Development of a Compact, Energy-positive Food Waste Treatment Process

By

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Author’s declaration

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners.

I understand that my thesis may be made electronically available to the public.

Abstract

This study developed an innovative bioprocess for food waste (FW) treatment by combining a leach bed reactor (LBR) with an anaerobic membrane bioreactor (AnMBR). The two bioreactors were consistently operated at neutral pH and room temperature. Best performance was observed in the LBR run at the conditions of inoculum to substrate ratio 10% and leachate circulation rate 4.4 L/h without any clogging issues in the LBR. In this operating condition, removal of volatile solids (VS) of 88±2%, H_2 production of 3.45 L/ kg VS_{added} and volatile fatty acid yield of 571 g chemical oxygen demand (COD)/kg VS_{added} were observed only in a reaction time of 14 d. Part of FW remained in the LBR, accounting for 13-20% of initial FW. LBR leachate (i.e., effluent from the LBR) was further stabilized in the AnMBR, and hydraulic retention time (HRT) related to membrane flux and solid retention time (SRT) were optimized in the AnMBR. Only in 13 d HRT and 75 d SRT, the AnMBR achieved 85% chemical oxygen demand or COD (an indicator of the amount of organic matters present) removal and complete solid reduction due to membrane separation, along with the specific methane yield of 0.3 L/g $COD_{removed}$. Under this condition, membrane flux was approximately 6 L/m^2-h (LMH) and maintenance cleaning once every five days was required to maintain the flux. Energy balance showed that the combined FW bioprocess is energy-positive with net energy benefit up to 841 kWh/ton $FW_{treated}$. The study proved that the newly developed FW process could achieve 79% VS removal of FW only in 20 d of overall reaction time in energy-positive manners.

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Dedication

To my parents and my lovely wife who supported me throughout the journey.

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List of abbreviations

FW	Food waste
AD	Anaerobic digestion
CSTR	Continuous stirred-tank reactor
LBR	Leach bed reactor
AnMBR	Anaerobic membrane bioreactor
HRT	Hydraulic retention time
SRT	Solid retention time
ISR	Inoculum to substrate ratio
OLR	Organic loading rate
COD	Chemical oxygen demand
TS	Total solids
TSS	Total suspended solids
VS	Volatile solids
VSS	Volatile suspended solids
VFA	Volatile fatty acid
TVFA	Total volatile fatty acid
AR	Acidogenic reactor
MR	Methanogenic reactor
EDTA	Ethylene-Diamine-Tetra-Acetic acid
TCOD	Total chemical oxygen demand
SCOD	Soluble chemical oxygen demand
TKN	Total kjendhal nitrogen
TP	Total phosphate
RP	Reactive phosphate
TMP	Transmembrane pressure

Chapter 1 : Introduction

1.1. Motivation

Food waste (FW) has become a severe economic and environmental issue that has gained tremendous attention in recent years. FW represents a significant proportion of organic material generated primarily by the residential, and industrial, commercial and institutional sectors (Li, Peng, Wang, & Wu, 2018). It can be originated from pre-consumers, such as distribution and retail agents, or post-consumers i.e. residential and commercial kitchens (Li et al., 2018; Ren et al., 2018). Improper management, rapid growth of population and economy found to be the main reasons for the increase in FW every year (Li et al. 2018; Ren et al., 2018; Braguglia, Gallipoli, Gianico, & Pagliaccia, 2018).

Currently Canada holds the second place in per capita FW generation in the world, just after USA (Worldometers, 2019; CBC, 2018). The economic value of FW is estimated at around $31 billion each year (CBC, 2018; Ontario Ministry of the Environment and Climate Change, 2017). Moreover, improper treatment and disposal of FW are also causing serious environmental hazards. Due to high moisture content and biodegradability, inappropriate disposal of FW leads to generation of leachate, odour and green-house gas like methane during collection, transportation and storage which can significantly pollute air, soil and water (Han & Shin, 2004; He et al., 2012; Zhou et al., 2018; Ahmed & Sulaiman, 2001; Krcmar et al., 2018; Vadillo, Andreo, & Carrasco, 2005).

Anaerobic digestion (AD) is the most common biotechnology due to its high treatability and energy recovery capability, potentially improving the sustainability of FW treatment (Xu et al., 2018; Wainaina et al., 2019). But AD of FW can be inhibited by formation of different toxins, mainly ammonia and medium to long chain fatty acids (Xu et al., 2018). Adopting multiple stages is a way to mitigate inhibitions in AD (Wu et al., 2015; Xu et al., 2012), called two-stage AD. Apart from increased stability at higher organic loading rates, two-stage AD also enables the capture of higher energy-containing hydrogen (141.7 kJ/g) along with methane (55.5 kJ/g) rather than methane alone (Shen et al., 2013; Voelklein et al., 2016; Wu et al., 2015).

A two-stage AD process consists of a hydrolytic-acidogenic reactor and a methanogenic reactor. Although continuously stirred tank reactors (CSTRs) are commonly used for two-stage AD, they are not ideal for high solid organic waste (20-30%), such as FW because of extremely high operating cost for mixing (Li et al., 2017; Xiong et al., 2019); hence, cost-intensive CSTRs are less attractive for FW treatment. Alternatively, leach bed reactors (LBRs) have gained attention in recent times due to its low operation and maintenance cost, treatability of high solid content, and solid-liquid separation capacity (Hussain et al., 2017; Li et al., 2017; Xiong et al., 2019). However, LBRs have mainly employed for fermentation, which means accumulation of simple acids. AD can be integrated with LBRs to further stabilize the simple acids as two-stage AD for FW treatment, but information on two-stage AD using LBRs is limited. Typical CSTR-type AD reactors can be coupled to LBRs, but effluent quality from the AD is poor. Anaerobic membrane bioreactors (AnMBRs) can improve the final effluent quality and methane production over the AD due to membrane separation (Charfi, Ben Amar, & Harmand, 2012; Galib et al., 2016). However, no literature has reported LBRs coupled to AnMBRs for FW treatment to the author's knowledge.

1.2. Scope and objectives

The overall study primarily focused on optimization of several operating parameters in the proposed two-stage FW process at room temperature. This study contributes in three research areas:

(a) Optimization of inoculum to substrate ratio (ISR), leachate recirculation rate and reaction time on fermentation of FW in an LBR.
(b) Optimization of hydraulic retention time (HRT), solid retention time (SRT) and membrane cleaning to maintain membrane flux in treating FW leachate using AnMBR followed by an LBR.
(c) Optimization of the two-stage FW process based on treatment efficiency and energy benefits.

1.3. Thesis outline

This dissertation is divided into five chapters. Chapter 1 discusses on the current challenges and a proposed solution along with the objectives and scopes of this study. Chapter 2 provides an

overview of available literature related to the study. Chapter 3 and 4 present the outcomes of experimental works on bioreactors by evaluating the performance of an LBR as hydrolytic-acidogenic bioreactor and an AnMBR as methanogenic reactor in treating FW at room temperature (22°C); Chapter 4 discusses the optimization of the two-stage FW process. Chapters 3 and 4 are both presented in article format. Chapter 5 summarizes the research results.

Chapter 2 : Background and Literature review

2.1. Food waste (FW) problem in Canada

Management of food waste (FW) has been a growing issue to be addressed all over the world as FW can affect economy and environment significantly (Tonini, Albizzati, & Astrup, 2018). According to the literature, generation of municipal solid waste (MSW) all over the world is currently about 1.3 billion tons per year and is anticipated to rise to 2.2 billion tons by 2025, among which one-third will be FW (Zhou, M., Yan, Wong, & Zhang, 2018; Wainaina et al., 2019).

