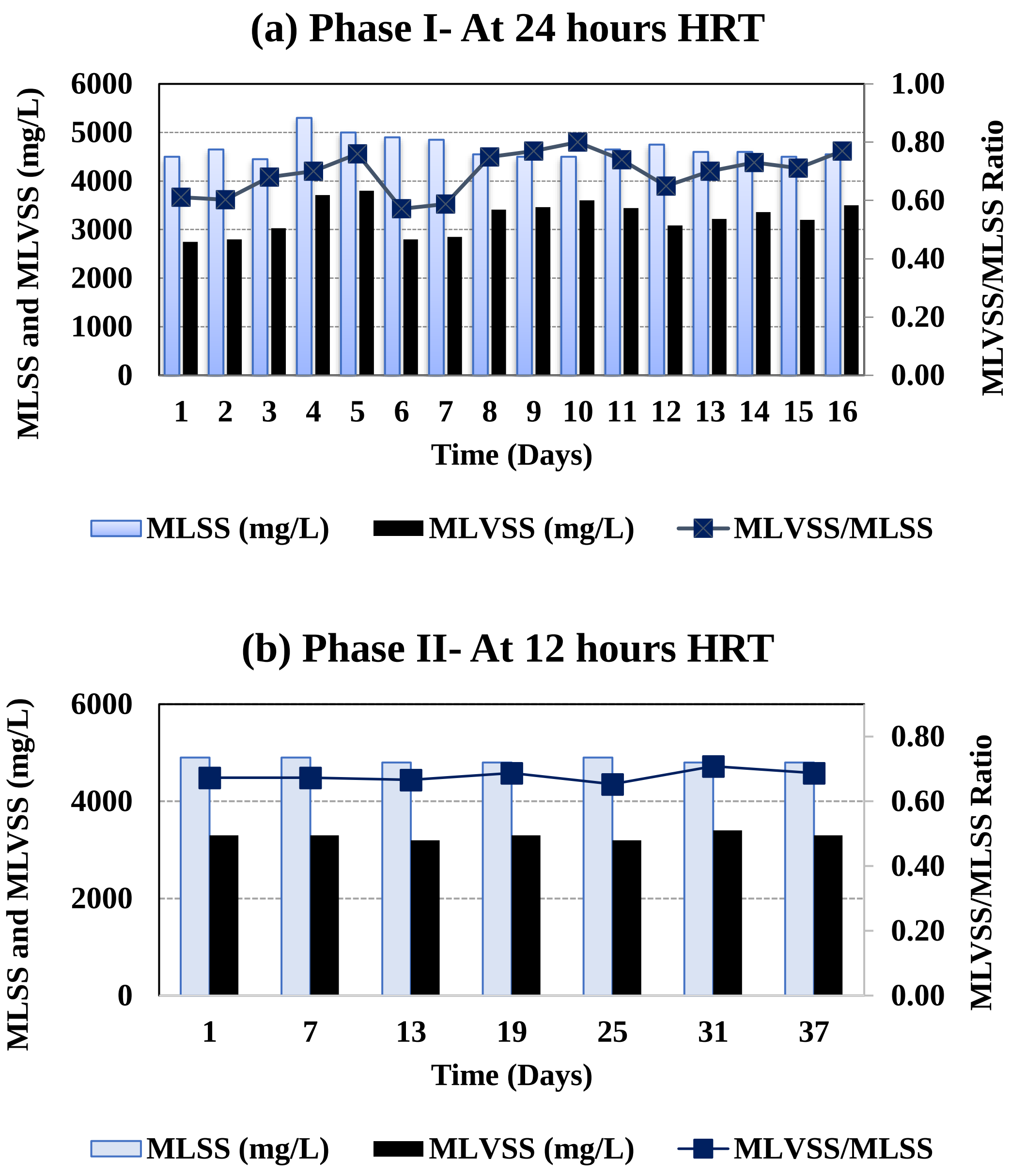


Figure 4.4. MLSS and MLVSS (a) at 24 hours HRT (b) at 12 hours HRT

4.1.4 Summary of Reactors Performance Stability Indicators at various HRTs

Organic removal efficiencies of anaerobic digestion tank (CSTR) were investigated to be excellent in terms of COD removal and biogas production while ensuring reactor's operational stability in the same time as reported in Table 4.1 for both phases.

Table 4.1. Operational stability or reactor

OLR (kg/m^3-day)	pH Range	VFAs (mg/L)	Alkalinity (mg/L)	Biogas Yield (L/day)	VFAs/Alkalinity
0.5	6.2- 8.0	110- 1,040	1,250- 2,500	2.4- 2.7	0.05- 0.46
1	6.7- 7.8	212.5- 290.0	525.0- 592.5	4.7- 5.7	0.38- 0.55

4.2 Phase B: Anaerobic Membrane Bioreactor (AnMBR)

4.2.1 COD Removal in AnMBR

A summary of COD removal and the concentrations of stability parameters during the operation of AnMBR has been illustrated in Figure 4.5. The COD removal varied from 88.0 (±1.5) to 96.7 (±1.4) in Phase I to Phase V while the OLR decreased from 0.78 to 0.46 kgCOD/m^3-day. The COD removal in Phase I was less making effluent concentration of 56.9 mg/L with average removal of 88.0 %. The decreased efficiency of the system was due to less concentration of MLSS and MLVSS (5.08 and 3.68 g/L respectively) in initial phase (acclimation period) in which microbes took time to grow and adapt to environmental conditions. The system may also show less efficiency due to decreased HRT (15.3 hr.) at net flux of 10.3 LMH in which microbes don't contact fully with biomass resulting in enhanced effluent COD.

The COD removal showed a significant increase in Phase II and averaged 93.6% with permeate concentration of 31.6 mg/L at slightly higher HRT of 17.9 h leading to a lower organic loading (0.66 kgCOD/m^3-day) followed by an increase in the sludge recirculation ratio (R=2). In Phase III, more COD removal was observed with same HRT as Phase II but at higher sludge recirculation ratio (R=3). The COD removal observed in Phase III was 95.8% with permeate COD of 20.5 mg/L. This improved efficacy of the system was related with the enhanced adaptation of microbes and enough contact time in phase II and III (HRT 17.9 h). The concentrations of MLSS and MLVSS in Phase II and III also showed increased trend with average concentrations of 5.75 (±0.17) and 4.24 (±0.21) to 5.99 (±0.13) and 4.64 (±0.06) respectively. The increased sludge recirculation ratios (R=2 and R=3) in Phase II and III respectively led to the increase in MLSS and MLVSS concentrations which also added to the increased organic removal.

