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Sequential Anaerobic and Algal Membrane Bioreactor (A2MBR) System for Sustainable Sanitation and Resource Recovery from Domestic Wastewater

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Sequential Anaerobic and Algal Membrane Bioreactor (A2MBR) System for

Sustainable Sanitation and Resource Recovery from Domestic Wastewater

by

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A dissertation submitted in partial fulfillment
of the requirements for the degree of
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ABSTRACT

An innovative wastewater treatment technology was developed to recover renewable resources, such as water, energy and nutrients, from sewage. First, a novel synthetic sewage was evaluated for its suitability to serve as an alternative substrate for lab-scale wastewater treatment (WWT) research. Based on granular dried cat food, Complex Organic Particulate Artificial Sewage (COPAS) is a commercially-available, flexible, and easy to preserve feed. Characteristics of COPAS, namely chemical composition, disintegration/dissolution kinetics, and anaerobic biodegradability, were determined. Anaerobic bioassays indicate that COPAS is highly biodegradable at the concentration used to simulate household sewage (1000 mg/L), with more than 72% of the theoretical methane content reached after 30 d of incubation. Results indicate that COPAS is a suitable substrate as a surrogate of domestic sewage.

In the second stage of the research, a lab-scale, 10L gas-lift anaerobic membrane bioreactor (Gl-AnMBR) was designed, fabricated and tested. The AnMBR is a hybrid treatment technology that combines anaerobic biological treatment with low-pressure membrane filtration. Although AnMBR has been used in many instances for the treatment of high strength industrial or agricultural wastewater, relatively little has been reported about its application for the treatment of domestic sewage and further conversion and recovery of resources embedded in sewage, such as energy and nutrient enriched water. The 10L column reactor uses a tubular PVDF ultrafiltration membrane

(with biogas as sparge gas) for sludge/water separation. COPAS was used as synthetic feed (at 1000 mg/L) to represent household wastewater. The configuration showed excellent removal efficiencies of organic matter (up to 98% and 95% in COD and TOC removal, respectively) while producing energy in the form of methane at quantities suitable for maintaining membrane scrubbing (4.5 L/d of biogas). Soluble nutrients were recovered in the effluent in the forms of NH_4, (9.1±4.2 mg/L), NO_3 (2.2±0.9 mg/L) and PO_4 (20±7.13 mg/L). The energy footprint (net energy) of this reactor was evaluated and the energy requirements per volume of permeate produced was found to be in the range of -1.2 to 0.7 kWh/m^3, depending on final conversion of methane to electric or thermal energy respectively. These values could potentially be improved towards energy surplus (-2.3 to -0.5 kWh/m^3) if applied to plant scale operation, which would employ more efficient pumps than those used in the lab. Results from this study suggest that the Gl-AnMBR can be applied as a sustainable treatment tool for resource recovery from sewage, which can further be optimized for large scale operation.

In the final stage of this research, further resource recovery from sewage was investigated by coupling the Gl-AnMBR with an innovative gas-lift algal photo MBR (APMBR). To our knowledge, this is the first reported application of membranes (in particular gas-lift tubular) for separation of algal cells from effluent in a continuous-flow photobioreactor. Nutrient rich effluent (9 mg/L NH_4-N and 20 mg/L PO_4-P) from the Gl-AnMBR treating domestic wastewater was used as substrate to grow the biofuel producing microalgae *Chlorella sorokiniana* (Cs). The initial set of operational conditions tested in this study (HRT of 24 hours, operational flux of 4.5 LMH, air-lift flow rate (Q_a) of 0.1 L/min and 0.1 bars of membrane inlet pressure), achieved 100% removal

efficiencies for NH_4 and PO_4. Flux remained constant during the experimental period which demonstrated the efficacy of gas lift as a membrane fouling control strategy for an algae bioreactor. Because the algae is photoautotrophic, little removal of organic carbon was expected nor observed. Further studies are required to better understand the fate and cycling of carbon in the APMBR. Limited information is available in the literature regarding biofuel-producing, algal photo MBRs utilizing anaerobic effluents as feedstock, which makes this study an important step in understanding the design and performance of combined anaerobic/algal biotechnology for large scale application of wastewater resource recovery.

1 INTRODUCTION

The availability of indispensable resources for our modern societies has reached a critical point in this century. Hubbert's global peak oil prediction has already surpassed (i.e. May 2005) and oil price has particularly increased during 2008 to highest historical values (e.g. $146.69 in UK and $145.85 in US as July 3, 2008) (McPherson and Weltzin, 2008; BBC, 2008). More than 1.1 billion people do not have access to safe water supply and 2.6 billion lack of adequate sanitation around the world (UNDP, 2006). Availability of fresh water sources is declining at an overwhelming rate and around 20% of world's population already lives in water stressed areas (FAO, 2007). Growing societies demand from agricultural production to accelerate its pace and around 197 trillion tons of fertilizers are used in the world for this purpose (FAO, 2008). Additionally, green house gases emissions due to human activity promote global warming and its devastating environmental effects. It is evident that what once was a concern about depletion of natural resources today is a latent crisis that requires immediate and sustainable solutions.

To address the current situation, broad-based commitments towards sustainable development were established in the Kyoto Protocol and the United Nations' Millennium Development Goals to counterbalance global warming and improve quality of life for people in developing countries, respectively. Specifically, research has been directed towards alternative, environmentally-friendly and sustainable ways to overcome

resources depletion, and a wide range of possibilities can be found within the waste and wastewater field. Typically, wastewater represents a problem for urban development and a risk for public health, but if adequate and sustainable treatment is provided, it could also represent an invaluable resource. Wastewater is a renewable material and full realization of its maximum potential is a pending subject that offers great opportunity for research and future implementation. Currently, conventional technologies for wastewater treatment (i.e., activated sludge) comply with regulatory requirements for discharge into the environment, or for possible reuse activities. However, these technologies are typically centralized in wastewater treatment plants (WWTP) that consume large amounts of energy (e.g. aeration of the activated sludge basin). Also, significant portion of the wastewater biodegradable organic matter is converted to sludge, which requires further treatment and disposition. Growing populations also exert more pressure to the WWTPs by discharging more spent water into the sewerage collection system. Furthermore, sprawling and expanding urban regions oblige developers and municipalities to incur additional costs of infrastructure to adequately serve remote areas with water and wastewater networks. In developing nations, the lack of infrastructure to centralized wastewater treatment plants leaves isolated rural areas with inadequate or altogether lacking potable water supply and/or basic sanitation conditions. Finally, the quality of surface water bodies is compromised when wastewater is inadequately treated before final discharge (Anh et al., 2002). Overall, conventional treatment does not fully take advantage of wastewater as a renewable resource.

To improve effluent quality of wastewater treatment and further applications of reclaimed wastewater, advanced technologies such as the membrane bioreactor (MBR) have been developed. MBRs couple biological treatment with a membrane filtration unit and their major advantage over conventional treatment is related to its effluent quality. Additionally, retention of biomass by the membrane allows separation of sludge retention time (SRT) and hydraulic retention time (HRT), which are basic parameters in conventional wastewater treatment operation. As a result, rapid sewage treatment and smaller space requirement are obtained from this technology. However, these advantages could be offset by the greater energy requirement to drive membrane filtration (Judd, 2006).

In the search of alternative technologies to improve recovery of wastewater intrinsic resources and provide efficient sanitation as well, anaerobic membrane bioreactor (AnMBR) is a treatment processes that should be investigated in more depth. Although MBR is generally known for its high quality effluent and small footprint, AnMBR has the additional benefits of energy generation (e.g. biogas), fertilizer recovery (e.g. nutrients), and low sludge generation. Under optimum operational conditions, an AnMBR can be used not only for on-site wastewater treatment, but generation of reusable water for agricultural applications. Additionally, biogas produced in the anaerobic process could potentially more than satisfy energy requirements of the system (Liao, 2006). More information, however, is required regarding maximization of the overall energy balance (energy footprint) in AnMBR. Recent studies have demonstrated improved energy efficiency of membrane technology by enhancing shear over membrane surface in vacuum-driven modules using air scouring (e.g. reducing cake layer deposition in

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submerged membranes). This approach is also applied to sidestream membrane configurations in aerobic airlift supported modules, which has gained increased attention for municipal wastewater treatment, but little is known about the application of this configuration in anaerobic mode by using biogas for gas-lift.

In this work, a decentralized anaerobic treatment process to treat wastewater generated by a community (hundreds to thousands) is developed. The aim is to reduce, eliminate and even generate surplus energy from wastewater treatment, with a focus on resource recovery (energy via methane, N and P for fertilizer, and clean water) rather than removal. An anaerobic bioreactor coupled with a gas-lift supported membrane unit (Gl-AnMBR) is used to treat synthetic sewage, which combines anaerobic digestion with low pressure membrane filtration. The ultrafiltration membrane used has micropores small enough such that practically all pathogens are removed (four log removal for virus, six log for bacteria and 8 log for helminths). This system has a small physical and ecological footprint, and focuses on routing the embedded energy in waste organic matter to methane, while liberating organically bound N and P. Additional value is added to this innovative wastewater treatment system by adding a gas-lift Algal Photo MBR (APMBR). The APMBR not only provides a polishing step (tertiary treatment) for Gl-AnMBR effluent by fixing downstream nutrient in algal cells, but increases the potential of energy recovery through carbon conversion to algal biofuel. Preliminary finding of this second stage of the two stage treatment process called Anaerobic-Algal MBR (A2MBR) will be shown in the subsequent chapters.

2 BACKGROUND

2.1 Literature Review

2.1.1 "Waste" Water: Problem or Solution

In 2005, a total of 410 billion gallons per day were withdrawn from different sources or fresh water in the US and 44.2 billion gallons per day were used for public supply and domestic uses (USGS, 2005). It is safe to assume that almost the totality of domestic water served is used and disregarded as wastewater. Massive amounts of water are treated every day in centralized wastewater treatment plants (WWTP) to remove organic matter, nutrients, pathogens and other contaminants; while expending equally massive amounts of energy at increased operational costs.

From a sustainable point of view, a WWTP is a factory of embedded wastewater resources (Table 2.1). Through the optimization of the treatment processes within the plant, the life cycle of many materials added upstream into the drinking water distribution system, can be extended to the point where the resulting waste products are minimal. Howard F. Curren WWTP is an excellent example of wastewater resources recovery. While providing effective sanitation to the Tampa Bay Area, nutrients are recovered as Class A pelletized fertilizer, clean water is returned to the grid for recycle, and energy is reused onsite by using the digesters biogas to generate electricity. However, some of these resource recovery practices can be maximized by including other techniques such as cultivation biofuel producing algae. At different treatment

stages, soluble forms of nitrogen and phosphorous are readily available for algal proliferation, which naturally grows on site and constitutes a problem for WWTPs in terms of maintenance (figure 2.1). Parallel and controlled cultivation of algae within a wwtp represents an opportunity to maximize downstream resources recovery.

Table 2.1: Water quality parameters in the treatment stages of conventional WWTP (Howard. F Curren AWTP, Tampa, FL) for June 2009. Concentrations reported in mg/L.

Parameter	Plant Influent	Primary settling effluent	CBOD removal effluent	Nitrification effluent	Denitrification Effluent	Final Effluent
NO3+NO2			10.71	24.31		1.22
TOTAL N						2.32
NH3		26.96	13.90	0.04		0.07
TKN	26.77	38.16	16.22	1.34		1.10
SS	97.17	99.00	9.91	8.01	0.37	0.42
TOTAL P	5.12					3.00
PO4	4.02	4.02	4.02			2.91
BOD	158.87	134.77	25.92		1.58	1.46
CBOD	122.80					1.29
COD						18.27
TOC						9.48
ALK	281.73					188.67

Figure 2.1: Native algae growing at different stages of treatment train at the Howard F. Curren AWTP. Post carbonaceous BOD removal clarifier (left), post nitrification clarifier (center) and denitrification filter (right).

2.1.2 Centralized vs. Decentralized Wastewater Treatment

Wastewater treatment in centralized facilities is a successful approach and a common denominator in urban areas of developed nations. Centralized systems represent a robust and effective treatment technology that has provided adequate sanitation for urban centers with a relatively small treatment capacity per inhabitant (Otterpohl et al., 1997). In spite of being well established technologies, centralized systems have certain characteristics that make them less desirable from a perspective of sustainability. According to Otterpohl et al. (1997), the traditional wastewater treatment causes a linear material flow that produces accumulation of contaminants (e.g. P, N, K and C) in water and food natural cycles. Also, high volumes of potable water are used to mobilize household wastewater to central facilities through extended sewage lines that require frequent maintenance. Recovery of nutrients for fertilization purposes is less feasible due to their dilution in large sewage volumes and mixed influent characteristics (Kujawa-Roeleveld and Zeeman, 2006). Conventional wastewater treatment is known for its large consumption of energy and resources. Activated sludge utilizes large amounts of energy for aeration (0.25 to 1.0 kWh/m3 for a wastewater with 500 mg/L COD) (Speece, 1996), and depending on influent strength, primary and tertiary treatment consume chemicals for solids precipitation and further disinfection (Kujawa-Roeleveld and Zeeman, 2006). Table 2 presents an example of energy consumption of a municipal treatment plant (Nouri et al., 2007). Moreover, isolated areas and new urban developments require extension of current sewer infrastructure to reach centralized WWTPs. This additional investment might not be a feasible solution for remote rural areas in developing nations.

Table 2.2: Average energy consumption in various processes of WWTP. Adapted from Nouri et al. (2007).

Process	Average energy consumption (kWh) of m³ of crude sewage)
Primary Treatment	0.01267
Primary sedimentation	0.00091
Recirculation pumping of activated sludge	0.03419
Aeration (Mixing and pumping)	0.23084
Digestion tank	0.02086
Final sedimentation	0.00068
Total Input	**0.30015**

Decentralized systems on the other hand, could overcome these problems because, besides providing efficient sanitation, they offer the possibility of on-site water reclamation, energy generation (anaerobic processes) and nutrients recovery (Kujawa-Roeleveld and Zeeman, 2006). Additionally, cluster systems should be able to avoid problems to the WWTP operation due to shock loads generated in certain points of the sewage net. Unfortunately due to the public misperception of system performance and discouraging liability laws from regulatory agencies (EPA, 2002), decentralized treatment is restrained to non-potable uses, and their potential as a water conservation alternative have been underestimated. Alternatives technologies or improvement of the current ones is necessary to assert feasibility of decentralized wastewater treatment towards water and energy conservation.

2.1.3 Anaerobic Biological Treatment

For decades, anaerobic biological treatment has been used to treat all type of waste streams. Implementation of anaerobic biotechnology presents numerous advantages over aerobic processes such as process stability, reduction of produced biomass (5% to 20% of aerobic process), smaller footprint, energy bio-generation (12×10^6 BTU per 1000 kg of COD), less maintenance requirement, reduced endogenous

decay during starvation, among others (Speece, 1996). Most of the decentralized treatment technologies currently in use are anaerobic units. In the United States, 1990 census showed that around 24% of wastewater treatment was accomplished using septic tanks (EPA, 2002). Thirty percent (30%) of sanitation in Latin American countries is achieved using septic tanks and pit latrines (Noyola, 2007). Also, countries like Brazil, Colombia and India have relied on anaerobic technologies for the treatment of domestic sewage because of their lower cost, low or no energy demand and simpler operation (Foresti, 2002; Chernicharo, 2006).

Although there are many configurations for decentralized anaerobic treatment units, septic tanks (anaerobic baffled reactors - ABR) and upflow anaerobic sludge blanket (UASB) are most commonly found in literature for real and laboratory scale applications. Besides all the advantages offered by anaerobic processes, a comparison of the two systems is summarized in Table 2.3.

Table 2.3: Comparison between ABR and UASB

Configuration	Advantages	Disadvantages	Source
ABR	High stability to hydraulic and organic influent shocks	Shallow depth of reactor to maintain acceptable upflow velocities	Barber and Stuckey, 1999
	Longer SRT due to baffle elongation of water path	Larger footprint due to shallow deep of reactors at large scale	
	Separation of anaerobic metabolism phases in baffled compartments	Uneven distribution of influent	
UASB	Increased contact of biomass with influent stream due to sludge blanket's depth	Dependence on upflow velocity for solids removal	Seghezzo et al., 1998
	Higher depth allows less footprint and reactor size	Shorter SRT due to reactor's column configuration	
	Natural mixing due to influent flow and biogas generation	No separation of acidogenesis and methanogenesis	

Energy generation is one of the most attractive characteristics of anaerobic processes. Biogas produced from influent wastewater contains about 50% of methane (Emcon Associates, 1982), which is considered the cleanest combustible fuel available. Additionally, methane has been applied for direct electricity generation as hydrogen source for fuel cells (AMI, 2000); also this gas is vastly used in industrial applications to propel pneumatic devices. Over 500,000 pneumatic devices are used in gas industry in the United States and some examples are liquid level controllers, pressure regulators, and valve controllers (EPA, 2006; Kirchgessner et al., 1997). Nevertheless, there is little information regarding application of biogas pneumatic potential within the wastewater treatment field. Biogas has been examined as a future alternative source of energy and promising opportunity for energy conservation.

In spite of these advantages, anaerobic processes are not known to reach acceptable quality level for immediate reuse and a post-treatment is required to meet water quality standards for reclamation. Table 2.4 summarizes the most relevant values of contaminants in wastewater after treatment with BAF and UASB.

Table 2.4: Example values of contaminant concentration in ABR and UASB effluents. Adapted from EPA (2002) and Chernicharo (2006).

Parameter	BOD$_5$	COD	TSS	Ammonia	TKN as N	TP as P	O&G	Fecal coliforms (log#/L)	Helminthes (egg/L)
Septic Tank (ST)	132 - 217	327-445	49-161	-	39-82	11-21.8	36-37	4.6 - 8.2	-
UASB	70–100	180–270	60–100	>15	>20	>4	-	7 - 8	> 1

*All units in mg/L unless indicated

2.1.4 The Anaerobic Membrane Bioreactor (AnMBR)

By coupling an anaerobic bioreactor with a membrane filtration unit, effluent quality of solely anaerobic treatment can be significantly improved. AnMBRs have been studied in the past decade, and their potential as sustainable sanitation and wastewater reclamation solution have been highlighted (DiGiano et al., 2004; Hu and Stuckey, 2006). In either submerged or sidestream configuration, a supplementary ultrafiltration (UF) (average pore diameter of 10 to 1000 Å) or microfiltration (MF) (average pore diameter of 0.1 to 10 um) (Baker, 2000) membrane, will provide tertiary treatment to wastewater by removing remaining pathogens and organic matter from anaerobic effluent. However, applications of AnMBRs with no auxiliary nutrient removal process (i.e. subsequent aerobic or anoxic treatment) are scarce and limited to high strength wastewaters. Table 2.5 summarizes some important application of AnMBR for wastewater treatment in the last decade.

Although MBR technology is widely known for providing excellent quality effluent, additional energy is required to drive filtration through membrane units (Zhang et al., 2003), and constant maintenance has to be performed to prevent and control membrane fouling. These two aspects represent the major challenges within the membrane biotechnology field and newer and more efficient mechanisms have to be developed to increase accessibility to MBR processes. Membrane fouling in wastewater treatment is basically produced by cake layer deposition on the membrane surface (Chang and Judd, 2002; Saddoud and Sayadi 2007; Jeison and van Lier, 2008). Many mechanisms of fouling prevention and cleaning have been developed to improve MBRs

performance. According to Yang et al. (2005), the techniques that have been used to prevent and control membrane fouling can be categorized as:

- Modification of membrane module design by optimizing the packing density of hollow fibers or flat sheets, the location of aerators, the orientation of fibers and diameters of fibers.

- Reduction of cake formation on membrane surfaces by controlling the filtration process below the critical flux, by air-sparging in the vicinity of membranes, and by operating in intermittent mode.

- Improvement of the filtration characteristics of the mixed liquor by adding powdered activated carbon (PAC).

- Removal of the fouling material after its formation by back-washing, by back-pulsing, and by chemical cleaning. But majority of these methods are chemically and energy intensive.

Table 2.5: Various applications of AnMBR for wastewater treatment

Research Description	Biological Treatment	Configuration	Membrane	Influent	In-situ Fouling Control Mechanism	Reference
Performance evaluation of anaerobic/aerobic staged reactor treating high strength wastewater with high concentrations of ammonium	UASB/Activated sludge	Submerged in aerobic zone	Capillary membrane polyethersulfone (Membrane Gmbh Company, Germany)	Three kinds of synthetic wastewater: 1300 mg COD L−1 and 110 mg NH4+-N L−1, 5250 mg COD L−1 and 610 mg NH4+-N L−1, and 10 500 mg COD L−1 and 1220 mg NH4+-N L−1	Air backwash and aeration around the membrane module	2002 - Zhang and Verstraete
Comparison between fine and coarse bubbles for air scouring in MBR	Denitrication/nitrification tank	Submerged	flat-sheet polyoletine microfiltration membrane (Kubota Co. Japan). PS = 0.4 um	Raw domestic sewage	Air sparging for nitrification	2004 - Sofia et al.
Eavalution of jet-loop circulating system to increase nitrification	Aerobic tank	Submerged in aerobic zone	Sterapore-L, Mitsubishi	Synthetic medium for nitrifying microorganisms growth. Mineral salts (NH4Cl, KH2PO4, MgSO4·7H2O, CaCl2 and FeSO4·7H2O) = 0.48 g/L	Air blowing and recirculated water (jet-loop)	2005 - Kouakou et al.
Performance of AnMBr for sulfate reduction of high salinity wastewater	Anerobic tower	Submerged	Cylindrical polysulfone membranes (Triqua B.V., Wageningen, The Nethelands). PS = 0.2 um	Acetate and ethanol as the sole electron donors operated at high salinity (50 g NaCl/L and 1 g MgCl2·6H2O/L)	External supply and recycled N2, relaxation and backflush	2005 - Vallero et al.
Performance of a SND-MBR system treating domestic sewage	Microaerobic tower (mixed microorganisms)	Submerged	U-shaped hollow-fiber membranes of polyethylene (Daiki, Japan). PS = 0.1 μm	Synthetic wastewater consisting of sugar, potato starch, peptone, meat extract, urea, NH4Cl, KH2PO4 and a mineral solution containing MgSO4 H2O, CaCl2 H2O, and FeSO4 H2O	None	2006 - Chu et al.
Performance of an Airlift External Circulation-MBR sytem for treatment and resue of toilet sewage	AEC/MBR	Submerged in aerobic zone	Hollow fiber PVDF (Tianjing Motimo Membrane Technology Ltd., China). PS = 0.2 um	Raw toilet wastewater	Air blowing and PAC	2006 - Fan et al.
Performance of and AnMBr for dilute wastewater treatment	Anaerobic baffled (1) tank	Submerged	Hollow-fiber membranes (Mitsubishi Rayon, Tokyo, Japan) and flat sheet membrane (Kubota membranes). PS = 0.4 um.	glucose, peptone 0.2 g L−1 , meat extract 0.14 g L−1 , urea 0.01 g L−1 , and NaHCO3 300 mg L−1 .	Biogas recirculation	2006 - Hu and Stuckey
Anoxic/oxic MBR retention time evaluation	Anoxic tank	Submerged in aerobic zone	A flat sheet MF membrane. PS = 0.4um	Raw domestic sewage	Air sparging for nitrification	2006 - Ng et al.

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Table 2.5: Various applications of AnMBR for wastewater treatment (cont.)

Research Description	Biological Treatment	Configuration	Membrane	Influent	In-situ Fouling Control Mechanism	Reference
Hydrogen dependant denitrification	Anaerobic tank	Submerged	Zenon ZW1	Synthetic: tap water, was composed of 25 mg l−1 NO3–N, 1000 mg l−1 NaHCO3, 25 mg l−1 KH2PO4, 5 mg l−1 CaCl2, 25 mg l−1 MgSO47H2O, and 1 mg l−1 FeSO4.	Nitrogen gas produced during denitrification was internally recycled to the membranes for scouring and reactor mixing	2006 - Rezania et al.
NORIT Arilift MBR system application	Activated slude	Sidestream	Norit X-Flow 8"	Raw municipal wastewater	Airlift, and a combination of forward and back flushing	2007 - Futselaar et al.
Application of MBR Technology to treat glue and dye wastewater	UASB/Activated sludge	Submerged in aerobic zone	UF capillar membrane module (Green Environmental Technology Company). PS = 0.036 um	wastewater from the liquid crystal display (LCD)-related industry	None (but aeration was supplied below membrane)	2007 - You et al.
Determination of optimal carbon surce	SAAR/AR (Sequencing anoxic/anaerobic and aerobic reactor)	Submerged	Flat sheet membrane	Acetate, propiuonate, glucose and methanol	Airlift was installed underneath the membrane module	2008 - Ahmed et al.
Treatment of municipal wastewater for the comparison of recirculation configurations in MBR	Anoxic/anaerobic/aerobic	Submerged in aerobic zone	Double-sided plate-frame cellulose membrane (Kubota Co., Japan). PS = 0.2 um	Medium-strength synthetic municipal wastewater	Air was introduced using filtered in-house compressed air via air diffusers placed at the bottom of the oxic compartment of the	2008 - Ersu et al.
Comparison of different AnMBR configurations for treatment of high strengh VFA wastewater	UASB	Sidestream	Polymeric inside/out microfiltration tubular membrane (Norit, The Netherlands). PS = 0.2 um	Highly saline acidified VFA stream (acetate, propionate andbutyrate). COD = 10 g/L	Biogas was recirculated and sparged inside membrane tube	2008 - Jaison and Van Lier
Performance of a SND (simultaneous nitrification and denitrification) process with an internal-loop airlift MBR	AS-MBR and BPAC-MBR	Submerged in aerobic zone	Hollow fiber PVDF. PS = 0.2 um (Tianjing Motimo Membrane Technology Ltd., China)	Synthetic wastewater was used as the influent with glucose, starch, ammonium chloride and sodium bicarbonate being the macro nutrients while peptone, KH2PO4, MgSO4-7H2O, MnSO4-7H2O, CaCl2 and FeSO4 were used as the trace nutrients.	Air blowing and PAC	2008 - Li et al.
Application of AnMBR to treat high strength dissolved petrochemical effluent	Anaerobic tank	Submerged	Flat panel Kubotaw membranes. PS = 0.45 um	High strengh Fischer-Tropsch Acid Water. COD = 18 g/L)	Compressed biogas recycle	2008 - Van Zyl

2.1.5 Airlift Fouling Control

Recently, special attention has been given to physical fouling prevention through membrane scouring with air bubbles for sidestream MBRs. Table 2.5 also presents the fouling control mechanism selected by several researchers. In those, variations in the bubble scrubbing system, according to membrane configuration, can be highlighted as the primary fouling control technique. From these methodologies, airlift membranes (using ultrafiltration capillary tubes) present a promising approach to prevent fouling, increase membrane flux and decrease energy consumption. By arranging the sidestream capillary membrane tubes vertically and supplying pressurized air at the bottom, right where the bioreactor's effluent gets into the membrane; the sparged air is responsible for sludge recirculation through the membrane (inside-out filtration) and it also increases turbulence and shear over membrane surface.   Results of airlift MBR configurations indicate longer operation (up to 8 months) before off-site cleaning (Sofia et al., 2004), up to 43% improvement of permeate flux (Chan and Judd, 2002), decreased transmembrane pressure requirement, and considerably less energy used for pumping (Table 2.6) (Yeh et al, 2006). Commercially available airlift membrane modules (e.g., Norit X-Flow[*] and HyperFlux[†]) are evidence of efficient performance of airlift system. Nevertheless, there is no published information regarding performance of biogas gas-lift for domestic wastewater treatment in an AnMBR.

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* Norit X-Flow company. http://www.norit.com/?RubriekID=2029

† Berghof Filtration. http://www.berghof-filtration.de/en/Products+and+Solutions/Tubular+Modules.html
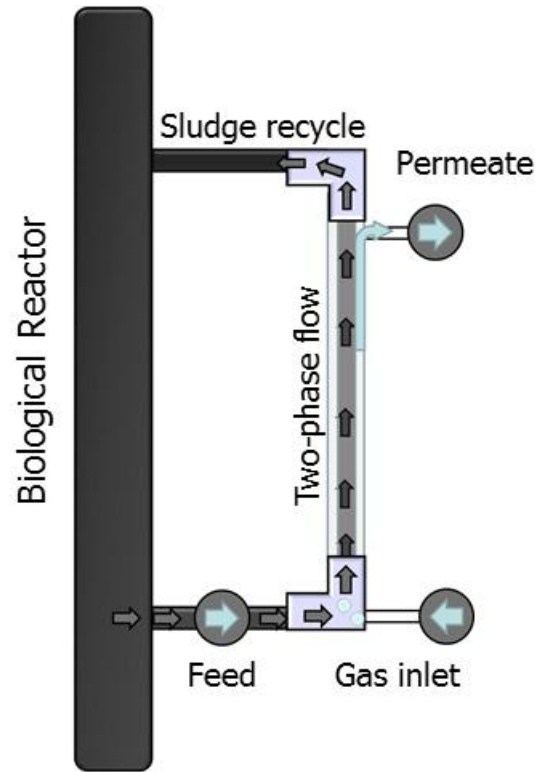
Figure 2.2: Example of air-lift MBR system using capillary membranes (inside-out filtration) by Norit X-Flow (Futselaar et al., 2007). Air introduced at the bottom of the membrane module creates two phase gas/liquid flow inside the capillary membrane tubes, thereby generating lift for the recirculating sludge and turbulence to mitigate membrane fouling.

Table 2.6: Comparison of external power consumption of tubular membrane configurations for treatment capacity of 100,000 GPD. Adapted from Yeh et al, 2006

Item	Cross-Flow	Airlift
Membrane pumps (bHP)	65	4.0
Membrane blower (bHP)	-	6.0
Backwash pump (bHP)	-	0.2
Permeate pump (bHP)	-	0.1
Total power (bHP)	65	10.3
Annual power (0.11/kWh)	$46,976	$7,444
bkW/m³	3.1	0.5

Table 2.7: Removal efficiencies and energy demand in different WWT technologies

Parameter	Removal Efficiency (%)									
	ST[1]	UASB[2]	CAS[3]	Sub-AnMBR[4]	Sub-AnMBR (gas-scouring)[5]	Cf-AnMBR[6]	Sub-MBR[7]	Sub-MBR (air-scour)[8]	AL-MBR[9]	Cf-MBR[10]
SS	83	80	100	100	100	100	100	100	100	100
COD	72	70	88	97	95	96	96	95	96	98.4
Nitrogen	26	60	81	60	60*	60*	90	80	80*	80*
Phosphorus	--	35	90	35*	35*	35*	30	40	40*	40*
Parasites	--	75	--	100	100	100	100	100	100	100
Bacteria	90	90	99.99	100	100	100	100	100	100	100
Viruses	--	--	99.9	100	100	100	100	100	100	100
Energy (KWh/m3)	0	0	0.30	--	--	--	0.55	0.45	0.5	3.5

Data from: 1) EPA; 2) Chernicharo (2006); 3) Lopez-Vazquez et al. (2008), Zhang and Farahbakhsh (2007), Nouri et al. (2007); 4) Wen et al. (1999), Anh et al. (2003); 5) Hu and Stuckey (2006); 6) Saddoud and Sayadi (2007); 7) Judd (2006); 8) Rosenberg (2002), Cote (2000); 9) Judd (2006), Yeh et al. (2006); 10) Judd (2006).
*Nutrient removal efficiencies for anaerobic and aerobic MBRs were assumed to follow the same pattern as UASB and CAS respectively

Various studies have looked into low strength wastewater treatment using AnMBRs and reported successful control of membrane fouling by gas scouring (Valero et al., 2005; Hu and Stuckey, 2006; Rezania et al., 2006; Jeison and Van Lier, 2008; Van Zyl et al., 2008). Still, there is little discussion regarding effect of biogas bubbling on the chemistry of anaerobic sludge recycled through a *gas-lift anaerobic MBR* (GL-AnMBR) system. Decreased pH by gas bubbling has been reported by Lei et al. (2007) as part of the pretreatment for an anaerobic digestion effluent. This is due to the dissolution in water of the CO_2 contained in the biogas. Additionally, there are no reports related to stripping of volatile organic carbons (VOC) by biogas produced in anaerobic reactors (Farhadian et al., 2008). Change in sludge composition and characteristics due to gas-lift configuration, is another potential issue requiring investigation.