Shockingly, Canada is the second largest food waste producer per capita in the world, just after the United States of America (CBC, 2018). Every year, each Canadian is producing around 400 kg which is estimated approximately $868 worth of FW (Ontario Ministry of the Environment and Climate Change, 2017; The Globe and Mail, 2018; CBC, 2018). This implies that $31 billion is being wasted (CBC, 2018; Ontario Ministry of the Environment and Climate Change, 2017).

Moreover, improper disposal of FW poses serious threats to health and environment. FW includes uneaten food residues and discarded food causing odor and leachate during the collection, transportation and storage (Han & Shin, 2004; He et al., 2012; Zhou et al., 2018). Common FW treatment includes feeding animals, composting, landfilling and incineration with landfilling being the most common practice of FW disposal in Canada. These FW treatment methods, however, are not considered sustainable solutions. For instance, untreated FW may lead to infection in animals; composting and landfilling need a large amount of valuable land and release green-house gases; and incineration is energy-intensive due to high moisture content of FW and causes air pollution (Zhou et al., 2018). Leachate produced from landfills also creates severe soil and groundwater contamination causing dangers to human health and the environment (Ahmed & Sulaiman, 2001; Krcmar et al., 2018; Vadillo et al., 2005).

Diverting FW from disposal stream to recycle and recovery gives large environmental and economic benefits. Not only FW can be turned into valuable products (e.g., compost and digestate, renewable natural gas and biofuels) but also can boost circular economy and increase employment opportunities. For instance, current efforts by different municipalities in Ontario to divert source separated municipal organic waste supported up to 1,682 direct and indirect jobs and generated

over $100 million in gross domestic product (Ontario Ministry of the Environment and Climate Change, 2017).

Anaerobic digestion (AD) is not only the most well-known biological treatment process among the biotechnologies capable of producing different value-added products, but also an effective diversion process. In the next section, AD of FW will be discussed in detail.

2.2. Fundamentals of anaerobic digestion (AD)

AD is a well-established but complex process involving diverse microorganisms working syntrophically (McCarty & Smith, 1986; Zamanzadeh et al., 2016). A simpler version can also be found in the literature to understand the process (McCarty & Smith, 1986; Parkin & Owen, 1986), as shown in Figure 2.1. The disintegration of organic matters can be divided into four steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis. They are presented in more detail in the following paragraphs.

In the first step, complex organic matters break down into simple monomers for microbial consumption. Carbohydrates, proteins and fats are transformed into simple sugars, amino acids and long-chain fatty acids. This step is conducted by extracellular reactions, called hydrolysis. This is generally the rate-liming step in AD (Hussain et al., 2017; Kim et al., 2014; Lee et al., 2010; Shen et al., 2013; Ventura et al., 2014; Voelklein et al., 2016; Wu, Kobayashi, Li, & Xu, 2015; Zhou et al., 2018).

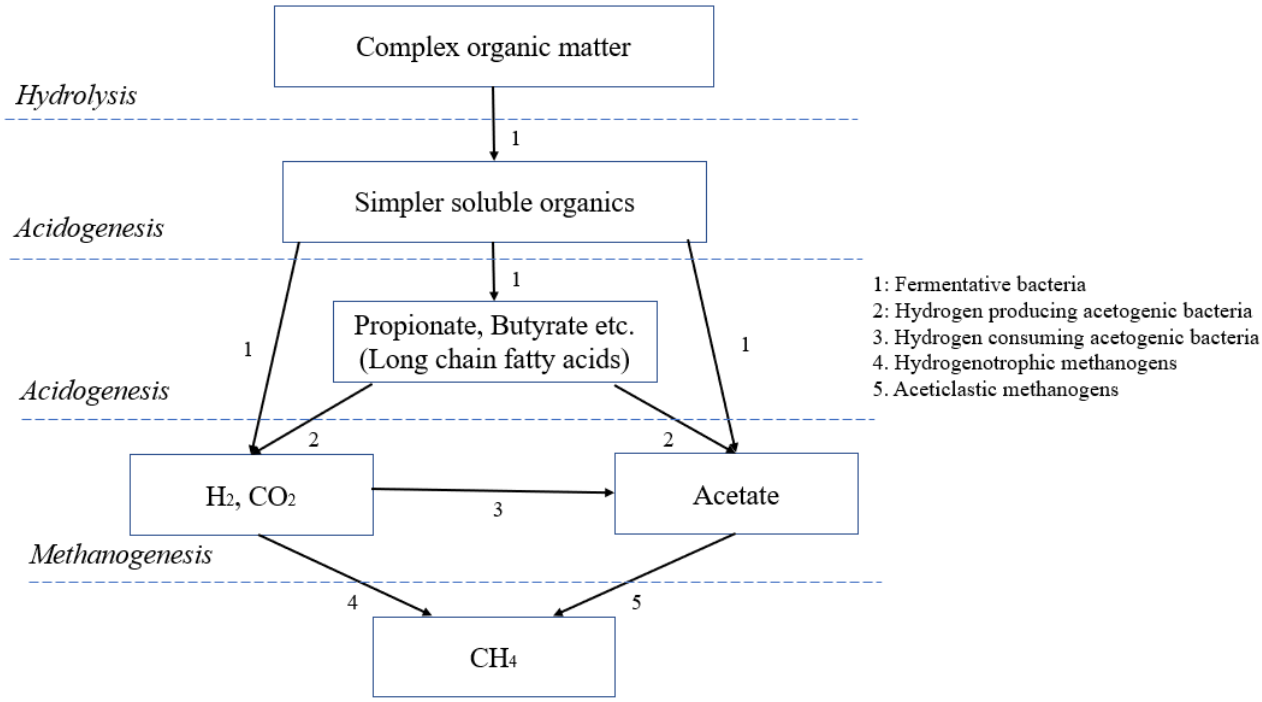


Figure 2.1: Metabolism steps and types of microorganisms involved in anaerobic digestion.

In the second step, these simple monomers are further disintegrated into volatile fatty acids (acetic acid, propionic acid, butyric acid etc.), small alcohols, aldehydes and ketons, and biogas (CO_2 and H_2) by fermenting bacteria. Due to acid accumulation, this step is called acidogenesis.

In the third step, simple organics (mainly acids) accumulated in acidogenesis are transformed into acetic acid, H_2 and CO_2. This step is called acetogenesis because of acetate accumulation. This step is thermodynamically unfavourable under standard conditions at pH 7 and high partial pressure of H_2 (see Equations 2.1-2.3). But consumption of H_2 by homoacetogens and hydrogenotrophic methanogens keeps the partial pressure of H_2 extremely low, allowing the acetogenesis thermodynamically favourable. For instance, the standard Gibbs free energy values ($\Delta G^{\circ\prime}$) of acetogenesis reactions for ethanol, propionate and butyrate become -91.55, -62.22 and -88.73 kJ, respectively, at partial pressure of H_2 of 10^{-6} to 10^{-4} atm (McCarty & Smith, 1986).