The COD removal varied further and showed a significant increase from Phase IV to V with permeate COD of 46.3 and 17.7 mg/L resulting in average removals of 95.4 and 96.7% respectively. The concentration of MLSS and MLVSS also showed an increasing trend and observed to be 6.15 (±0.1) and 4.75 (±0.05) and 6.23 (±0.08) and 4.83 (±0.03) g/L followed by an increase in sludge recirculation ratio in Phase V (R=3) than in Phase IV (R=2). The observed efficiency was almost same (87-95%) as supported from the literature for the treatment of wastewater by single staged anaerobic fluidized membrane bioreactor (Aslam et al., 2017) and observed for the treatment of organic and inorganic mixed synthetic wastewater (Harb et al., 2016) where a COD removal above 90% was observed in anaerobic membrane reactor. The results prove that the anaerobic fermentation process can significantly be improved employing sludge recirculation which is supported by other researches from the literature (Gnanapragasam et al., 2010; Lee et al., 2010). Thus, it can be concluded that the effluent from the AnMBR meets the National Environmental Quality Standards of Pakistan of COD<150 mg/L.

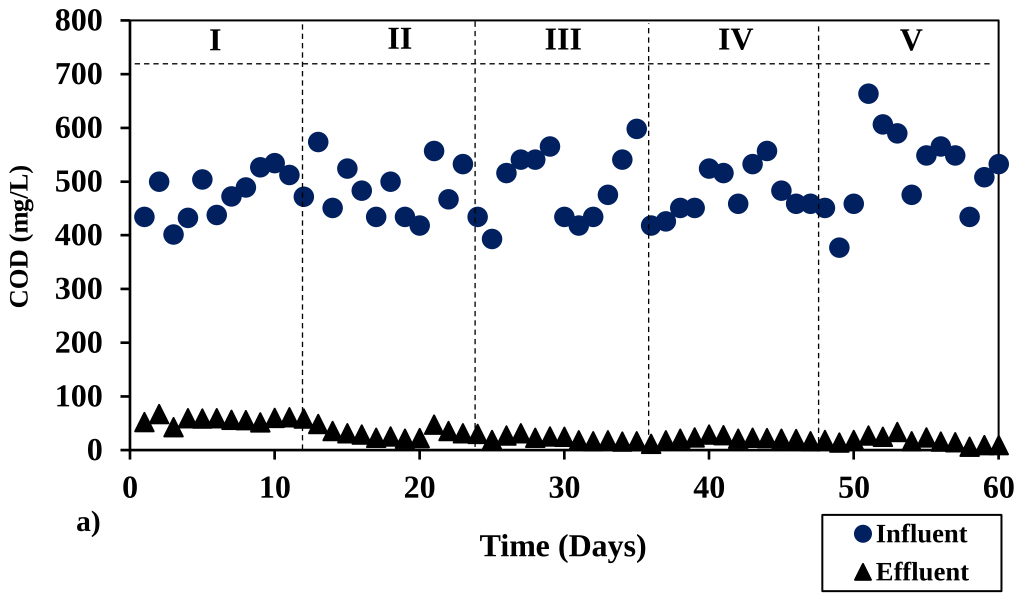
Overall the MLVSS/MLSS ratio was less in the beginning of the process in Phase I while the ratio slightly increased in Phase II to 0.74 and later it got stable to 0.77 in Phase III to Phase V. The maintained stable MLVSS to MLSS ratio under varied operational conditions reflects operational stability of the AnMBR (Baek et al., 2010;

Zhang et al., 2015) which results in significant COD removal and proves sustainability of the system. The higher MLVSS to MLSS ratio depicts activity of the biomass for degradation which may lead to efficient COD removal (Santos et al., 2017) and also the results were consistent with the observation in literature where a higher COD removal for anaerobic ceramic membrane bioreactor (AnCMBR) was observed associated with the greater concentration of both MLSS and MLVSS (Jeong et al., 2018). The concentration of MLSS and MLVSS in the entire experimental study, (Phase I to Phase V) are comparable to 5.8 ± 0.8 g/L and 4.7 ± 0.6 g/L respectively reported in the literature for the treatment of domestic wastewater by anaerobic ceramic membrane bioreactor (AnCMBR) (Jeong et al., 2018).

4.2.2 Biogas production and VFA's and Alkalinity Accumulation

The amount of biogas production in comparison to the VFAs and alkalinity concentrations in the reactor are depicted in Figure 4.5 (b). The biogas yield was very low in Phase I of the reactor and averaged 0.22 L/g $COD_{removed}$ while the concentration showed a tremendous shift in Phase II of reactor with an average yield of 0.38 L/g $COD_{removed}$. The observed very low yield of biogas in Phase I was due to low conversion of organics into biogas or it might be low due to being dissolved into the reactor effluent. The observed transformation of biogas production in Phase II could be attributed to enhanced growth of methanogens and improved reactor stability indicators as reflected by increased MLSS and MLVSS in this phase. The biogas yield gradually increased over time and averaged 0.41 L/g when sludge recirculation rate was increased to 3 in Phase III. Increased sludge recirculation ratio along with the stable reactor stability indicators (VFA and alkalinity) as depicted by Figure 4.5 (b) added a major contribution to the enhanced biogas yield over time. Similar biogas production was observed in Phase IV and V with average yield of 0.43 and 0.44 L/g $COD_{removed}$ at sludge recirculation rate of 2 and 3 respectively. It was observed that the increase in sludge recirculation didn't affect biogas production significantly in Phase IV and V indicating a stable yield in these operational phases. The similar trend of highest biogas production in Phase IV and Phase V of this study at lowest OLR (0.46 kgCOD/m^3-day) was observed in literature for biogas production for mesophilic screw anaerobic digester (Vongvichiankul et al., 2017). The stable biogas production in Phase III to Phase V was a result of optimal pH of 7.3 (±0.1) of the system while the ORP ranged from −324.4 (±28.5) to −324.4 (±14.4) mV which are important parameters to govern in anaerobic system (Vongvichiankul et al., 2017). The biogas production rate in the optimum phase (Phase V) was a little less than that reported in the literature for the treatment of synthetic wastewater by up-flow anaerobic sludge blanket (UASB)-SAnMBR where the observed production rate was 0.46 ± 0.1 L/g $COD_{removed}$ (Mahmoud & Liao, 2017). While the biogas yield measured in Phase III to Phase V was in accordance with the production of 0.4- 0.6 L/g $COD_{removed}$ found in literature (Song et al., 2016). The biogas production in Phase II to phase V was also higher than that reported in literature for the treatment of malting wastewater by submerged anaerobic membrane bioreactor (Maleki et al., 2018). Generally, the biogas production rate increased with the increase in COD removal as the degradation of organics resulted in biogas production. The similar fact is well known in the literature (Maleki et al., 2018).