2.1.6 Energy Considerations in a MBR System

Zhang et al. (2003) evaluated the energy requirement of a transverse flow membrane module by identifying the principal points of energy demand in this type of aerobic MBR. Using a similar approach and including other considerations related to MBR's usual requirements, the energy demand in an AnMBR compared to an aerobic MBR can be discriminate as follows:

Aerobic MBR

E1 = energy consumption by oxygen supply in aeration tank
E2 = energy consumption by pipe system
E3 = velocity energy loss
E4 = energy consumption by membrane module (function of permeate and recirculation flow rates)
E5 = energy consumption for air scrubbing
E6 = energy consumption by pump
E = E1 + E2 + E3 + E4 + E5 + E6

Anaerobic MBR

E1 = energy consumption by heating bioreactor
E2 = energy consumption by pipe system
E3 = velocity energy lost
E4 = energy consumption by membrane module
E5 = energy consumption for gas scrubbing
E6 = energy consumption by pump
E7 = energy produced in biogas
E = E1 + E2 + E3 + E4 + E5 + E6 − E7

In the case of an AnMBR, energy for aeration was replaced by heating demand in the bioreactor and an additional term is added to account for biogas production. It has been stated that produce biogas can offset heating requirement, which reduced total energy demand in and AnMBR to five terms (E = E2 + E3 + E4 + E5 + E6). Additionally, operational parameters adapted from optimized aerobic MBR systems to anaerobic conditions (i.e. AL-MBR to GL-AnMBR), could potentially decrease energy requirement even further:

- For an air-lift supported configuration, energy loss due to friction in the pipeline is minimal due to very low recycle flows (van 't Oever, 2007), therefore E3 decreases.

- Two-phase flow in an air-lift supported membrane allows turbulence to create shear over membrane surface which improves membrane flux, and decreases energy demand for sludge recirculation, bubbling and filtration (i.e. E4, E5 and E6) (Futselaar et al., 2006)

Presently, overall energy requirements for AL-MBR (NORIT X-Flow) are in the range of 0.4 - 0.7 kWh/m³ and recent optimization of the system at pilot plant scale (i.e. San Diego, California) has reached 0.25 KWh/m^3 (Miller et al., 2008); which is lower than current energy values for conventional activated sludge systems (0.3 - 0.4KWh/m^3) (Zhang et al., 2003; Nouri et al., 2007). If AL-MBR concepts are applied in anaerobic conditions to GL-AnMBR, this technology could result in a sustainable solution for low strength wastewater treatment and resource recovery.

2.1.7 Alternative Energy Production from Anaerobic WWT

Besides the mentioned advantage of anaerobic WWT for energy production through methane generation, an indirect source of energy derived from this process could be localize in biomass growth. As it was mentioned before, the effluent from anaerobic processes are characterized by high concentrations of nutrients that usually require a further polishing step for either reuse applications or to meet local legislation discharge requirements. Taking advantage of the fertilizing potential of wastewater

embedded nutrients, is a task that can be easily fulfilled by photosynthetic organisms (i.e. biomass growth). Further conversion of biomass into energy such as biofuel and biogas, provides an additional value to the resource recovery cycle and opens an opportunity in the blooming market of renewable energy.

In the United States, the current clean energy market has localized its efforts in the production of biofuel from feedstock related technologies that include corn, soy, woodchips and algae (F2F Summit, 2009). Within this range of possibilities, algae provide a unique opportunity of making clean energy development possible. The aquatic plant naturally stores its energy as lipids, which can eventually be converted into various types of fuel. Algae can be artificially induced in freshwater or wastewater therefore it does not take up valuable cropland or conflict with food prices. Also, algae can be grown very fast which can enhance production efficiency over time. Growth is stimulated through the introduction of nutrients which facilitate artificial development of cultures. In fact, algae can produce a number of different energy fuel types and can be refined using existing oil infrastructure. Additionally, harmful global warming emissions are mitigated, as algae consume carbon dioxide.

Although different types of algae have been proven to grow in aggressive and very diverse environments (Table 2.8), the potential of biofuel producing algae to grow from wastewater has recently gained a lot of interested since it has been highlighted as one of the most sustainable source of clean energy (Farm to fuel summit, 2009). Readily available nutrients, water and carbon in a wastewater treatment plant, make it an ideal location for cultivation of biofuel producing algae, but information about the feasibility of

implementing an algal photoreactor in an AWTP scenario is limited, specifically referring to nutrient recovery, savings on chemical demand and energy consumption. Table 2.8 summarizes some of the most relevant studies on biofuel algal growth from wastewater. Although several studies have emphasized on the effectiveness of microalgae to remove nutrients, carbon and even organic matter through heterotrophic growth (Hammouda et al., 1995; Travieso et al., 2006; Wang et al., 2010a and 2010b), the application of continuous-flow algal bioreactor configurations present some limitation for microalgal growth since optimal operation of these systems with such slow growing organism requires extended acclimation stages, enhanced cell biomass concentration (e.g. overcome wash out of algal cells) and steady nutrient removal performance with variable influent conditions (Mallick, 2002).

Table 2.8: Algal production form wastewater effluents

Substrate /Pretreatment	Reactor type (batch/flow through)	Algal strain	Removal efficiencies	Reference
Cheese factory anaerobic/aerobic digestion effluent	B – 1.8 L	*Phormidium bohneri Micractinium pusilum*	100% NH4 removal and 2.9 and 2.5 mg P-PO4/L-d respectively	Blier et a., 1995
Aquaculture wastewater	B and F – 15 and 16 L respectively	*Chlorella sp Scenedesmus sp.*	83% BOD. ~90% COD and 100% NO3 and NH4	Hammouda et al, 1995
Agroindustrial waste	B – 4 L and 50 L	*Chlorella vulgaris Scenedesmus dimorphus*	---	Gonzalez et a.,l 1997
Synthetic wastewater	B - 500-mL	*Rhodobacter sphaeroides Chlorella sorokiniana Spirulina platensis*	---	Ogbonna et al., 2000
WW from ethanol and citric acid production		*Chlorella vulgaris Lemna minuscula*	73% NH4 and 51% PO4	Valderrama et al., 2003
Secondarily treated sewage/AS	B - 3 L	*Botryococcus braunii*	Nitrate: 99.9% Phosphate: from 0.02 to <0.01 mg P/L	Tsukahara and Sawayama, 2005
Settled and diluted piggery waste	B – 1 L	*Chlorella vulgaris*	COD removed at 190h HRT: were 88.0%, 57.5%, 55.6%, 56.5%, 60.6% and 20.6% for initial COD concentrations of 250, 400, 520, 650, 800 and 1100 mg/l, respectively.	Travieso et al., 2006
Sterilized effluent from two-stage AD of two-phase olive mill solid waste (OMSW)	B – 500 ml	*Chlorella zofingiensis*	TCOD and SCOD removals of 37% and 45% respectively at HRT 11 days	Travieso et al., 2008
WW obtained from the inlet channel to the Nehru Vihar OPS at Delhi		*Chlorella minutissima*	---	Bhatnagar et al., 2009
Effluent of AD of livestock waste	B – 1 L	*Scenedesmus sp*	---	Park et al., 2010
AD of dairy manure	B – 250 ml	*Chlorella sp.*	Removal of NH4, TN, TP and COD:100%, 75.7–82.5%, 62.5–74.7%, and 27.4–38.4%, respectively	Wang et al, 2010a
Effluent from WWTP. Before primary settling (#1), after primary settling (#2), after activated sludge tank (#3), and centrate (#4)	B – 250 ml	*Chlorella sp.*	Removals of NH3-N, PO4-P, TN and COD respectively: #1: 82.4%, 83.2%, 68.4, 50.9% #2: 74.7%, 90.6%, 68.5%, 56.6% #3: n/a (62.5% removal of NO3-N), 4.69%, 50.8%, -22.7% #4: 78.3%, 85.6%, 82.8%, 83.0%	Wang et al., 2010b

In terms of energy generation, algal biofuel extraction and processing, and anaerobic digestion of algal biomass are established methodologies that have been extensively reported (Golueke et al., 1956; Sialve et al., 2009). Other techniques such as bio-hydrogen generation will not be discussed in detail since limitations regarding specificity of algal strains and growth conditions (Li, 2008), establishes a gap between this particular energy generation process and the intrinsic randomness of wastewater based media. Table 7 also presents some of the most studied microalgal species either acclimated to, or isolated from wastewater substrates. The first species of green algae, *Botryococcus braunii*, can contain up to 75% of its dry weight as hydrocarbon oil (Chisti, 2007). This oil is similar enough in composition to crude oil, that it can be processed in the same cracking facilities, to yield 67% gasoline, 15% aviation fuel, 15% diesel, and 3% residual oil (Hillen et al., 1982). The latter one, *Chlorella sorokiniana*, have been widely known for its capacity to grow from sub optimal substrates such as wastewater and produce lipids at concentrations up to 32% of its dry weight (de Bashan et al., 2008; Chisti, 2007). However, quantification of the specific algae derived energy depends on the initial cultivation methods, nutrient demand and final conversion approach. Table 2.9 summarizes the overall energy inputs and outputs for algal oil production (Chisty, 2008).

Table 2.9: Generalized inputs and outputs for algal biofuel production. Adapted from Chisty (2008)

Inputs	MJ/Kg oil	kWh/kg oil
Energy in fertilizers	14.12	3.92
Energy for cultivation	8.77	2.44
Energy for harvesting	0.30	0.08
Energy for oil recovery	3.17	0.88
Energy for biogas production	0.88	0.24
Energy for construction (entire facility including maintenance)	4.00	1.11
Energy embodied in equipment (including maintenance)	0.00	0.00
Outputs		
Energy in algal oil	37.90	10.53
Energy in biogas from residual biomass digestion	50.00	13.89

2.2   Problem Statement

From the above review, the following challenges regarding domestic sewage treatment using AnMBR and algal photobioreactor are identified:

- For laboratory bioreactor studies, access to actual sewage is often not possible. Yet, there is a lack of suitable synthetic surrogates which resemble the major properties of actual sewage, namely complex particulate organic matter.

- Although energy production is an attractive outcome of anaerobic treatment, AnMBRs are generally not yet optimized to decrease energy consumption associated with membrane operation. Typically, the energy balance of inputs and output has not been optimized towards energy surplus.

- Analogous to air scouring, biogas scouring can potentially improve membrane flux in anaerobic systems. However, there is little knowledge regarding effects of extensive biogas bubbling on solution chemistry and anaerobic process stability. For example, possible loss of substrate compounds such as volatile fatty acids

(VFA) and CO_2 can be caused by stripping. The potential CO_2 loss in this type of systems can lead to pH changes and system instability.

- There is lack of information related to maximizing biogas production rate in an AnMBR treating low strength wastewater for the specific purpose of satisfying gas-lift system bubbling demand. Furthermore, membrane fouling in GL-AnMBR systems is not at all characterized.

- The design and operation of AnMBR have not yet been optimized to fully recover wastewater intrinsic resources (e.g. water, energy and nutrients). Also, a suitable decision making process has not yet been established for this purpose.

- Relatively little information is available on the use of anaerobic effluent, especially from AnMBR, to support the growth of algae for further energy recovery and nutrient utilization.

- Cell separation (for biomass retention and clarified effluent) remains a challenge in continuous flow algae photobioreactors used for wastewater treatment, thereby limiting hydraulic loading. The application of cell-separation membranes in an algal photobioreactors, or algae photo MBR, has not been reported.

2.3   Research Hypotheses and Objectives

The overall goal of this investigation is to develop a combined process, utilizing AnMBR and algae photobioreactor, to treat domestic wastewater for the targets of improving sanitation and recovering and reusing wastewater intrinsic resources (water, energy and nutrients). Research is driven by the following hypotheses:

- For laboratory studies, sewage can be represented by a surrogate material that is readily available commercially and represents the salient complexities of actual sewage.

- A tubular gas lift ultrafiltration membrane can be used in an anaerobic membrane bioreactor to create a low energy footprint approach to recover energy from wastewater, and potentially be energy surplus.

- Anaerobic MBR effluent, which is nutrient rich and optically clear, can be a suitable feedstock for growing and sustaining biofuel-producing microalgae in a photobioreactor.

- Similar to aerobic and anaerobic MBRs which separate sludge from effluent, solid/liquid-separation ultrafiltration membranes (in particular gas lift) can be combined with an algal photobioreactor to create an algal photo MBR. The continuous flow system will be characterized by concentrated algal biomass and high quality effluent.

- Anaerobic and algae bioprocesses can be combined to maximize resource recovery from wastewater

The overall research goal is pursued through the following research objectives:

The *first objective* is to identify, evaluate and characterize a synthetic surrogate for domestic sewage which is suitable for laboratory wastewater treatment studies.

The *second objective* is to construct a prototype GL-AnMBR for domestic wastewater treatment. Specific objectives consist of:

- Characterization of treatment performance using the synthetic sewage

- Evaluation of effluent quality for different reuse applications

The *third objective* is to evaluate suitability of using biogas for gas-lift membrane filtration in an anaerobic bioreactor. Specific objectives consist of:

- Determining the sufficiency of the produced biogas to sustain membrane gas scrubbing

- Determining the effects of short- and long-term gas-lift operation on possible membrane fouling under anaerobic conditions

- Devising strategies for maintaining sustainable membrane flux using gas-lift

The *objective four* corresponds to the exploration of the Gl-AnMBR design and operation to minimize energy footprint

- Minimize energy consumption (kWh/m^3) for gas-lift membrane operation

- Identify and harness available energy associated with system to offset treatment demands

Lastly, the *fifth objective* is to develop a continuous-flow algal photobioreactor using a cell-separation membrane to retain biomass and clarify the effluent

- Couple the APMBR to the AnMBR to grow algae using AnMBR effluent for further resource recovery

- Evaluate APMBR performance and effluent quality

- Determine overall performance of combined A2MBR

2.4   Phases of Study

The performance of the sequential anaerobic and algal membrane bioreactor system for treatment of domestic wastewater for resource recovery was conducted in three major study phases:

2.4.1 Phase 1: Substrate Characterization for Anaerobic Biodegradability

During this phase, a synthetic domestic sewage was characterized and evaluated as a surrogate of domestic wastewater. Complex organic particulate artificial sewage (COPAS) was used for its similarities in COD, nutrients and particulate matter content to actual sewage. Since this proposed substrate is introduced as solid granules, the suitability of COPAS as a reliable carbon and nitrogen source was evaluated through dissolution tests. Additionally, biological degradation of COPAS was investigated based on methanogenic activity and hydrolysis (i.e. VFAs production) of the compound in an anaerobic environment. Acclimation of anaerobic sludge flora to COPAS was also performed during this phase. Results from this phase were used for later on in the research for mass balance calculations in lab scale operation and further applications.

2.4.2 Phase 2: GL-AnMBR Design, Fabrication and Performance Evaluation

An anaerobic bioreactor column coupled with a sidestream gas lift membrane unit (i.e., Norit X-Flow) was selected as the most advantageous configuration for this study. A gas-lift system was evaluated for its performance in domestic wastewater treatability. Preliminary sludge filterability tests defined the hydraulic conditions for the continuous operation of the membrane module. Results from the extended operation of the Gl-AnMBR are also presented during this phase. Average biogas production was evaluated in this stage and obtained values were used in for energy estimations of net power demand.

2.4.3 Phase 3: Development of a Proof-of-Concept APMBR

In this stage, the resource recovery potential of the Gl-AnMBR is highlighted in an application that includes photosynthetic biomass growth and generation of green energy. Batch experiments using Gl-AnMBR permeate for the growth the biofuel producing microalgae *Chlorella sorokiniana* were performed. Nutrient consumption/removal efficiencies of this algal strain are reported. A gas-lift APMBR was designed, fabricated, and tested, using AnMBR effluent as feedstock, to demonstrate the feasibility of the integrated system for resource recovery from wastewater.

3 ANALYTICAL METHODS AND GENERAL PROCEDURE

3.1   Analytical Methods

In all samples, *pH* was measured with a digital pH meter (Corning pH/ion analyzer 350) and a gel-filled combination pH electrode (Model 2411-10, Cole Palmer, Vernon Hills, IL). The meter was calibrated before every measurement with standard buffer solutions of pH 4.0, 7.0 and 10.0 (Fisher Scientific, Pittsburg, PA). The electrode was rinsed with distilled water and dried with a tissue before and after every sample measurements.

Chemical oxygen demand *(COD)* values were obtained using Hach HR COD digestion vials (Hach Company, Loveland, CO). Each vial contains a 5 ml of reagent solution ready to be used. The main ingredients of the reagent solution are mercuric sulfate, silver sulfate, chromic acid, sulfuric acid and demineralized water in proportions described somewhere else (MSDS for Digestion Solution for COD 20-1500 mg/l Range, Hach Company). For COD measurement, 2 ml of sample should be added to each vial and digested for 2 hours at a temperature of 150°C. During digestion, oxidizable organic compounds react reducing the dichromate ion ($Cr_2O_7{}^{2-}$) to green chromic ion (Cr^{3+}) (HACH procedures). The concentration of Cr^{3+} is determined using colorimetric method

at a wavelength of 620 nm in a spectrophotometer (Model DR/4000U, Hach Company, Loveland, CO). This method is approved by USEPA (USEPA, 1980).

DOC and DN content in liquid samples were measured using a Total Organic Carbon analyzer (Shimadzu TOC-V CSH) coupled with a Total Nitrogen detector (Shimadzu TNM-1) (Shimadzu Scientific Instruments, Inc., Columbia, MD). Carbon analysis (Total, Organic and Inorganic) were based on catalytic combustion of sample at $680^{o}C$ and Non-Dispersive Infrared (NDRI) method. Nitrogen measurement was based on detection of produced NO (nitrogen monoxide) from combusted sample by chemiluminescence method. Depending on their initial solids concentration, liquid samples were taken for DOC measurement after filtration through a 0.45 um filter (TS<1000 ppm), or from the supernatant after centrifugation (e.g. Sludge samples). Acidification was done externally by adding 1N HCl to a pH below 2. Samples were sparged with Ultra Zero Grade Air for 2 minutes, to remove inorganic carbon (i.e. CO2) before measuring. Calibration curves for organic carbon and nitrogen were done using standard solutions of reagent grade potassium hydrogen phthalate (KHP) and potassium nitrate respectively (KNO_3).

POC was measured on the fraction of suspended solids in samples with high content of organics. A Shimadzu TOC-V CSH coupled with a Solid Sample Module (Shimadzu SSM-5000A) was used for this purpose (Shimadzu, Columbia, MD). Carbon analysis (Total, Organic and Inorganic) were based on catalytic combustion of sample at $900^{o}C$ and Non-Dispersive Infrared (NDRI) method. Before analysis, samples of known volume (0.3 ml) were filtered through a 2.5 mm diameter Whatman GF 934/AH glass

fiber filter (Whatman Ltd. 2007-2009) as recommended by the ALPHA (2005) standard methods for separation of suspended solids in wastewater samples. To correct for the additional carbon content available on the filter, clean filters were combusted prior to each set of measurements and the obtained value was used as blank for each run.

The total *biogas production* in batch experiments (i.e. serum bottles) was measured using the volume displacement method in a graduated burette. The accumulated pressurized gas in the headspace of the sealed bottles is released through rubber tubing connected to the top of a graduated burette. The burette, acting as a manometer, is filled with water to a known initial volume. The gas is left to equilibrate to atmospheric pressure while displacing the water in the burette to a final volume. The change is volume (DV) is quantified as produced gas (generally in ml). To measure the produced gas in a larger scale (e.g. bioreactor), a wet tip meter (WTM) was used (www.wettipgasmeter.com). A submerged double sided inverted tipping bucket receives the raising gas produced in the reactor. When one side of the bucket is filled (calibrated to a volume of 100 ml/tip), it tips to allow the other side to be filled. Every tip generates a pulse that is quantified over time by an on line data collection system.

Gas composition was analyzed for carbon dioxide (CO_2) and methane (CH_4) using a gas chromatography (GC) unit (Agilent 7820A) equipped with a thermal conductivity detector (TCD) and a 30-m J&W 113-3133 GS-CarbonPLOT, 0.32 mm diameter column (Agilent Technologies, Lexington, MA). The inlet, oven and detector temperature were set at 185°C, 50°C and 160°C respectively. Helium was used as carrier gas at 1.3 mL/min. A volume of 200 ul of gas samples was manually injected to the instrument

using a 500 ul glass gas-tight syringe (National Scientific, Rockwood, TN). Calibration curves were done using CO_2 and CH_4 with a purity >99.9%. A sample of atmospheric air was injected before every run to check for anaerobic conditions in each bottle. The output signal of the instrument was processed in personal computer using the GC Chemstation software (Agilent Technologies, Lexington, MA) software included with the instrument.

The volatile fatty acids (*VFAs)* (i.e. acetic, propionic, butyric and valeric acids) were monitored in liquid samples using a GC unit equipped with a flame ionization detector (FID) and a 30-m Restek 11025 Stabilwax DAm 0.53 mm ID column (Restek Corp. Bellefonte, PA). The inlet and detector temperature were both set at 250°C. Helium was used as carrier gas at 4.5 mL/min and the following program was used for the oven temperature: 90 °C for 0.5 min, 2 °C/min to 100 °C, 6 °C/min to 120 °C, 30 °C/min to 230 °C for 15 min. The total run time was 27.5 min. Before measurement, samples were filtered through 0.22 um membrane filter and acidified with equal volumes of 2.5% phosphoric acid. Calibration curves for VFAs were done using pure acids (i.e. >99.0% purity acetic, propionic, butyric and valeric acids) dissolved in deionized water with 1.25% phosphoric acid.

Ammonia *(NH$_4^+$)* values were determined using Hach Test'N Tube™ Nitrogen-Ammonia vials, Salicylate Method for 0.4-50 mg/L range concentration (Hach Company, Loveland, CO). Each vial contains 5 ml of demineralized water, to which 0.1 ml of sample be added. Ammonia Salicylate reagent powder and Ammonia Cyanurate reagent powder are added to each vial to react with the ammonia present in the sample. The

main ingredients of the Ammonia Salicylate reagent are Sodium Salicylate and Sodium Nitroferricyanide in proportions described somewhere else (MSDS for Ammonia Salicylate and Ammonia Cyanurate Reagents for NH_3-N 0.4-50 mg/l Range, Hach Company). For the Ammonia Cyanurate reagent, the main ingredients are Sodium Dichloroisocyanurate, Lithium Hydroxide, Sodium Citrate and Sodium Tartrate; in proportions described somewhere else (MSDS for Ammonia Salicylate and Ammonia Cyanurate Reagents for NH_3-N 0.4-50 mg/l Range, Hach Company). Prior to NH_3 measurement, prepared vials should be allowed to react during 20 minutes where the chloramines present in solution react with salicylate to form 5-aminosalicylate that oxidizes in the presence of a sodium nitroprusside catalyst. The product of the reaction is a blue colored compound that, combined with the remaining excess reagent, turns into a green colored solution that can be measured through colorimetric. A spectrophotometer is used for this purpose at a wavelength of 655 nm (Model DR/4000U, Hach Company, Loveland, CO).

Nitrate *(NO₃)* values were determined using Hach Test NitraVer® X Test'N Tube™ vials, for 0-30 mg/L range NO_3 concentration (Hach Company, Loveland, CO). Each vial contains a solution of demineralized water and sulfuric acid, to which 1 ml of sample is added. This sample is measured as the blank. In a following step, a reagent powder is added to each vial to react with the NO_3 present in the sample. The main ingredients of the powder reagent are Urea, Chromatropic Acid (disodium salt), White Quartz Sand and Sodium Metabisulfite in proportions described somewhere else (MSDS for Nitrate NitraVer® X Nitrogen, Nitrate Reagent B, Hach Company). Prior to NO_3 measurement, prepared vials should be allowed to react during 5 minutes where the

NO_3 present in solution reacts with the chromotropic acid under strongly acidic conditions. The product of the reaction is a yellow colored compound that can be measured through colorimetric methods. A spectrophotometer is used for this purpose at a wavelength of 410 nm (Model DR/4000U, Hach Company, Loveland, CO).

Phosphate *(PO4)* values were determined using Hach Test Total Phosphorus High Range (HR) Test 'N Tube^{TM} vials, for 1-100 mg/L range PO_4^{3-} concentration (Hach Company, Loveland, CO). Each vial contains a solution of demineralized water and sulfuric acid, to which 5 ml of sample is added. In a following step, Potassium Persulfate powder is added to each vial to react with the organic and condensed inorganic forms of PO_4^{3-} present in the sample. Prepared vials should be digested during 30 minutes allowing the persulfate, heat and acid conditions to convert the organic PO_4^{3-} into orthophosphates. After digestion, 2.0 mL of 1.54 N Sodium Hydroxide and 0.5 mL of Molybdovanadate Reagent are added to each vial to allow orthophosphates to react with molybdate in an acid medium to produce yellow molybdovanadophosphoric acid forms in the presence of vanadium. The product of the reaction can be measured through colorimetric methods. A spectrophotometer is used for this purpose at a wavelength of 420 nm (Model DR/4000, Hach Company, Loveland, CO). The main ingredients of the Molybdovanadate Reagent are Ammonium Molybdate, Ammonium Metavanadate, Sulfuric Acid and Deminerilized Water, in proportions described somewhere else (MSDS for Phophorous Total Phosphorus HR, Molybdovanadate Reagent, Hach Company). This method is an adaptation of the 4500-P B-C ALPHA methods (ALPHA, 2005).

Solid concentrations in different samples were measured according to the 2540 ALPHA standard methods for the examination of water and wastewater (ALPHA, 2005). For TS, a pre-weighted aluminum plate was filled with a known volume of sample and then dried at a temperature of $105^{o}C$ in an oven. Dried samples are then removed from the oven, allowed to cool down in a desiccator and weighted again. The difference between the final minus the initial weight, divided by the sample volume results in the TS concentration. This value is usually corrected to have mg/L o ppm units. For VS, the sample is then ignited in a muffle furnace at a temperature of $550^{o}C$. After 5 to 15 minutes, the sample is removed from the furnace, cooled down in a desiccator and weighted again. The difference between the final weight of the plate after dried at $105^{o}C$ and the final weight after ignited at $550^{o}C$ divided by the sample volume results in the VS concentration (usually corrected to mg/L or ppm).

For TSS, a similar procedure to the TS concentration is followed. The sample has to be prepared to separate the suspended solids and dissolved solids by filtration. A 1.5 um fiber glass filter (2.5 mm diameter Whatman GF 934/AH - Whatman Ltd. 2007-2009) is used for this purpose. In this case, the aluminum plate and the filter (pre-dried in an oven during 2 hours at $105^{o}C$) are weighted together on a balance (AE 260 DeltaRange, Mettler Toledo Inc., Columbus, OH). The filter is then used to filter a known volume of sample. After filtration is complete, suspended solids remain on the filter that is put back into the aluminum plate and dried at a temperature of $105^{o}C$ in an oven. Dried samples are then removed from the oven, allowed to cool down in a desiccator and weighted again. The difference between the final minus the initial weight, divided by the sample volume results in the TSS concentration. This value is usually corrected to have mg/L o

ppm units. For VSS, the dried plate-filter is then ignited in a muffle furnace at a temperature of 550°C. After 5 to 15 minutes, the plate is removed from the furnace, cooled down in a desiccator and weighted again. The difference between the final weight of the plate-filter after dried at 105°C and the final weight after ignited at 550°C, divided by the sample volume results in the VSS concentration (usually corrected to mg/L or ppm).

3.2   Membrane Filtration System

3.2.1 The Ultrafiltration Membrane

Ultrafiltration membrane tubes (e.g., X-Flow by Norit Membrane Technology, Enschede, NL) have been used in this investigation, as they have been successfully applied in airlift MBR systems (e.g., Dynalift MBR). The membrane used is a 5.2 mm diameter polyvinylidene fluoride (PVDF) tubular membrane (Norit X-Flow, F4785) (Norit Membrane Technology, Enschede, NL) with a mean pore size of 0.3 um and active filtration area of 0.013 m^2. Custom membrane modules were fabricated in the lab to better control the membrane performance. The main components of the membrane module are showed in Figure 3.1.

3.2.2 The Experimental Set Up

The Gl-AnMBR experimental set-up shown in Figure 3.1 consists of an 8.5 L Anaerobic Bioreactor, coupled with a sidestream gas-lift ultrafiltration module. Due to the particulate nature of the influent, the reactor was fed in batches every 6 hours using a piston pump (FMI Q pump with a 3/8" diameter piston, Fluid Metering Inc., Oysterbay,

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NY). Filtration is mainly driven by applying vacuum to the membrane permeate side with a variable speed pump (Masterflex L/S, Cole Palmer, Vernon Hills, IL). Membrane Cross flow velocity (CFV) was controlled using a peristaltic pump (Masterflex L/S, Cole Palmer, Vernon Hills, IL). Membrane effluent was measured with an on-line rain gage and transmembrane pressure (TMP) was measured by placing on-line pressure transducers at the feed (P_f), permeate (P_p) and recycle (P_r) lines of the membrane module. Gas from the head space was compressed using a peristaltic pump (Masterflex 7520-25, Cole Palmer, Vernon Hills, IL) and applied for membrane scrubbing. The gas flow rate (Q_g) was controlled visually with a gas flow meter and a needle. Total biogas exiting the MBR was measured using a wet tip meter (WTP). The retentate is separated from the gas in an intermediate tank prior to recycling it back to the anaerobic reactor. Temperature at the bioreactor was regulated with hot water circulating in a hose around the column and continuously monitored inside the reactor and at the membrane feed line using on-line sensors.

Originally, the anaerobic reactor configuration to be used in these studies was an Upflow Anaerobic Sludge Blanket (UASB). However, several modifications to the original airlift filtration concept had to be considered for its application to an anaerobic bioreactor such as the UASB. First, the supernatant of the UASB is used as influent for the membrane, which requires the placement of a recycle pump that controls membrane feed the flow rate (Q_f) and CFV. The hydrostatic head available from the reactor column becomes irrelevant for this particular UASB-membrane application. Second, Q_f has to be maintained at a point where upflow velocity of the reactor and the CFV are within acceptable operational conditions for both UASB and membrane. For these reasons and

to maintain the MBR operation at minimum energy consumption, the biological reactor

was mainly maintained as a complete mixed anaerobic column.
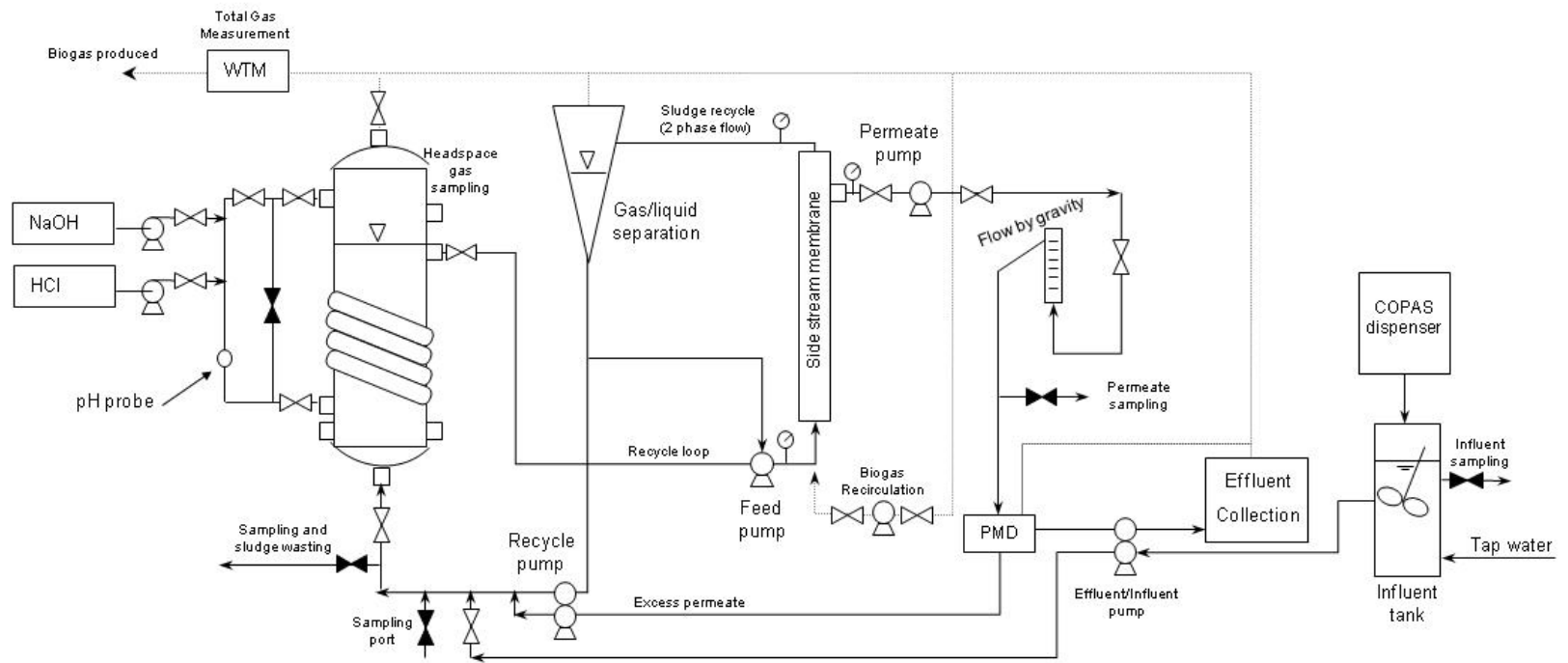
Figure 3.1: Schematic of the final GI-AnMBR configuration

3.2.3 Data Collection and Processing

During continuous MBR operation, the on-line sensors were connected to a real time data acquisition device (HOBO® Weather Station Data Logger model H21-001, ONSET Computer Corporation, Cap Cod, MA), which was connected to a personal computer at all times. Since the on-line sensors only transmit voltage differentials (e.g. pressure transducers and temperature probes) and/or pulse outputs (e.g. rain gage and WTM), each sensor was calibrated for the desired measurement before allocating it on the reactor.