Ethanol:

$$CH_3CH_2OH + H_2O = CH_3COO^- + H^+ + 2H_2;\ \Delta G^{\circ\prime} = +9.65\ \text{kJ} \qquad (2.1)$$

Propionate:

$$CH_3CH_2COO^- + 2H_2O = CH_3COO^- + 3H_2 + CO_2;\ \Delta G^{\circ\prime} = +71.67\ \text{kJ} \quad (2.2)$$

Butyrate:

$$CH_3CH_2CH_2COO^- + 2H_2O = 2CH_3COO^- + H^+ + 2H_2;\ \Delta G^{\circ\prime} = +48.30\ \text{kJ} \quad (2.3)$$

Finally, methanogens convert acetate and H_2 to CH_4. This process is called methanogenesis. Methanogens are strictly anaerobic archaea. In this process, methane can be produced in two ways: from acetic acids by acetoclastic methanogens (Equation 2.4) and from H_2 and CO_2 by hydrogenotrophic methanogens (Equation 2.5). The literature suggested that approximately 72% of the methane production could generate from acetate and the other 28% from H_2 in AD (McCarty & Smith, 1986), as illustrated in Figure 2.2.

Acetoclastic methanogenesis:

$$CH_3COOH = CO_2 + CH_4 \quad (2.4)$$

Hydrogenotrophic methanogenesis:

$$4H_2 + CO_2 = CH_4 + 2H_2O \quad (2.5)$$

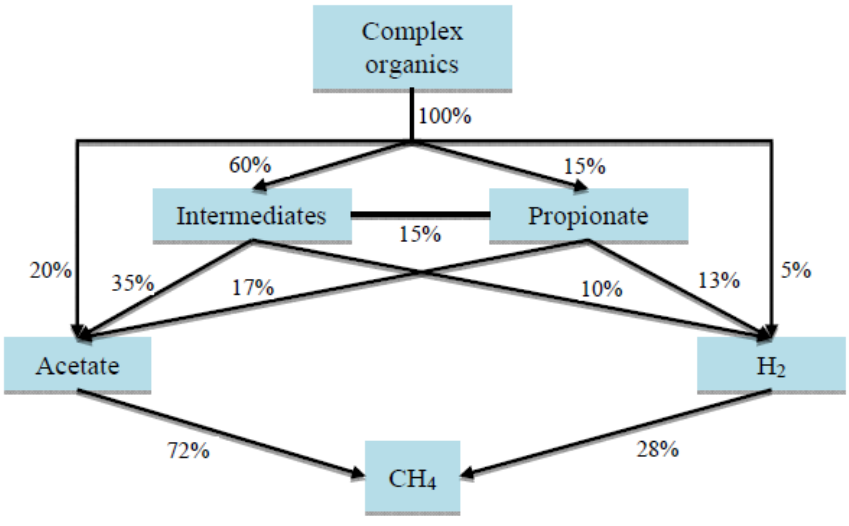


Figure 2.2: Electron flow in anaerobic digestion (McCarty & Smith, 1986).

2.3. Challenges in FW anaerobic digestion

The two most common problems of AD of FW are inhibitions caused by high concentrations of ammonia, and medium to long chain fatty acids (Xu et al., 2018). Dissolved ammonia is produced mainly via hydrolysis of proteins and urea. Excessive ammonia can inhibit methanogens by changing intracellular pH, increasing maintenance energy requirement and impeding specific enzyme reaction (Chen, Cheng, & Creamer, 2008). Free ammonia (aqueous ammonia) is one of inhibitors to microorganisms, since it can penetrate through the cell membrane (Wang et al., 2018; Chen et al., 2008; de Baere, Devocht, Van Assche, & Verstraete, 1984; Kroeker, Schulte, Sparling, & Lapp, 1979). After penetration, free ammonia shuttles protons between the two sides of cell membrane without energy consumption, thereby causing cell inactivation by breaking the proton and potassium balance inside the cell (Wang et al., 2018). Free ammonia concentrations above ~100 mg/L can adversely affect microbial metabolism, and total ammonia nitrogen (aqueous ammonia + ammonium) concentration over 1.7 g/L can inhibit methanogens in anaerobic digestion (Chen et al., 2008; Liu & Sung, 2002; Yenigün & Demirel, 2013).

Medium to long chain fatty acids (e.g., oleic acid, lauric acid, capric acid, myristic acid, etc.) are produced as intermediates during the early stage of AD (Hanaki, Matsuo, & Nagase, 1981). These acids have been reported to be inhibitory to gram-positive microorganisms, one of key players in AD, even at concentrations as low as 1.5 g COD/L (Palatsi et al., 2012). The cell wall of methanogens is close to that of gram-positive bacteria, and thus methanogenesis can be inhibited by the fatty acids through adsorption onto the cell wall/membrane and interference with the transport or protective function (Chen et al., 2008). The literature reported that excessive production of the fatty acids decreased methane production and caused foaming issue in AD (Xu et al., 2018).

2.4. Two-stage anaerobic digestion (AD)

One of the popular approaches to handle inhibition in FW anaerobic digestion is two-stage AD (Wu et al., 2015; Xu et al., 2012). In this process, the first reactor acts as a hydrolytic-acidogenic reactor and the second acts as a methanogenic reactor (see Figure 2.3) (Kim et al., 2014; Lee et al., 2010; Shen et al., 2013; Ventura et al., 2014; Voelklein et al., 2016; Wu et al., 2015). It is well known that growth rates of acidogens are about ten times higher than acetogens and methanogens

(0.05-1.79 h^{-1} vs 0.008-0.173 h^{-1}) (Xu et al., 2012). As a result, this stage-separation in anaerobic digestion enables enrichment of target microorganisms in individual reactors which can accelerate reaction time and improve organic loading rate (OLR) (Santos, Ricci, França Neta, & Amaral, 2017; Ventura et al., 2014). Moreover, hydrogen and methane gases can be captured separately in two-stage anaerobic digestion, potentially enhancing energy benefits from biogas reuse (Shen et al., 2013; Voelklein et al., 2016; Wu et al., 2015). Table 2.1 and 2.2 summarize the performance of FW anaerobic digestion in single and two-stage mode.