The concentration of mixed liquor VFA in Phase I was observed to be 660 (±114.8) mg/L while the concentration suddenly decreased in Phase II with an average concentration of 574.8 (±210.6) mg/L. The concentration of VFA in Phase III, IV and V was 678.8 (±25.8), 701.7 (±57.6), and 714.6 (±60) mg/L respectively. The concentration of VFA didn't indicate any rapid shock load or instability of the reactor. It should be noted that the concentration of VFA in all operational phases were significantly lower than 1000 mg/L which didn't inhibit the system throughout the experimental period and is also supported from the evolving biogas yield over VFA and alkalinity, which should have been maintained for the optimum operation of methanogenic archaea (Foresti, 2002). The mixed liquor alkalinity decreased in Phase II than in Phase I while the concentration showed a little increase in Phase III to Phase V while the results were comparable to accumulated, 2216 ± 220 mg/L in anaerobic membrane bioreactor-membrane distillation hybrid system (Song et al., 2018).



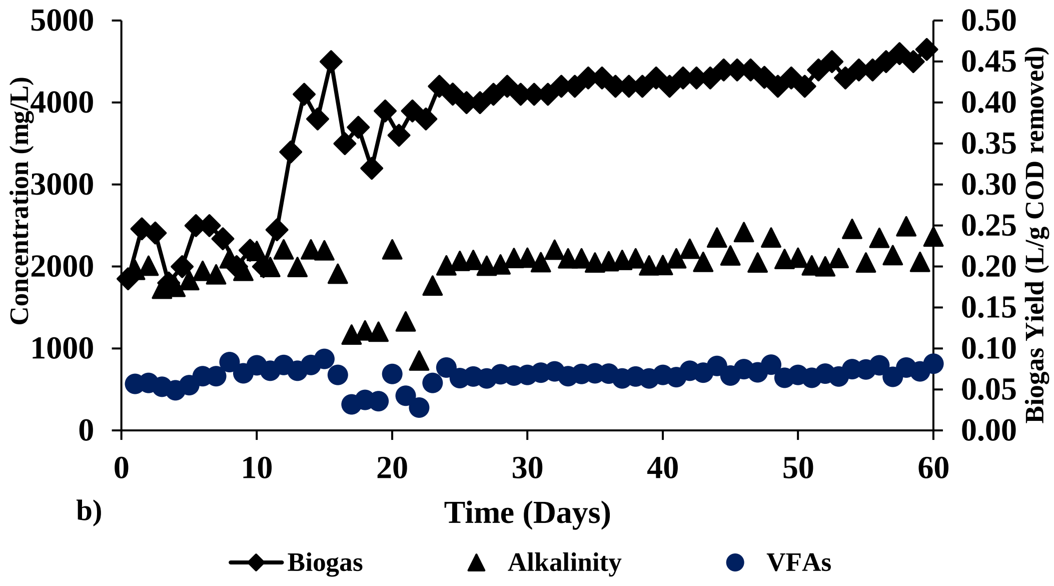


Figure 4.5. Summary of (a) COD removal and (b) stability indicators in AnMBR

4.2.3 Trans-membrane pressure (TMP) control in AnMBR

In this study of AnMBR, fouling limitation mechanism used was hydrodynamic control using different sludge recirculation ratios in side stream membrane configuration. In Phase I (acclimation period) of reactor operation, instantaneous flux of 11 LMH was set corresponding to net flux of 10.3 LMH. The sludge recirculation ratio (R=1) was employed and a sharp TMP rise was observed as compared to the other phases of the study. The steady TMP rise led to rapid membrane fouling leading to shorter membrane runs as is depicted from the Figure 4.6. The periodic membrane relaxation of 2 min after every 8 mins permeation didn't affect significantly to prevent membrane fouling. On day 4, the membrane was taken out from the reactor for recovery cleaning and the membrane tank was sealed to prevent the air from being entering the reactor. After performing cleaning, the membrane was again put into the reactor for operation after nitrogen purging of AnMBR system for ensuring the anaerobic environment. After the completion of every run, a resistance-in-series test was conducted to evaluate the relative contribution of intrinsic, pore and cake resistance to the total resistance offered by the membrane module. A total of 6 runs were performed in Phase I and the average membrane run time in Phase I was 2.3 days. The average cake layer resistance (R_c) was found to be 89% of the total resistance (R_t) experienced which indicated cake layer to be the major fouling factor for the membrane as Table 4.2 shows.