For the membrane module used in this configuration, the TMP was calculated using the equation:

$$TMP = \frac{Pf\text{-}Pr}{2} - Pp$$

Where Pf corresponds to the pressure at the membrane feed line, Pr corresponds to the pressure at the recycle/concentrate line, and Pp corresponds to the pressure at the permeate side. All measurements are reported in Bars. Since the membrane module is placed in a vertical fashion, the average of the feed and recycle pressures is a simplified calculation of the pressure differential throughout the length of the membrane before filtration. Since the permeate flowrate (Qp) is measured in real time with a rain gage, the permeate flux is calculated using the following equation:

$$J = \frac{Qp}{Am} \times \frac{60}{1000}$$

Where:

J = measured flux reported in units of L/m^2-hr (LMH)

Qp = permeate flow rate in units of mL/min

Am = active membrane filtration area in units of m^2

The total filtration resistance is calculated based on the measured TMP and J. A modified version of the Darcy's law for the discharge of liquid through a porous media, the total filtration resistance using a membrane given by:

$$Rt = \frac{TMP}{J \times \mu}$$

Where:

Rt = total resistance reported in units of 1/m

Although the permeate is assumed to have a viscosity close to pure water, it is also influence by temperature changes. In this study, the anaerobic reactor is maintained at thermophilic conditions at all times, the effluent temperature rapidly decreases after exiting the bioreactor. The feed temperature is continuously measured and used in the following empirical equation to correct the value of viscosity at a given temperature (USEPA, 2003).

$$\mu = 1.784 - (0.0575T) + \left(0.0011T^2\right) - \left(10^{-3}T^3\right) \tag{3.4}$$

In this case, dynamic viscosity has units of centipoises (cp) and temperature is in degrees Celsius.

3.2.4 Fouling Control Mechanisms

In this investigation, gas scrubbing was established as the main anti-fouling mechanism for continuous membrane performance. Declining operation of the membrane module was assessed by monitoring TMP and J. Due operational restriction described later in this document (Chapter 5), J was used as the main parameter determining membrane fouling. Forward flushing and Backwashing were applied weekly to maintain and improve the performance of the gas-lift filtration. However, for significant reduction in permeate generation (i.e. more than 30% reduction of

sustainable flux), the following cleaning protocol was adapted from Evenblij (2006) to recover membrane performance:

- Forward flush: tap water is used to flush the membrane at an increased CFV of 1 m/s during 15 minutes.

- Backwash: tap water is used to backwash the membrane at a constant flow rate of 2 L/hr during 15 minutes.

- Chemical cleaning: commercially available NaOCl (i.e. Bleach) is used to supply the active chlorine necessary to clean the membrane. A solution of 500 ppm is prepared and fed to the membrane at regular operational conditions during 15 minutes. In extreme cases of porous blockage, the same solution is used in an additional backwash step.

Before, in between and after each fouling control step, tap water is filtered through the membrane to assess the recovery in membrane total resistance and establish the effectiveness of the cleaning procedure.

3.3   Seed Sludge

Seed sludge from the anaerobic digesters of the Howard F. Curran Advanced Wastewater Treatment Facility (Tampa, FL) was used throughout this investigation. Typical values of VFA, TS, volatile fraction, alkalinity and pH for the seed sludge are 333 mg/L, 2.54%, 72.6%, 5673 mg/L, and 7.34-7.57; respectively (Plant's laboratory report as June 2007).

3.4   Synthetic Substrate

A new formulation developed between USF and Stanford University is used in this investigation. Complex Organic Particulate Artificial Sewage (COPAS), to simulate domestic wastewater.   This particular substrate presents advantages over other synthetic sewage such as:

- Low cost and commercially available substrate

- Ease of preparation

- No special conditions required to preserve its integrity (e.g. lower pH or refrigeration)

- Slowly-disintegrating and hydrolyzing particles with tunable particle distribution

- Contain complex organic matter typical of domestic sewage

Characterization of COPAS is presented later on in this thesis (Chapter 4), as well as its application as feed for the Gl-AnMBR (Chapter 5).

4 CHARACTERIZATION AND BIODEGRADABILITY ASSESSMENT OF COMPLEX ORGANIC PARTICULATE ARTIFICIAL SEWAGE (COPAS)

4.1 Introduction

Laboratory scale based research is commonly conducted to improve the understanding on physical, chemical and biological processes in WWT. Ideally, actual sewage should be used for these studies. However, constraints related to accessibility to the sewage, and even health considerations, prevent researchers from using actual sewage. Moreover, raw WW is highly variable in composition, which presents a reproducibility problem in laboratory-scale investigations. As a result, lab-scale investigations often use some sort of synthetic wastewater that is easy to prepare with a highly reproducible composition. Several studies use a readily biodegradable carbon source such as glucose or acetate; however, the simplicity in the composition of some synthetic WWs may not adequately represent actual sewage.

The main objective of synthetic sewage is to reproduce as close as possible the characteristics of domestic WW (Tables 4.1 and 4.2). For this purpose, commonly used ingredients for synthetic sewage include chemicals such as K_2HPO_4, $MgSO_4$ and Urea, food ingredients (e.g. starch, soy oil, beef extract, etc.) and even animal feed (e.g. canned dog food). In some cases, the ingredients in synthetic sewage are fine-tuned to obtain the desired concentration of chemical oxygen demand (COD) and nutrients (especially nitrogen and phosphorous) for a better process performance (Kato et al., 1997; Gao et al., 2004; Kurian et al. 2006, Kofina and Koutsoukos, 2005), thus

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increasing the non-uniformity among synthetic domestic sewage. Preparation of synthetic sewage recipes often also requires the use of expensive chemicals. Frequently, most of these recipes are prepared in concentrated solution and preserved by refrigeration or acidification (Nopens et al., 2001), bringing into question potential changes in chemical integrity during storage. It is important to highlight the presence of particulate matter in sewage, which sometimes cannot be simulated by combining soluble chemicals in water, and require the addition slowly degradable constituents (e.g. starch) to mimic particulate compounds in synthetic waters. However, mono-component particles such as starch do not adequately simulate the complex, heterogeneous compositions of natural sewage particles.

Several research labs have presented different combinations of chemicals, food ingredients and other constituents to imitate the composition of real domestic sewage. Although some of these formulas have similar characteristics to raw sewage, such as SYNTHO and SYNTHES (Boeije et al., 1998; and Aiyuk and Verstratete, 2004); others are customized to the treatment process under study (e.g., Iaquinta et al., 2006; and Lin et al., 2004). A summary of different synthetic sewage used for research is presented in Table 4.2. This table shows the composition, concentrations and specific application for every recipe.

Although standardization of synthetic sewage would be difficult to accomplish, the concept of an economical, consistent, and easy to obtain and preserve recipe is an attractive resource for laboratory research. In this study, a new option for domestic synthetic sewage is proposed. Based on dried granular cat food, *Complex Organic Particulate Artificial Sewage* (COPAS) is an unexploited material for mimicking domestic wastewater. The chemical composition, solution quality (COD, TOC and TN) and

anaerobic biodegradability of COPAS were determined in this investigation, in order to evaluate the suitability of COPAS for use in developing new lab-scale sewage treatment processes.

Dried granular cat food an inexpensive, commercially available product that does not require any special preparation and can be easily manipulated to reach desired particle size by simply grinding the pellets. The amount of cat food used for COPAS can be tailored to match the TS and COD concentrations typical of domestic sewage. Although dried dog food is another candidate, cat food kibbles are much easier to break than dog food kibbles, making cat food-based COPAS easier to prepare. Further, according to the Association of American Feed Control Officials, cat food is required to have more protein and fat than dog food due to the dietary necessities of felines (Dzanis, 1994). Also, the mineral composition of cat food differs from dog food in metals concentrations (i.e. iron, copper and manganese) and salts (i.e. sodium, chloride and magnesium) (Table 4.3). A main advantage of using dried granular pet food for COPAS is that no special preservation is required. A single batch of dried pet food can maintain its chemical composition during extended storage under ambient conditions in the lab (room temperature of 25 to 28°C). This statement was substantiated by testing and comparing the moisture, fat, protein, fiber, carbohydrates, ash, nitrogen and phosphorous from a brand new batch of cat food to one that has been used for lab experiments for more than a year. The characteristics varied for less than five percent (<5%) despite the elapsed time (Table 4.4). Using COPAS as synthetic sewage could avoid: preparing complicated and concentrated chemical solutions to be diluted in a further step (i.e. error accumulation in influent variables), adding additional particulate material to feed solution, and losing solution chemical integrity due to storage.

Table 4.1: Examples of actual domestic wastewater composition reported in treatment studies

Application	Water quality parameters of wastewater	Source
Evaluation of protein , carbohydrates and lipids content in domestic wastewater by using Lowry, phenol and anthrone, and infrared lipid methods	Percentages of total COD in the influent: Proteins: 28±4%; Carbohydrates: 18±6%; Lipids: 31±10%; Other organics: 23%; VFA within other organics < 1%	Raunkjaer et al., 1994
Comparison among CAS, Behrtest KLD4® and CAS-UCT for biological nutrient removal. Duffel WWTP (Belgium)	CODt: 400±200 mg/L; TN: 25±7 mg/L; TP: 7±3 mg/L; pH: 6.9-7.5	Rottiers et al., 1998
Anaerobic digestion of domestic sewage and black water	CODt: 634 mg/L and CODs: 217 mg/L	Elmiatwalli et al., 2000
Application of Molinga oleifera to UASB reactor for process enhancement. Ossermeersen WTP (Ghent, Belgium)	CODt: 320±58 mg/L; CODs: 140±35 mg/L; SS: 165±41 mg/L; VSS: 132±22 mg/L; TKN: 33±12 mg/L; NH4-N: 23±9 mg/L; TP: 10±1 mg/L; Alk: 412±45 mg CaCO3/L; pH: 7.7±0.2	Kalogo et al., 2001 This WTP influent was also used by Aiyuk et al., 2004;
Biodegradation of settleable COD by interpretation of hydrolysis rate in an aerated batch reactor. Atakoy WTP (Istanbul, Turkey)	CODt: 425 mg/L; CODs: 120 mg/L; SS: 240 mg/L; VSS: 150 mg/L	Orhon et al., 2002
Application of UASB for treating domestic sewage at moderate based on COD removal efficiencies (Salta, Argentina)	CODt: 224.2±10.1 mg/L and CODs: 65.4±5.5 mg/L	Seghezzo et al., 2002
Performance of a pilot-scale treatment wetland for low-cost domestic wastewater treatment (Santa Maria Nativitas, Mexico)	CODt: 1569.2±81.2 mg/L; TN: 164.9±14.3 mg/L; NH4-N: 66.3±4.5 mg/L; NO3: 28.4±7.3mg/L; DO: 1.9±0.2 mg/L; TSS: 406.1±33.4 mg/L; pH: 8.2±0.1	Belmonte et al., 2004
Evaluation of wastewater characteristics and treatment of domestic sewage in tropical monsoon areas. Ruamrudee sewer pipe (Bangkok, Thailand)	BOD5: 241.5 mg/L; CODt: 320.6 mg/L; TN: 42.4 mg/L; NO3: 0.9 mg/L	Giri et al., 2006
Evaluation of performance of a DHS system for treating UASB effluent (Japan)	BODt: 162±37 mg/L; BODs: 78±19 mg/L; CODt: 373±83 mg/L; CODs: 168±38 mg/L; SS: 134±48 mg/L; TN: 61±11 mg/L; NH4-N: 33±6mg/L; pH: 7.3	Tandukar et al., 2006
Performance of submerged NF MBR for treating domestic wastewater (Tokyo Bay, Japan)	TOC: 35.3-91.2 mg/L; SS: 40-180 mg/L; DO: 6.07-7.59 mg/L; TN (dissolved): 7.2-31.9 mg N/L; TP: 2.27-31.1 mgP/L; pH: 7.27-7.85	Choi et al., 2007
Characterization of domestic wastewater and treatability approach. Beishiqiao Wastewater Purification Center (Xi'an, China)	CODt:257.8 mg/L; BOD5: 134.7 mg/L; SS: 162.3 mg/L; TN: 38.8 mg/L; NH3-N: 26.2 mg/L; NO3-N: 0.48 mg/L; TP: 8.16 mg/L; pH: 7.6	Xiaochang et al., 2007

Table 4.2: Summary of representative synthetic wastewaters used in literature

Application	Composition	Water quality parameters	Source
Performance of an anaerobic-aerobic domestic sewage treatment using UASB and SBR	Meat extract, sucrose, starch, cellulose and vegetable oil	CODt: 422±68 mg/L; CODs: 169±45 mg/L; BOD5: 257±26 mg/L; TSS: 246±130 mg/L; VSS: 158±65 mg/L; TKN: 57±11 mg N/L; NH4-N: 26±7 mg/L; Alk: 288±85 mg CaCO3/L; pH: 7.0±0.36	Sousa and Foresti, 1996
Evaluation of degradation kinetics and heat and mass transfer in an aerated static bed reactor	Dry dog Food and hard maple wood chips as bulking agent and carbon source	Dog food C: 44.6% and N: 5.3%; Wood chips C: 29.7% and N: 4.1%	VanderGheynst et al., 1996
Performance of UASB and EGSB reactor for low strength wastewater treatment	Ethanol or whey	Whey CODt: 113 – 630 mg/L; Ethanol CODt: 146 - 722 mg/L	Kato et al., 1997
Development of a risk assessment tool for chemical fate prediction in aquatic environment	Syntho (precursor) Urea, ammonium chloride, uric acid, sodium acetate, dried yeast, lauric acid, diet fiber, LAS, AE, meat extract, peptone, potato starch, low fat milk powder, mineral salts and trace elements	CODt: 390 mg/L, TN:, 34.6 mg/L and TP: 7.9 mg/L; pH: 7.25	Boeije, 1998
Introduction of an improved option for synthetic sewage and potential applications for lab and pilot scale WWT.	SYNTHO	CODt: 470 mg/L, TN:, 31.6 mg/L and TP: 8.3 mg/L	Boieje et al., 1998
Immobilization of sludge using PVA and performance evaluation	Peptone, beef extract, NaCl, KCl, MgSO4-7H2O, Na2HPO4 and CaCl2-2H2O	COD: 360 mg/L, TKN: 48 mg/L, BOD: 240 mg/L and TOC: 150 mg/L	Chen et al., 1998
Comparison among CAS, Behrtest KLD4® and CAS-UCT for biological nutrient removal	BSR3 and SYNTHO Urea, ammonium chloride, uric acid, dried yeast, lauric acid, sodium acetate, diet fiber, LAS, AE, meat extract, peptone, starch, low fat milk powder, mineral salts and trace elements	BSR3 (Syntho precursor) CODt: 390 mg/L, TN:, 34.6 mg/L and TP: 7.9 mg/L SYNTHO CODt: 470 mg/L, TN:, 31.6 mg/L and TP: 8.3 mg/L	Rottiers et al., 1998
Evaluation of two-stage treatment configuration by comparison of CAS and MBR performance for reduced sludge production	Skimmed milk powder and antifoam	CODt: 360 - 1033 mg O2/L Average C/N/P ratio: 100/8.3/4.0 100 g of dry skimmed milk powder contains: Carbohydrates: 51.9 g; proteins: 35.5 g; lipids 1 g; minerals: 7.8 g	Ghyoot and Verstraete., 1999
Performance of ABR for treating complex (soluble and colloidal) dilute wastewaters	Semi-skimmed milk (soluble feed), dry dog food and rice (colloidal feed >500um) and trace chemicals	COD: 500 mg/L	Langenhoff et al., 1999
Evaluation of novel biosensor for BOD measurement using HCF(III) as mediator	OECD synthetic sewage (also adapted by the EPA) Peptone, meat extract, urea, NaCl, CaCl2-2H2O, MgSO4-7H2O and K2HPO4	BOD5 of solution: 14000 mg of O/L BOD values from 15 to 200 mg of O/L used to show sensor response	Yoshida et al., 2000
Evaluation of the reliability of synthetic wastewater for breeding stable activated sludge in an SBR	SYNTHO (modified) Urea, NH4Cl, Na-Acetate, Peptone, MgHPO4-3H2O, KHPO4, FeSO4-7H2O, starch, milk powder, yeast, soy oil, trace metals	COD: 439.47 mg/L, N: 60.23 mg/L and P: 9.42 mg/L	Nopens et al., 2001
pH effect on anaerobic solubilization of synthetic and domestic sludge	Dry dog food	Protein: 21%, Fat: 8% and Fiber: 5% VS: 90% of TS; VSS: 30000 mg/L	Gomec et al., 2002

Table 4.2: Summary of representative synthetic wastewaters used in literature (Cont.)

Application	Composition	Water quality parameters	Source
Process optimization of a trickling filter by using off-gas analysis	SYNTHO (Boeije et al., 1998)	Same as Boeije et al. (1998)	Vanhooren et al., 2002
Evolution of the sludge bed sedimentology for a UASB	SYNTHES Urea, NH4Cl, Na-Acetate, Peptone, MgHPO4-3H2O, KHPO4, FeSO4-7H2O, CaCl2, starch, milk powder, dried yeast, soy oil, trace metals	CODt: 500±50 mg/L; CODs: 170±40 mg/L; TKN: 49±8 mg/L; NH4-N: 27±7 mg/L; PO43-P: 21±2 mg/L; SS: 200±50 mg/L; COD/N/P ratio: 30/3/1	Aiyuk and Verstraete, 2004
Performance of an SMBR for the treatment of highly concentrated ammonia influent	NH4HCO3, K2HPO4, MgSO4-7H2O, MnSO4-4H2O, FeCL3-6H2O and NaCl	NH4-N: 180 – 1300 mg/L	Gao et al., 2004
Performance evaluation of BASR for wastewater treatment application	C2H5OH, K2HPO4, MgSO4, NH4Cl and trace elements	SCOD: 298-694 mg/L COD/NH4-N: 6/1 COD/P: 78/1	Lin et al., 2004
Study of struvite kinetics for salt precipitation	MgSO4-7H2O, NH4H2PO4, glucose, NaHCO3, NaCl, NaNO3 and NaSO4	Not reported	Kofina and Koutsoukos, 2005
Performance of microarobic MBR and anaerobic granular sludge domestic wastewater treatment	Sugar, potato starch, peptone, meat extract, urea, NH4Cl, KH2PO4, MgSO4-H2O, CaCl2-H2O, FeSO4-H2O and trace metals	COD: 500±46 – 214±30 mg/L TN: 45.1±2.2 – 18.9±0.4 mg/L	Chu et al., 2006
Performance of and AnMBr for dilute wastewater treatment	glucose, peptone , meat extract, urea, and NaHCO3	COD: 460±20 mg/L	Hu and Stuckey 2006
Determination of biokinetics of aerobic biomass in MBR using oily wastewater	Ammonium sulphate, K2HPO4, MgSO4-7H2O, CaCl2-2H2O, glycerol, FeCl3, CuSO4-5H2O, NaMoO4-2H2O, MnSO4-2H2O, ZnCl2, CoCl2 and NaHCO3	TCOD: 18700±3100 mg/L; SCDO: 15000±1900 mg/L; TBOD: 9050±1510 mg/L; SBOD: 7200±900 mg/L; Oil and grease: 670±86 mg/L; TSS: 1750±890 mg/L; VSS: 1400±780 mg/L	Kurian et al., 2006
Evaluation of alternating pumped sequencing batch biofilm reactor performance	Glucose, yeast, extract, dried milk, NH4Cl, urea, Na2HPO4-12H2O, NaHCO3, MgSO4-7H2O, MnSO4-H2O, CaCl2-6H2O and KHCO3	CODt: 346±32 mg/L; CODs: 319±25 mg/L; TN: 33±1.3 mg/L; P: 18±2.7 mg/L	Rodgers et al., 2006
Evaluation of befouling in attached and suspended growth media MBR	Glucose, soy starch, NH4Cl, KH2PO4, CaCl2, MgSO4-7H2O, FeCl3 and NaHCO3	COD: 500 mg/L COD/N/P ratio: 100/10/2	Sombatsompop et al., 2006
Performance improvement of an aerobic MBR system by ozone gas backwashing as fouling control	Glucose, Poly-peptone, NH4Cl, CaCl2· 2H2O, FeCl3· 6H2O, MgSO4· 7H2O, NaHCO3 and KH2PO4	BOD: 250 mg O2/L, Alkalinity: 270 mg CaCO3/L, NH4+ -N: 22.3 mg N/L, TN: 42.3 mg N/L, TP: 6.8 mg P/L	Kim et al., 2007
Evaluation of the degradation of non-ionic surfactants by activated sludge's bacterial community	peptone, yeast extract, urea, NaCl, CaCl2.2H2O, MgSO4. 7H2O, K2HPO4, KH2PO4, and nonylphenol ethoxylates (NPE)	COD: 190 mg/L	Lozada et al., 2007
Determination of optimal carbon source in a SAAR/AR (Sequencing anoxic/anaerobic and aerobic reactor)	Acetate, propionate, glucose and methanol. Combinations of VFAs at different ratios.	COD from 250 to 400, TN from 5.2 to 6.2 and TP from 9 to 41.	Ahmed et al. 2008
Performance of a SND (simultaneous nitrification and denitrification) process with an internal-loop airlift MBR	Glucose, starch, ammonium chloride, NaHCO3, peptone, KH2PO4, MgSO4-7H2O, MnSO4-7H2O, CaCl2 and FeSO4	TN: ~60 mg/L	Li et al. 2008
Assessment of the fate of PPCPs in aerobic MBR treating domestic wastewater	AcNa · 3H2O, NH4Cl, Na2HPO4, KH2PO4, NaHCO3, and PPCPs	Not reported	Reif et al., 2008

Table 4.3: Nutrient profile for dog food and cat food. Adapted from Dzaniz, 1994. AAFCO dog and Cat

Nutrients profiles (minimum values for adult pet maintenance)

Feeding component	Units dry matter	Dog food	Cat food
Protein	g/Kg	180	**260**
Fat	g/Kg	50	**90**
Minerals			
Ca	g/Kg	6	6
P	g/Kg	5	5
K	g/Kg	6	6
Na	g/Kg	0.6	2
Chloride	g/Kg	0.9	3
Mg	g/Kg	0.4	0.4
Fe	mg/Kg	80	80
Cu	mg/Kg	7.3	5
Mn	mg/Kg	5	7.5
Zn	mg/Kg	120	75
I	mg/Kg	1.5	0.35
Se	mg/Kg	0.11	0.1

Table 4.4: Comparison between old and new bags of COPAS

Concentration	As specified by manufacturer**	Old lot* (No. 71230850318L02)	New lot* (No. 71240850554L07)
Moisture	12% (max)	1.49%	2.56%
Fat	11.5% (min)	15.17%	15.50%
Protein	31% (min)	36.69%	35.35%
Fiber	4.5% (max)	1.70%	2.40%
Ash		8.11%	8.23%
Carbohydrates		38.54	38.27%
Nitrogen		5.87%	5.66%
NFE (Nitrogen free extract)		36.84%	35.87%
Phosphorous	1% (min)	1.46%	1.40%
Urea		0.14%	0.12%
Linoleic Acid (Min)	1.50%		
Calcium (Min)	1.20%		
Zinc (Min)	125 mg/kg		
Vitamin A (Min)	15000 IU/kg		
Vitamin E (Min)	60 IU/kg		
Taurine (Min)	0.12%		

*Analysis performed by Borrow-Agee Laboratories, LLC. Memphis, TN
**COPAS Ingredients: Ground yellow corn, corn gluten meal, poultry by-product meal, meat and bone meal, corn germ meal, animal fat preserved with mixed-tocopherols (form of Vitamin E), ocean fish meal, soybean meal, brewers dried yeast, phosphoric acid, animal digest, potassium chloride, tetra sodium pyrophosphate, salt, choline chloride, tuna meal, salmon meal, added color (Yellow 6, Red 40, Yellow 5), taurine, zinc sulfate, ferrous sulfate, Vitamin E supplement, niacin, manganese sulfate, calcium carbonate, Vitamin A supplement, calcium pantothenate, thiamine mononitrate (Vitamin B-1), copper sulfate, riboflavin supplement (Vitamin B-2), Vitamin B-12 supplement, pyridoxine hydrochloride (Vitamin B-6), folic acid, Vitamin D-3 supplement, calcium iodate, biotin, menadione sodium bisulfite complex (source of Vitamin K activity), sodium selenite. D-5007

4.2   Materials and Methods

The cat food used for this study was a commercial brand (Purina Friskies® Ocean Fish Flavor) obtained from a popular discount superstore (Walmart). To assess its nutritional composition, a sample of ground cat food (particle size 0.472 to 1.7 mm) was sent to a registered animal feeding analysis laboratory (Borrow-Agee Laboratories, LLC. Memphis, TN). Chemical composition of COPAS was calculated based on dry weight of COPAS sample. Chemical oxygen demand (COD) values were measured using Hach HR COD digestion vials (Hach Company, Loveland, CO). Dissolved organic carbon (DOC) and dissolved nitrogen (DN) content in liquid samples were measured using a Total Organic Carbon analyzer (Shimadzu TOC-V) coupled with a Total Nitrogen detector (Shimadzu TNM-1). A Solid Sample Module (Shimadzu SSM-5000A) coupled to the TOC-V was used for particulate organic carbon (POC) in solid phase samples, (Shimadzu, Columbia, MD).   Biological Oxygen Demand BOD) was measured in an external laboratory (Howard Curren AWT Environmental Laboratory, Tampa, FL) using the method 5210 for BOD5 and BOD20 described in ALPHA 2008.

To evaluate the feasibility of COPAS as a readily available carbon and nitrogen source, a *dissolution test* was performed and the DOC and DN concentrations were closely monitored during a 24 hours period. For this test, two 1 liter batch reactors were filled with a solution of 500 mg/L of ground COPAS (particle size between 0.425 to 1.7 mm). For homogenization, the reactors were mixed at controlled speeds of 20, 50 and 100 rpm. Samples were filtered through a 0.45 um SFAC syringe filter and acidified with 1 N HCl to a pH of 2 to 3 to avoid biological activity and to facilitate removal of inorganic carbon. DOC and DN were measured using the Shimadzu TOC-V/TNM-1 analyzer.

The effect of COPAS on solution pH was evaluated by closely monitoring this parameter during the first hour of the dissolution test. The initial pH of DI water was 7.10 and decreased after cat food addition to a minimum value of 6.52 at t = 30 min. The pH then stabilized in the range of 6.52 to 6.56. These pH values do not differ significantly from the initial pH concentration and are acceptable for wastewater treatment.

Anaerobic *biodegradability* of the proposed synthetic sewage was assessed through serum bottle assays seeded with anaerobic digester sludge seed from a local wastewater treatment plant (Howard Curren AWT, Tampa, FL.). For the serum bottles (clear 118 ml glass bottles, 60 ml liquid sample) (Fisher Scientific, Pittsburg, PA), the sludge was previously screened using 1 mm wire mesh to remove grit, hair, and other things that might interfere in the biodegradability assay. The serum bottles were maintained at $37^{o}C$ and fed with COPAS at different solids concentrations within a range typical of domestic wastewater (i.e., 100, 200, 500, 1000 and 2000 mg/L). Chemical oxygen demand (COD), volatile fatty acids (VFAs), biogas production and particulate organic carbon (POC) were continuously measured in each bottle. Sludge samples were centrifuged at 3000 rpm for 20 minutes and supernatant was used for COD and VFA measurements. Dissolved COD was measured using HACH HR COD digestion vials (HACH Company, Loveland, CO). POC was measured on filtered sludge samples (i.e., 1.5 um fiber glass filters) using the Shimadzu TOC-V/SSM-5000A. Biogas volume was determined using a water displacement burette. Gas composition was analyzed for CO_2 and CH_4 using a gas chromatography (GC) unit (Agilent 7820A) equipped with a thermal conductivity detector (TCD) and a 30-m J&W 113-3133 GS-CarbonPLOT, 0.32

mm diameter column (Agilent Technologies, Lexington, MA). The inlet, oven and detector temperature were set at 185°C, 50°C and 160°C respectively. Helium was used as carrier gas at 1.3 mL/min. VFAs (i.e. acetic, propionic, butyric and valeric acids) were monitored using a GC unit equipped with a flame ionization detector (FID) and a 30-m Restek 11025 Stabilwax DAm 0.53 mm ID column (Restek Corp. Bellefonte, PA). The inlet and detector temperature were both set at 250°C. Helium was used as carrier gas at 4.5 mL/min and the following program was used for the oven temperature: 90 °C for 0.5 min, 2 °C/min to 100 °C, 6 °C/min to 120 °C, 30 °C/min to 230 °C for 15 min. The total run time was 27.5 min.

4.3   Results and Discussion

On dry weight basis of the organic portion, COPAS granules are mainly composed by proteins (40%), carbohydrates (43%) and fats (17%). Other constituents such as metals are found in trace concentrations and are accounted in the ash fraction of COPAS particles. Elemental constituents such as carbon, nitrogen and phosphorus were found in COPAS in proportions of 48.1% C, 6.35% N and 1.57% P of the organic fraction. Theoretically, an organic molecule of domestic wastewater is represented by the formula $C_{10}H_{18}O_3N$ (Rittman and McCarthy, 2001), where the C:N ratio is about 5:1, and a COD/wt and COD/TOC ratios of approximately 2 and 2.5 respectively. COPAS has a similar C:N ratio to wastewater and direct measurement of COPAS total COD/wt and COD/TOC resulted in values of 1.5 and 2.6 respectively. Figure 4.1 and Table 4.5 summarize the composition of COPAS, with emphasis on the organic fraction. However, this particulate substrate is intrinsically heterogenic and a definite empirical formula

cannot be established. The heterogenic nature of COPAS substrate can be easily assessed by direct observation of the individual particles (Figure 4.2).
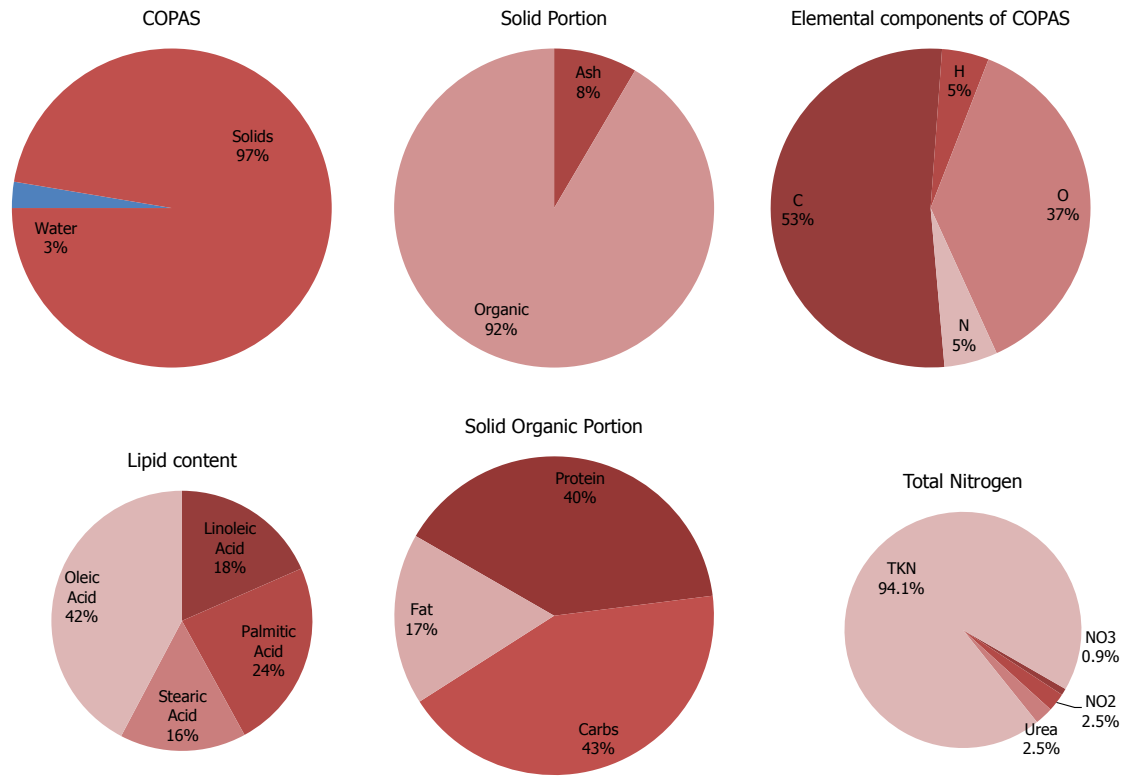
Figure 4.1: Characterization of COPAS

Table 4.5: COPAS composition

Characteristics	Total weight	Dry wt. solids	Organic solids
% of total weight	100%	97.35%	91.55%
Moisture (%)	2.65%	n/a	n/a
Ash (%)	8.23%	8.45%	n/a
Carbohydrates (%)	38.27%	39.31%	42.94%
Protein (%)	35.35%	36.31%	39.67%
Fat (%)	15.50%	15.92%	17.39%
Fiber (%)	2.40%	2.47%	2.69%
Phosphorous (%)	1.40%	1.44%	1.57%
TKN (%)	n/a	4.65%	5.08%
Nitrates (%)	0.0019%	0.002%	0.002%
Nitrite (%)	0.0040%	0.004%	0.004%
Nitrogen Free Extract (%)	35.87%	36.85%	40.25%
Urea (%)	0.12%	0.12%	0.13%
COD/wt (g/g)		1.15	1.26
OC/wt (g/g)		0.48	0.52
COD/OC (g/g)			2.5
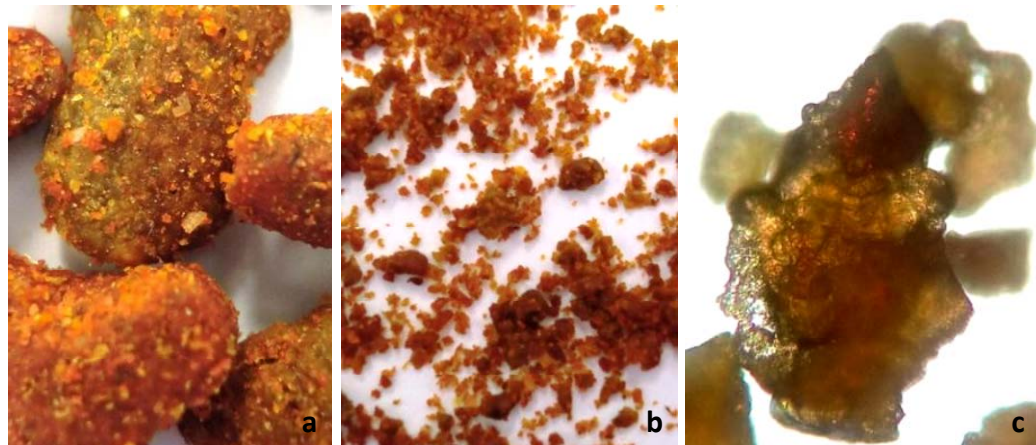
Figure 4.2: Image of COPAS substrate from kibble (a) to ground particle (b). Heterogenic sub-particles can be identified in a closer view (10X) (c).