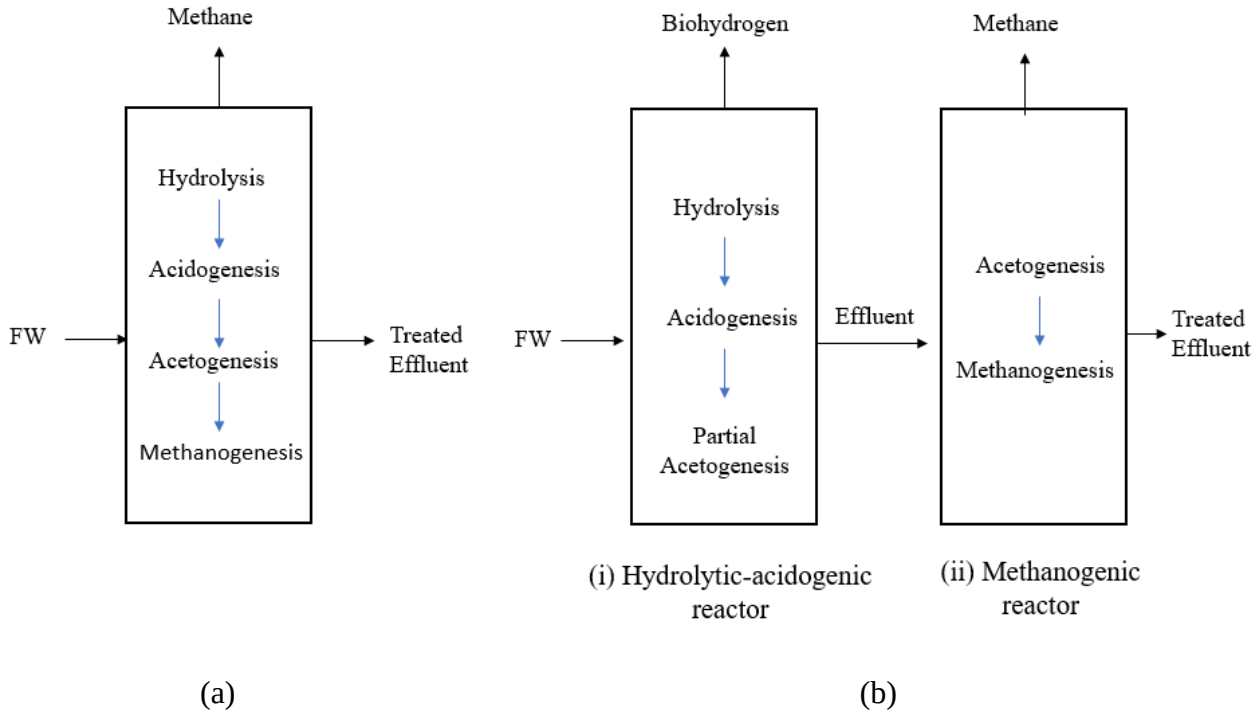


Figure 2.3: Schematics of (a) single stage and (b) two-stage anaerobic digester

Table 2.1: Optimum performance of single stage anaerobic digestion in treating FW.

Reactor	Feed type	Temperature range	Optimum OLR	HRT	Maximum COD removal (%)	Maximum VS reduction (%)	Maximum Specific Methane yield	References
CSTR	FW	38 °C	3 g VS/L/d	16 d	-	-	326.6 ± 26.2 L/kg VS_{added}	Voelklein et al. (2016)
CSTR	FW	37 °C-55 °C	3.08 g VS/L/d	20 d	-	-	480 ± 33 L/kg VS_{added}	Zamanzadeh et al. (2016)
CSTR	FW:FVW (8:5)	35 °C	< 2 g VS/L/d	30 d	-	-	544 ± 6 L/kg VS_{added}	Shen et al. (2013)
CSTR	Kitchen FW	37.40 ± 3.61 °C	3.79 g VS/L/d	-	93%	96%	380 L/kg VS_{added}	Grimberg, Hilderbrandt, Kinnunen, & Rogers (2015)
CSTR	Diluted FW	35 °C	2.4±0.1 g COD/L/d	30 d	82.8±1.5%	74.1±1%	440±20 L/kg VS_{added}	Wu et al. (2015)

- not reported, FW= FW, FVW= Fruit and vegetable waste

Table 2.2: Optimum performance of two-stage anaerobic digestion in treating FW.

Feed type	Temperature (AR+MR)	OLR	HRT	COD removal (%)	VS reduction (%)	Specific hydrogen yield	Specific Methane yield	References
FW	38 °C+38 °C	5.3 g VS/L/d	16 d	-	-	11.8 ± 2 L/kg VS_{added}	419± 23.2 L/kg VS_{added}	Voelklein et al. (2016)
FW slurry	55 °C+55 °C	7.04 g VS/L/d**	17.3 d	89%	88.1%	2.5 mole/mole $hexose_{added}$	287 ± 2 L/kg VS_{added}	Lee et al. (2010)
Diluted FW	35°C+55°C	4.4 g VS/L/d**	25 d	86.6%	81.7%	-	440 L/kg VS_{added}	Ventura et al. (2014)
FW leachate	40.7°C+37°C	2.36 g VS/L/d	30 d	-	82.6%	-	550 L/kg $VS_{removed}$	Kim et al. (2014)
FW:FVW(8:5)	35°C+35°C	≥ 2 g VS/L/d	20 d	-	-	16± 0.3 L/kg VS_{added}	455± 2 L/kg VS_{added}	Shen et al. (2013)
Diluted FW	55°C+35°C	2.4±0.1 g VS/L/d	30 d	88.2 ± 1.4%	80.1 ± 0.9 %	50 L//kg VS_{added}	450± 1 L/kg VS_{added}	Wu et al. (2015)

*- not reported, FW= Food waste, FVW= Fruit and vegetable waste, AR= Acidogenic reactor, MR= Methanogenic reactor

** Calculated assuming density of FW= 1 kg/L

2.5. Performance of leach bed reactor (LBR)

Continuous stirred-tank reactors (CSTRs) are widely used for bioreactors including FW anaerobic digestion, but these bioreactors are not ideal for FW having high solid content (20-30% TS) (Li et al., 2017; Xiong et al., 2019). Hence, FW is typically diluted at 8-10% and treated in CSTRs. Despite the dilution, mixing FW is highly energy-intensive accounting for approximately 30% of the operating cost (personal communication with an engineering and consulting firm, GHD). Leach bed reactors (LBRs) are suitable for treating high solid organic waste because FW percolates with microbial cocktails and thus mixing is minimized. Due to FW leaching feature, LBRs need little to no water for operation (Li et al., 2017). For these reasons, mixing cost for LBRs is minimal, (Hussain et al., 2017; Li et al., 2017; Xiong et al., 2019). A study on FW digestion with LBRs is limited probably due to clogging issues and complex operating conditions. Instead, several studies tested LBRs for FW fermentation prior to methanogenesis (Hussain et al., 2017; Xu et al., 2012; Cysneiros, Banks, Heaven, & Karatzas, 2012; Li et al., 2017; Xiong et al., 2019). Although several LBRs have been tested for FW fermentation, effects of mixing conditions, inoculum to substrate ratio (ISR) and reaction time are yet to be optimized.

The LBR consists of a leachate container at the bottom, a solid holding vessel at the top and a leachate recirculation system (see Figure 2.4). Inoculum-to-substrate ratio, leachate circulation and reaction time are crucial parameters for optimization of LBRs. For instance, an appropriate ratio of ISR should be used to improve FW hydrolysis, accelerate biogas production rate, and consequently reduce reaction time (Xu et al., 2012). Leachate circulation rate, on the other hand, mainly determines the contact between microbes and FW (Cadavid-Rodríguez & Horan, 2014). Insufficient circulation may lead to poor hydrolysis and acidogenesis. But clogging in the food basket may occur at high circulation of leachate (Xiong et al., 2019). Reaction time is another important parameter that determines the performance of LBRs, as well as scale of the reactor. Inadequate reaction time will lead to incomplete fermentation of FW. Therefore, further disintegration will be necessary in the second stage of anaerobic digestion, and longer reaction time will be required for the second-stage reactor. On the other hand, a long reaction time means a large footprint of LBRs, demanding high investment costs. Study of ISR, reaction time and leachate circulation effects on LBR performance is very limited in literature. There are several LBR studies, as summarized in Table 2.3, but FW compositions, LBR design, and operating

conditions are not comparable in literature as the operating conditions were not the same. Hence, it is challenging to conclude optimal conditions for FW fermentation in LBRs.