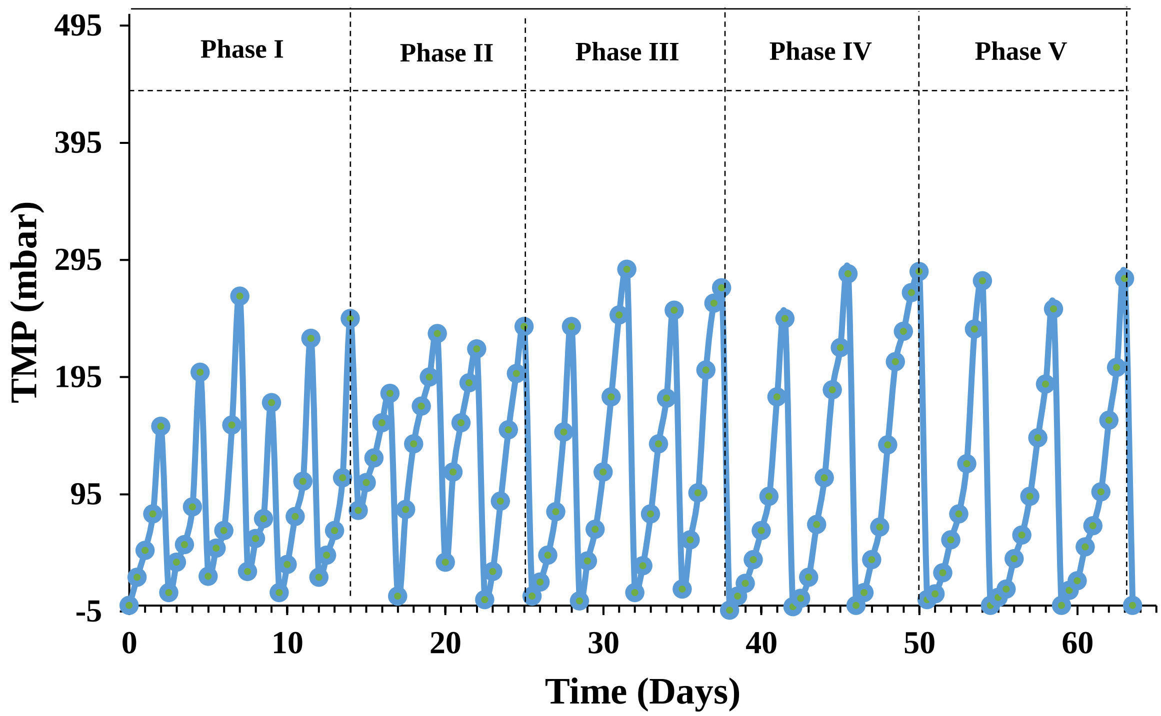


Figure 4.6. TMP profile of all operational phases

In Phase II of the study, membrane flux was reduced and sludge recirculation was increased (R=2) which resulted in longer membrane runs as compared to the Phase I. The cake layer resistance (R_c) in this phase was found to be 87.4% while pore resistance (R_p)was 11.4%. To check the prolong run time for the enhanced sludge recirculation, another phase of rector operation (Phase III) was performed on the same flux (8.8 LMH) while R was increased to 3. The result was slow TMP rise and a clear increase in membrane run time. It proved enhancing sludge recirculation can substantially enhance membrane filtration time while ensuring reasonably stable flux. The increased recirculation ratio (R=3) played a vital role in the fouling mitigation as is supported from the cake layer resistance (R_c) of 86.3% and pore resistance (R_p) of 12.6%. In Phase IV and V, the membrane instantaneous flux was further decreased to 6.9 and 6.8 LMH respectively yielding a net flux of 6 LMH in both phases while the sludge recirculation

(R=2) and (R=3) was employed. The similar transmembrane pressure cycles and membrane fouling was observed for these both phases as was for Phase II and Phase III. The total resistance offered was 88.0% and 87.7% respectively while the pore resistances (R_p) were 10.6% and 10.9% respectively.

The deposition of cake layer over membrane was the main reason of membrane fouling since the cake layer resistance (R_c) was >80% in all the phases performed. The similar results were found in the past study where the resistance experiments revealed cake layer resistance to be 80% and the reason of severe membrane fouling (Meng et al., 2009). The increase in sludge recirculation resulted in enhanced membrane filtration time causing less membrane fouling which otherwise would cause sharp TMP rise. The results show that membrane fouling was significant at low sludge recirculation rates which can be controlled employing lower operational flux and higher sludge recirculation and would incur low maintenance cost.

Table 4.2. Membrane fouling resistances in phases I to V during the operation of AnMBR system.

	Resistances (E12 m^{-1})																			
	Phase I				Phase II				Phase III				Phase IV				Phase V			
	Rm	Rp	Rc	Rt	Rm	Rp	Rc	Rt	Rm	Rp	Rc	Rt	Rm	Rp	Rc	Rt	Rm	Rp	Rc	Rt
Mean	0.11	0.90	8.21	9.23	0.12	1.10	8.46	9.67	0.11	1.25	8.60	9.96	0.13	1.02	8.42	9.57	0.12	0.99	7.93	9.04
Standard Deviation	0.05	0.03	0.29	0.33	0.05	0.14	0.27	0.15	0.02	0.02	0.03	0.02	0.00	0.23	0.54	0.38	0.01	0.21	0.91	0.73
Rc/Rt (%)	89.0				87.4				86.3				88.0				87.7			
Rp/Rt (%)	9.8				11.4				12.6				10.6				10.9			

Chapter 5

CONCLUSIONS AND RECOMMENDATIONS

The feasibility of anaerobic membrane bioreactor (AnMBR) for domestic wastewater treatment was evaluated in this study and the following conclusions were drawn based on the research outcomes.

- In batch study, the average COD removal was found 64.43% at OLR of 0.5 kgCOD/m^3-day while the effluent contained COD of 177.84 mg/L. The COD removal was almost the same at OLR of 1.0 kgCOD/m3-day with average of 64.78% when the HRT was reduced to 12 hours. The decline in HRT didn't affect the performance of the reactor and maintained the operational stability.
- The biogas production substantially increased and averaged 5.15 L/day in second phase of batch study (OLR 1.0 kgCOD/m^3-day) when the production of VFAs and alkalinity stabilized in the reactor than the first phase of operation (OLR=0.5 kgCOD/m^3-day).
- In AnMBR, the performance of the reactor remained excellent in terms of COD removal with the variation from 88.0 (±1.5) to 96.7 (±1.4) in Phase I to Phase V with the increase in sludge recirculation from membrane tank to anaerobic digestion tank. The increased concentration of MLSS and MLVSS in the anaerobic digestion tank led to better degradation of organic matter and hence COD removal.
- The biogas production in AnMBR showed an increasing trend with the increase in sludge recirculation between membrane and anaerobic digestion tanks and the optimum yield observed was 0.44 L/gCOD$_{removed}$ in Phase V (Flux= 6 LMH and R=3).

- The optimized condition for AnMBR operation was found at R=3 and flux= 6 LMH since the it also resulted in longer membrane runs owing to less membrane fouling.

Recommendations

- Performance evaluation of coupled system- Anaerobic membrane bioreactor (AnMBR) and pre-concentration system for the treatment of domestic wastewater.
- The same can also be investigated to check the treatment efficiency for textile wastewater.

REFERENCES

Abbasi, T., Tauseef, S., Abbasi, S. 2012. A brief history of anaerobic digestion and "biogas". in: *Biogas Energy*, Springer, pp. 11-23.

Achilli, A., Marchand, E., Childress, A. 2011. A performance evaluation of three membrane bioreactor systems: aerobic, anaerobic, and attached-growth. *Water Science and Technology*, **63**(12), 2999-3005.