4.3.1 Availability of COPAS as Substrate

A summary of the dissolution profiles for DOC and DN in COPAS is presented in Figure 4.3. The available concentration of organic carbon in water showed a rapid increment of about 20 mg/L during the first 1 hour of each experiment followed by slower DOC increment over the following 23 h. The first stage of this dissolution behavior corresponds to the breakage of COPAS particles (i.e. secondary particles) into smaller primary particles due to the dissolution of the gelling agent (e.g. gums, gelatin, carrageenan, or other starches and thickeners) used to aggregate the COPAS ingredients. In this case, this readily available fraction of COPAS is assumed to be soluble substrate incoming the reactor (S_o). The following stage would be the result of carbon leaching from the COPAS primary particles plus additional DOC from the gelling agent (Figure 4.4).
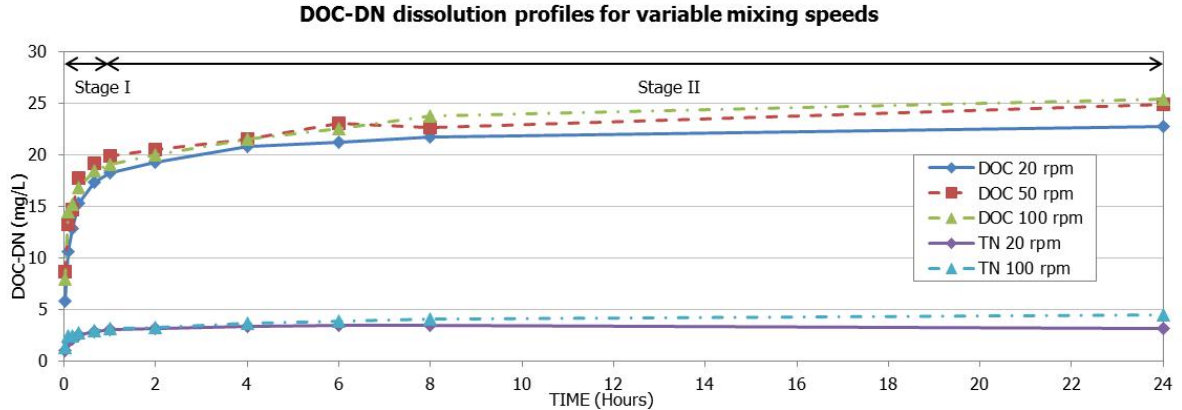
Figure 4.3: Summary of COPAS dissolution curves for different mixing conditions. Dissolution stages correspond to I) Breakage of secondary particle (i.e. gelling agent dissolving in water), II and III) Gradual disintegration of primary particle
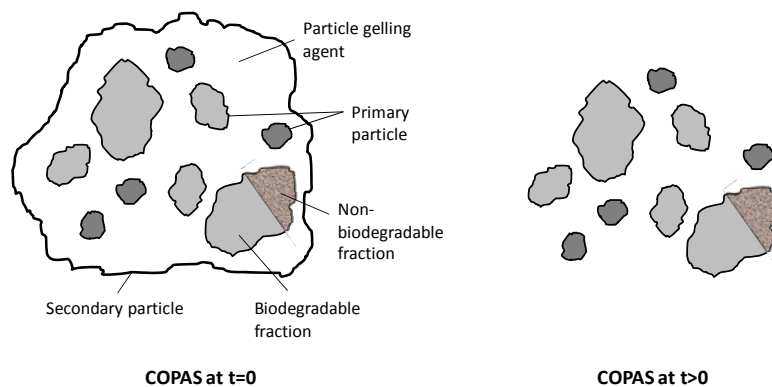
Figure 4.4: Schematic of COPAS particle and different components (left). At t>0 the gelling agent dissolves and the the primary particles are released (i.e. particle disintegration starts) (right).

While the dissolution of carbon and nitrogen observed in this study seems to asymptotically approach a limit around 25 mg DOC/L and 5 mg TN/L, these values only correspond to 10% and 17% of the organic carbon and nitrogen present in the COPAS sample. This means that the remaining 90% of TOC and 83% of TN still available in particulate form either for dissolution or biodegradation. It is also possible that the dissolution curves have reached a saturation level that is function of the total concentration of carbon or nitrogen (C_T), and their solubility limit in water (C_s). Due to the heterogeneity of the carbon and nitrogen forms in COPAS, Cs cannot be assumed to correspond to those of carbon or nitrogen. Instead, an approximation of those can be derived from data fitting using a pre-defined dissolution model. In the pharmaceutical field, dissolution models are widely used to quantitatively interpret the results from dissolution assays and to define drug release kinetics. For this study, the Weibull model has been used for its simple application to time-dependant dissolution assays where variables such as particle surface area, length of diffusion layer, and diffusion coefficient

are unknown. Costa et al., (2001) explains a modified version of the Weibull dissolution profile in terms of the fraction of solute present in water (m) and is defined as:

$$C(t) = mC_T$$

$$m = 1 - \exp\left[-(t-T_i)^b/a\right]$$

Where T_i represent a lag time before dissolution starts, b is a shape parameter, and a defines the time scale of the process. By applying this equation to obtained DOC data and using $C_{T,DOC}$=248 mg/L, an approximate dissolution profile of carbon in COPAS was obtained (Figure 4.5).
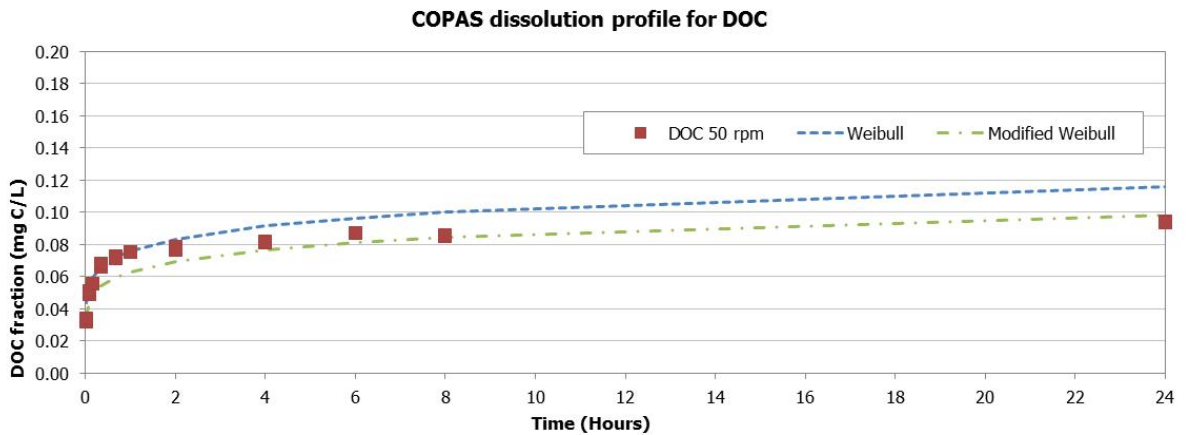
Figure 4.5: COPAS dissolution data fitted to Weibull and modified Weibull dissolution models

Although this experiment was conducted at different mixing speeds, the dissolution/hydrolysis rate of COPAS does not seem to be affected by this variable, which highlights the possibility of avoiding extra cost or effort in homogenizing influent with reactor content. It was also observed that the nitrogen released into water followed the same dissolution pattern as carbon.

The COD contained in the fraction of particulate matter in domestic sewage (Sp^o) is usually difficult to account for at lab scale with other synthetic substrates. However for COPAS, hydrolysis of the particulate fraction is evident through the slow dissolution of the particulate substrate during the second stage of the dissolution profile. Assuming a first order reaction kinetics for the hydrolysis of the particulate COPAS, its hydrolysis rate can be identified as follows:

$$S_p = S_p^o e^{-k_{hyd}t}$$

Where S_p^o is the initial biodegradable fraction of COPAS, k_{hyd} is the constant of hydrolysis (1/hr) and S_p is the concentration of particulate COPAS available in the water phase over time (mg COD/L). Additionally, the initial substrate available in biodegradable COPAS can be expressed as:

$$S_p^o = \gamma\beta C_{in}$$

Where γ is the COD/wt in COPAs, β is the percentage biodegradability of the substrate and C_{in} is the concentration of COPAS in the influent. Data fitting of the DOC dissolution profile as COD to first order hydrolysis kinetics results in k_{hyd} of -0.9×10^{-3} hr^{-1}.

By considering the above hydrolysis kinetics in a bioreactor model (e.g. steady state continuous flow complete mixed reactor), a mass balance of the soluble COD can be expressed as:

$$In - Out + Sources - Sinks = 0$$

[Incoming COD] − [Effluent COD] + [Particulate COD hydrolysis] − [Biological utilization] = 0

The available COD for biological utilization is provided by the incoming COD to the reactor and the hydrolyzing COD from the particulate fraction. The effective substrate (S_{eff}^o) available for biodegradation can be expressed as:

$$S_{eff}^o = S^o + \frac{k_{hyd}\theta_x}{1+k_{hyd}\theta_x} \; S_p^o$$

Where S_{eff}^o is the effective substrate available for biodegradation and θ_x is the sludge retention time. The effective substrate available for COPAS biodegradation at different SRT is summarized in Figure 4.6.
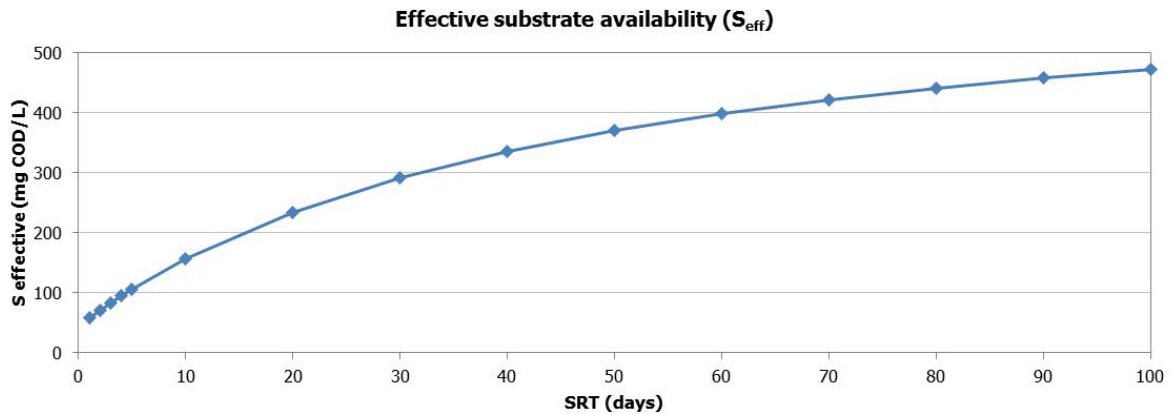
Figure 4.6: Effective substrate availability (Seffo) for COPAS at a TS of 500 mg/L

4.3.2 Biodegradability

Table 4.6: Characterization of COPAS as synthetic sewage

Sample	TS mg/L	TSS mg/L	COD mg/L	BOD$_5$	BOD$_{20}$	TKN mg/L
	500	215	625	191	540	19.8
COPAS	1000	520	1250	382	906	53.9
	2000	1184	2500	680	3,144	92.1
HFC AWTP Primary Influent	1200	190	401.9	254	716	43.7
HFC AWTP Primary Effluent	1200	132	150	198	538	45.8

* As reported by the HCF Environmental lab

COPAS not only contains the particulate fraction missing in other synthetic recipes but has environmental characteristics very similar to actual sewage. A summary of these characteristics for COPAS solution is presented in Table 4.6. By comparing it to raw sewage (raw and primary effluent), available nutrients and organic matter in COPAS are found in similar concentrations. COPAS has also been demonstrated as a highly biodegradable substrate under anaerobic conditions. The distribution of the available COPAS for degradation was evaluated by continuously measuring methane production as well as the fraction of substrate dissolved in the liquid and remaining in the biomass. Measurements were corrected for the background activity of the sludge. For the particulate COPAS, POC was measured in the biomass and converted to COD equivalents. Results for the specific methane production of COPAS at different concentrations suggested that at least 50% of methane can be obtained from this substrate. Up to 72% of the biogas produced from the anaerobic biodegradation of COPAS was methane, even at concentrations as low as 200 mg/L. These values were obtained daily and converted to COD equivalents as well. Dissolved COPAS was assessed by direct measurement on the liquid phase after centrifugation. A mass balance of the substrate partitioning into different phases is expressed as:

$$\text{Total COD}_{COPAS} = \text{COD}_{particulate} - \text{COD}_{dissolved} - \text{COD}_{methane}$$

Where:

$$\text{Total COD}_{COPAS} = \gamma \times \text{COPAS dry sample}$$

$$\text{COD}_{particulate} = \omega \times \text{POC}$$

The COD corresponding to CH_4 generation were calculated based on the theoretical COD equivalence:

$$CH_4 + 2O_2 = CO_2 + 2H_2O$$
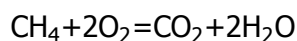
Since one mole of CH_4 as an ideal gas is equivalent to 22.4 L at STP (i.e. 0^oC and 1 Atm), approximately 350 ml of CH_4 are produced per 1 gram of COD available for biodegradation at STP. By correcting this value for an incubation temperature of 37^oC, the COD equivalents corresponding to methane generation can be expressed as:

$$COD_{methane} = V_{CH_4} \div 397.4 \text{ ml } CH_4/g \text{ COD}$$

The methane production profile for different COPAS concentration is presented in Figures 4.7, 4.8 and 4.9. At lower COPAS concentration (100 and 200 mg COPAS/l), the substrate is efficiently converted to methane by day 5 (more than 90% COD conversion) and little remains in the dissolve and particulate fractions. However, at concentrations above 200 mg COPAS/l, a more defined COD phase distribution can be observed since the methane generation declines with increasing substrate availability. Due to the heterogeneous composition of COPAS, identification of the specific component affecting biodegradability at higher concentrations was not assessed in this study. However, the biodegradable COD fraction of COPAS at higher concentration was about 90%, 65%, and 60% for COPAS concentrations 500, 1000 and 2000 mg/L respectively. A summary of the COD balance for COPAS concentrations of 500, 1000 and 2000 mg/l is presented in Figures 4.10 and 4.11.
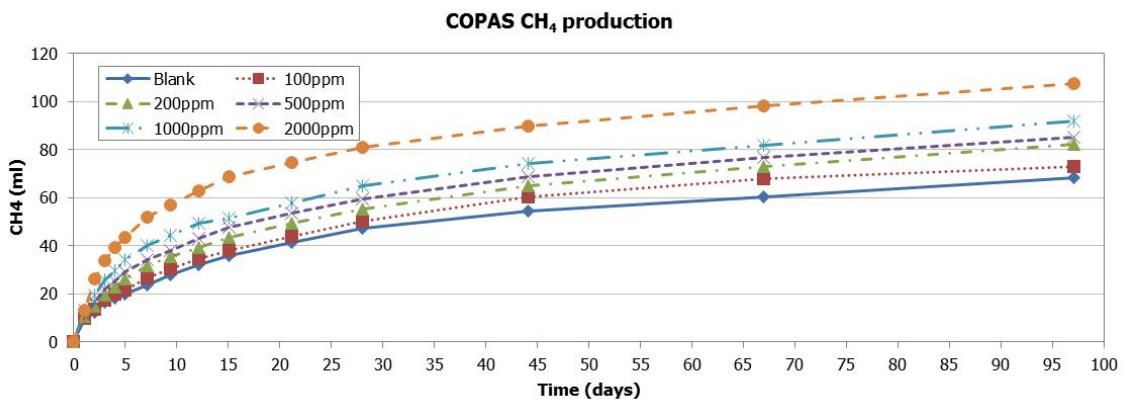
Figure 4.7: Methane production from COPAS digestion at different concentrations
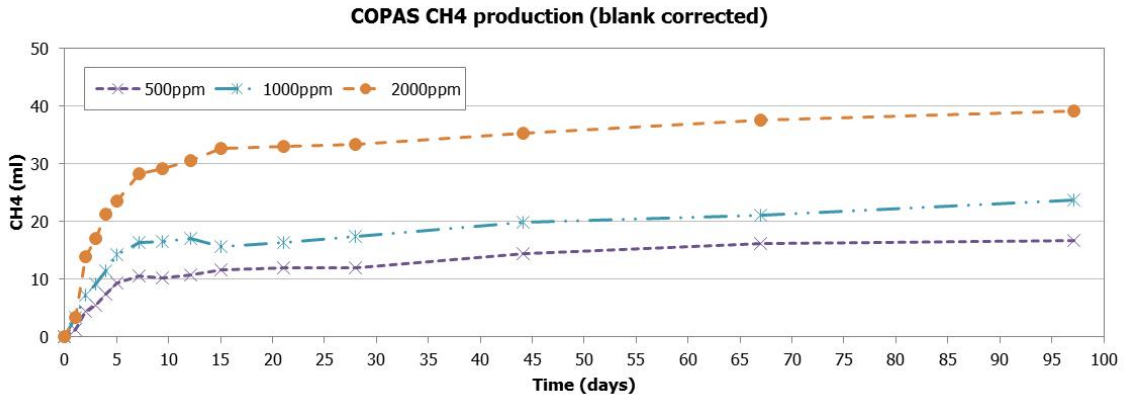
Figure 4.8: Blank corrected methane production from COPAS digestion at different concentrations
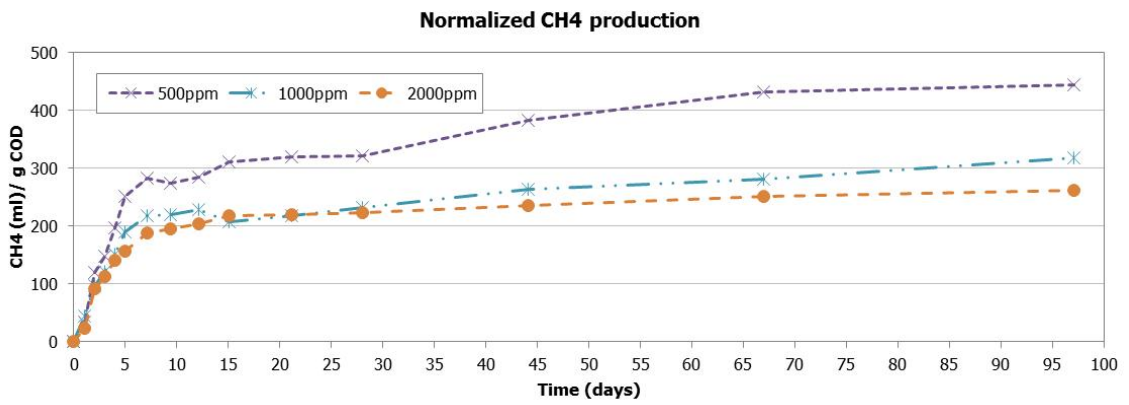
Figure 4.9: Specific methane production from COPAS digestion at 500, 1000 and 2000 mg/L
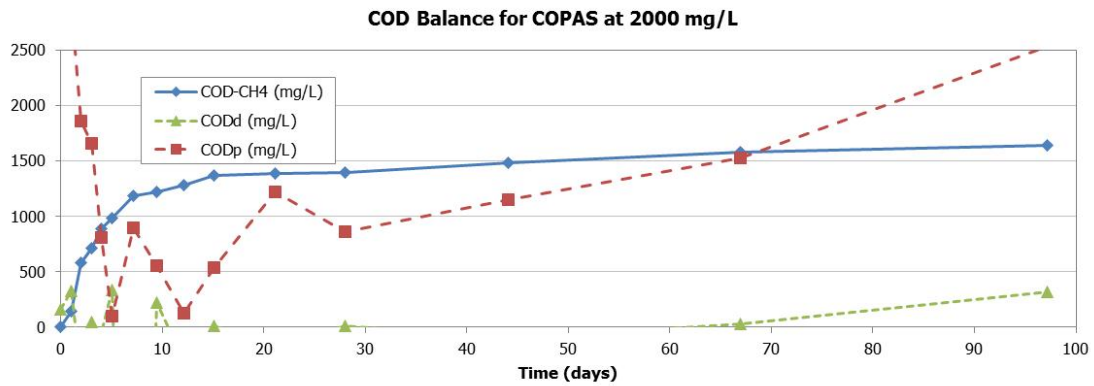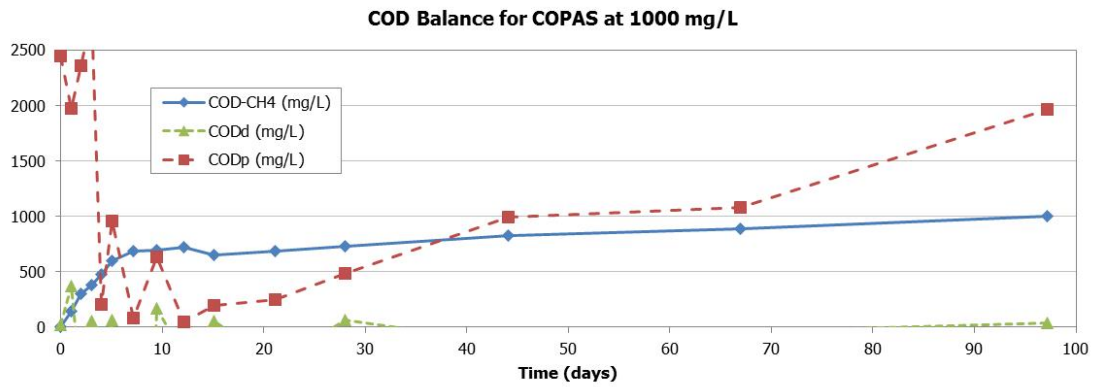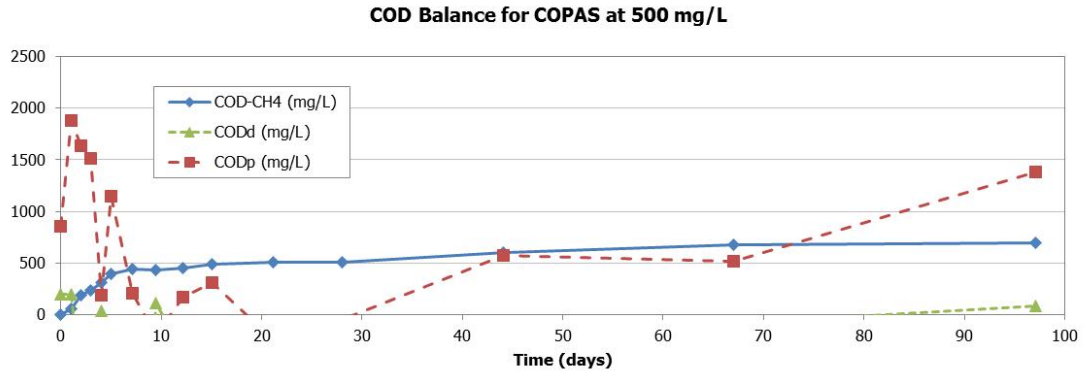
Figure 4.10: COD distribution of COPAS in the liquid, gas and particulate phases at 500, 1000 and 2000 mg/L. COD values below zero were obtained at 500 mg/L after correcting for sludge background activity.
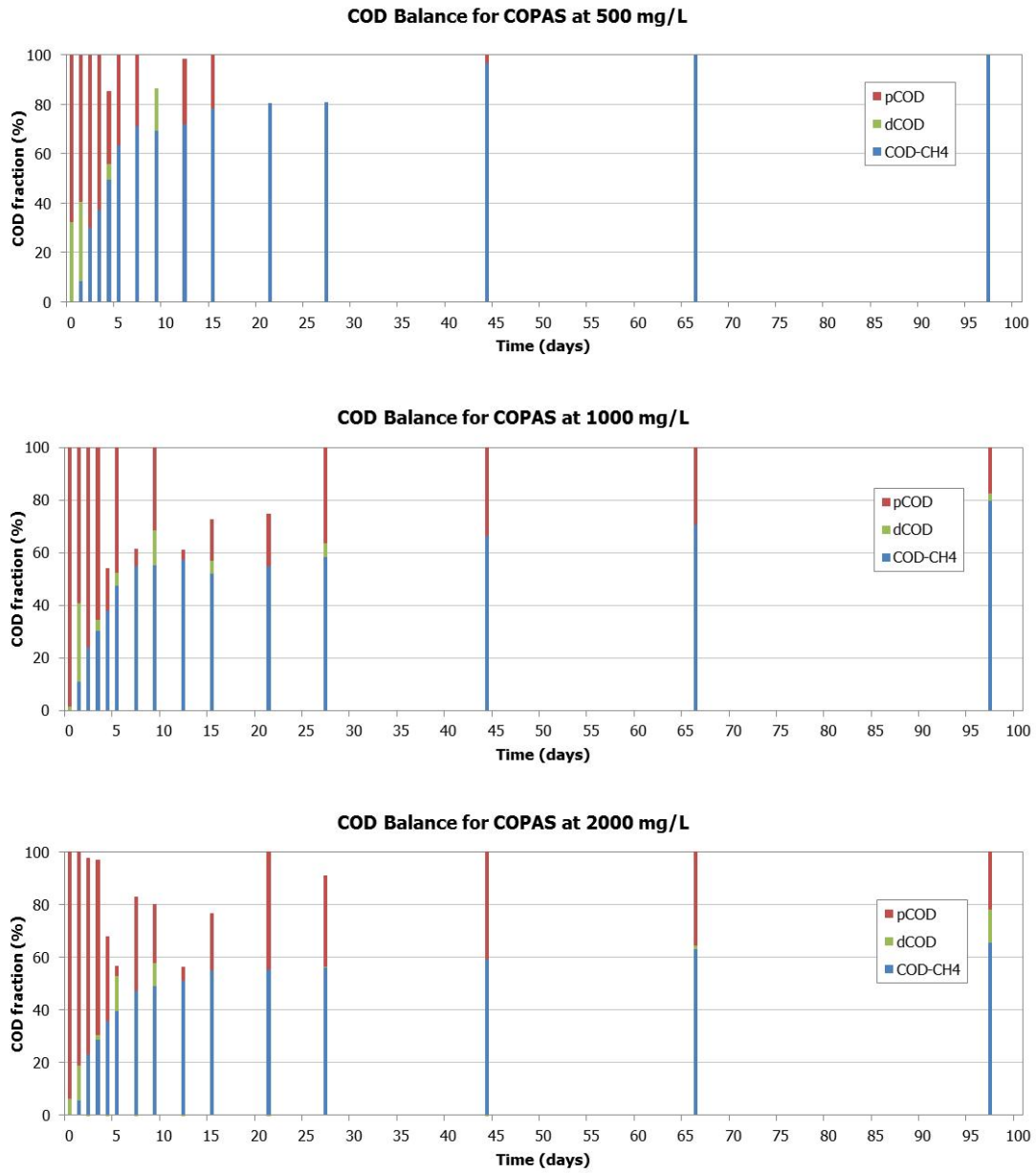
Figure 4.11: COD distribution of COPAS in the liquid, gas and particulate phases at 500, 1000 and 2000 mg/L. Data presented as percent of total COD added.

4.4 Conclusions

Besides the satisfactory characteristics of COPAS for representing domestic wastewater, it also can be manipulated to simulate complex particulate organic matter in any desired particle size range, without presenting extra cost or effort in homogenization of substrate with bioreactor content. The following conclusion could be assessed during this study:

- COPAS mimicked sewage well in terms of its composition of complex organic matter (proteins, carbohydrates and lipids) from animal and plant origins.

- The biodegradable fraction of COPAS was estimated at 65% of available COD in the COPAS sample. In comparison, the recalcitrant content in COPAS (35%) is similar to those exerted by actual sewage (i.e Primary influent BOD_5/COD = 0.63).

- COPAS is a highly biodegradable compound that can be used as a bioreactor substrate for anaerobic activity evaluation. The substrate was completely utilized at TS concentration lower than 500 mg/L, and up to 47% conversion of COPAS into CH_4 could be assessed at higher concentration (e.g. 500, 1000 and 2000 mg/L). In batch experiments, the anaerobic degradability was more limited at high COPAS concentrations. Since average concentrations of solids in domestic sewage usually do not exceed 2000 mg/L, this limiting factor should not restrict the use of COPAS in continuous flow bioreactors.

- The slow hydrolysis of particles and good biodegradability make COPAS an ideal surrogate for raw sewage for lab-scale wastewater treatment applications targeting complete resource recovery, such as anaerobic MBR.

5 GAS-LIFT ANAEROBIC MEMBRANE BIOREACTOR (GL-ANMBR) FOR CONVERSION OF SEWAGE TO ENERGY, WATER AND NUTRIENTS

5.1   Introduction

Although MBR is generally known for its high quality effluent and small footprint, AnMBR has the additional benefits of energy generation (e.g. biogas), fertilizer recovery (e.g. nutrients), and low sludge generation. Under optimum operational conditions, an AnMBR can be used not only for on-site wastewater treatment, but generation of reusable water for agricultural applications. Additionally, biogas produced in the anaerobic process could satisfy the energy requirements of the system (Liao, 2006). More information, however, is required regarding maximization of the overall energy balance (energy footprint) in AnMBR.   Recent studies have demonstrated improved energy efficiency of membrane technology by enhancing shear over membrane surface in vacuum-driven modules using air scouring (e.g. reducing cake layer deposition in submerged membranes).   This approach is also applied to sidestream membrane configurations in aerobic airlift supported modules, which has gained increased attention for municipal wastewater treatment, but little is known about the application of this configuration in anaerobic mode by using biogas for gas-lift.