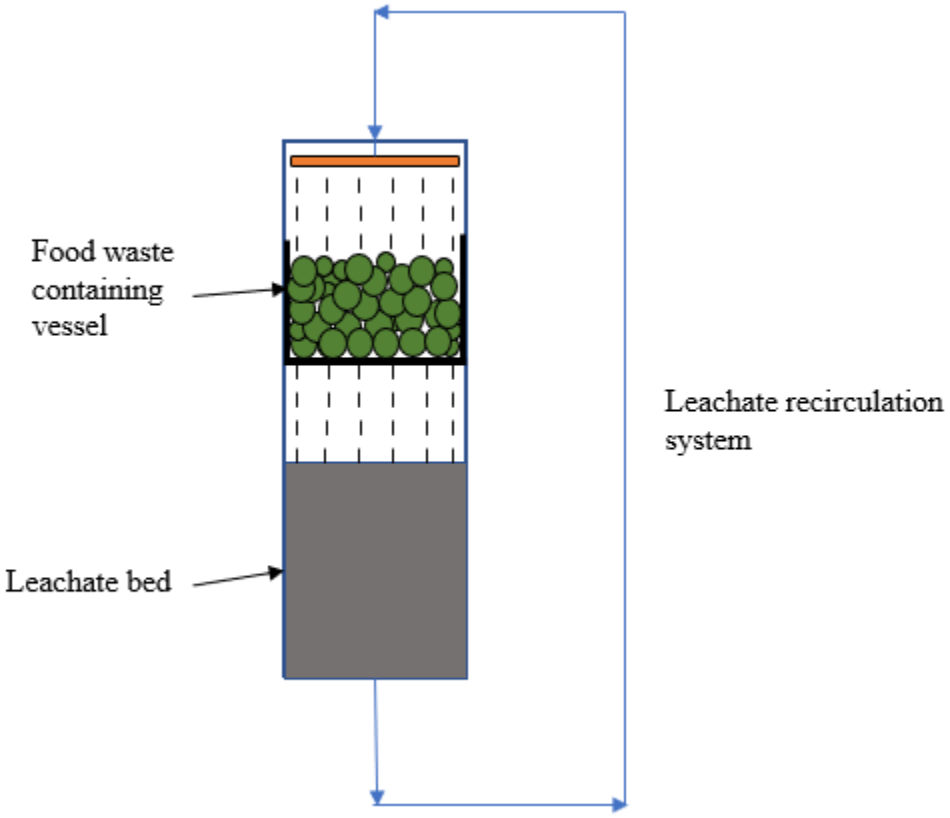


Figure 2.4: Schematics of a leach bed reactor (LBR).

Table 2.3: Optimal conditions in hydrolytic-acidogenic leach bed reactor treating FW.

Feed type	**ISR (%) (vs/vs)**	**Reaction time (days)**	**Leachate recirculation rate**	**VS reduction (%)**	**Hydrolysis yield (g COD/kg VS_{added})**	**VFA yield (g COD/kg VS_{added})**	**Reference**
Cafeteria FW	4*	14	0.375 L/hr	72	565	330	Hussain et al. (2017)
Simulated FW	6.9	17	-	71.7 ± 2.8	640 ± 70	180±30	Xu et al. (2012)
Whole-crop of maize	2.5*	28	0.083 L/hr	89	935	840	Cysneiros et al. (2012)
Vegetable waste	10	4-8	-	-	450	425	Li et al. (2017)
Cafeteria waste	5	14	4.4 L/hr	87	883	762	Xiong et al. (2019)

- not reported, *reported as w/w

2.6. Background information on anaerobic membrane bioreactors (AnMBRs)

Slow growth of methanogens is a drawback in conventional anaerobic digestion (Zayen et al., 2010), which means that retention of methanogens is key for achieving high methane yield and improving effluent quality. Membrane separation in AnMBRs enables a high concentration of methanogens to be kept in bioreactors (Galib, Elbeshbishy, Reid, Hussain, & Lee, 2016). For this reason, AnMBRs have the benefits of better effluent quality, higher methane production, lower sludge production and smaller footprint than conventional anaerobic digestion (Charfi, Ben Amar, & Harmand, 2012; Galib et al., 2016).

2.6.1. Membrane configurations in AnMBR

There are two basic configurations of membrane system in AnMBRs (Aslam et al., 2018; Zayen et al., 2010), depending on the position of membrane modules. As shown in Figure 2.5, membrane modules can be directly submerged into a bioreactor, like typical aerobic membrane bioreactors, or can be contained in a separate tank from the bioreactor. Submerged membrane system is known for its low energy requirement but prone to high fouling potential and complexity in cleaning. In contrast, external setup provides easier membrane replacement, maintenance and higher fluxes, but needs higher energy input as an additional pump is required to put the retentate back in the bioreactor.

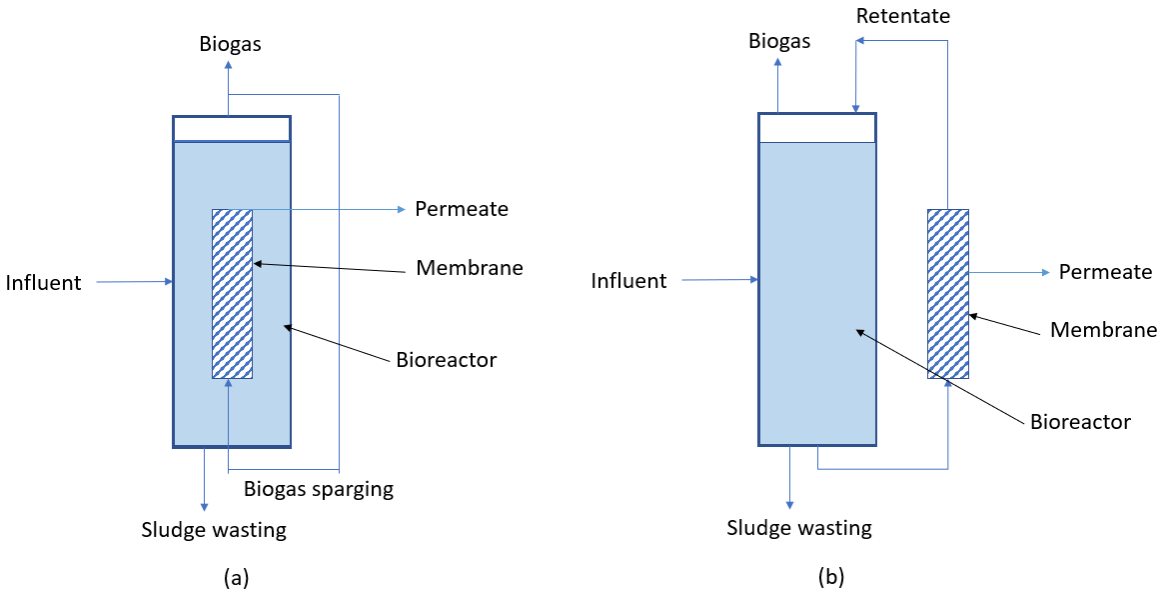


Figure 2.5: Configurations of anaerobic membrane bioreactors (AnMBRs): (a) a submerged AnMBR and (b) an external AnMBR.

2.6.2. Membrane fouling and mitigation

Membrane fouling is one of the major drawbacks of AnMBR application (Dong, Parker, & Dagnew, 2016; Galib et al., 2016; Meng et al., 2017). All membrane processes have fundamental self-conflict because clean water production through membranes stimulates membrane fouling, while the processes attempt to mitigate or completely stop the fouling. Membrane fouling determines the suitability of membrane cleaning methods and eventually its lifespan. Hence, it is one of the important cost factors in AnMBRs.

Membrane fouling can be classified as removable, irremovable and irreversible fouling (Meng et al., 2009). As shown in Figure 2.6, removable fouling can be easily eradicated by using physical cleaning methods such as backwashing with permeate. To eliminate irremovable foulants, chemical cleaning (e.g. backflushing with or submerging in chemical solutions) is needed. In general, removable fouling and irremovable fouling occurs due to formation of cake layer and pore blocking respectively (Meng et al., 2009). Irreversible fouling is a permanent fouling which cannot be removed even with chemical cleaning. This kind of fouling usually occurs due to exposure to

cleaning agents like NaOCl. As a result, irreversible fouling ultimately determines the membrane life (Gkotsis, Banti, Peleka, Zouboulis, & Samaras, 2014).