Akamatsu, K., Lu, W., Sugawara, T., Nakao, S.-i. 2010. Development of a novel fouling suppression system in membrane bioreactors using an intermittent electric field. *Water research*, **44**(3), 825-830.

Akram, A., Stuckey, D.C. 2008. Flux and performance improvement in a submerged anaerobic membrane bioreactor (SAMBR) using powdered activated carbon (PAC). *Process Biochemistry*, **43**(1), 93-102.

Alvarez, J., Armstrong, E., Gómez, M., Soto, M. 2008. Anaerobic treatment of low-strength municipal wastewater by a two-stage pilot plant under psychrophilic conditions. *Bioresource Technology*, **99**(15), 7051-7062.

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

An, Y., Yang, F., Bucciali, B., Wong, F. 2009b. Municipal wastewater treatment using a UASB coupled with cross-flow membrane filtration. *Journal of environmental engineering*, **135**(2), 86-91.

Andrews, J.F., Cole, R., Pearson, E.A. 1964. *Kinetics and characteristics of multi-stage methane fermentations*. Sanitary Engineering Research Laboratory, College of Engineering and School of Public Health, University of California.

Aslam, M., McCarty, P.L., Shin, C., Bae, J., Kim, J. 2017. Low energy single-staged anaerobic fluidized bed ceramic membrane bioreactor (AFCMBR) for wastewater treatment. *Bioresource Technology*, **240**, 33-41.

Bae, J.-H., Kim, J.-h., McCarty, P.L. 2013. Fluidized membrane bioreactor, Google Patents.

Bae, T.-H., Kim, I.-C., Tak, T.-M. 2006. Preparation and characterization of fouling-resistant TiO2 self-assembled nanocomposite membranes. *Journal of membrane science*, **275**(1-2), 1-5.

Baek, S.H., Pagilla, K.R. 2006. Aerobic and anaerobic membrane bioreactors for municipal wastewater treatment. *Water environment research*, **78**(2), 133-140.

Baek, S.H., Pagilla, K.R., Kim, H.-J. 2010. Lab-scale study of an anaerobic membrane bioreactor (AnMBR) for dilute municipal wastewater treatment. *Biotechnology and Bioprocess Engineering*, **15**(4), 704-708.

Baêta, B., Ramos, R., Lima, D., Aquino, S. 2012. Use of submerged anaerobic membrane bioreactor (SAMBR) containing powdered activated carbon (PAC) for the treatment of textile effluents. *Water Science and Technology*, **65**(9), 1540-1547.

Bal AS, D.N. 2001. *Indian Journal of Environtal Health*, **43**(2), 1-83.

Bandara, W.M., Satoh, H., Sasakawa, M., Nakahara, Y., Takahashi, M., Okabe, S. 2011. Removal of residual dissolved methane gas in an upflow anaerobic sludge blanket reactor treating low-strength wastewater at low temperature with degassing membrane. *Water research*, **45**(11), 3533-3540.

Berube, P., Hall, E., Sutton, P. 2006. Parameters governing permeate flux in an anaerobic membrane bioreactor treating low-strength municipal wastewaters: a literature review. *Water environment research*, **78**(8), 887-896.

Boonyaroj, V., Chiemchaisri, C., Chiemchaisri, W., Theepharaksapan, S., Yamamoto, K. 2012. Toxic organic micro-pollutants removal mechanisms in long-term operated membrane bioreactor treating municipal solid waste leachate. *Bioresource Technology*, **113**, 174-180.

Brockmann, M., Seyfried, C.F. 1997. Sludge activity under the conditions of crossflow microfiltration. *Water Science and Technology*, **35**(10), 173-181.

Bryant, M. 1979. Microbial Methane Production—Theoretical Aspects1, 2. *Journal of Animal Science*, **48**(1), 193-201.

Calderón, K., Rodelas, B., Cabirol, N., González-López, J., 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.

Cases, V., Alonso, V., Argandoña, V., Rodriguez, M., Prats, D. 2011. Endocrine disrupting compounds: A comparison of removal between conventional activated sludge and membrane bioreactors. *Desalination*, **272**(1-3), 240-245.

Chan, P.C., de Toledo, R.A., Shim, H. 2017. Anaerobic co-digestion of food waste and domestic wastewater – Effect of intermittent feeding on short and long chain fatty acids accumulation. *Renewable Energy*.

Chernicharo, C., Van Lier, J., Noyola, A., Ribeiro, T.B. 2015. Anaerobic sewage treatment: state of the art, constraints and challenges. *Reviews in Environmental Science and Bio/Technology*, **14**(4), 649-679.

Chernicharo, C.A.L. 2001. *Pós-tratamento de efluentes de reatores anaeróbios*. PROSAB.

Choo, K.-H., Lee, C.-H. 1998. Hydrodynamic behavior of anaerobic biosolids during crossflow filtration in the membrane anaerobic bioreactor. *Water research*, **32**(11), 3387-3397.

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

Dagnew, M., Hong, Y., Adams, N., Fonseca, N., Cumin, J. 2014. Zeeweed AnMBR: Advancing anaerobic digestion for industrial application. *Proceedings of the Water Environment Federation*, **2014**(13), 479-487.

Dagnew, M., Parker, W., Seto, P., Waldner, K., Hong, Y., Bayly, R., Cumin, J. 2011. Pilot testing of an AnMBR for municipal wastewater treatment. *Proceedings of the Water Environment Federation*, **2011**(11), 4931-4941.

De Man, A. 1988. The use of EGSB and UASB anaerobic systems for low strength soluble and complex wastewaters at temperatures ranging from 8 to 30°C. *Proceeding of the Fifth International Symposium on Anaerobic Digestion*. pp. 197-209.

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

Elango, D., Pulikesi, M., Baskaralingam, P., Ramamurthi, V., Sivanesan, S. 2007. Production of biogas from municipal solid waste with domestic sewage. *J Hazard Mater*, **141**(1), 301-4.

Ellouze, M., Saddoud, A., Dhouib, A., Sayadi, S. 2009. Assessment of the impact of excessive chemical additions to municipal wastewaters and comparison of three technologies in the removal performance of pathogens and toxicity. *Microbiological research*, **164**(2), 138-148.

Field, R.W., Wu, D., Howell, J.A., Gupta, B.B. 1995. Critical flux concept for microfiltration fouling. *Journal of membrane science*, **100**(3), 259-272.

Foresti, E. 2002. Anaerobic treatment of domestic sewage: established technologies and perspectives. *Water Science and Technology*, **45**(10), 181-186.