In this in chapter, the performance of the Gas-lift Anaerobic MBR (Gl-AnMBR) to treat domestic water has been tested and evaluated for its energy footprint. Analog to

the air-lift MBR, this system uses biogas to provide two phase flow through the vertically placed tubular membranes. By including biogas bubbles into the membrane feed, the potential of membrane fouling is decreased due to additional shear over the membrane surface provided by the raising bubbles. Additionally, concentrate recirculation is improved by the gas-lift and less crossflow velocity is necessary to drive filtration. Pumping requirements for recirculation and filtration are minimized allowing less energy consumption. In this study, a preliminary filterability assessment defined the operational parameters for subsequent operation. Extended performance of the Gl-AnMBR is reported as prove of concept for the application of this technology in the treatment of low strength streams (i.e sewage). The concept of energy footprint for this treatment technology is evaluated as indicator of the feasibility of this system compared to its aerobic counterparts in terms of energy efficiency for water treatment.

5.2   Construction of the Gl-AnMBR

5.2.1 Materials and Methods

The Gl-AnMBR experimental set-up shown in Figure 5.1 consists of an 8.5 L anaerobic bioreactor column, coupled with a sidestream gas-lift ultrafiltration module. The membrane used is a 5.2 mm diameter polyvinylidene fluoride (PVDF) tubular membrane (Norit X-Flow, F4785) with a mean pore size of 0.03 um and active filtration area of 0.013 m^2.   As a starting point, the membrane influent and scouring gas flow rates were set to assure cross flow velocities (CFV) of more than 0.3 m/s as reported optimal for airlift operation (Futselaar et al., 2009). Filtration was driven by applying negative pressure to the membrane permeate side with a variable speed pump (Masterflex I/P). Membrane effluent was measured with an in-line rain gage and

transmembrane pressure (TMP) was measured by placing pressure gauges at the influent (P_{in}), effluent (P_{eff}) and recycle (P_r) lines of the membrane module. Compressed helium supplied for membrane scrubbing and the gas flow rate (Qg) was controlled visually with a gas flow meter and a needle. The retentate is separated from the gas in an intermediate tank prior to recycling it back to the bioreactor. Sludge temperature was regulated with a heat exchanger around the column and continuously monitored inside the reactor and at the membrane feed line using in-line sensors.

Several modifications to the original airlift filtration concept had to be considered for its application to an anaerobic bioreactor such as the UASB. First, the supernatant of the UASB is used as influent for the membrane, which requires the placement of a recycle pump that controls membrane feed the flow rate (Q_f) and CFV. The hydrostatic head available from the reactor column becomes irrelevant for this particular UASB-membrane application. Second, Q_f has to be maintained at a point where upflow velocity of the reactor and the CFV are within acceptable operational conditions for both UASB and membrane. Additionally, a recycle loop was introduced to decrease the bioreactor effluent flow rate by half, while providing adequate membrane shear and controllable CFV. The CFV was set at 0.3 m/s during the length of the preliminary filterability assessment. Since gas scrubbing is the main anti-fouling method considered for gas-lift filtration, backwashing and relaxation were also evaluated as support mechanisms.
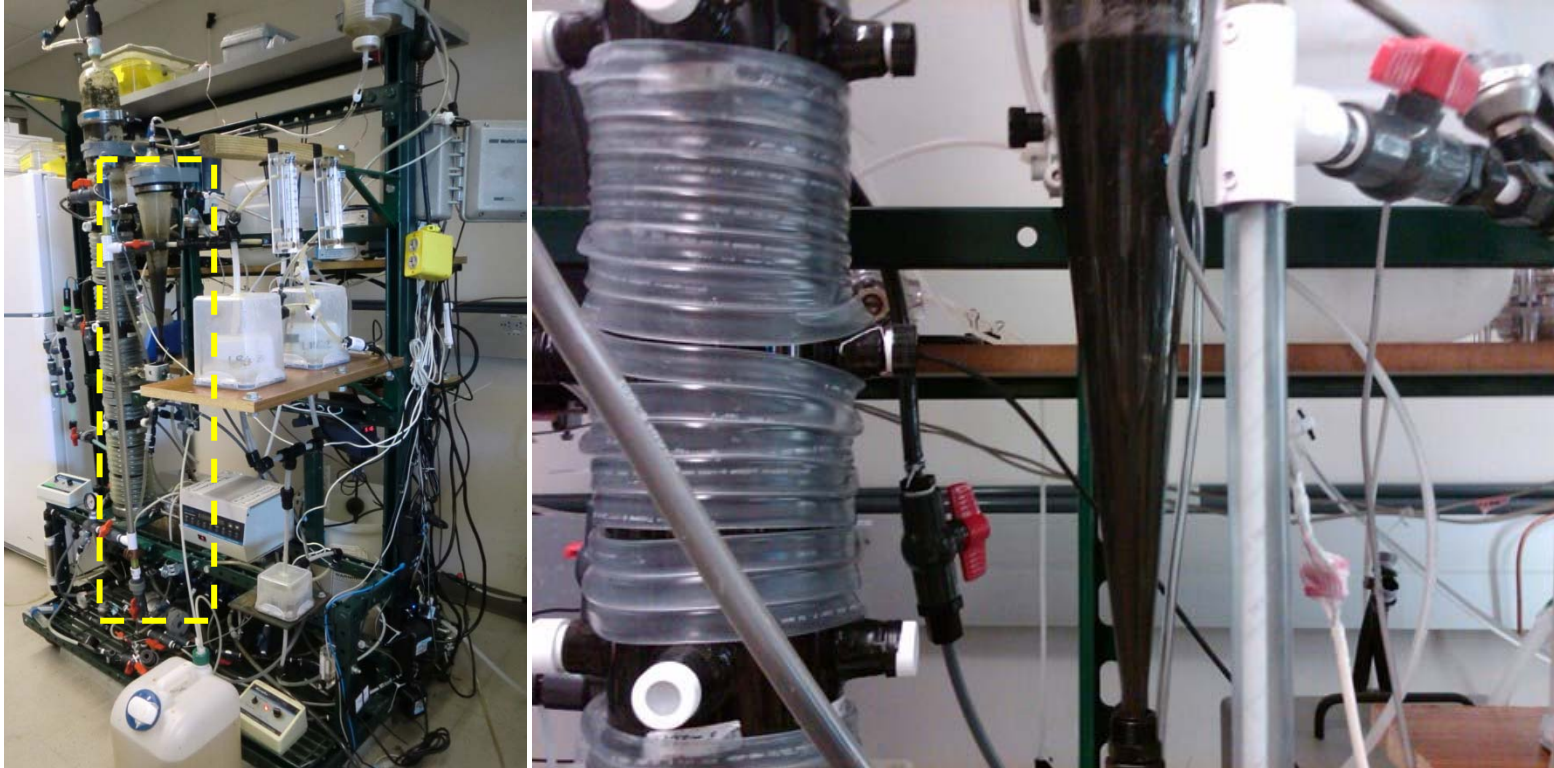
Figure 5.1: Gl-AnMBR configuration. Detail of the membrane (right) shows the complete retention of sludge while providing high quality permeate.

Digester sludge from the local municipal wastewater treatment plant in Tampa Bay Area (Florida, US) was used as seed for Gl-AnMBR operation. The seed anaerobic sludge was obtained from the solids digester at the. For the preliminary assessment, the raw sludge was diluted to a concentration comparable to MLSS in aerobic configuration (Judd, 2006). Sludge and Gl-AnMBR effluent characteristics are summarized in Table 5.1. Solids characterization was performed as described in the Standard Methods (APHA, 2005). Dissolved organic carbon and dissolved nitrogen was measured with a Shimadzu TOC V-CSH Analyzer.

Table 5.1: Anaerobic sludge and Gl-AnMBR effluent characteristics

Parameter	UASB	Permeate
Reactor volume	8.5 L	
MLSS	8105 mg/L	----
TOC	1088 mg/L	487.9 mg/L
TN	516.1 mg/L	466.9 mg/L
Turbidity	197 NTU (supernatant)	6.5 NTU
Temperature	22-25 $^{\circ}$C	----

5.3   Filterability Tests Using Anaerobic Mixed Liquor

Full scale operation of airlift membrane filtration systems has proven to be successful by decreasing energy consumption while showing increased flux and lower membrane fouling. Under aerobic conditions, the membrane configuration used in this study has presented optimal performance at CFV between 0.3 – 0.5 m/s, while having air flow velocities in the same range (Futselaar et al., 2007 and 2009). Taking these values as a precedent and knowing that the two-phase flow pattern (slug flow) in this type of configuration is characterized by ratio of an injection factor (ε) from 0.2 to 0.9

(Cabassaud et al., 2001; Chang and Judd., 2002), the membrane was tested at the conditions shown in Table 5.2.

Table 5.2: Experimental conditions for two-phase flow in anaerobic sludge filterability

Liquid velocity (CFV), m/s	Gas velocity, m/s	ε*	Flux, Lm⁻²hr⁻¹ Mean	Membrane Resistance, 10¹²x m⁻¹
	0.00	0.0	34.9	5.14
0.31	0.31	0.5	17.5	13.8
	0.47	0.6	16.0	15.1
	0.78	0.7	17.5	13.2

*Where $= \dfrac{Q_g}{Q_g+Q_i}$, or fraction of gas in two-phase flow.

The effect of increasing gas flow rate on sludge filterability is almost unperceivable. By maintaining the injection factor (ε) within the two-phase flow range, the membrane flux can be sustained at values up to 20 LMH with no significant effect in TMP (Figure 5.2). However, it could be observed that the measured membrane resistance was affected by increasing Qg. This was originally accounted to the resistance of the cake layer (Rc), but after quantifying Rc by filtering distilled water, this value represented only 12% of the Rt. As discussed in other air-lift studies (Chang and Judd, 2002), the cake layer is not offering much resistance since the bubbles in the two-phase flow are continuously scrubbing the membrane, nevertheless this statement should be confirmed in extended operation since continuous sludge pumping could shear sludge flocs and increase colloidal deposition on membrane surface. Additionally, it was observed that the scrubbing gas was drawn to the permeate side in the filtration process, therefore decreasing the permeate volume and increasing the Rt value.
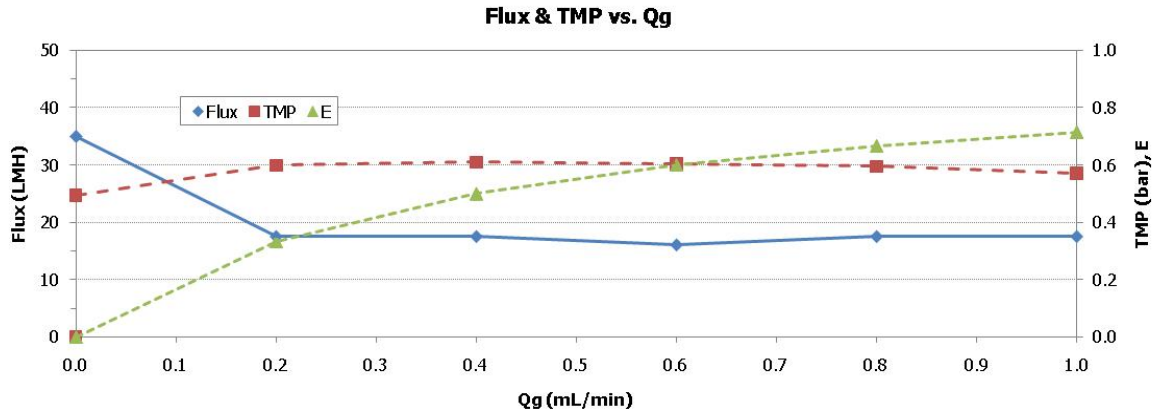
Figure 5.2: Effect of Qg on Flux and TMP

5.3.1 Critical Flux

Determination of the critical flux for was carried out as described in other studies (Defrane and Jaffrrin, 1999; Cho and Fane, 2002). Flux was increased in steps while monitoring TMP and a sudden jump in TMP was expected to be indicator of the critical flux. However, it was observed that the flux reached a steady condition soon after starting gas-lift assisted filtration (Figure 5.3). This behavior has been identified by Defrane and Jaffrrin (1999) when operating at constant CFV and the flux is mainly imposed by the permeate pump. Their system consists on solely cross flow filtration and larger fluxes could be obtained by increasing the permeate flow rate. Nevertheless, the presence of gas in the membrane significantly drops the flux to a stable value below the maximum flux allowed by the permeate pump (Figure 2a). Even though a flux higher 20 LMH could not be obtained under the conditions set in this experiment (i.e. CFV = 0.3 m/s and ε = 0.5), the TMP stabilizes and remains constant during operation. This fact indicates that the Gl-AnMBR is being operated at sub-critical conditions (Cho and Fane,

2002), which is expected to maintain reliable flux before irreversible fouling occurs. A *sustainable flux* of 20 LMH was obtained while maintaining constant TMP of 0.6 bar.
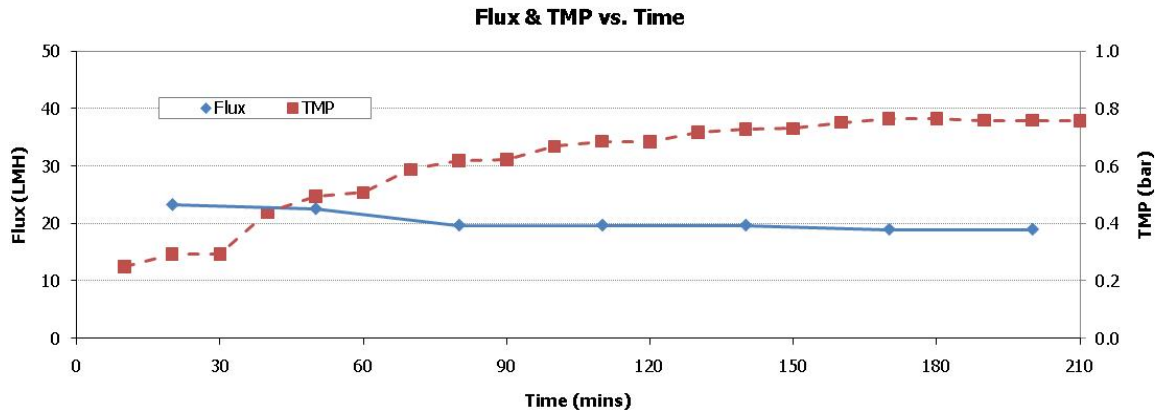
Figure 5.3: Sustainable flux during this study

5.3.2 Temperature Influence in Membrane Performance

Although all the others test in this study were performed at room temperature (22-25°C), the effect of temperature in the sludge filterability was assessed by the temperature in the reactor column (Figure 5.4). No significant change in flux was observed at mesophilic conditions in the bioreactor; however TMP decreased with ascending temperature. Higher temperatures for the membrane influent were not reached since the heat loss from the bioreactor to the membrane feed is significant. For the extended performance, the temperature will be set at 35°C to 40°C, which is expected to allow maximum flux without affecting the biological reactions that favor biogas production.
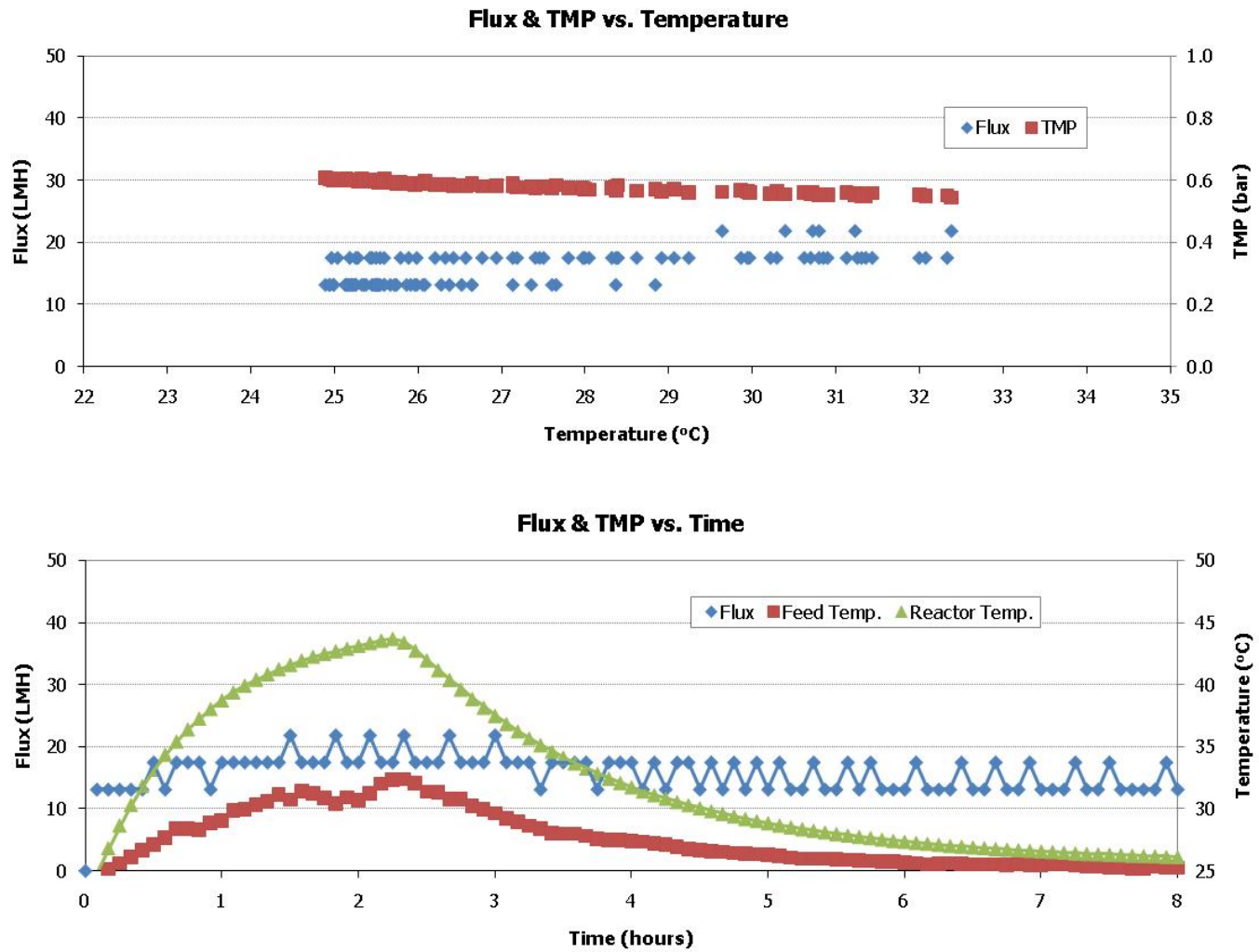
Figure 5.4: Influence of temperature in sludge filterability

5.3.3 Additional Fouling Control Mechanisms

Although the initial 20 LMH were maintained for the most part of the experimental runs, flux progressively declined due to sludge deposition o (cake layer formation) on the membrane surface. This contradicts the statement above presented for short term filtration and cake deposition. As reported in other studies (Table 2.5), the most common fouling control mechanisms, besides gas scrubbing, are relaxation and backwashing. In gas assisted filtration, relaxation is applied by periodically ceasing filtration to allow the cross flow and bubbles to scrub the membrane lumen. As a more aggressive mechanism, the permeate flow is reversed towards the lumen side during backwash. Theoretically, the majority of the reversible fouling should be remediated during the process. To evaluate their effect on filterability, these two mechanisms were applied and compared to continuous gas-lift operation. In the case of relaxation, the permeate pump was stopped every hour during 15 minutes. Likewise, backwashing was applied hourly for 15 minutes. Results from these experiments are presented in Figure 5.5. It could be observed that relaxation and backwash have similar effect in gas-lift filtration. After relaxation, 75% of the maximum flux (40 LMH) was recovered to but rapidly decayed to the flux before relaxation. There was not any effect on TMP during the testing period. Even though it had a very similar effect on membrane flux, backwashing demonstrated to slightly improve TMP while decreasing membrane Rt. These results confirm that for the set operational conditions, flux is completely independent from TMP. Relaxation and backwash did not have a significant consequence on flux improvement in the short term, which also confirms the two phase flow is the limiting factor for higher flux.
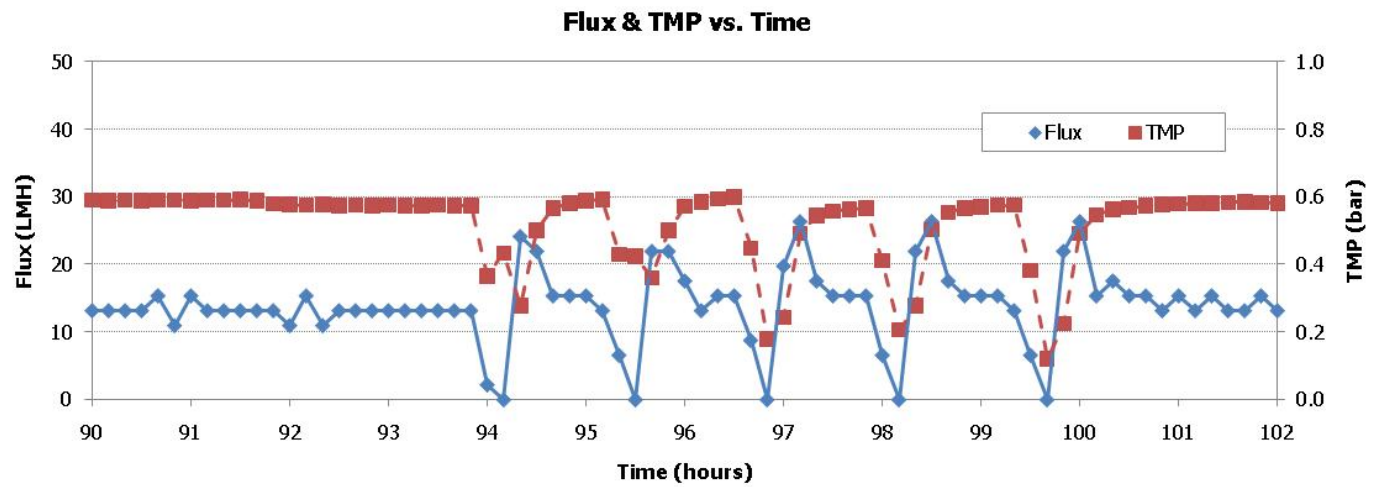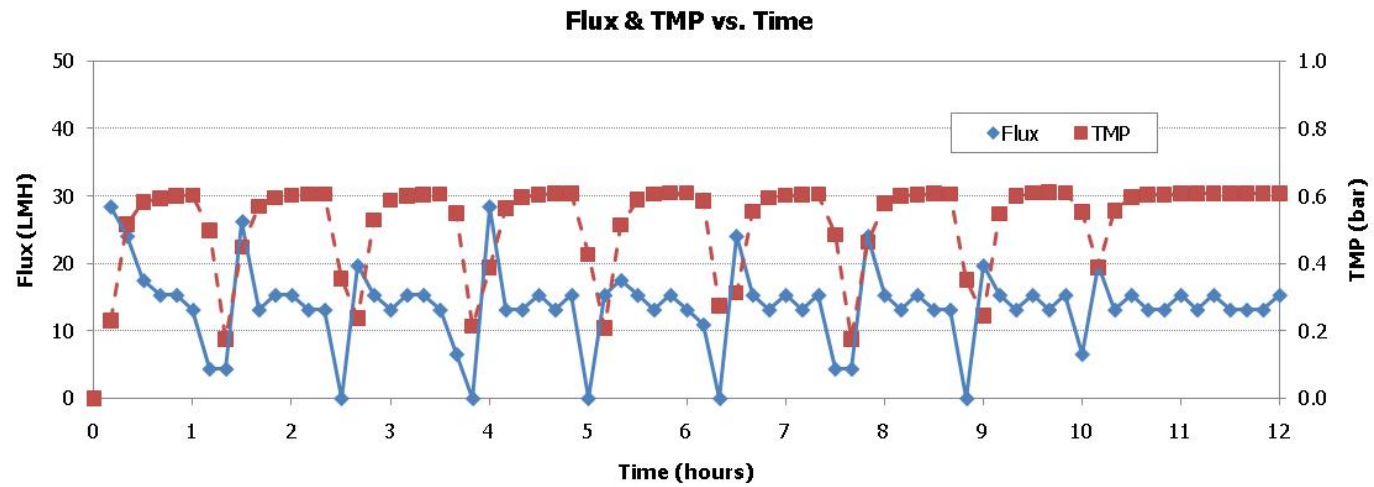
Figure 5.5: Effect of additional fouling control mechanism in Gl-AnMBR filtration
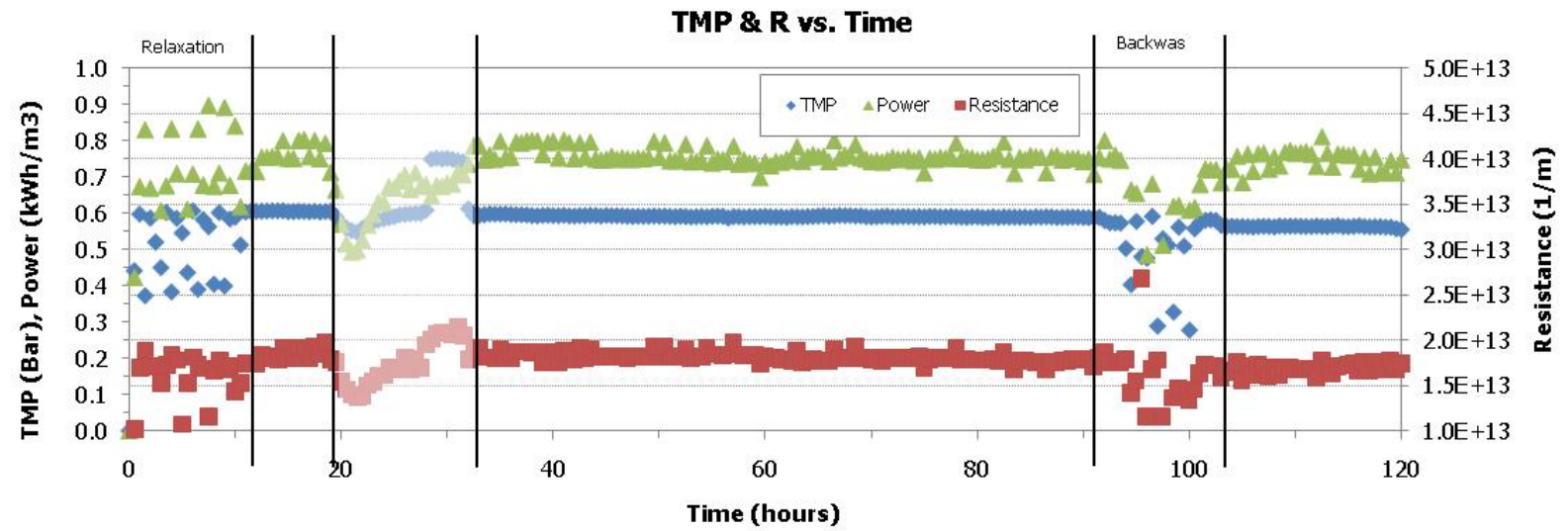
Figure 5.6: Influence of additional fouling control mechanism on Rt and power demand. Lighter area corresponds to a minor disruption of permeate pump.

5.3.4 Conclusions

A Gl-AnMBR was tested to identify its optimal operational conditions. Some of the most important conclusions from this study are listed:

- Critical flux during short term operation is mainly governed by the fraction of gas phase in the two phase flow. Further testing during extended periods should be done to evaluate the sustainability of the limited flux established in this preliminary study.

- When coupled with an UASB, minimum CFV through the membrane had to be applied to avoid disturbance of the bioreactor and guarantee minimum shear over the membrane surface. However, the applied CFV was not sufficient to drive high flux/low TMP filtration as described in previous air-lift and cross flow filtration studies. Additional changes to the flow rates in the membrane feed line (e.g. recycle loop) had to be done to provide higher CFV.

- Temperature proved to favor filtration at mesophilic conditions, which is an intrinsic advantage for anaerobic biological reactions.

- The power required for membrane operation under the tested operational conditions is comparable with those in literature for air-lift systems. However, the total energy consumption of the Gl-AnMBR still not quantified. Further studies with optimized membrane operation will be used for this purpose.

- Additional fouling control mechanism to gas-lift filtration did not improve significantly the flux values under the conditions tested in this study. Backwashing the membrane yet decreased TMP and total resistance, which could help to maintain a stable flux in the long run before irreversible fouling occurs.

5.4   Performance Evaluation: UNESCO-IHE Case Study

A similar GL-AnMBR was constructed at UNESCO-IHE (Delft, The Netherlands) to assess the reproducibility in the performance of Gl-AnMBR and to evaluate additional conditions in order to improve previous results. This study was done as a part of the International Research Experience for Students (IRES) program during 12 weeks.

5.4.1 Methodology

The experimental set-up at UNESCO-IHE lab consists of a 4 L complete mixed anaerobic bioreactor, coupled with a sidestream gas-lift ultrafiltration module (Figure 5.7). The membrane used is a 5.2 mm diameter polyvinylidene fluoride (PVDF) tubular membrane (Norit X-Flow, F4785) with a mean pore size of 0.03 um and active filtration area of 0.013 m^2.   Previous work had shown that maintaining a cross flow velocity of 0.3 m/s for the liquid side did not provide flux values larger than 20 LMH. In this case, the CFV was tested at higher values to improve cross flow filtration and membrane surface shear.   Although filtration was possible without additional filtration drivers except for gas lift and CFV, vacuum was applied to the permeate side to increase permeate production. A variable speed peristaltic (Masterflex I/P) was used to control permeate flow rate. Permeate volume over time was measured with an in-line rain gauge and transmembrane pressure (TMP) was measured by placing pressure gauges at the feed (Pf), permeate (Pp) and recycle (Pr) lines of the membrane module.   For this study, compressed hydrogen gas was used for membrane scrubbing since it was readily available at the IHE facility.   The gas flow rate (Qg) was controlled visually with a gas flow meter.   The retentate is recycled back to the reactor by a peristaltic pump. Temperature of the membrane feed was continuously monitored using in-line sensors.

The membrane was thoroughly cleaned after finishing sludge each test as described in Chapter 3, section 3.2.4. Gas-lift was the main antifouling mechanism used in this study.

The set-up was fed with flocculent anaerobic digestion sludge from a local wastewater treatment plant in Delft (Hoek van Holland, The Netherlands). Raw sludge had a solids concentration of around 29 g/L and the reactor was operated under room temperature during the summer season (23°C to 25°C) at neutral pH (6.5 to 7.5). The sludge was diluted to a concentration of 17g/L, similar to the maximum concentration of solids attainable in the previous filterability assessment. Table 5.3 summarizes the set of operational conditions evaluated. An additional scope of this study was to evaluate the effect of high oil content on the membrane performance since COPAS, as well as domestic wastewater effluents, are usually rich in oil and fats that can interfere with the performance of conventional MBR (Chang et al., 2002; Cheryan and Rajagopalan, 1998). Once the reactor was operating at stable conditions (constant flux and TMP), oleic acid in the form of sodium oleate was added to the sludge to mimic the content of oily compounds in wastewater. Oleic acid is one of the main long chain fatty acid (LCFAs) present in wastewater (both municipal and food processing), and concentrations of 100, 300 and 600 mg/L were selected to be tested for filterability purposes. Even though these final experiments could not be concluded due to time limitations (e.g. length of research experience at IHE was only 12 weeks), early results are presented in Appendix A.1.