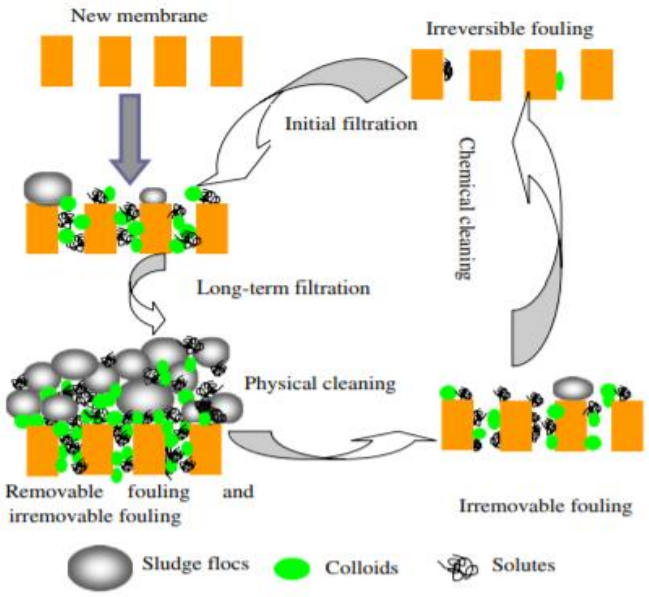


Figure 2.6: Schematic illustration of removable, irremovable and irreversible fouling (Meng et al., 2009).

Among the several maintenance techniques, chemical cleaning is considered the most critical step in managing fouling in AnMBRs (Meng et al., 2017). Chemical cleaning is usually carried out by caustic solutions, acids, Ethylene-Diamine-Tetra-Acetic acid (EDTA), enzymatic cleaners or chlorine (Dong et al., 2016; Zhang, J. et al., 2007). Table 2.4 summarizes cleaning agents and optimized cleaning frequency from the literature (Zhang et al., 2007; Ramos, Zecchino, Ezquerra, & Diez, 2014; Xiao et al., 2015; Galib et al., 2016; Dong et al., 2016; Santos et al., 2017). Citric acid and NaOCl are commonly used for cleaning fouled membranes and show higher foulant removal efficiency, as compared to other cleaning agents (Dong et al., 2016; Galib et al., 2016). But too high dosage and frequent use of these chemicals can adversely impact the physicochemical properties of membranes and microbial metabolism (Meng et al., 2017). Therefore, membrane cleaning should be delicately optimized without having adverse effect on AnMBRs.

Table 2.4: Survey of recommended cleaning processes.

Cleaning duration	Cleaning agents	Cleaning frequency	Reference
1 hr for each step at 50°C	0.5% EDTA+1% Na_3PO_4 or 0.1 N HNO_3	After 135 days of operation	Zhang et al. (2007)
3 hrs	1000 ppm NaOCl	Minimum 2 weeks interval	Ramos et al. (2014)
-	0.2% NaOCl+ pH 2 HCl+1% EDTA	Once every 10 days	Xiao et al. (2015)
45 mins	200 mg/L NaOCl + 2000 mg/L Citric acid	Twice a week	Galib et al. (2016)
16 hrs	2000 mg/L Citric acid + 2000 mg/L NaOCl	Once a week	Dong et al. (2016)
20 min	500 ppm NaOCl+ Citric acid (pH 2.5)	Whenever flux declines	Santos et al. (2017)

- not reported

2.7. Performance of AnMBR in treating food related high strength wastewaters

AnMBRs have been applied for treating high strength organic wastewaters, such as landfill leachate, municipal solid waste leachate and FW slurries. Employment of AnMBRs as the second phase of two-stage FW anaerobic digestion has not yet been reported to the author's knowledge. Table 2.5 summarizes performance of AnMBRs treating high strength organic wastewater, including operating conditions.

Unlike conventional AD, high COD removal efficiency can be achieved in AnMBRs when operated at high OLRs and low HRTs. One of key features in AnMBRs is decoupling of SRT from HRT, meaning a compact process with small wasted sludge. Long SRTs also help AnMBRs to have steady performance. For example, Trzcinski & Stuckey (2010) showed that COD removal efficiency over 95% was achieved at OLR as high as 11.75 kg COD/m^3/d and HRT as low as 1.5

days when SRT of 300 days was maintained in an AnMBR. Cheng et al. (2018) also reported high treatment efficiency at SRT 500 days. In both cases, high methane yields were observed. However, maintaining high SRT and short HRT increased membrane fouling events (Huang, Ong, & Ng, 2011), and hence high permeate flux is hard to be kept in AnMBR run. Trzcinski & Stuckey (2010) reported that the highest flux was only 4.5 LMH in a submerged AnMBR treating high strength wastewater, implying extensive fouling above 4.5 LMH. Chemical cleaning can alleviate membrane fouling maintaining high flux, but Zayen et al. (2010) reported that even after chemical cleaning membrane flux declined from 8.3 to 2.5 LMH.

HRT and SRT are two important operating parameters to be optimized for AnMBR. Shorter HRTs and longer SRTs are preferred for improving AnMBR benefits (compact system with high performance), but these two factors should be compromised with membrane fouling and flux. In literature, a very wide range of HRTs (1.1-30 d) and SRTs (30-500 d) can be found in AnMBR treating high strength organic wastewater, which implies that these parameters significantly depend on various factors of feed, organic loading rate, microorganisms, and so on. The two parameters can become incomparable, and hence, they need to be understood well to have an optimized AnMBR system.

Table 2.5: Performance of AnMBR in treating food related high strength wastewater.

Reference	**Zayen et al. (2010)**	**Bohdziewicz et al. (2008)**	**Trzcinski & Stuckey (2010) ***		**Cheng et al. (2018)**	**Xiao et al. (2015)**	**Taskan & Hasar (2012)**
Type of substrate	Landfill leachate	Diluted landfill leachate	Municipal solid waste leachate	Municipal solid waste leachate	FW slurry	KW slurry	Landfill leachate
Pore size of membrane	100 kDa	0.1 µm	0.4 µm	0.4 µm	0.2 µm	100 kDa	0.1 µm
Reactor volume	50 L	29 L	3 L	3 L	15 L	1200 L	-
Configuration	External	Submerged	Submerged	Submerged	External	External	Submerged
pH	7.5	8.18	7.3	7.3	7.5	7.6-7.8	7.5-8.3
Temperature	37°C	35 °C	35°C	35°C	-	39±1°C	37°C
HRT	7 d	2 d	1.1 d	1.5 d	30 d	-	-
SRT	-	-	30 d	300 d	500 d	60 d	100 d
OLR (kg COD/m^3/d)	6.27	2.5	8	11.75	2.43	5.9	0.54
COD removal efficiency (%)	90.7%	90%	> 95%	> 95%	92.9%	> 99%	75%
Specific methane yield	-	-	0.24-0.28 L/ g COD_{fed}	0.25 L/g COD_{fed}	≥ 0.33±0.05 L/g $COD_{removed}$	-	0.28 L/g $COD_{removed}$
Average flux (LMH)	~4 LMH	-	~0.5 LMH	~4.5 LMH	2.4 LMH	12-15 LMH	0.39 LMH

* best performance, - not reported

Chapter 3 : FW fermentation using an LBR

3.1. Introduction

Proper disposal of food waste (FW) has become a pressing issue as environmental pollution, economic efficiency and sustainability issues are growing. The two well-known methods to dispose FW are landfilling and composting in North America (The Atlantic, 2016; National Zero Waste Council, 2018; Waste today, 2018; CEC, 2017; Statistics Canada, 2013). However, these methods are neither economical nor sustainable due to large land requirements and the generation of odor, secondary contaminants such as leachate and greenhouse gas like methane. Anaerobic digestion (AD) can be a sustainable, cost-effective, and environment-friendly solution to FW treatment due to highly efficient stabilization of FW and reuse of recovered biogas energy.