Gangagni Rao, A., Venkata Naidu, G., Krishna Prasad, K., Chandrasekhar Rao, N., Venkata Mohan, S., Jetty, A., Sarma, P.N. 2005. Anaerobic treatment of wastewater with high suspended solids from a bulk drug industry using fixed film reactor (AFFR). *Bioresour Technol*, **96**(1), 87-93.

Gao, D.-W., Zhang, T., Tang, C.-Y.Y., Wu, W.-M., Wong, C.-Y., Lee, Y.H., Yeh, D.H., Criddle, C.S. 2010. Membrane fouling in an anaerobic membrane bioreactor: differences in relative abundance of bacterial species in the membrane foulant layer and in suspension. *Journal of membrane science*, **364**(1-2), 331-338.

Giménez, J., Robles, A., Carretero, L., Durán, F., Ruano, M., Gatti, M.N., Ribes, J., Ferrer, J., Seco, A. 2011. Experimental study of the anaerobic urban wastewater treatment in a submerged hollow-fibre membrane bioreactor at pilot scale. *Bioresource Technology*, **102**(19), 8799-8806.

Gizgis, N., Georgiou, M., Diamadopoulos, E. 2006. Sequential anaerobic/aerobic biological treatment of olive mill wastewater and municipal wastewater. *Journal of Chemical Technology & Biotechnology*, **81**(9), 1563-1569.

Gnanapragasam, G., Senthilkumar, M., Arutchelvan, V., Sivarajan, P., Nagarajan, S. 2010. Recycle in upflow anaerobic sludge blanket reactor on treatment of real textile dye effluent. *World journal of microbiology and biotechnology*, **26**(6), 1093-1098.

Gooch, C., Pronto, J., Labatut, R. 2011. Evaluation of Seven On-Farm Anaerobic Digestion Systems Based on the ASERTTI Monitoring Protocol: Consolidated Report and Findings Cornell University, Department of Biological and Environmental Engineering, Cornell University Ithaca, NY.

Grundestam, J., Hellström, D. 2007. Wastewater treatment with anaerobic membrane bioreactor and reverse osmosis. *Water Science and Technology*, **56**(5), 211-217.

Harb, M., Wei, C.-H., Wang, N., Amy, G., Hong, P.-Y. 2016. Organic micropollutants in aerobic and anaerobic membrane bioreactors: Changes in microbial communities and gene expression. *Bioresource Technology*, **218**, 882-891.

Hatamoto, M., Yamamoto, H., Kindaichi, T., Ozaki, N., Ohashi, A. 2010. Biological oxidation of dissolved methane in effluents from anaerobic reactors using a down-flow hanging sponge reactor. *Water research*, **44**(5), 1409-1418.

Herrera-Robledo, M., Cid-León, D., Morgan-Sagastume, J., Noyola, A. 2011. Biofouling in an anaerobic membrane bioreactor treating municipal sewage. *Separation and Purification Technology*, **81**(1), 49-55.

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

Ho, J., Khanal, S., Sung, S. 2007. Anaerobic membrane bioreactor for treatment of synthetic municipal wastewater at ambient temperature. *Water Science and Technology*, **55**(7), 79-86.

Ho, J., Sung, S. 2010. Methanogenic activities in anaerobic membrane bioreactors (AnMBR) treating synthetic municipal wastewater. *Bioresource Technology*, **101**(7), 2191-2196.

Hu, A.Y., Stuckey, D.C. 2007. Activated carbon addition to a submerged anaerobic membrane bioreactor: effect on performance, transmembrane pressure, and flux. *Journal of environmental engineering*, **133**(1), 73-80.

Huang, Z., Ong, S.L., 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.

Jeison, D. 2007. *Anaerobic membrane bioreactors for wastewater treatment: feasibility and potential applications*.

Jeison, D., Plugge, C.M., Pereira, A., Van Lier, J.B. 2009a. Effects of the acidogenic biomass on the performance of an anaerobic membrane bioreactor for wastewater treatment. *Bioresource Technology*, **100**(6), 1951-1956.

Jeison, D., Telkamp, P., Van Lier, J. 2009b. Thermophilic sidestream anaerobic membrane bioreactors: the shear rate dilemma. *Water environment research*, **81**(11), 2372-2380.

Jeison, D., Van Lier, J. 2006. On-line cake-layer management by trans-membrane pressure steady state assessment in anaerobic membrane bioreactors for wastewater treatment. *Biochemical Engineering Journal*, **29**(3), 204-209.

Jeong, Y., Kim, Y., Jin, Y., Hong, S., Park, C. 2018. Comparison of filtration and treatment performance between polymeric and ceramic membranes in anaerobic membrane bioreactor treatment of domestic wastewater. *Separation and Purification Technology*, **199**, 182-188.

Jeris, J.S., McCarty, P.L. 1965. The Biochemistry of Methane Fermentation Using \rmC^14 Tracers. *Journal (Water Pollution Control Federation)*, 178-192.

Kang, I.-J., Yoon, S.-H., Lee, C.-H. 2002. Comparison of the filtration characteristics of organic and inorganic membranes in a membrane-coupled anaerobic bioreactor. *Water research*, **36**(7), 1803-1813.

Kaspar, H.F., Wuhrmann, K. 1978. Kinetic parameters and relative turnovers of some important catabolic reactions in digesting sludge. *Applied and Environmental Microbiology*, **36**(1), 1-7.

Kataoka, N., Tokiwa, Y., Tanaka, Y., Fujiki, K., Taroda, H., Takeda, K. 1992. Examination of bacterial characteristics of anaerobic membrane bioreactors in three pilot-scale plants for treating low-strength wastewater by application of the colony-forming-curve analysis method. *Applied and Environmental Microbiology*, **58**(9), 2751-2757.

Kato, M.T., Field, J.A., Lettinga, G. 1997. The anaerobic treatment of low strength wastewaters in UASB and EGSB reactors. *Water Science and Technology*, **36**(6-7), 375-382.

Keating, C., Chin, J.P., Hughes, D., Manesiotis, P., Cysneiros, D., Mahony, T., Smith, C.J., McGrath, J.W., O'Flaherty, V. 2016. Biological phosphorus removal during high-rate, low-temperature, anaerobic digestion of wastewater. *Frontiers in microbiology*, **7**, 226.