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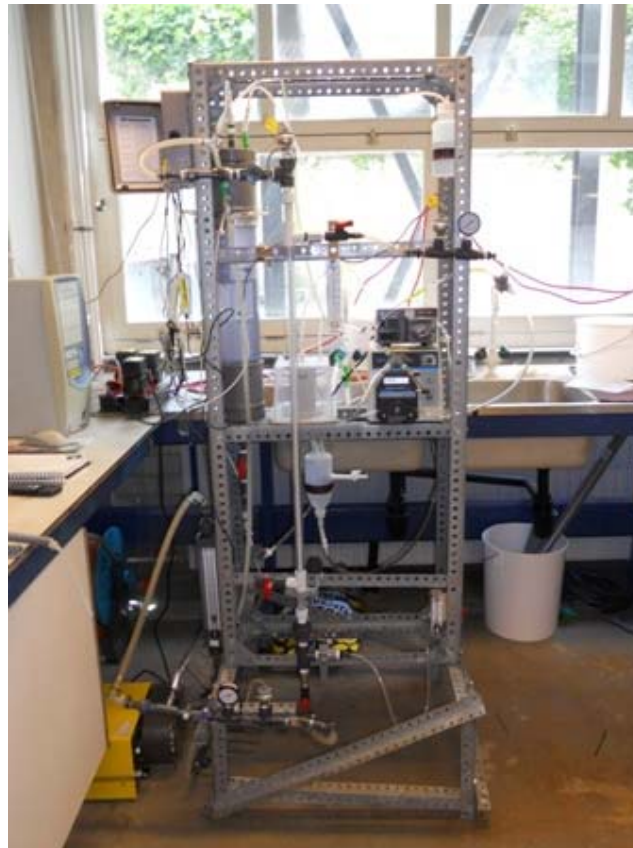
Figure 5.7: Gl-AnMBR configuration for UNESCO-IHE case study

Table 5.3: Summary of operational conditions tested on the Gl-AnMBR at UNESCO-IHE

Experiment	Feed	CFV (m/s)	Qg (LMP)	Qp
Membrane start-up				
1) Rm calculation/Qp det.	Tap water	0.3, 0.5, 0.75 and 1	Not applied	Increasing values per CFV
2) Rm calculation	Tap water	0.3, 0.5, 0.75 and 1	Not applied	No permeate pump
3) Gas-lift effect – CW1	Tap water	0.3, 0.5, 0.75 and 1	0, 0.2, 0.4, 0.6, 0.8 and 1	No permeate pump
4) Gas lift effect – CW2	Tap water	0.3, 0.5, 0.75 and 1	0, 0.2, 0.4, 0.6, 0.8 and 1	Qp det. In 1)
5) Critical flux	Raw sludge	0.3, 0.5, 0.75 and 1	Not applied	Increasing values per CFV
6) Gas-lift effect – RS	Raw sludge	CFV det. In 5)	0, 0.2, 0.4, 0.6, 0.8 and 1	Qp det. In 5)
7) Synthetic sludge 1	Raw sludge	CFV det. In 5)	Qg det. In 6)	Qp. det in 5)
Synthetic sludge filterability				
8) Synthetic sludge 2	Raw sludge + oleic acid	CFV det. in 5)	Qg det. in 6)	Qp. det in 5)
9) Synthetic sludge 3	Raw sludge + palmitic acid	CFV det. in 5)	Qg det. in 6)	Qp. det in 5)
10) Synthetic sludge 4	Raw sludge + stearic acid	CFV det. in 5)	Qg det. in 6)	Qp. det in 5)
11) Synthetic sludge 5	Raw sludge + 3 oily compounds	CFV det. in 5)	Qg det. in 6)	Qp. det in 5)

5.4.2 Influent of Operational Parameters in Membrane Flux

A maximum *operational flux* of 20 LMH could be maintained for MLSS of around 17 g/L at CFV of 0.52 m/s. These operational conditions were achieved after comparing the reactor performance at CFV of 0.52, 0.75 and 1.05 m/s (Figure 5.8). Selection of CFV was based on the lowest observed TMP value that allowed a maximum possible flux. Fluxes larger than 30 LMH were not attainable during the length of the operation due to intrinsic limitations of the MBR configuration (Defrance L. and Jaffrin M.Y., 1999). In this case, the maximum possible flux depends upon the flow rate established by the permeate pump and only lasts a few minutes after reaching a stable state. For this set up, 20 LMH could be maintained for at least 72 hours without applying any fouling control mechanism such as backwashing or relaxation. TMP for this type of configuration was maintained at 1 Bar. This value could not be decreased at the set operational condition without sacrificing the permeate production (Figure 5.9). A minimum TMP of 0.7 Bar was obtained by decreasing permeate flow rate, therefore decreasing flux.
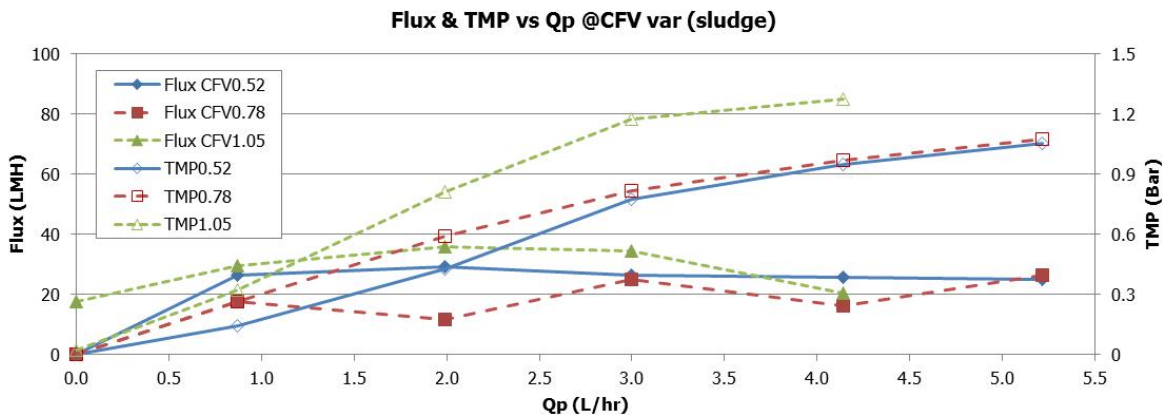
Figure 5.8: Comparison of CFV for sludge filterability
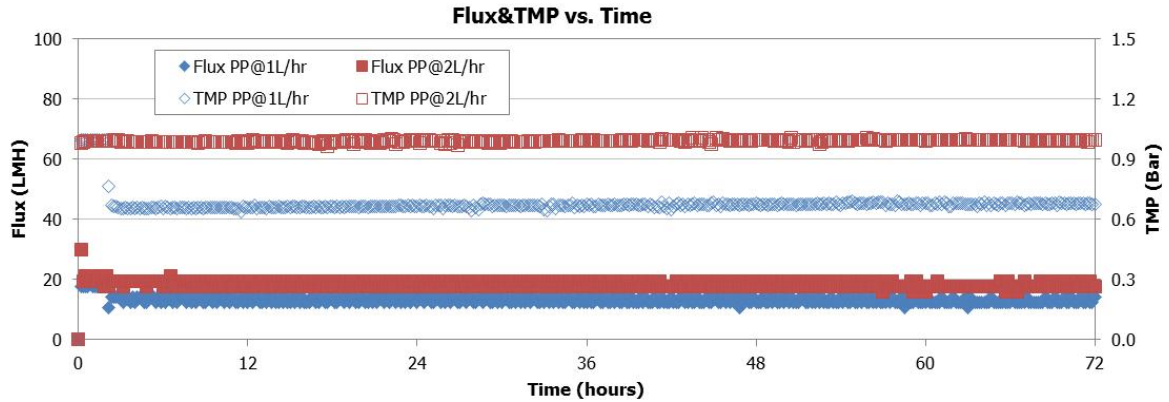
Figure 5.9: Effect of permeate pump flow rate on flux for 72 hours operation

Variation in the gas flow rate for larger values of E did not favor significantly filterability of sludge as established in previous work (Figure 5.10). Increasing the fraction of gas in the gas-sludge mixture does not considerably improve membrane flux but increased filtration resistance. Table 5.4 summarizes the effect of increasing gas fraction on resistance while operating at CFV of 0.52 m/s. A gas fraction for the two phase flow of 0.1 was sufficient for the MBR operation. This result suggests that the gas present in the gas-liquid mixture is only scrubbing the membrane as it rises and not lifting the fluid according to the gas-lift concept. In this case, the performance of the GL-AnMBR in the long run (i.e. more than 72 hours) has to be evaluated to assess the effectiveness of the "gas-lift" to improve membrane operation and decrease irreversible fouling.
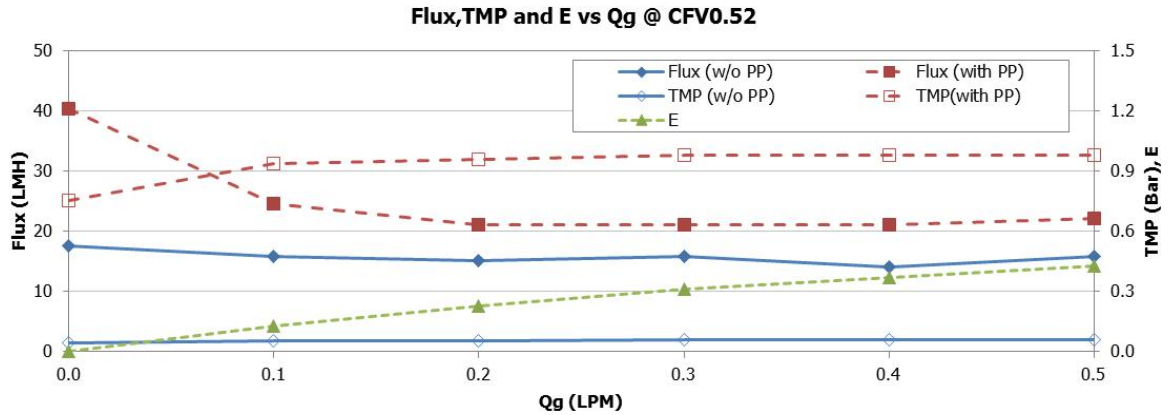
Figure 5.10: Effect of gas fraction in two-phase flow filtration. Notice the effect of permeate pump (PP) on membrane TMP and Flux.

Table 5.4: Effect of gas fraction in filtration resistance

ε	Tap water		Sludge	
	Avg. Flux (LMH)	Rt (1/m x 10^{12})	Avg. Flux (LMH)	Rt (1/m x 10^{12})
0	105.59	0.14	40.32	9.04
0.13	97.47	0.15	24.54	16.38
0.23	101.53	0.14	21.04	19.42
0.31	101.53	0.14	21.04	19.57
0.37	97.47	0.15	21.04	19.54
0.43	97.47	0.16	22.21	19.63

5.4.3 Summary and Conclusions

A lab scale Gl-AnMBR was built at UNESCO-IHE and tested to identify its optimal operational conditions and the following statements were confirmed during this study:

- An operational flux of 20 LMH could be maintained for a solids concentration of 17 g MLSS/L at a CFV of 0.52 m/s and permeate flow rate (Qp) of 2 L/hr. A gas fraction of 0.13 was sufficient for GL-AnMBR operation. The concept of gas-lift should be reevaluated and better described as gas scrubbing.

- More testing on the long run (i.e. more than 72 hours) should be performed to assess the effectiveness of gas-lift/scrubbing as fouling control mechanism.

- Although other studies have reached TMPs as low as 0.1 bars in aerobic configurations (Futselaar et al., 2007 and 2009), the current set-up does not allow TMP lower than 0.7 bars without sacrificing permeate production. However, fouling control mechanisms coupled with lower Qps should be evaluated to overcome increased TMP.

- Future work includes testing of filterability at higher concentrations of solids to evaluate its effect on membrane performance and operational parameters.

5.5   Extended Operation of The Gl-AnMBR

By considering the operational conditions with potential of least energy consumption in Gl-AnMBR operation, the CFV, Qp and temperature were established at 0.5 m/s, 1 L/hr and 37°C respectively for extended operation of the reactor. Under these conditions, continuous operation of the Gl-AnMBR was evaluated during three months where the performance is only interrupted by short periods of membrane cleaning. Cleaning of the membrane module was performed weekly as indicated in Chapter 3, Section 3.2.4. Although similar MBR configurations require more frequent and intensive cleaning protocols to maintain operational fluxes much larger than the ones presented in this work, this specific configuration was operated under suboptimal conditions to evaluate the sustainability of long term/low cost operation of this reactor.

Since full operation of the Gl-AnMBR is assessed in this stage of the research, biogas produced form the anaerobic digestion of COPAS is used for membrane scrubbing. Gas from the headspace was continuously recirculated to provide a gas

fraction in the two-phase (ε) flow of about 0.1. Additional challenges to the Gl-AnMBR operation were assessed in this stage, specifically related to the reactor feeding with particulate substrate, sufficiency of biogas to support gas scrubbing and system gas leaks.

5.5.1 Feed Sewage and Seed Sludge

For the extended operation of the Gl-AnMBR, COPAS was used at a TS concentration of 1000 mg/L. The reactor was restarted with fresh flocculant digester sludge from the local WWTP (i.e. Howard F. Current AWTP). In this case, the sludge was used as it was obtained from the plant with a TS concentration of 17 g/L and 70% TSS.

Environmental characteristics of the feed are summarized in Table 5.5, however a more detail description of the COPAS feed is presented in Chapter 4. The sludge was sieved through the No. 20 mesh to remove any debris that could clog the membrane lumen or block in the reactor tubing. Influent flow rate was determined by the limiting flux assessed in previous studies (i.e. 20 LMH). For conservative purposes, an operational flux of 10 LMH was assumed and a feeding flow rate 2.1 mL/min was set. Since COPAS is a particulate substrate, there is an additional challenge to reactor's feeding protocol. As common practice at lab scale, soluble synthetic influents are easily pumped into the biological at the desired flow rate. In our case, COPAS had to be pumped in batches every 6 hours at a higher rate so particulate matter do not precipitate within the feed line and become remain.

Table 5.5: Environmental characteristics of the influent, effluent and liquid fraction of the Gl-AnMBR MLSS.

Concentrations are reported in units of mg/L.

Parameter	Influent		Effluent	MLSS Supernatant
	Total	**Soluble**		
CODt	1267±6.4			
CODs		257.3±195.5	70±22.1	438.5±198.3
TOC	528.5±2.7	73.29±44.29	25.44±4.5	118.16±2.38
TKN	54.3±0.3	21.7±9.5	75.43±9.7	
NH4-N		5.7±3.3	9.1±4.2	
NO3-N		1.8±0.7	2.2±0.9	
PO4-P		15.5±7.8	20±7.13	

Concentrations are reported in mg/L

5.5.2 Filtration Performance

As expected, a sustainable flux of 20 LMH was achieved during the start of the extended operation period. However, this performance could not be maintained for more than 24 hours since the flux rapidly declined to a stable value of 12 LMH (Figure 5.11). During the first month of operation, filtration was aided only by gas scrubbing generated from the gas-lift and no other anti-fouling mechanism was applied. Only when an increment of TMP of more than 15% was observed, a weekly additional cleaning protocol was applied to the module to sustain filtration. Such protocol is described in Chapter 3 and consists of forward flushing, backwashing and relaxation. Chemical cleaning was only applied when severe fouling was observed (i.e. more than 30% increase in TMP).

A maximum flux of 30 LMH was obtained momentarily during the first hour of operation when using a new membrane. This value has been reported elsewhere as a sustainable flux with analog aerobic configurations (Futselaar et al., 2007 and 2009; Evenblij, 2006), yet requiring a more intensive cleaning protocol. Overall hydraulic performance of the Gl-AnMBR treating domestic synthetic sewage was satisfactory.

Under suboptimal operational conditions, filtration of flocculent anaerobic sludge could be sustained by allowing gas scrubbing to be the main fouling control mechanism.

5.5.3 Biological Treatment

After reaching stable performance, total COD removal efficiencies up to 98% were assessed with average values of 93±6%. Average soluble COD (CODs) in the influent comprises about 23% of the total and varies upon feed sampling. Since the majority of the COD in COPAS is located in its particulate fraction, freshly prepared feed have very low concentrations of CODs compared to those of feed samples taken after more than 24 hours of remaining on the feed tank under continuous mixing. A summary of the environmental characteristics of the influent, effluent and liquid fraction within the reactor are presented in table 5.5.

As discussed in Chapter 4, biogas composition from the digestion of COPAS is characterized by 47% CH_4 and 37% CO_2 at a MLSS of about 2%. Since the reactor was seeded with the same sludge, these values were used to calculate the COD equivalence of methane in the biogas generated from the Gl-AnMBR. At steady state, average biogas production was about 4.5 L/day which was more than enough biogas to sustain membrane gas-lift. Although the biogas produced with this configuration was not used for any other purpose than membrane scrubbing, the potential applications of this digestion product as recovered resource (i.e. energy) are to be evaluated in further research.

The overall COD mass balance of the Gl-AnMBR can be represented with the following expression:

$$dM(total)/dt = dM(effluent)/dt + dM(CH_4)/dt$$

Where dM(total)/dt the total COD mass load on COPAS influent, dM(effluent)/dt
is the effluent soluble COD, and COD-CH$_4$ is the COD embedded in methane production.
COD assimilated for cell growth is not taken into account for this calculation since
anaerobic biomass growth is a very slow process and direct measurement of this
fraction in MLSS was usually covered by the background COPAS particulate fraction
accumulated within the reactor. A similar approach to the COD balance in Chapter 4 is
done to calculate each term of the equation. In this occasion, the values are reported in
g/d and are expressed as follows:

$$\frac{dM(total)}{dt} = Q_{influent} \times C_{COPAS} \times \gamma$$

Where Q$_{influent}$ is 3 L/d, C$_{COPAS-In}$ is 1 g/L, and γ corresponds to the COD/wt ratio in
COPAS of 1.26.

$$\frac{dM(effluent)}{dt} = Q_{effluent} \times CODs\text{-}Eff(t)$$

Where Q$_{effluent}$ is 3 L/d and CODs-Eff(t) is the soluble COD in the effluent at time t
(g/L).

$$\frac{dM(CH_4)}{dt} = \frac{P_{CH4} \times Q_{biogas}}{(0.35 \times (273^{o}K + T(t)/273^{o}K))}$$

Where P$_{CH4}$ is the concentration of CH4 in the biogas form COPAS degradation
(i.e. 47%), Q$_{biogas}$ is the daily biogas production (L/d), 0.35 is theoretical production of
CH$_4$ per g of COD (L CH$_4$/g COD), and T(t) is the sludge temperature at time t (K).

The estimated difference between the inflows and outflows of the Gl-AnMBR is
less than 15%, corroborating the assumptions taken into account in this balance.
Nevertheless, further research should include a more detailed COD balance that account

for biological growth. A summary of the COD profiles of the influent, effluent and sludge

liquid fraction is presented in figure 5.12.
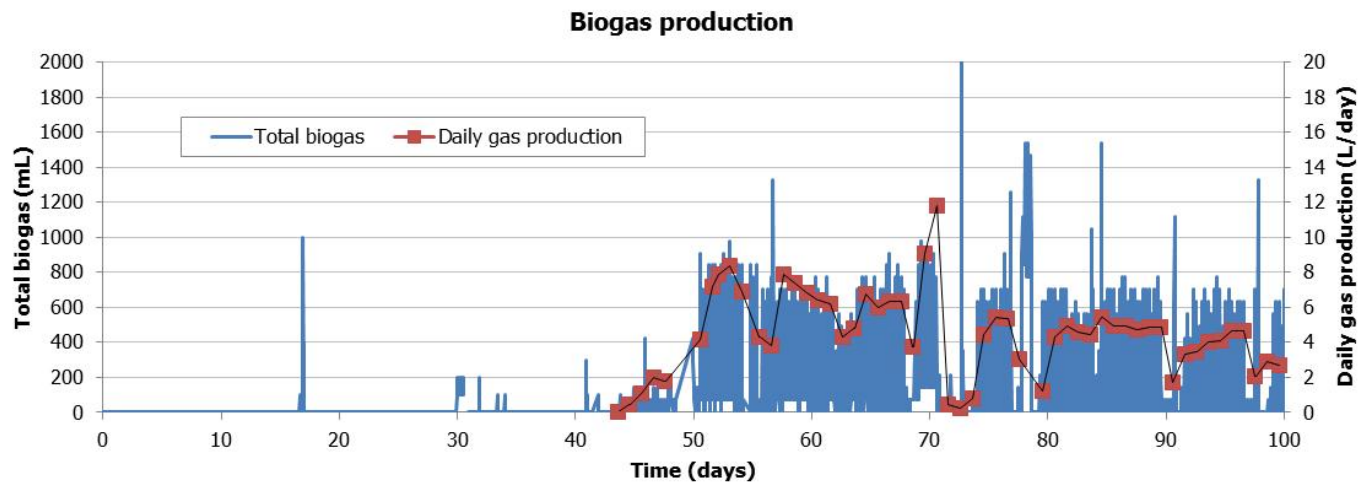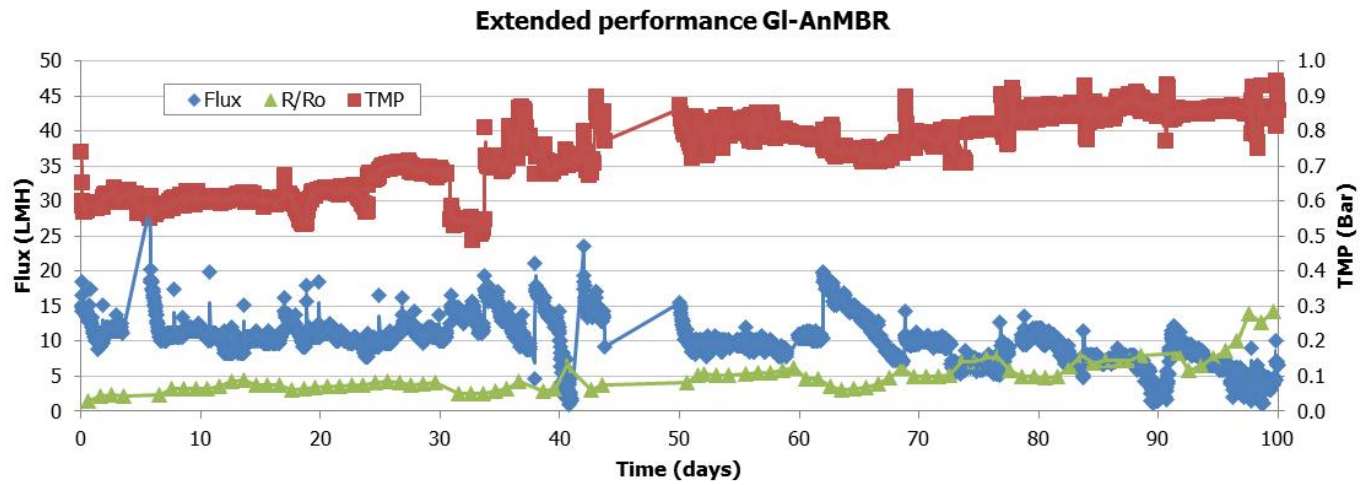
Figure 5.11: Gl-AnMBR extended operation performance
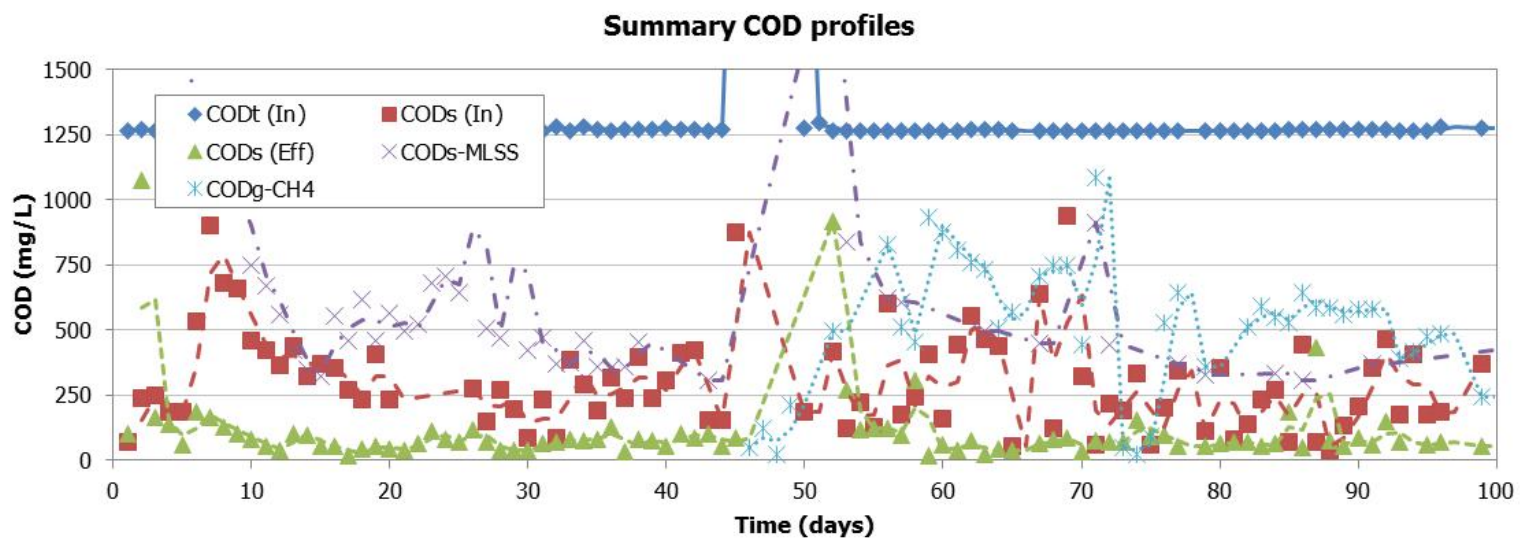
Figure 5.12: COD profiles of the influent, effluent and sludge liquid fraction of the GI-AnMBR during extended operation. The peak concentrations from

day 45 to 50 are due to excess addition of COPAS to recover biogas production.

Although anaerobic processed are very efficient in the removal of organic matter, nutrients on the other hand are hardly utilized in the biological process and represent a weakness of this type of treatment application. Although TOC was successfully removed with an average efficiency of 95.2±0.9% (Figure 5.13), results for nitrogen removal are almost unnoticeable. The apparent removal of nitrogen and phosphorous is mainly due to the retention of particulate COPAS within the reactor (Figure 5.14). Membrane filtration is therefore, the principal mechanism for nutrients removal. Retention of biomass and particulate matter allows more time for degradation of complex forms of nitrogen and carbon in proteins and fats respectively. However, soluble forms of nitrogen and phosphorous (i.e. NH_4, NO_3 and PO_4) simply pass through the system, providing a nutrient rich effluent. Average concentrations of specific nutrient species are presented in Table 5.5.
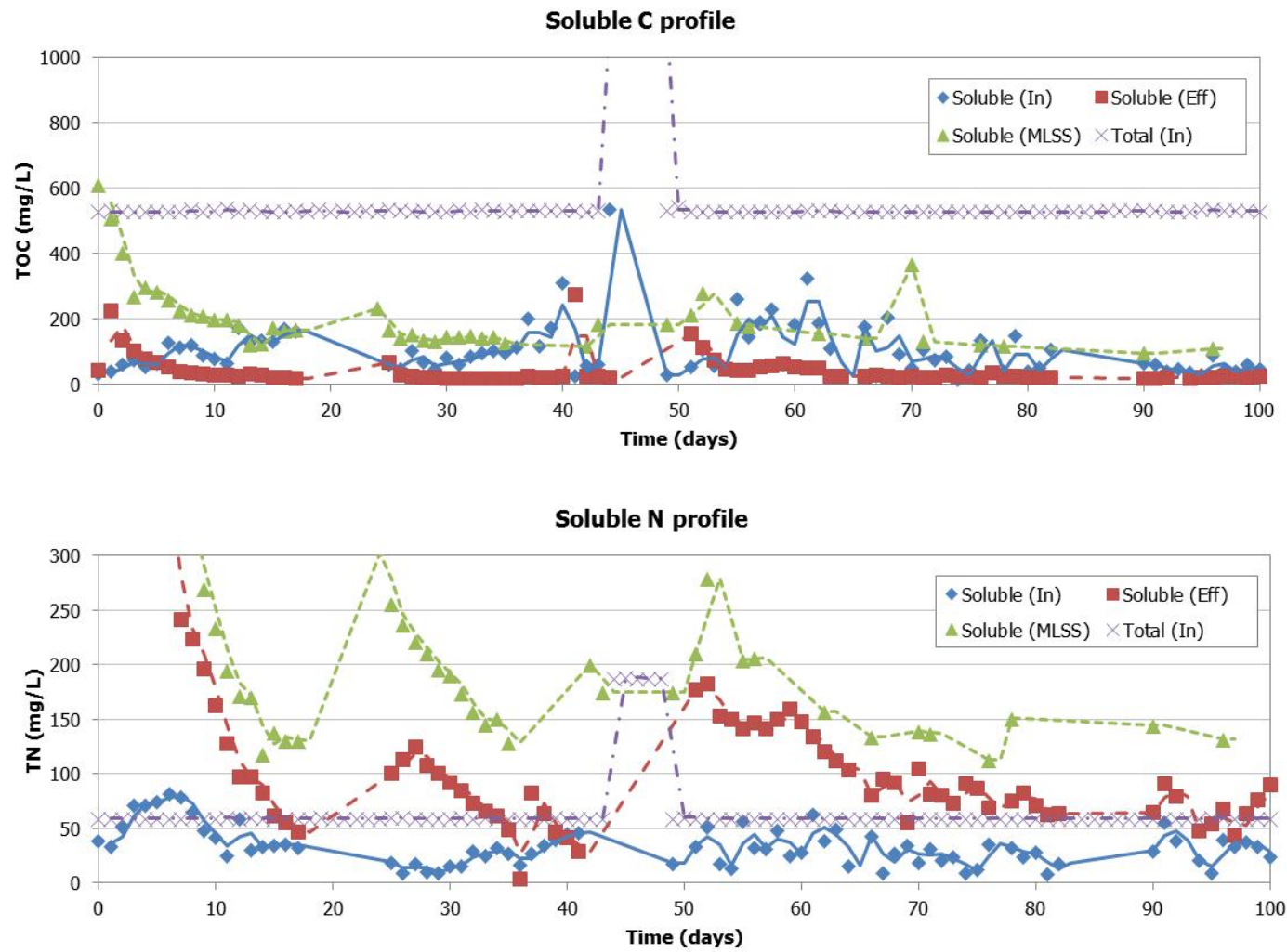
Figure 5.13: Organic carbon (top) and Nitrogen (bottom) profiles of the influent, effluent and sludge liquid fraction of the Gl-AnMBR during extended operation.

Figure 5.14: NH$_4$, NO$_3$ and PO$_4$ profiles of the influent, effluent and sludge liquid fraction of the Gl-AnMBR during extended operation.

5.6   Estimation of Energy Footprint in a Gl-AnMBR

In Chapter 3, the general energy demand for an AnMBRs was expressed as:

$$E_{net} = E_h+E_p+E_v+E_m+E_s+E_{pp}-E_g$$

Where E_h corresponds to bioreactor heating, E_p is the pipe system energy loss, E_v is the velocity energy loss, E_m is the energy required for membrane module operation, E_s is the energy required for gas scrubbing, E_{pp} is the energy for permeate pump operation, and E_g is the energy produced in biogas. Additionally some considerations regarding Gl-AnMBR were discussed in terms of overall energy production:

- For an air/gas lift supported configuration, energy loss due to friction in the pipeline is minimal due to very low recycle flows (van 't Oever, 2007)

- Energy loss for low recirculating flow rates (i.e. low CFV) can be negligible (Zhang et al.,2003)

- Gas scrubbing improves membrane flux, and decreases energy demand for sludge recirculation, bubbling and filtration (Futselaar et al., 2006)

- Power requirements for biogas recycle/scrubbing (E_s) are defined depending on the type of recirculation device (pump or compressor)

These considerations reduce the overall energy expression to:

$$E_{net} = E_h+E_m+E_s+E_{pp}-E_g$$

Where the overall energy demand per treated effluent for the system is given by:

$$E_T = E_{net}/Q_p/3600; E_T = KWh/m^3$$

Power demand due to membrane operation, pumping and biogas recycle has to be calculated to evaluate the overall energy consumption of the Gl-AnMBR. Additionally, energy generated through biogas production has to be quantified depending upon on-site application (e.g. biogas for sludge recirculation) and/or potential uses. Based on

Zhang et al. (2003) and Judd (2006), equations related to power requirements in an air-lift filtration system are stated as follows:

5.6.1 Power for Membrane Operation

$$E_m = Q_r(P_{in} - P_r) \times 100 + Q_p(TMP) \times 100$$

Where E_m is the power for membrane operation (KW), Q_r is the recycle flow rate (m^3/s), Q_p is the permeate flow rate (m^3/s), P_{in} is the liquid pressure in the membrane influent (bar), P_r is the liquid pressure in the recycle line (bar), TMP is membrane trans membrane pressure, and P_{out} is the liquid pressure in the membrane effluent.