AD is a well-established biotechnology that recovers methane from FW. But higher value-added products can also be obtained from FW using different microbial metabolism and related bioreactors. Fermentation is a great example of such processes. FW fermentation has high potential to recover biochemicals including H_2, acetate, butyrate, butanol, etc. (Dahiya et al., 2018; Hussain et al., 2017; Xiong et al., 2019). Conventionally, continuously stirred tank reactor (CSTRs) have been used for FW fermentation which demand substantial maintenance and operating costs due to the high solid content of FW ranging from 20 to 30% (Browne, Allen, & Murphy, 2013; Xiong et al., 2019). High operation and maintenance costs can reduce energy benefits of recovered bioenergy and biochemical, and hence bioreactor suitable for high solid feedstock like FW should be adopted to improve energy benefits. Several studies have reported that leach bed reactors (LBRs) designed for low energy input can improve the benefit of recovered bioenergy from FW, since mixing cost for high solid FW, one of the main operating and maintenance costs, is minimal in LBRs (Hussain et al., 2017; Li et al., 2017; Xiong et al., 2019). As a result, LBRs can maximize the profit of bioenergy and biochemical recovered from FW.

Since hydrolysis is the rate-limiting step in the fermentation process, inoculum to substrate ratio (ISR), leachate circulation rate and reaction time can be the key parameters to improve hydrolysis of FW and eventually boost generation of value-added products by increasing microbial contact with FW and consequently biochemical reactions. Hussain et al. (2017) reported that VS reduction,

hydrolysis yield and VFA yield increased by 7%, 10% and 33%, respectively, in a LBR when leachate circulation rate was increased from 6 L/d to 9 L/d during fermentation of cafeteria FW. Xu et al. (2012) also showed that VS reduction, COD removal and VFA production increased by 8.5%, 14% and 80%, respectively, when ISR was incremented from 1.7% to 6.9% (vs/vs). The literature indicated better hydrolysis and fermentation at higher ISR, leachate circulation rate or reaction time (Hussain et al., 2017; Xu et al., 2012). However, there is limited information on optimization of these parameters on FW fermentation in LBRs, although the parameters can affect each other. For instance, reaction time required for FW fermentation can decrease at higher ISR or leachate circulation rate. On the other hand, ISR would need to be in an optimum range of leachate circulate rate, due to potential clogging events in LBRs.

This study systematically evaluated the effects of ISR, leachate circulation rate, and reaction time on FW fermentation, and optimized in a LBR operated at room temperature (22°C) and neutral pH (7±0.1).

3.2. Materials and methods

3.2.1. Characterization of FW and inoculum

FW was collected once every three months from a cafeteria at University of Waterloo. After collection, it was manually sorted to remove non-biodegradable materials, such as egg shells and bones. The sorted FW consisted of vegetables, fruits, meat and other carbohydrate-rich foods, such as bread, pasta, potatoes, waffles, etc. They were diced approximately into approximately 1 cm cubes using a commercial chopper (Starfrit, Canada). The chopped FW was homogenized and put at -20°C in airtight bags to avoid any deterioration. The samples were thawed at 4°C for 24 hours prior to the experiments. In this study, FW was classified based on collection time, and the composition and characteristic of FW was summarized in Table 3.1 and Table 3.2.

AD sludge sampled from Kitchener wastewater treatment facility (Ontario, Canada) was used as inoculum to the LBR. The sludge was heated for 15 mins at 75°C to deactivate all methanogens before use. Initial characteristics of the inoculum are also shown in Table 3.2.

Table 3.1: Composition of FWs collected from the cafeteria.

	Vegetables (%)	Fruits (%)	Meat (%)	Other carbohydrate-rich food (%)
FW_{Feb}	32	52	6	10
FW_{May}	26	70	4	0
FW_{Aug}	11	1	1	87
FW_{Nov}	34	40	0	26

FW_{Feb}: FW collected in February 2018; FW_{May}: FW collected in May, 2018; FW_{Aug}: FW collected in August, 2018; FW_{Nov}: FW collected in November, 2018.

Table 3.2: Initial characteristics of FW and inoculum.

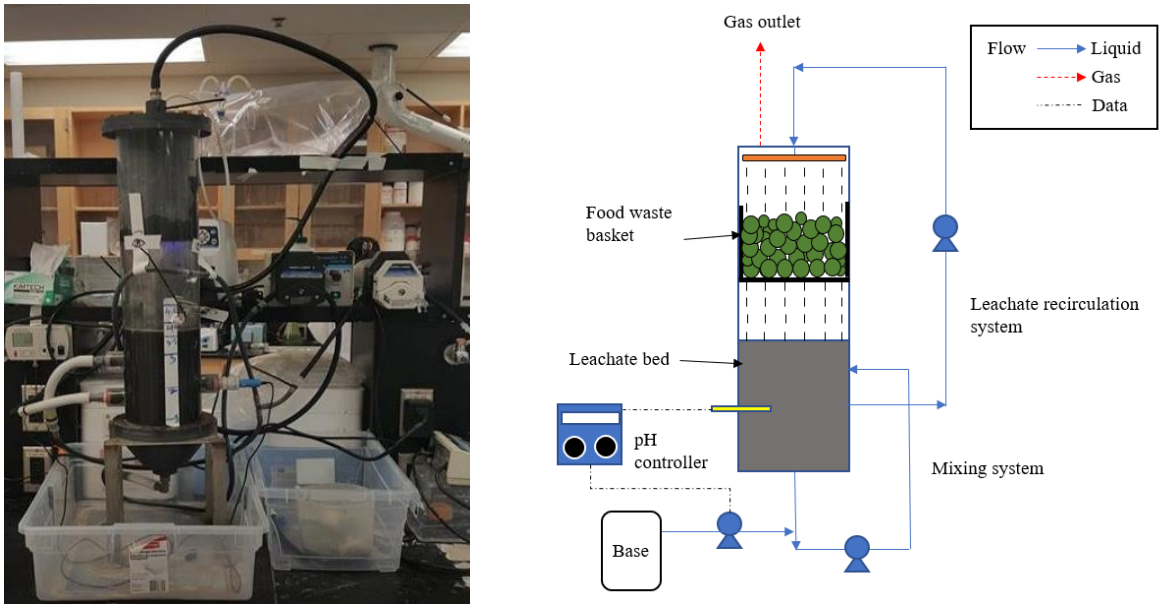
Parameters[a]	**Unit**	**FW_{Feb}**	**FW_{May}**	**FW_{Aug}**	**FW_{Nov}**	**Inoculum**
TCOD	g/L	210±15	145±10	354±29	352±26	22.3±1.9
sCOD	g/L	-	-	-	-	3.15±0.27
TS	g/L	204±18	126±9	319±23	173±11	14.15±0.9
VS	g/L	188±11	118±9	306±25	165±6	12.33±1.1
VS/TS	%	92	93	96	95	86
Specific COD	g COD/ g VS	1.12	1.15	1.11	2.13	1.81

[a] All the parameters were tested at least in triplicates. Values are reported with their standard deviation. FW_{Feb}: FW collected in February 2018; FW_{May}: FW collected in May, 2018; FW_{Aug}: FW collected in August, 2018; FW_{Nov}: FW collected in November, 2018.