Kleerebezem, R., Macarie, H. 2003. Treating industrial wastewater: anaerobic digestion comes of age: anaerobic treatment systems offer important advantages over conventionally applied aerobic processes for removing organic pollutants from water-based streams.(Cover Story). *Chemical Engineering*, **110**(4), 56-65.

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

Labatut, R.A., Gooch, C.A. 2014. Monitoring of anaerobic digestion process to optimize performance and prevent system failure.

Lawrence, A.W., McCarty, P.L. 1969. Kinetics of methane fermentation in anaerobic treatment. *Journal (Water Pollution Control Federation)*, R1-R17.

Le Clech, P., Jefferson, B., Chang, I.S., Judd, S.J. 2003. Critical flux determination by the flux-step method in a submerged membrane bioreactor. *Journal of membrane science*, **227**(1-2), 81-93.

Lee, D.-Y., Ebie, Y., Xu, K.-Q., Li, Y.-Y., Inamori, Y. 2010. Continuous H2 and CH4 production from high-solid food waste in the two-stage thermophilic fermentation process with the recirculation of digester sludge. *Bioresource Technology*, **101**(1), S42-S47.

Lefebvre, O., Uzabiaga, A., Chang, I.S., Kim, B.-H., Ng, H.Y. 2011. Microbial fuel cells for energy self-sufficient domestic wastewater treatment—a review and discussion from energetic consideration. *Applied microbiology and biotechnology*, **89**(2), 259-270.

Lefebvre, O., Vasudevan, N., Torrijos, M., Thanasekaran, K., Moletta, R. 2006. Anaerobic digestion of tannery soak liquor with an aerobic post-treatment. *Water Res*, **40**(7), 1492-500.

Lettinga, G., Rebac, S., Parshina, S., Nozhevnikova, A., Van Lier, J.B., Stams, A.J. 1999. High-rate anaerobic treatment of wastewater at low temperatures. *Applied and Environmental Microbiology*, **65**(4), 1696-1702.

Lettinga, G., Rebac, S., Zeeman, G. 2001. Challenge of psychrophilic anaerobic wastewater treatment. *TRENDS in Biotechnology*, **19**(9), 363-370.

Lettinga, G., Van Velsen, A., Hobma, S.W., De Zeeuw, W., Klapwijk, A. 1980. Use of the upflow sludge blanket (USB) reactor concept for biological wastewater treatment, especially for anaerobic treatment. *Biotechnology and bioengineering*, **22**(4), 699-734.

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

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

Lin, H., Gao, W., Meng, F., Liao, B.-Q., Leung, K.-T., Zhao, L., Chen, J., Hong, H. 2012. Membrane bioreactors for industrial wastewater treatment: a critical review. *Critical Reviews in Environmental Science and Technology*, **42**(7), 677-740.

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

Mahmoud, I., Liao, B. 2017. Effects of sludge concentration and biogas sparging rate on critical flux in a submerged anaerobic membrane bioreactor. *Journal of water process engineering*, **20**, 51-60.

Maleki, E., Catalan, L.J., Liao, B. 2018. Effect of organic loading rate on the performance of a submerged anaerobic membrane bioreactor (SAnMBR) for malting wastewater treatment and biogas production. *Journal of Chemical Technology & Biotechnology*, **93**(6), 1636-1647.

Manariotis, I.D., Grigoropoulos, S.G. 2003. Anaerobic treatment of low-strength wastewater in a biofilm reactor. *Journal of Environmental Science and Health, Part A*, **38**(10), 2057-2068.

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

Martinez-Sosa, D., Helmreich, B., Horn, H. 2012. Anaerobic submerged membrane bioreactor (AnSMBR) treating low-strength wastewater under psychrophilic temperature conditions. *Process Biochemistry*, **47**(5), 792-798.

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

Martinez-Sosa, D., Helmreich, B., Netter, T., Paris, S., Bischof, F., Horn, H. 2011b. Pilot-scale anaerobic submerged membrane bioreactor (AnSMBR) treating municipal wastewater: the fouling phenomenon and long-term operation. *Water Science and Technology*, **64**(9), 1804-1811.

McCarty, P. 1966. Kinetics of waste assimilation in anaerobic treatment. *Devs ind. Microbiol. l 965(*144-155.

McCarty, P.L., Bae, J., Kim, J. 2011. Domestic wastewater treatment as a net energy producer–can this be achieved?, ACS Publications.

McCarty, P.L., Jeris, J.S., Murdoch, W. 1963. Individual volatile acids in anaerobic treatment. *Journal (Water Pollution Control Federation)*, **35**(12), 1501-1516.

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

Moharram, M.A., Abdelhalim, H.S., Rozaik, E.H. 2016. Anaerobic up flow fluidized bed reactor performance as a primary treatment unit in domestic wastewater treatment. *HBRC Journal*, **12**(1), 99-105.

Ozgun, H., Dereli, R.K., Ersahin, M.E., Kinaci, C., Spanjers, H., 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.

Pavlostathis, S., Giraldo-Gomez, E. 1991. Kinetics of anaerobic treatment: a critical review. *Critical Reviews in Environmental Science and Technology*, **21**(5-6), 411-490.

Rebac, S., van Lier, J.B., Lens, P., Stams, A.J., Dekkers, F., Swinkels, K.T.M., Lettinga, G. 1999. Psychrophilic anaerobic treatment of low strength wastewaters. *Water Science and Technology*, **39**(5), 203-210.

Rinzema, A., Alphenaar, A., Lettinga, G. 1989. The effect of lauric acid shock loads on the biological and physical performance of granular sludge in UASB reactors digesting acetate. *Journal of Chemical Technology and Biotechnology*, **46**(4), 257-266.

Rogers, H.R. 1996. Sources, behaviour and fate of organic contaminants during sewage treatment and in sewage sludges. *Science of The Total Environment*, **185**(1-3), 3-26.

Saddoud, A., Abdelkafi, S., Sayadi, S. 2009. Effects of domestic wastewater toxicity on anaerobic membrane-bioreactor (MBR) performances. *Environmental technology*, **30**(13), 1361-1369.

Saddoud, A., Ellouze, M., Dhouib, A., Sayadi, S. 2006. A comparative study on the anaerobic membrane bioreactor performance during the treatment of domestic wastewaters of various origins. *Environmental technology*, **27**(9), 991-999.

Salazar-Peláez, M., Morgan-Sagastume, J., Noyola, A. 2011. Influence of hydraulic retention time on UASB post-treatment with UF membranes. *Water Science and Technology*, **64**(11), 2299-2305.