During preliminary operation (i.e. performance at room temperature, CFV = 0.3 m/s and ε = 0.5), the energy required for membrane performance reached an estimated maximum of 0.9 kWh/m^3, which is comparable to those obtained in full scale application. Although these findings are not conclusive, extended operation of the Gl-AnMBR at upgraded operational conditions (i.e. thermophilic operation, CFV= 0.5 m/s and ε = 0.1) did not show major improvement in power demand for membrane filtration since this estimation directly depends on produced permeate and pressure (Figure 5.15). At an operational flux of 20 LMH, an energy demand of about 1.4 kWh per cubic meter of permeate is estimated under suboptimal operational conditions. In this case, higher fluxes should be obtained to decrease power demand which can be easily attained by applying frequent fouling control techniques.
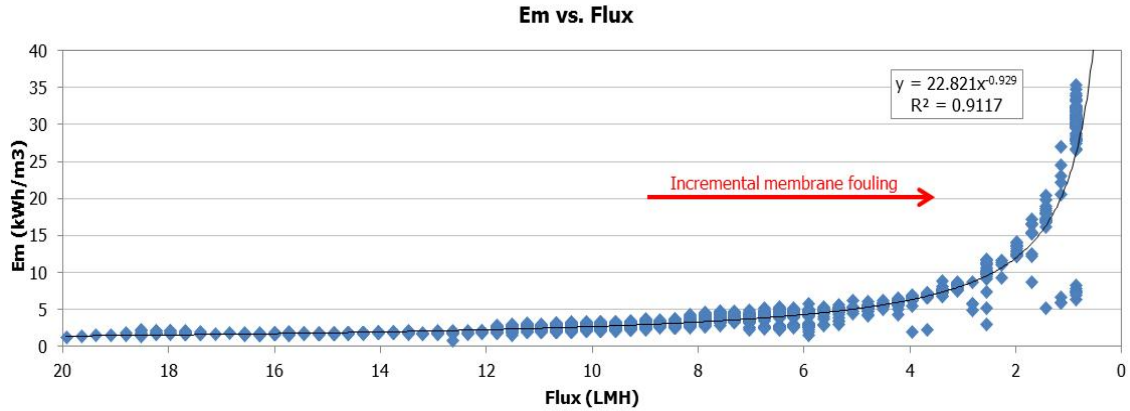
Figure 5.15: Relation between power demand for membrane operation and permeate production during extended operation.

5.6.2 Power for Pumping Requirements

$$E_p = \rho g H_T Q_{pump}/1000\eta$$

Where E_p is the power for liquid pumping (KW), ρ is the liquid density (kg/m^3), g is 9.81 m/s^2, H_T is the pump head including system losses (m), Q_{pump} is the pump capacity (m^3/s), and η is the pump efficiency.

Since the gas recycle in the Gl-AnMBR is done by pumping the headspace gas back to the membrane module, the above equation applies for the calculations of E_s. However, energy for pumping requirements depends of the choice of pump, and efficiency (η) and density of the pumped fluid. For practical purposes the assessment of the latter variable is not attained in this work therefore; any pumping energy consumption at lab scale is monitored using a commercial electricity load meter. Average daily power demand for pumping requirements in the Gl-AnMBR is presented in Table 5.6.

Table 5.6: Average daily power demand for pumping requirements in the Gl-AnMBR. Values measured with

a load meter at lab scale

Item	Power demand (kW)
Permeate pump	0.9×10^{-2}
Sludge recycle pump	1.2×10^{-2}
Gas recycle pump	1.6×10^{-2}
Total power for pumping	3.7×10^{-3}

If normalized to the permeate generated from the lab scale set-up, the values of power demand for pumping requirements are largely overestimated and they are not representative of large scale operation. Actual values for backwash and permeate pump requirements in a large scale Air-lift MBR (i.e. 100000 GPD treatment capacity) were reported by Yeh et al. (2006) as 0.2 HP (0.9×10^{-2} kWh/m^3) and 0.1 HP (0.5×10^{-2} kWh/m^3) respectively.

5.6.3 Power for Reactor Heating

Although it have been stated in previous chapters that the produced biogas in anaerobic digestion can offset reactor heating requirement, the E_h factor have been included in the E_{net} estimations during extended operation to evaluate the certainty of this statement. Edelman et al. (2000), established the energy demand for reactor heating at mesophilic conditions in about 50 kWh/ton of waste. In our case, the daily load of COPAS to the Gl-AnMBR is about 3.5 g/day and the energy requirement for heating can be estimated in 1.5x10-4 kWh/d or 0.05 kWh/m^3 of permeate.

5.6.4 Power from Biogas Production

On the other hand, power generation from biogas can be accounted depending upon final application. Lubken et al. (2007) defines power generation for electricity and/or thermal application as:

$$E_g = Q_g P_{CH4} H_c \eta$$

Where Q_g is the biogas flow rate (m^3/d), P_{CH4} is the percentage of CH_4 in biogas (%), H_c is calorific value of methane (kWh/m^3 of methane), and η is the efficiency of the final conversion process. According to Zupancic and Ros (2003) the value of H_c is approximately 35800 kJ/m^3 of CH_4 at STP (11.2 kWh/m^3 at 35^oC). Lubken also identifies the efficiency of methane conversion to electricity in about 35% and the efficiency of methane conversion to thermal in about 55% (for a combined heat and power unit). For an average biogas flow rate of 4.5 L/d and 47% CH_4 content, an estimated value of 0.01 KWh/d is obtained for the power generated through produced methane conversion to electricity. This value is surprisingly small compared to the actual energy embedded in CH_4. An estimation of potential in power generation from Gl-AnMBR biogas is summarized in Table 5.7. As discussed in Chapter 4, COPAS exerts about 1.26 g of COD per g of sample (i.e., γ = 1.26) and about 65% of this substrate is easily biodegradable to methane. Rows 3 and 4 were calculated using the conversion factors for methane equivalence to COD of 0.25 g CH_4 /g COD an 0.35 L CH_4/g COD respectively. The fifth row is obtained using H_c in units of kJ/g CH_4 at 35^oC.

Table 5.7: Potential power generation from COPAS in the Gl-AnMBR

	Concentration (unit /m3 of sewage)		Load normalized rate (unit/day)		Load normalized rate per reactor volume	
Total COD	1260.0	g COD/m3	3.8	g COD/day	381.0	g COD/m3-d
Biodegradable COD	819.0	g COD/m3	2.5	g COD/day	247.7	g COD/m3-d
Methane equivalent	204.8	g CH4/m3	0.6	g CH4/day	61.9	g CH4/m3-d
Volume equivalent at 35°C	497.5	L CH4/m3	1.5	L CH4/day	150.5	L CH4/m3-d
Power from combustion (η=100%)	10258.0	kJ/m3	31.0	kJ/day	3102.0	kJ/m3-d
	2.8	kWh/m3	8.6E-03	kWh/day	0.9	KWh/m3-d
Power from CHP conversion (η=55%)	5641.9	kJ/m3	17.1	kJ/day	1706.1	kJ/m3-d
	1.6	kWh/m3	4.7E-03	kWh/day	0.5	KWh/m3-d
Power from electric conversion (η=35%)	3590.3	kJ/m3	10.9	kJ/day	1085.7	kJ/m3-d
	1.0	kWh/m3	3.0E-03	kWh/day	0.3	KWh/m3-d

In summary, the total power (E_T) or energy footprint of the Gl-AnMBR is summarized in Table 5.8. An energy footprint in the range of *-1.2 to 0.7 kWh/m³* and *-2.3 to -0.5 kWh/m³* was determined for lab-scale and full-scale systems, respectively, under different methane to energy conversion options. The energy requirement to operate lab-scale membrane systems (1.4 kWh/m³ as directly measured using plug-in watt meters) is higher than that expected for full-scale systems (0.2 kWh/m³ as reported by equipment vendor Dynatech) due to the inherent inefficiency of small peristaltic pumps. The values presented in Table 5.8 are based on conservative estimations of itemized energy input requirements. However, these values can be easily improved in a large scale scenario by improved biogas generation and conversion mechanisms, optimized permeate production for lower TMP and higher flux. Meanwhile, biogas production can offset reactor's heating requirements and moreover, can lower the overall energy footprint and even shift it to energy surplus. A number of options are available for methane conversion to energy. The most efficient is complete combustion for heat (η=100%), followed by combined heat and power (η=55%), with electricity conversion (η=35%) the least efficient. CHP could be a suitable option, using a portion

of the produced methane to heat the reactor and the rest to generate electricity to run the pumps.

Table 5.8: Comparison of energy footprint of lab-scale and full-scale Gl-AnMBR under different methane-to-energy conversion options

Gl-AnMBR energy requirements	Case based Net Energy (kWh/m^3)					
	Full biogas conversion		**CHP conversion**		**Electricity Conversion**	
Membrane operation	1.4[a]	0.2[b]	1.4[a]	0.2[b]	1.4[a]	0.2[b]
Pump requirements [c]	0.2	0.2	0.2	0.2	0.2	0.2
Reactor heating[d]	0.05	0.05	0.05	0.05	0.05	0.05
Power from biogas	-2.8[e]	-2.8[e]	-1.6[f]	-1.6[f]	-1.0[g]	-1.0[g]
Energy footprint	**-1.2**	**-2.3**	**0.1**	**-1.1**	**0.7**	**-0.5**

a) Energy required for membrane operation at lab-scale; b) Energy required for membrane operation at plant-scale (Yeh et al., 2006); c) Energy for pumping at plant-scale; d) Energy required for mesophilic digestion at plant-scale; e) Energy from full conversion of methane in combustion; f) Energy from CHP conversion of methane; and g) Energy from electricity conversion of methane

5.7 Summary and Conclusions

- Although air-lift aided filtration have been widely used to decrease membrane fouling and increase permeate production, its application in anaerobic conditions is still under development and more research is required to get this MBR configuration to a commercial stage.

- The set of operational parameters established in this study allowed the reactor to sustain filtration at suboptimal conditions for an extended period while providing a maximum operational flux of 20 LMH.

- Under the tested operational conditions, the Gl-AnMBR present excellent removal efficiencies of organic matter (i.e. up to 98% and 95% in COD and TOC removal respectively) while producing energy in the form of methane at a amounts suitable for maintaining membrane scrubbing (4.5 L/d of biogas). Removal of

nutrient is less relevant since the effluent of this reactor is suitable for immediate reuse applications, allowing the recovery of soluble fertilizers from sewage.

- Depending on methane conversion options, the energy footprint of this configuration ranged from -1.2 to 0.7 kWh/m^3 and -2.3 to -0.5 kWh/m^3, for lab-scale and full-scale systems, respectively. These are values comparable to actual energy consumption from anaerobic and aerobic commercial MBRs discussed in Chapter 3.

- Energy demand per treated sewage can be easily improved in a plant scale scenario by using more efficient pumps, improved biogas generation rates and energy conversion mechanisms, and optimized permeate production for lower TMP and higher flux.

- Results from this study suggest that the Gl-AnMBR can be applied as sustainable treatment tool for wastewater resources recovery, which can further be optimized.

- Further research should include comparison of the obtained values to actual domestic sewage in terms of organic matter removal, sufficiency of biogas production for membrane scrubbing and methane production, and recovery of soluble forms of nutrients for further reuse applications.

6 REUSE OF GL-ANMBR EFFLUENT BY ALGAL-PHOTO MEMBRANE BIOREACTOR (APMBR)

6.1   Introduction

Among the innumerable reuse applications for wastewater, biofuel producing algal growth has become an extremely attractive option to maximize the utilization of wastewater embedded materials. In 2008, an article written by Clarens et al. (2010) highlighted the weak points of the current practices on biofuel algae mass production, pointing put the high demand of resources like water and fertilizers and the low efficiency in the current harvesting methods. On the other hand, algal growth from wastewater has been used as treatment technique for decades and its application towards biofuel production has gained a lot of attention lately since algae not only provide an additional polishing step to ww treatment, but wastewater itself provides a fertile medium for algal development (Table 2.8). An established limitation of algal growth form wastewater rests on the necessity of a "clean", particle free medium that allows algae maximum uptake of soluble nutrients in wastewater. Moreover, adequate light penetration should be allowed to the algal reactors, while minimizing the exposure of the algal cultures to exogenous microorganisms that could outcompete the algae for the available nutrients. With the aim to overcome some of these challenges, membrane filtration units have been identified as a feasible pre/post treatment technology to algal reactors. Concentration of algal slurry using membrane filtration provides excellent results in terms of retention of algal cell and high quality effluent (Danquah et al., 2009;

Zhang et al., 2010; Zou et al., 2011). More specialized membrane applications to algal biotechnology include, and are not limited to, energy recovery and improvement of operational parameters within algal reactors. For example, gas permeable/selective membranes have been wildly used for recovery of gaseous algal byproducts such as hydrogen (Teplyakov et al., 2002), as well as to improve algal growth through optimized CO_2 mass transfer using capillary membranes (Kumar et al., 2010). Table 6.1 present some examples of membrane filtration recently applied in algal technology.

Table 6.1: Applications of membrane technologies to algal bioreactors

Application	Algal strain	Membrane module	References
Recovery of pigment from marine algae	*Haslea ostrearia*	Flat sheet ultra-filtration membrane (Rayflow, rhodia-Orelis Co., Maribel, France)	Rossinnol et al., 2000
Filterability of algal monocultures responsive to seasonal variation of temperature and radiation	*Chlorella sp.*	PVDF Disc filters 0.45 um (hydrophilic Durapore membrane; Millipore HVLP 090-50)	Babel et al., 2002
Recovery of high quality fuel gases using an active membrane systems (membrane contactors)	Algae or Cyanobacteria (not specified)	0.2 um flat sheet asymmetric polyvinyltrimethylsilane (PVTMS) membrane	Teplyakov et al., 2002
Optimizing CO2 mass transfer to algal cultures grown on industrial wastewater	*Spirulina platensis*	Composite laminated hollow fibers (MHF200TL; Mitsubishi Rayon, Tokyo, Japan)	Kumar et al., 2010
Membrane filtration for harvesting of algal biomass	*Scenedesmus quadricauda*	Polyvinylchloride (PVC) hollow fiber ultrafiltration (UF) membrane module (LU8A-4A, Litree Co., Hainan, China)	Zhang et al., 2010
Separation of biofuel algae cultures using FO	*Chlorella sorokiniana*	flat-sheet FO membrane from Hydration Technology Inc. (Hydrowell Filter, HTI, Albany, OR).	Zou et al., 2011

Major limitations regarding membrane filtration systems are localized in their energy consumption and membrane fouling propensity. In the case of algal filtration, fouling mechanisms have been extensively studied, especially when applied to surface

water filtration. As part of the organic matter in surface water, algae actively participates in membrane biofouling, which is characterized cake layer deposition and adsorption of intercellular and/or extracellular organic matter (EOM) (proteins, polysaccharides or polysaccharide-like substances) on the membrane surface (Babel et al., 2002; Lee et al., 2006; Zhang et al., 2010). In this case, controlled environmental conditions within closed algal reactors might have a great effect in algal biofouling since EOM characteristics depend on nutrient concentrations, CO_2 availability, temperature and light exposure (Babel et al., 2002).

On the other hand, only a few investigations have focused on decreasing membrane filtration energy consumption in algal related applications. Zou et al., (2011) recently evaluated the use of forward osmosis as a mean to improve algal dewatering while decreasing the energy demand related to pressurized filtration (e.g. power for pumping requirements). In this case, fluxes higher than 35 LMH could be obtained by using only the concentration differential between a low concentration feed water and a high concentration draw solution. Zhang et al., (2010), evaluated a more conventional approach using cross flow filtration with hollow fiber membranes. Although low TMP of 0.3 bars (CFV 0.17 m/s) at fluxes up to 45 LMH were obtained, rapid fouling occurred in all experiments and concerns related to algal cells integrity are not considered in this study since dewatering is the main objective. This investigation however, highlights the use of gas scrubbing to improve extended membrane operation.

Although gas-lift assisted filtration has been proven as a successful approach to overcome both, fouling and energy demand problems of conventional MBRs (Table 2.8, Chapter 2), little information is available in its application to algal-MBR systems. Troubleshooting membrane operational constrains while providing optimum conditions

for biofuel algal growth, is task that gas lift filtration can easily target since it provides membrane scrubbing (i.e. fouling control mechanism), gentle recycle of algal liquor (i.e. pumping requirement and biomass shearing is avoided). In the meantime, complete retention of algal biomass allows extended contact times for carbon and nutrient removal.

In this chapter, a gas-lift algal photo membrane bireactor (APMBR) has been developed as a new tool for algal growth/harvesting and polishing treatment of reusable wastewater. Details about the reactor configuration, algal flora and further uses of algal biomass and reactor effluent will be discussed. Performance of flow through extended operation is presented, while identifying mayor limitation of this treatment application.

6.2   Materials and Methods

The green algae *Chlorella sorokiniana* (Cs) (UTEX 2805), obtained from the Culture Collection of Alga at the University of Texas (Austin, Texas), was used in these experiments for its rapid adaptability to harsh environments such as sewage (Ogbonna et al., 2000; de Bashan et al., 2002; de Bashan et al., 2008; Muñoz 2006). A pure culture of Cs was acclimated in batch configuration (500 ml bottles) to sterilized effluent of the Gl-AnMBR during a period of 1 month (Figure 6.1). Light and temperature conditions were maintained at 12 W/m^2 and 25°C respectively. The bottles were exposed to atmospheric CO2 at all times without additional enriched gas was provided. After reaching medium saturation (i.e. plateau of exponential growth), the acclimated culture was used for seeding the APMBR. Characterization of the Gl-AnMBR permeate experiments are summarized in Table 5.5.
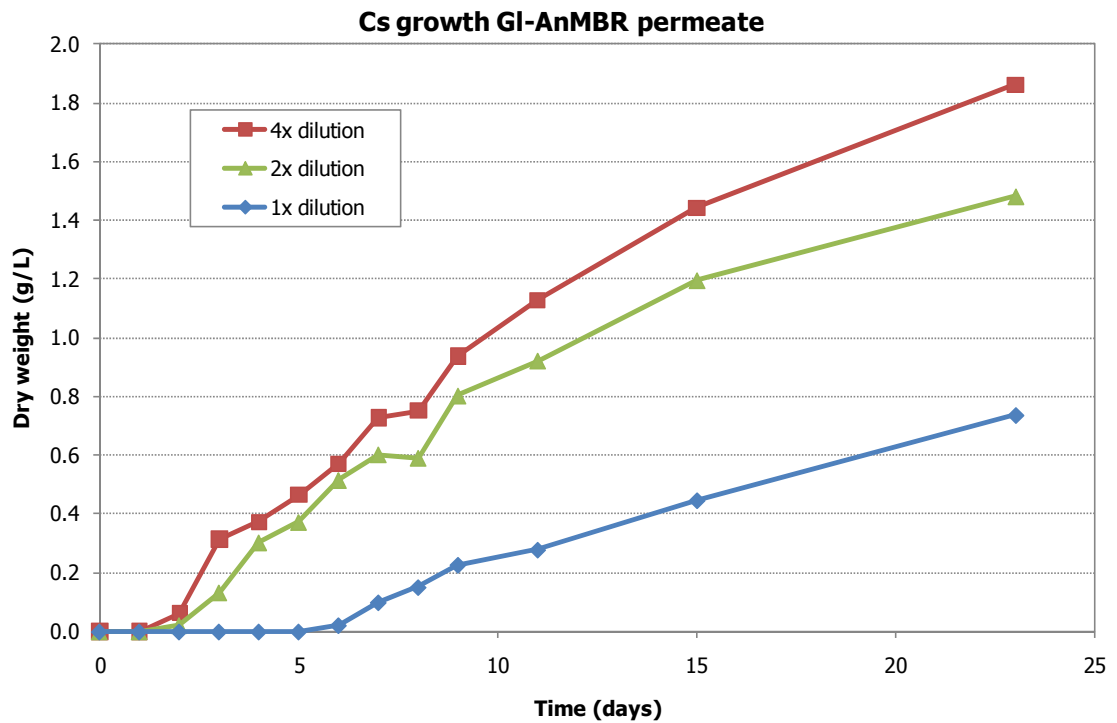
Figure 6.1: Batch acclimation of *Chlorella sorokiniana* in diluted concentration of Gl-AnMBR effluent

The experimental set-up shown in Figure 6.2 consists of a 1.8 L photoreactor column, coupled with a sidestream gas-lift ultrafiltration module. Filtration is mainly driven by applying vacuum to the membrane permeate side with a double head variable speed pump (Masterflex L/S, Cole Palmer, Vernon Hills, IL). This pump also controls the incoming flow rate to the reactor (Qin). Membrane cross flow velocity CFV is controlled by the gas lift resulting from pumping atmospheric air at the bottom of the membrane module. Air is compressed using a peristaltic pump (Masterflex 7520-25, Cole Palmer, Vernon Hills, IL) and applied for membrane scrubbing. The air flow rate (Qa) was controlled visually with a gas flow meter and a needle valve. Membrane effluent was measured with an on-line rain gauge (Model WS-9004U-IT, La Crosse Technology, La Crosse, MA) and transmembrane pressure (TMP) was measured by placing on-line

pressure transducers at the feed (Pf), permeate (Pp) and recycle (Pr) lines of the membrane module (Model EW-68075-32, Cole Palmer, Vernon Hills, IL). The two phase (gas/liquid) retentate is recycled back to the top of the column, providing continuous mixing to the algal suspension. Temperature at the bioreactor was maintained at room temperature and continuously monitored at the membrane feed line using an on-line sensor (Model S-TMB-M002, Onset Computer Corporation, Bourne, MA).
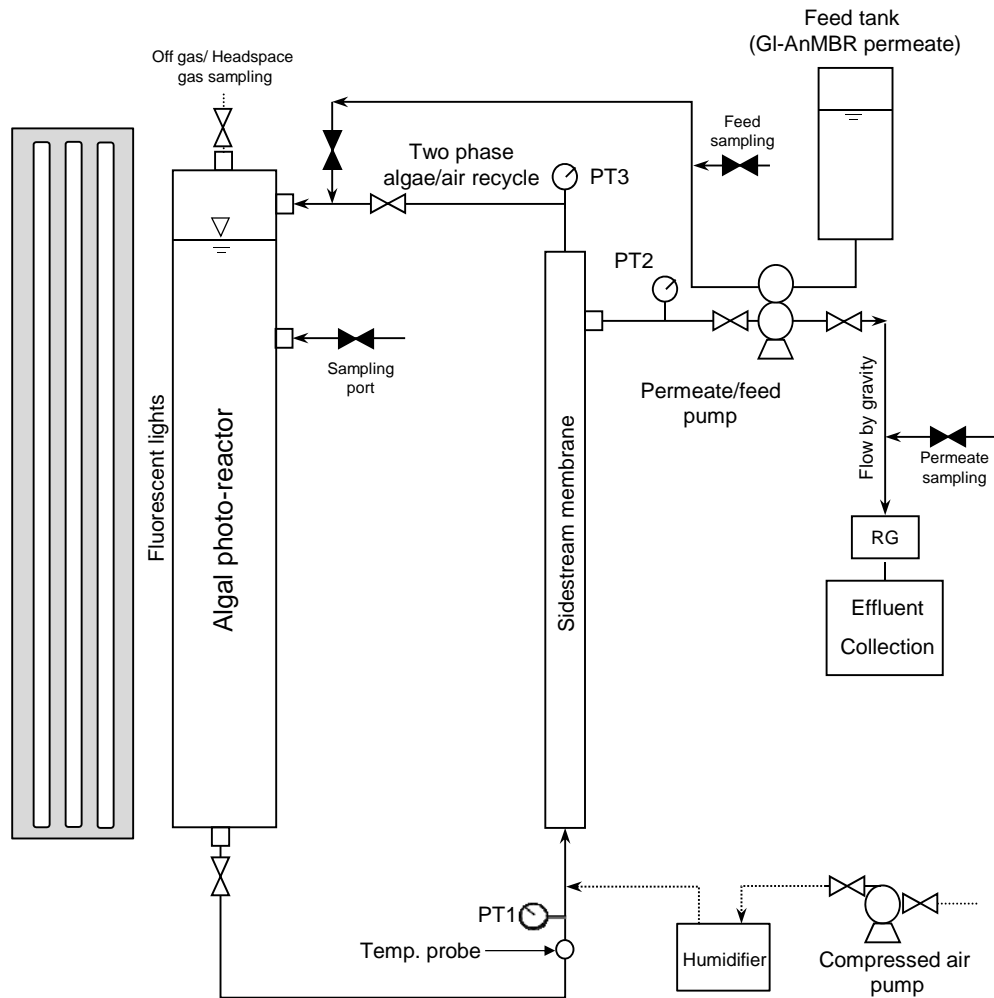

Figure 6.2: Schematic of the gas-lift APMBR

6.3   Algal Photo-MBR Performance

Several considerations were taken into account to define the operational parameters during the startup of the algal photo-MBR:

Typical *HRT* values for wastewater treatment are located between 6 to 24 hours depending on the type of stream to be treated. In the case of algal bioreactors, the HRT not only depends on nutrient concentrations, but on levels of dissolved oxygen which have been reported to be toxic at values of 29 mg OD/L (Munoz and Guieysse, 2006). Several studies have shown that shorter SRTs (i.e. less than 2 days) allow to maintain these values under dangerous/toxic levels for algae and, moreover, under favorable DO concentration for exogenous bacteria (Munoz and Guieysse, 2006). For this configuration, a HRT of 1 day was selected as starting point.

For a biofuel-algal photoreactor, *SRT* is directly related to harvesting events. While maintaining exponential growth and a considerably high algal biomass concentration within the reactor, harvesting should occur before light penetration and self-shading become limiting factors for growth (Figure 6.3). However, it was observed that the algae tend to attach to surfaces of lower turbulence within the reactor while remaining at exponential growth. Additionally, growing biomass stars to agglomerate and settles to the bottom of the column. In this case, light penetration becomes less of a concern since the actual concentration of suspended biomass is about 75% lower than the total algal biomass. On the other hand, higher removal of nutrients is accounted when having larger concentrations of algal biomass. For this specific configuration, harvesting was provided after reaching a biomass density of 0.5 kg/m^3 (OD larger than 0.9), which translated in an SRT of 15 days.
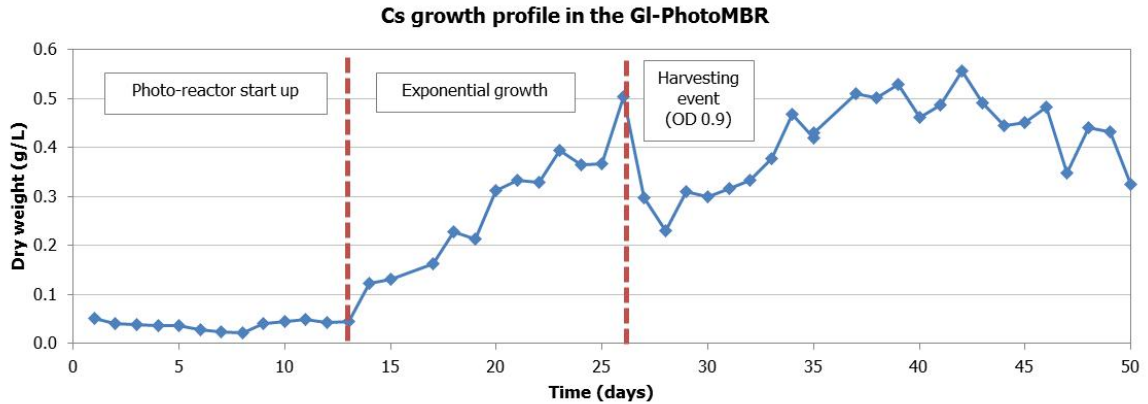
Figure 6.3: Algal biomass growth in the Gl-Photo MBR

Since one pump controls the flow rate incoming and leaving the reactor, the operational flux of the MBR was initially set at constant 4.5 LMH; allowing an HRT of 1 day. Inlet pressure was provided by the hydrostatic head of the photoreactor and maintained constant around $0.1\pm0.9\%$ Bar. Shear and recirculation of membrane concentrate was controlled using air-lift, at a rate of 0.1 L/min.

6.3.1 Biomass Retention and Algal Growth

Exponential growth was reached after a short acclimation period of 2 weeks. Complete retention of the algal biomass was assessed without significantly affecting membrane performance. Algal biomass concentration reached a maximum of 0.5 g/L after leaving the reactor to run without harvesting. This maximum also determined a short period of stable biomass density (plateau) and subsequent decrement in suspended algal biomass (Figure 6.4). At this point, severe aggregation of algal cells and attachment to photo reactor walls were the dominant limitations to reactor operation. On the other hand, biomass density under flow through operation did not reach

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concentrations superior to those in batch configuration. Expected biomass concentrations were about 50% larger than those obtained in continuous operation, which might be the response of algae to the drastic change in growth conditions when in the photo reactor.

In terms of membrane operation, even after reaching higher algal biomass density (i.e. VSS higher than 500 mg/L), the TMP at the membrane module remained constant at average value of 0.06 Bar. This value for pressure differential is extremely low compared to dewatering applications of membrane filtration in literature (Zhang et al., 2010; Danquah et al., 2008; Danquah et al., 2009), as well as those reported for low pressure/low energy MBR systems in wastewater treatment. TMP values of 0.1 Bars have been reported for higher solids concentrations in activated sludge treatment coupled with air-lift filtration (Futselaar et al., 2008), leaving some room for increasing concentrations of algal biomass before low pressure filtration is affected. Although close monitoring of TMP is necessary to assess membrane fouling, shear provided by the air bubbles in the two-phase flow have satisfied the need for additional antifouling mechanisms. For this type of configuration, the CFV will be controlled by the provided air flow rate (Q_a) which did not show any advantage for filtration if increased (results not presented). On the contrary, higher Q_a tends to increase TMP and the air in the two-phase flow competes with the liquid to exit the membrane. A summary of the preliminary operation of the APMBR are presented in Figure 6.5.

Cs growth profile in Gl-PhotoMBR
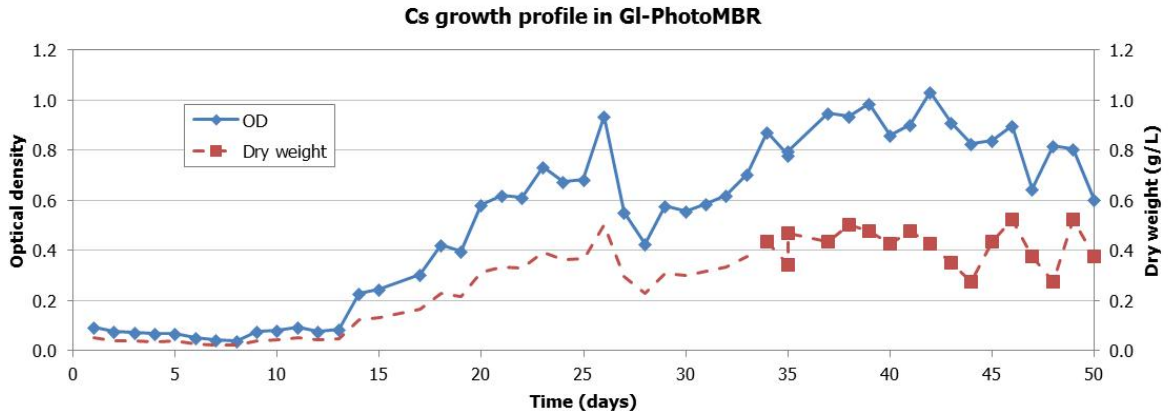
Figure 6.4: Cs growth profile during extended operation of the Gl-APMBR. The dashed line to the left corresponds to back-extrapolated values of dry weight based on actual data (square dots).

Algal photo-mbr performance
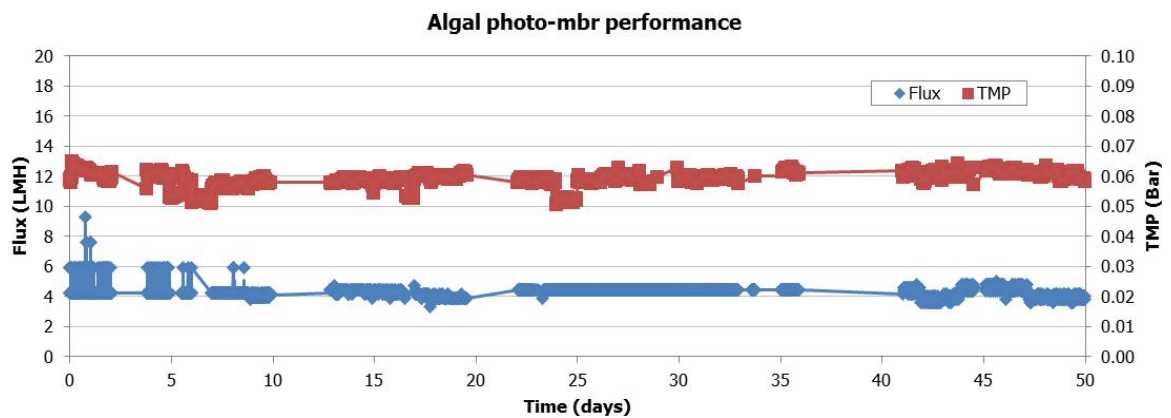
Figure 6.5: Extended performance of the Gl-PhotoMBR

Dissolved oxygen within the reactor did not exceed the 4.1 mg DO/L during the exponential growth or after harvesting events. This value is found in literature to be below toxic levels for algae cultures and was observed not to affect algal growth. In the case of pH, the reactor was maintained below 8.5 by acidifying the feed to a neutral value with 1 N hydrochloric acid. Acidification was necessary since the reactor feed reached basic pH values while stored exposed to atmospheric air for periods longer than 6 hours.