Santos, A., Ma, W., Judd, S.J. 2011. Membrane bioreactors: two decades of research and implementation. *Desalination*, **273**(1), 148-154.

Santos, F.S., Ricci, B.C., Neta, L.S.F., Amaral, M.C. 2017. Sugarcane vinasse treatment by two-stage anaerobic membrane bioreactor: Effect of hydraulic retention time on changes in efficiency, biogas production and membrane fouling. *Bioresource Technology*, **245**, 342-350.

Sato, T., Qadir, M., Yamamoto, S., Endo, T., Zahoor, A. 2013. Global, regional, and country level need for data on wastewater generation, treatment, and use. *Agricultural Water Management*, **130**, 1-13.

Seghezzo, L., Zeeman, G., van Lier, J.B., Hamelers, H., Lettinga, G. 1998. A review: the anaerobic treatment of sewage in UASB and EGSB reactors. *Bioresource Technology*, **65**(3), 175-190.

Shafi, E.A., Halim, H.S.A., Mostafa, M.H., Nazih, M. 2009. COST EFFECTIVE WASTEWATER TREATMENT PROCESSES: ANAEROBIC DOMESTIC WASTEWATER TREATMENT USING FIXED FILM REACTOR AS A LOW-COST TREATMENT ALTERNATIVE.

Smith, A., Love, N., Skerlos, S., Raskin, L. 2011. Role of membrane biofilm in psychrophilic anaerobic membrane bioreactor for domestic wastewater treatment. *Proceedings of the Water Environment Federation*, **2011**(11), 4948-4952.

Smith, A.L., Stadler, L.B., Love, N.G., Skerlos, S.J., Raskin, L. 2012. Perspectives on anaerobic membrane bioreactor treatment of domestic wastewater: a critical review. *Bioresource Technology*, **122**, 149-159.

Song, X., Luo, W., McDonald, J., Khan, S.J., Hai, F.I., Price, W.E., Nghiem, L.D. 2018. An anaerobic membrane bioreactor–membrane distillation hybrid system for energy recovery and water reuse: Removal performance of organic carbon, nutrients, and trace organic contaminants. *Science of The Total Environment*, **628**, 358-365.

Song, X., McDonald, J., Price, W.E., Khan, S.J., Hai, F.I., Ngo, H.H., Guo, W., Nghiem, L.D. 2016. Effects of salinity build-up on the performance of an anaerobic membrane bioreactor regarding basic water quality parameters and removal of trace organic contaminants. *Bioresource Technology*, **216**, 399-405.

Sponza, D.T., Uluköy, A. 2005. Treatment of 2, 4-dichlorophenol (DCP) in a sequential anaerobic (upflow anaerobic sludge blanket) aerobic (completely stirred tank) reactor system. *Process Biochemistry*, **40**(11), 3419-3428.

Sui, P., Wen, X., Huang, X. 2008. Feasibility of employing ultrasound for on-line membrane fouling control in an anaerobic membrane bioreactor. *Desalination*, **219**(1-3), 203-213.

Tchobanoglous, G., Burton, F., Stensel, H. 2003a. Metcalf and Eddy Wastewater Engineering: Treatment and Reuse McGraw Hill. *New York, NY*, **384**.

Tchobanoglous, G., Burton, F.L., Stensel, H.D. 2003b. *Wastewater engineering treatment and reuse*. Boston, US: McGraw-Hill Higher Education.

Tiranuntakul, M., Schneider, P., Jegatheesan, V. 2011. Assessments of critical flux in a pilot-scale membrane bioreactor. *Bioresource Technology*, **102**(9), 5370-5374.

van Lier, J.B., Mahmoud, N., Zeeman, G. 2008. Anaerobic wastewater treatment. *biological wastewater treatment, principles, modelling and design*, 415-456.

Van Lier, J.B., Rebac, S., Lettinga, G. 1997. High-rate anaerobic wastewater treatment under psychrophilic and thermophilic conditions. *Water Science and Technology*, **35**(10), 199-206.

Visvanathan, C., Aim, R.B., Parameshwaran, K. 2000. Membrane separation bioreactors for wastewater treatment. *Critical Reviews in Environmental Science and Technology*, **30**(1), 1-48.

Vongvichiankul, C., Deebao, J., Khongnakorn, W. 2017. Relationship between pH, Oxidation Reduction Potential (ORP) and Biogas Production in Mesophilic Screw Anaerobic Digester. *Energy Procedia*, **138**, 877-882.

Vyrides, I., Stuckey, D. 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.

Wen, C., Huang, X., Qian, Y. 1999. Domestic wastewater treatment using an anaerobic bioreactor coupled with membrane filtration. *Process Biochemistry*, **35**(3-4), 335-340.

Wu, B., An, Y., Li, Y., Wong, F.S. 2009. Effect of adsorption/coagulation on membrane fouling in microfiltration process post-treating anaerobic digestion effluent. *Desalination*, **242**(1-3), 183-192.

Xu, M., Wen, X., Yu, Z., Li, Y., 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.

Yeoh, B. 1995. Anaerobic treatment of industrial wastewaters in Malaysia. *Post conference seminar on industrial wastewater management in Malaysia, Kuala Lumpur, Malaysia*.

Yoo, R., Kim, J., McCarty, P., Bae, J. 2014. Effect of temperature on the treatment of domestic wastewater with a staged anaerobic fluidized membrane bioreactor. *Water Science and Technology*, **69**(6), 1145-1150.

Yoo, R., Kim, J., McCarty, P.L., Bae, J. 2012. Anaerobic treatment of municipal wastewater with a staged anaerobic fluidized membrane bioreactor (SAF-MBR) system. *Bioresource Technology*, **120**, 133-139.

Zehnder, A.J., Mitchell, R. 1978. Ecology of methane formation. *Water pollution microbiology*, **2**, 349-376.

Zhang, X., Yue, X., Liu, Z., Li, Q., Hua, X. 2015. Impacts of sludge retention time on sludge characteristics and membrane fouling in a submerged anaerobic–oxic membrane bioreactor. *Applied microbiology and biotechnology*, **99**(11), 4893-4903.

Zoutberg, G.R., de Been, P. 1997. The Biobed® EGSB (Expanded Granular Sludge Bed) system covers shortcomings of the upflow anaerobic sludge blanket reactor in the chemical industry. *Water Science and Technology*, **35**(10), 183-188.