6.3.2 Nitrogen, Phosphorous and Carbon Removal

Removals of nutrients depend on biomass density. A TN removal efficiency of 30% was observed when reaching VSS concentrations larger than 0.5 g/L. Although removal of TN seems not to be as remarkable as those obtained in other batch studies (Wang et al., 2010a and 2010b), up to 100% removal of specific nitrogen species such NH4 were obtained after maximum biomass concentration was achieved within the reactor (Figure 6.6). A sudden drop in nitrogen TN concentration results from the change in the feed. Permeate from the GI-AnMBR is stored and sterilized to avoid contamination in the reactor. TN for the 30 to 50 corresponds to a new batch of GI-AnMBR permeate after reached stable performance. Nitrate on the other hand, was observed not be utilized by the algae which elucidates the preference of Cs to ammonia as an N source for metabolic purposes. A more detailed characterization of the nitrogen uptake to the particulate fraction (biomass) should be done to assess the different pathways of the N within this reactor configuration, especially since previous studies have reported proteins as one of the mayor components of algal EOM contributing to membrane biofouling.

A similar behavior was observed for phosphate removal. After reaching maximum biomass concentration, up to 100% reduction of phosphate was observed (Figure 6.7). With improved nutrient removal efficiencies, a minor concern arises since nutrient availability might become a limiting factor for algal growth. To corroborate this statement, a comparison between the cumulative APMBR removal of ammonia and phosphate and the stoichiometric nutrient demand for algal growth is presented in Figures 6.8 and 6.9. Since the algal biomass nutrient requirements correspond to a small fraction of the NH_3 and PO_4 removed from the APMBR, nutrient limitation is not a

concern for this configuration and instead, nutrient removal can perhaps be attributed to NH_3 stripping and/or salt precipitation/filtration due to the high pH values (i.e. 8.5 to 9) observed during this study. The specific nutrient removal mechanisms of the APMBR are yet to be established in future studies.

At an HRT of 1 day, complete removal of ammonia and phosphate could be assessed by the APMBR. Reduction of HRT might be possible for this configuration, consequently reactor's permeate production (i.e. higher flux) can be increased as well. Further studies are necessary to optimize HRT with this configuration.
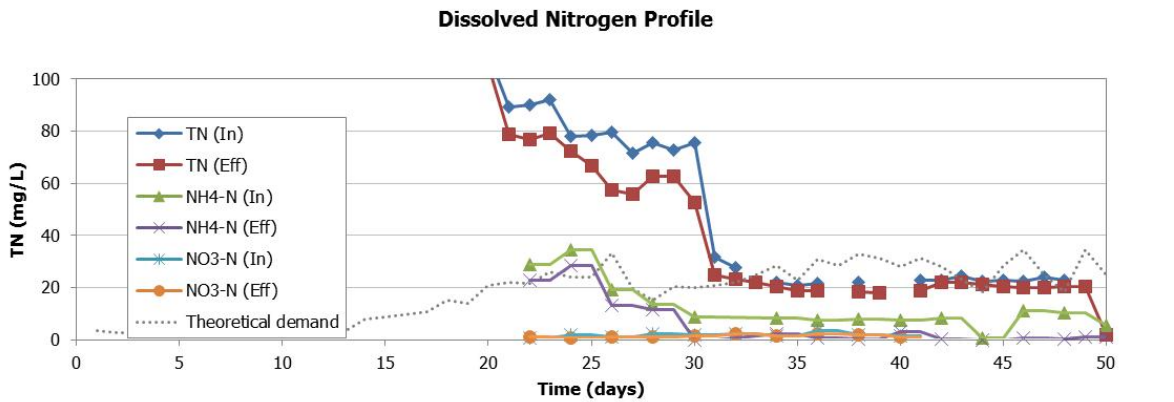
Figure 6.6: Soluble nitrogen profiles for the Gl-Photo MBR during extended operation. Sudden drop in TN concentration can be observed around day 30 after feed batch changed.
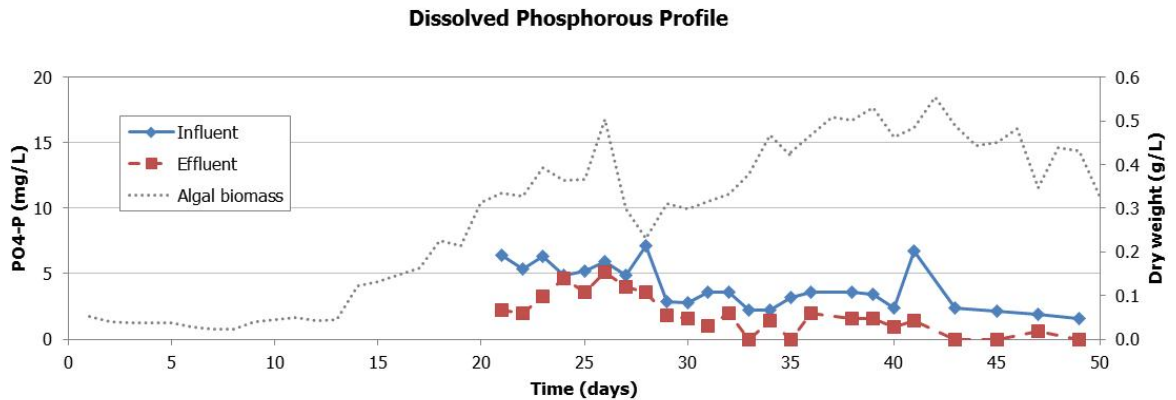
Figure 6.7: Soluble phosphorous profiles for the Gl-Photo MBR during extended operation. Sudden drop in TN concentration can be observed around day 30 after feed batch changed.

Removal vs. Uptake NH₃
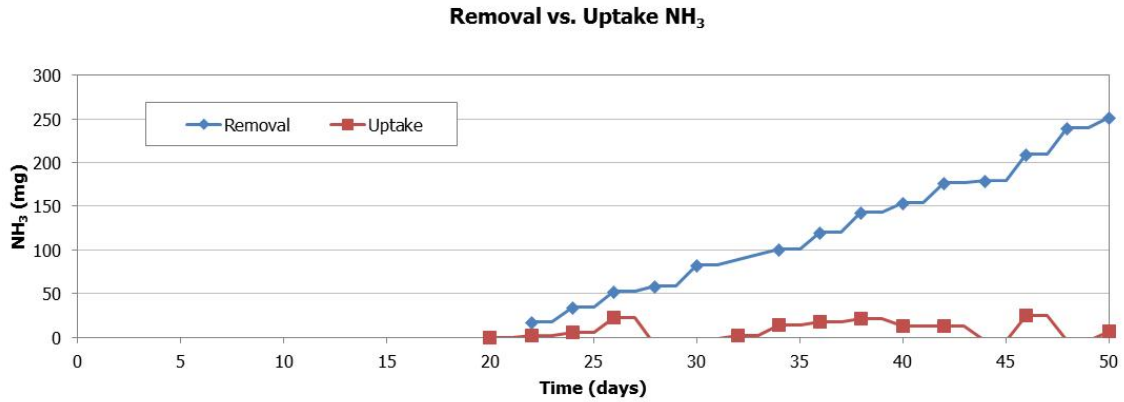
Figure 6.8: Comparison between cumulative ammonia removal of the Gl-Photo MBR and cumulative ammonia uptake for algal growth.

Removal vs. Uptake PO₄
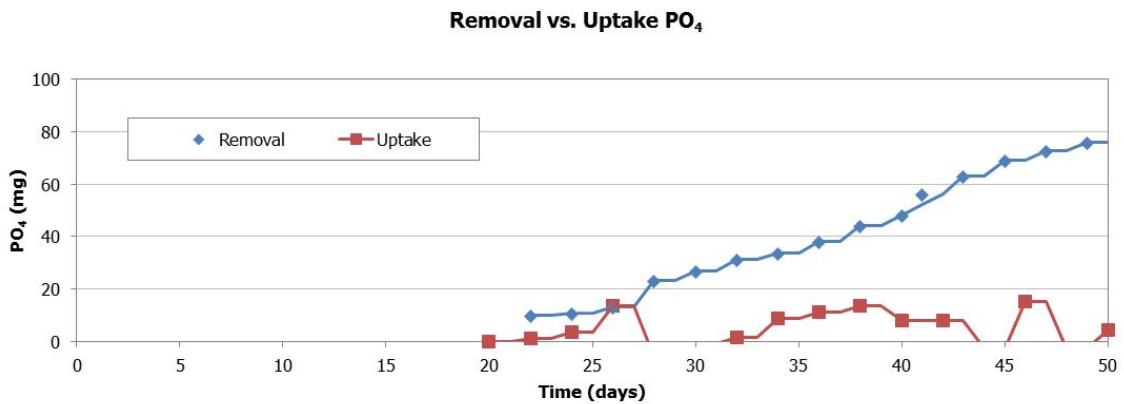
Figure 6.9: Comparison between cumulative phosphate removal of the Gl-Photo MBR and cumulative phosphate uptake for algal growth.

In the case of carbon utilization, COD and TOC were closely monitored mainly to assess dominant metabolic preferences (heterotrophic and/or autotrophic). During the first exponential growth phase, removal of organic carbon was not observed, but up to 50% removal COD was obtained. Results for TOC and COD profiles are presented in Figure 6.8. After day 30, only COD was monitored for carbon removal. Since membrane filtration has been uninterrupted and no additional antifouling methods have been used, removal of carbon might also be caused by membrane biofouling. In a worst case

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scenario, contamination of the algal culture might have cause removals of COD, however this statement is less probable since the algal photo reactor was closed at all times and measured DO and pH conditions were not favorable for bacterial proliferation. Furthermore, additional sources of carbon may come from algal lipids and EOMs due to the continuous changes in feed characteristics. In this study, the specific source of carbon preferred by the algal culture and specific COD and TC removal mechanisms taking place within the reactor were not conclusive. Further research has to be done to confirm the previous statements. Effect of variable alkalinity on algal growth should be investigated since this condition is intrinsic to anaerobic treatment. Lipid content is another parameter that should be assessed in future studies, especially if algal biomass is used for digestion experiments.

Dissolved Organic Carbon Profile

Figure 6.10: Soluble COD and carbon profiles. TOC and TC measurements are presented before and after day 30 respectively.
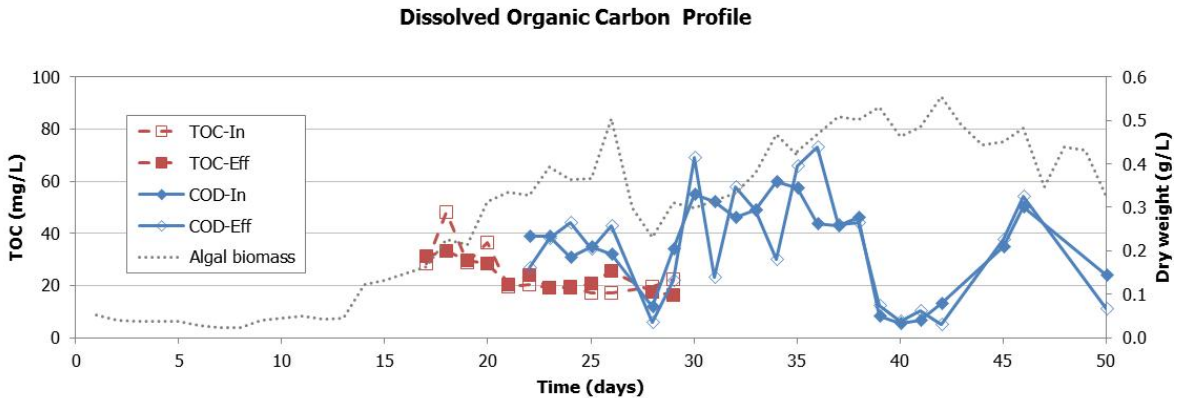
6.4 Conclusions

Limited information is available regarding in biofuel algae photo-MBRs treating anaerobic effluents, which makes of this study an important step in the feasibility of the gas-lift APMBR as an advanced wastewater treatment tool. Nutrient rich effluent from

the Gl-AnMRB treating domestic wastewater has been used for these experiments and the following conclusions are established:

- Although successful growth of the biofuel producing algae *Chlorella sorokiniana* was achieved in the continuous-flow photobioreactor, operational conditions are not yet optimized to yield biomass concentrations at similar or superior concentrations than those obtained in batch experiments.

- At the operational conditions tested in this study (HRT 24 hours, operational flux of 4.5 LMH, air-lift flow rate (Q_a) of 0.1 L/min and 0.1 Bars of membrane inlet pressure), complete removal of ammonia and phosphate was achieved by the APMBR. In this case, further concerns regarding nutrient limitation for algal growth can be easily targeted by decreasing the HRT, hence improving APMBR permeate production and overall efficiency in providing tertiary treatment to Gl-AnMBR effluent.

- The carbon removal mechanisms assessed by the gas-lift APMBR require further studies since removal of TOC, TC and COD were not conclusive in determining the dominant metabolic preferences of algae used (heterotrophic, autotrophic or mixotrophic growth). Besides algal carbon utilization, other biological and physical processes taking place in this reactor (membrane biofouling, EOM formation and air scrubbing) can contribute to the sources and/or sinks of carbon in this set up.

- Nutrient removal mechanisms in this configuration require further characterization specifically due to the influence of pH in the chemistry of NH_3 and PO_4. Removal of ammonia due to the intense scrubbing characteristic of this

configuration needs to be identified as the baseline for future biological removal assessments in the APMBR.

- Variability of reactor feed challenges the resiliency of algae to sustain growth while continuously adapting to variable growth conditions. Although successfully achieved, variable conditions also affect the characteristics of algal products such as lipid content, EOM and removal efficiencies.

- Light penetration did not represent a limiting factor during this study, mainly because algae naturally aggregate after reaching a critical biomass concentration. Continuous harvesting needs to be applied to maintain adequate biomass suspension during extended operation should be evaluated. Other factors such as higher gas-lift flow rate should be evaluated to improve biomass suspension.

7 CONCLUSIONS AND RECOMMENDATIONS

In the past two decades, efforts to recover intrinsic resources in sewage has steadily grown in the wastewater treatment industry. Biosolids are land applied and reclaimed water is piped throughout many municipalities. Methane recovery for energy production is a common practice at anaerobic digestion facilities throughout the developed world. However, most "recovery" efforts result from convenient byproducts of the removal process, and are not the focus of technology development. With rising energy costs, depletion of mineral reserves, increasing fertilizer costs, and increasing population stress on resources, alternative wastewater treatment technologies have to evolve to cope with resources depletion. Focused efforts to recover renewable resources such as energy, nitrogen, phosphorus, and clean water from sewage are now becoming the basis of new technology innovation. Perhaps society is on the brink of a paradigm shift where recovery of resources from wastewater is not only sustainable but also makes good business sense.

In this work, new wastewater treatment technologies were developed and evaluated for their potential to recover valuable resources from sewage such as water, energy and fertilizers. First, COPAS (complex organic particulate artificial sewage) was identified as a surrogate sewage organic material and characterized in its capability to mimic domestic wastewater in both particulate organic matter content and environmentally relevant parameters such as BOD, COD, OC and TN. Secondly, an

advance wastewater treatment technology based on low pressure membrane filtration and anaerobic bioprocess was used as a tool to recover embedded nutrients and energy from sewage. The gas lift anaerobic MBR (Gl-AnMBR) coverts N and P in sewage to mineralized soluble forms of nutrients (i.e. ammonium and phosphate), providing a clear, nutrient-rich effluent for direct reuse in applications as fertilizer. Additionally, anaerobic digestion of COPAS produces methane, which can be converted to different forms of energy (directly combusted, electricity and/or heat energy conversion). The energy footprint estimation from the lab-scale configuration ratified this system as a sustainable low energy treatment MBR technology, although performance at plant scale is necessary to establish the Gl-AnMBR's competitively among low energy aerobic counterparts.

Finally, the resource recovery cycle was closed by introducing a reuse application to the Gl-AnMBR effluent with a gas-lift algal photo membrane bioreactor (APMBR). Biofuel-producing microalgae utilized carbon-dioxide and AnMBR nutrients for biomass growth, which can be further converted to biofuels. Similar to the Gl-AnMBR, the principle of gas-lift was applied to the APMBR to decrease energy consumption associated with membrane filtration while providing continuous recycle of algal liquor for mixing and even a continuous source of atmospheric CO_2 for algae growth. As the objective of the present study was to demonstrate proof of concept for gas-lift APMBR, the dynamics taking place in this bioreactor (e.g., potential generation of algal metabolites) were not fully characterized and require further studies. Since limited information is available in the literature regarding biofuel-producing algal photobioreactors utilizing anaerobic process effluents, the present study provides an important contribution towards better understanding of the design and performance of

combined anaerobic/algal biotechnology for full-scale application of sewage resources recovery.

The sequential two phase treatment process, the anaerobic/algal MBR (A2MBR), is a promising treatment technology for closing the Water-Energy-Nutrient (WEN) cycle. The evolution of sewage through the A2MBR is graphically depicted and summarized in Figure 7.1.

Figure 7.1: Waste to Water in the A2MBR. From left to right: COPAS synthetic sewage, Gl-AnMBR sludge, Gl-AnMBR permeate, Gas lift APMBR mixed liquor and APMBR permeate and final product.

Table 7.1: Summary of the water quality changes in different treatment stages of the A2MBR. Values reported in mg/L.

Parameter	COPAS		Gl-AnMBR permeate	Gas-lift APMBR permeate
	Total	Soluble	Soluble	Soluble
CODt	1267±6.4			
CODs		257.3±195.5	70±22.1	33.24±21.9
TOC	528.5±2.7*	73.29±44.29	25.44±4.5	N/A
TKN	54.3±0.3	21.7±9.5	75.43±9.7	20±1.9
NH4-N		5.7±3.3	9.1±4.2	Non detected
NO3-N		1.8±0.7	2.2±0.9	1.8±0.5
PO4-P		15.5±7.8	20±7.13	Non detected
NTU	447±8.4		6.9±2.3	1.34±0.3

* Calculated from OC/wt ratio based on 1000 mg/L COPAS added

7.1 Intellectual Merit and Broader Impacts of Present Research

This investigation helped to expand the current knowledge regarding low strength wastewater treatment using AnMBR technology. Because it is an unexplored technology, Gl-AnMBR represents a novel alternative for sewage treatment, especially if its energy footprint is optimized for maximum resource recovery. Findings regarding optimization of energy balance within the GL-AnMBR served as a model to improve performance of similar configurations. Additionally, this study demonstrated the feasibility of a Gl-AnMBR to renovate low strength sewage and recover water, energy and nutrients. Extension of the nutrient and carbon cycle through algal growth in the proof-of-concept gas-lift APMBR demonstrated the paradigm shift of viewing wastewater as a matrix of valuable resources rather than a disposable commodity. The coupling of these two advanced processes in the A2MBR system established a novel approach to closing the WEN cycle, as depicted in Figure 7.2.

The innovative technology developed in this investigation could have many potential applications in different fields related to freshwater and natural environments. This technology could be applied to a variety of communities that consider decentralized wastewater treatment as a feasible way of recovering and reusing valuable resources. The performance of the GI-AnMBR in other scenarios such as remote communities (e.g. countryside populations, touristic resorts and suburban neighborhoods) and low income localities (i.e. developing regions) should be target of further studies. Extension of this research foresees application of the resulting reactor as a low-cost solution for the water and sanitation problem in developing countries. Contribution to Goal 7 of the UN's Millennium Development Goals regarding natural resources conservation and basic sanitation for less privileged communities around the world is an additional contribution of this study.
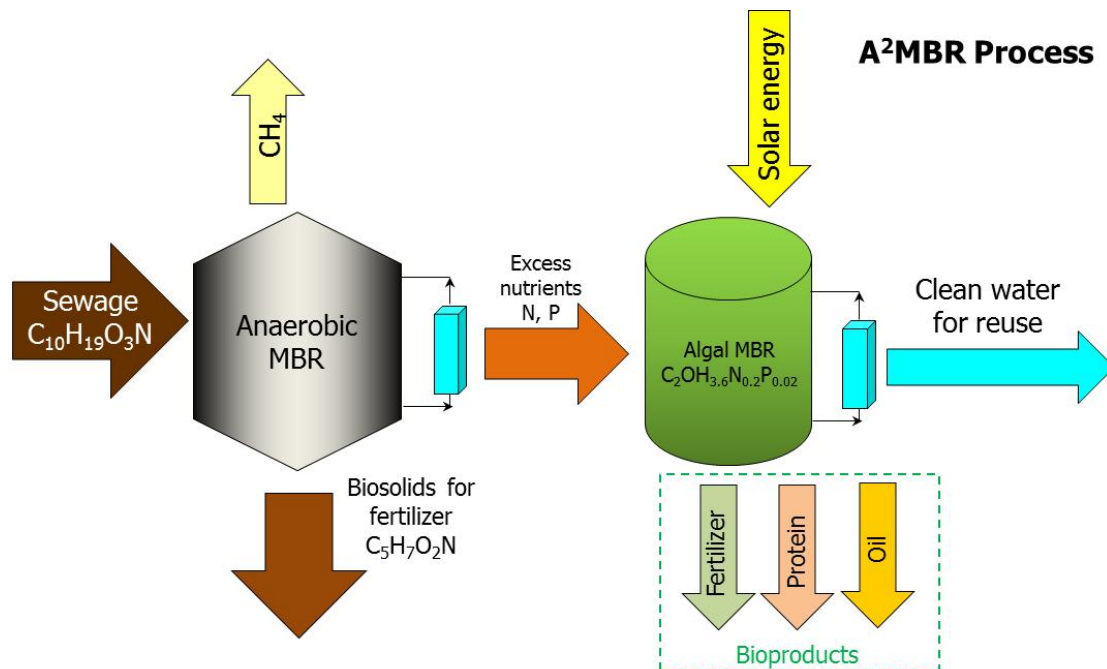
Figure 7.2: Flow of material in the sequential A2MBR

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APPENDICES

Appendix A. Gas-Lift Anaerobic Membrane Bioreactor (Gl-AnMBR): Filterability Of Anaerobic Mixed Liquor With High Content Of Oily Compounds

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A.1   Introduction

During this study, a gas lift anaerobic membrane bioreactor (GL-AnMBR) was constructed at UNESCO-IHE. Its performance in the filtration of flocculent anaerobic sludge with high concentration of oily compounds was evaluated. Although air-lift aided filtration have been widely used to decrease membrane fouling and increase permeate production, its application in anaerobic conditions is still under development and more research is required to get this MBR configuration to a commercial stage. Its performance under different wastewater scenarios is also under evaluation. With this premise, treatment of food processing effluents is one of the major applications for anaerobic technologies. Specifically, highly oily influents are often used since they favor the biogas production in anaerobic set ups. Problems regarding sludge settleability and inhibition of anaerobic processes due to highly oily streams have been highlighted as drawbacks for this type of biological treatment. Coupling a membrane separation unit to an anaerobic bioreactor could allow longer hydraulic retention times (HRT) that allow

Appendix A (Continued)

adequate digestibility of oily compounds. Filterability of highly oily compounds is evaluated in the short and long run.

A.2   Methodology

A GL-AnMBR was built at UNESCO-IHE (Figure 1). This set up was fed with flocculent anaerobic digestion sludge from a local wastewater treatment plant in Delft (The Netherlands). The sludge had a total solids concentration of around 32 g/L and MLSS concentrations of 7.3, 17 and 29.4 g/L were used to evaluate the effect of solids content on filtration. The reactor was operated under room temperature (23°C to 25°C) at neutral pH (6.5 to 7.5). During the preliminary operational stage of the MBR, the following conditions were established for its continuous operation:

- Cross flow velocity (CFV) = 0.5 m/s

- Fraction of gas in the two-phase flow (E) = 0.1

- Permeate flow rate (Qp) = 2 L/hr

- Backwash frequency = determined by increment in total resistance or start of new experiment

Once the reactor was operating at stable conditions (constant flux and TMP), oleic acid in the form of sodium oleate was added to mimic the content of oily compounds in wastewater. Oleic acid is one of the main long chain fatty acid (LCFAs) present in wastewater (both municipal and food processing), and concentrations of 100, 300 and 600 mg/L were selected to be tested for filterability purposes.
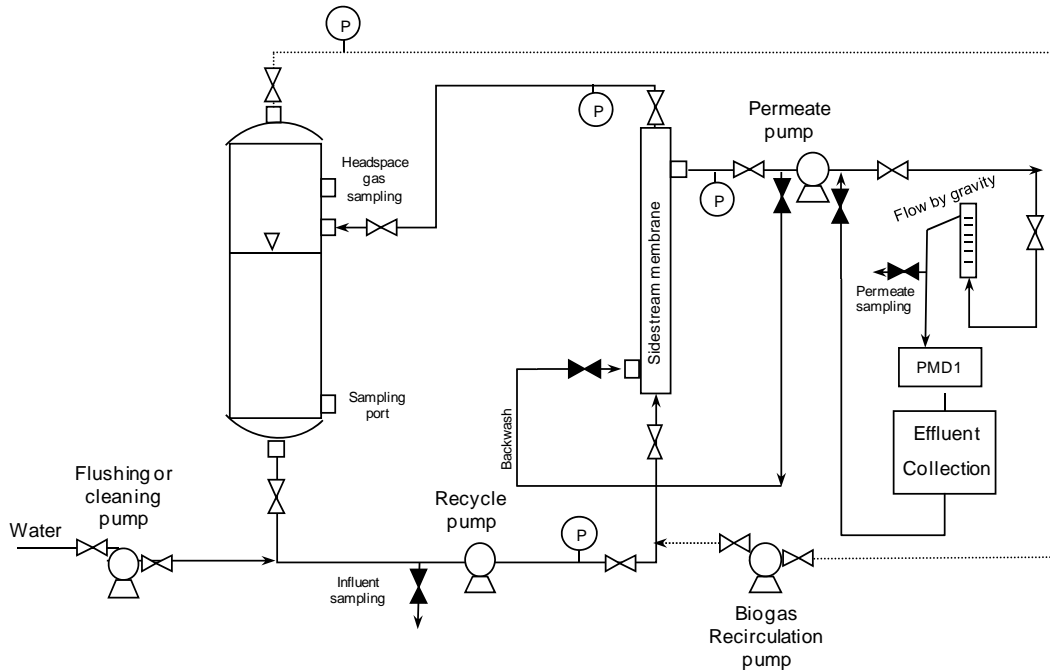
Appendix A (Continued)

Figure A.1: Schematic of GI-AnMBR configuration for this study

A.3   Results and Conclusions

A continuous flux of 20 LMH could be maintained for MLSS of around 17 g/L at CFV of 0.52 m/s. This CFV was selected since it was minimum maximum value of CFV that allows minimum TMP at a maximum flux of 20 LMH. Fluxes larger than 20 LMH were not attainable during the length of the operation. Variation in the gas flow rate for larger values of ε did not favor significantly filterability of sludge. TMP for this type of configuration was maintained at 1 Bar. This value could not be decreased at the set operational condition without sacrificing the permeate production. A minimum TMP of 0.7 Bar was obtained by decreasing permeate flow rate, therefore decreasing flux. Additionally, a gas fraction for the two phase flow of 0.1 was sufficient for the MBR operation.

Appendix A (Continued)

This result suggests that the gas present in the gas-liquid mixture is only scrubbing the membrane as it rises and not lifting the fluid according to the gas-lift concept. In this case, the performance of the GL-AnMBR in the long run has to be evaluated to assess the effectiveness of the "gas-lift" to improve membrane operation and decrease irreversible fouling. Future work includes testing of filterability of higher concentrations of oleic acid since no change was observed when filtering anaerobic sludge with an oleic acid concentration of 100 mg/L (Figure A2).

Results regarding filtration of different concentrations of MLSS are not conclusive yet. However, preliminary testing suggests that at the set operational conditions the permeate production was significantly affected by sludge solids concentration (figure A3). Further studies should be done to corroborate these statements.
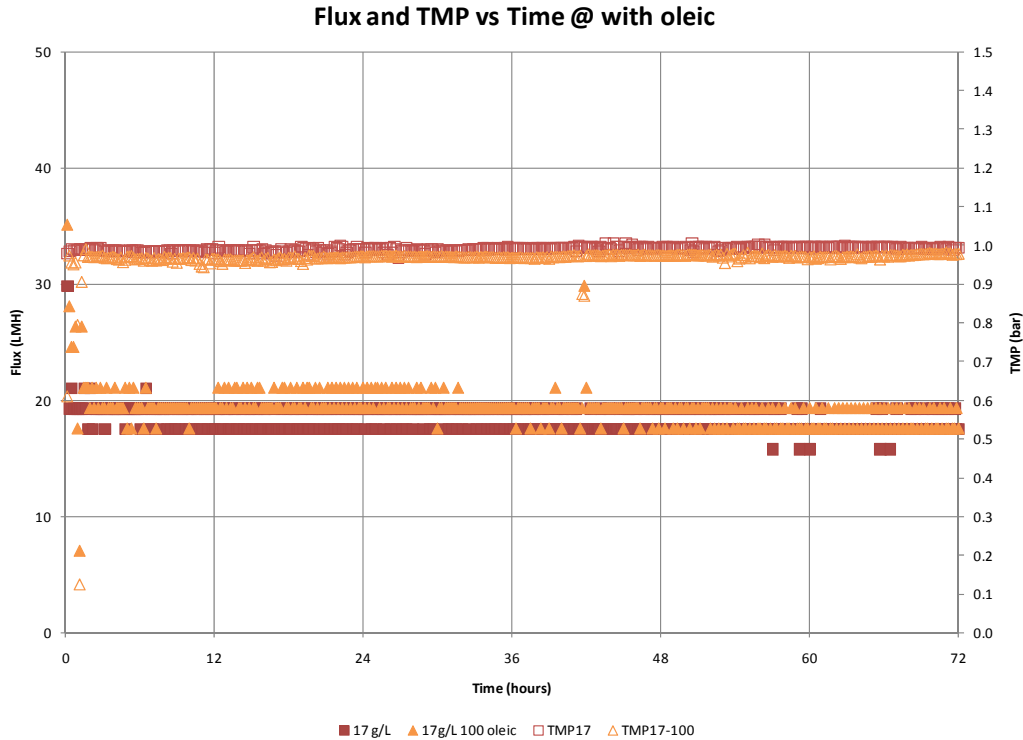

Figure A.2: Filtration of anaerobic sludge. Comparison between sludge with and without oleic acid
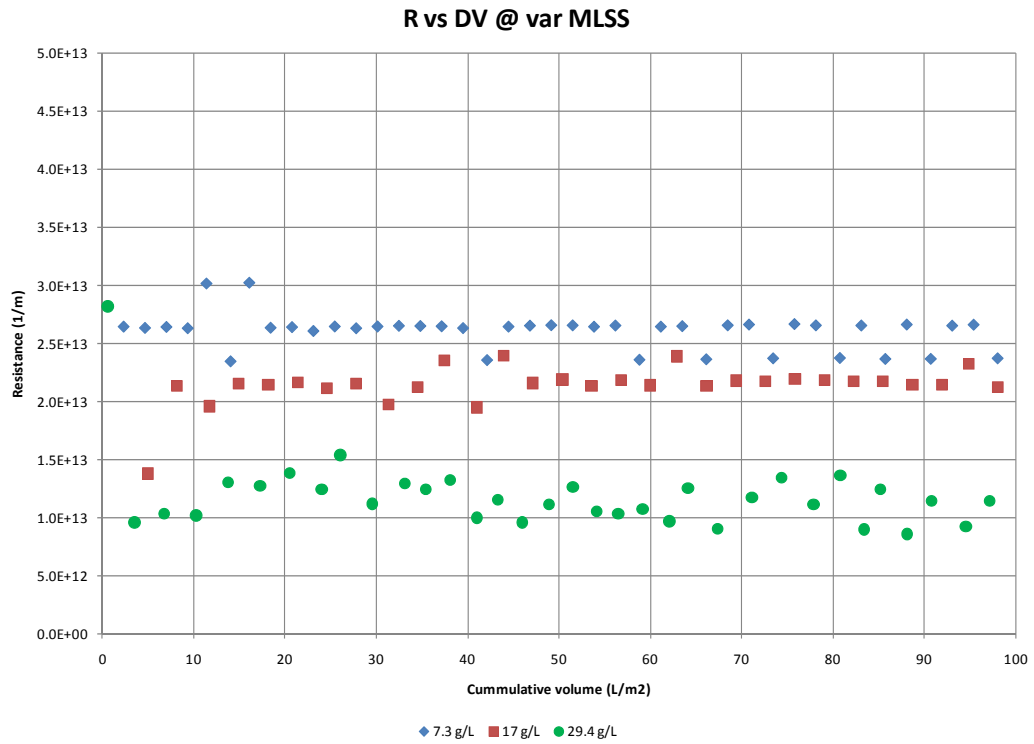
Appendix A (Continued)

Figure A.3: Membrane resistance development per volume of permeate produced for different MLSS

concentrations