3.2.2. Reactor design

A cylindrical LBR was made with acrylic materials and had a total volume of 11 L with an inner diameter of 14 cm, an outer diameter of 15.25 cm and a height of 72 cm. The reactor consisted of 3 sections: a removable top cover with a gas outlet and a sprinkling system to distribute leachate, a container in the middle section to hold FW and a leachate holding bed at the bottom (see Figure 3.1). The FW basket was made of PVC and had an inner diameter of 12.75 cm and a height of 21.6 cm (effective volume 1 L). The basket had a perforated bottom with a pore size of 4 mm that prevents food waste particles from falling into the leachate holding bed. The percolated leachate then sits in the 6.75 L leachate holding bed at the bottom of the reactor before it is recirculated by

a digital peristaltic pump (Masterflex L/S Digital Drive, 600 RPM, 115/230 VAC, Model no. 07523-80, USA). The leachate in the holding bed was continuously mixed using a peristaltic pump (Masterflex L/S Economy Drive, Model no. 07554-90, USA). A pH controller (Milwaukee, MC-122 pH meter) was coupled to an in-situ pH probe and a pump injecting 1M NaOH to keep neutral pH in the leachate. A gas counter (MilliGas counter, Ritter Apparatus, Bochum, Germany) was connected to the top cover of the LBR to measure biogas production. Liquid and gas samples were taken from a mixing line and gas line, respectively, for routine analysis (Figure 3.1b).



(a) (b)

Figure 3.1: (a) Photo of the leachate bed reactor (LBR) and (b) Schematic diagram of the LBR.

3.2.3. Experimental setup

The LBR was operated at room temperature (22°C). Literature suggests that FW hydrolysis improved in LBRs run at pH 7 (Xiong et al., 2019; Hussain et al., 2017), and hence neutral pH was maintained in the LBR throughout the experiments using the pH controller as described above. In this study, three operating parameters were assessed for the LBR: (1) ISR, (2) reaction time, and (3) leachate circulation rate. Table 3.3 summarizes the operating conditions. The LBR was initially

loaded with 1 kg of FW. To meet a desired ISR value, pre-heated AD sludge was added to the LBR with dilution with deionized water, and the initial sludge volume was fixed at 3.5 L. Before the start of each batch cycle of experiments, the LBR was sparged with nitrogen gas to create anaerobic condition at a flow rate of 0.5 L/min for 15 min. After every batch, the leachate was centrifuged at 9,500 rpm for 20 min and the centrifuged solids were reused as inoculum to the LBR in the next batch, which allowed enrichment of FW fermenting microorganisms in the LBR. The leachate in the holding bed was continuously mixed at a rate of 60 L/hr with a peristaltic pump (Model 07554-90, Cole-Parmer, USA). A leachate recirculation pump was programmed to be turned on for 15, 30 and 45 s at every 5 min to sprinkle the leachate on the top of the FW at a rate of 88 L/hr. They corresponded to leachate recirculation rates of 4.4, 8.8 and 13.2 L/hr, respectively.

Table 3.3: Operating conditions in the LBR.

Operating conditions	**ISR (%) (vs/vs)**	**Reaction time (days)**	**Leachate recirculation rate (L/hr)**	**Average OLR (g VS/L/d)**
$LBR_{5\text{-}4.4\text{-}14}$	5	14	4.4	2.31
$LBR_{10\text{-}4.4\text{-}14}$	10	14	4.4	2.31
$LBR_{10\text{-}4.4\text{-}7}$	10	7	4.4	4.62
$LBR_{5\text{-}8.8\text{-}7}$	5	7	8.8	4.62
$LBR_{10\text{-}8.8\text{-}7}$	10	7	8.8	3.76
$LBR_{10\text{-}13.2\text{-}7}$	10	7	13.2	4.82

Subscripts for LBR stand for ISR, leachate circulation rate, and reaction time. For instance, $LBR_{5\text{-}4.4\text{-}14}$ indicates that the LBR was operated with ISR 5% and leachate circulation rate 4.4 L/hr at reaction time 14 d.

3.2.4. Analytical methods

Concentrations of total solids (TS), volatile solids (VS), total suspended solids (TSS), total volatile solids (VSS), total chemical oxygen demand (TCOD) and soluble chemical oxygen demand (SCOD) were quantified according to the Standard methods (APHA, AWWA & WEF, 2005). TS and VS of FW were analyzed at the beginning and end of the experiments to determine solid

reduction. TSS, VSS, TCOD and SCOD were quantified daily during the experiments. Volatile fatty acids (VFAs) that include acetic acid, propionic acid, isobutyric acid, butyric acid, isovaleric acid, valeric acid, isocaproic acid, hexanoic acid and heptanoic acid were also analyzed at the beginning and end of the experiments. Liquid samples were first filtered through 0.2 µm membrane filters (Whatman, 6751-2502, USA) and then VFA concentrations were measured using a gas chromatography (GC) (HP 5890 Series II, Hewlett Packard, USA) equipped with a flame ionization detector (FID) and a capillary column (30 m x 0.53 mm x 0.5µm PAG, Supelco, Bellefonte, PA). Helium was used as a carrier gas (40 PSI) and hydrogen and air were used for ignition (15 PSI and 34 PSI respectively). The GC-FID was programmed to maintain an initial temperature of 150°C for 2 mins which increased to 190 °C at a slope of 4°C/min and maintained at that temperature for 3 mins.

To analyze the composition of biogases from the LBR, 0.5 mL of gas sample was collected using a gastight syringe (model 1005 GASTIGHT syringe, Hamilton, Reno, NV) and injected into a GC (model 310, SRI Instrument, 51 USA) equipped with a thermal conductivity detector (TCD) and a Porapak Q 80-100 mesh column (Supelco, Bellefonte, PA) using argon as a carrier gas. The GC-TCD was programed to have an initial oven temperature of 50°C which holds for 1 min and increases to 110 °C (hold for 1 min) in 8 min at a rate of 10°C/min. Total kjeldahl nitrogen (TKN), ammonium nitrogen (NH_4^+-N), total phosphate (TP) and reactive phosphate (RP) concentrations of the samples were determined using HACH vials (TNTplus™ 880 for TKN, TNTplus™ 832 for ammonium nitrogen and TNTplus™ 844 for phosphates) after filtering the samples with 0.45µm syringe filters. All chemical analyses were carried out in triplicates and average values are reported with standard deviations.

3.2.5. Calculation

To calculate hydrolysis yield, the change of SCOD mass (g) in leachate was determined at the beginning and end of the experiments by multiplying SCOD concentration (g/L) with the leachate volumes (L). Equation (3.1) shows the hydrolysis calculation used in this study.

$$\text{Hydrolysis yield } \left(\frac{\text{g SCOD}}{\text{kg VS}_{\text{added}}}\right) = \frac{\text{Final SCOD (g)-Initial SCOD (g)}}{\text{Initial VS of FW (kg)}} \quad (3.1)$$

VFA yield were expressed with VFA production per initial VS of FW (g COD/kg VS) using Equation (3.2). VFAs were expressed as COD using half reactions of individual short-chain fatty acids. For example, 1 mole of acetic acid (CH_3COOH) is equivalent to 64 g of COD ($CH_3COOH+2\ H_2O=2\ CO_2+8\ H^+ +8\ e^-$, 1 mole e^-=8 g COD).

$$\text{VFA yield}\left(\frac{\text{g COD}}{\text{kg VS}_{\text{added}}}\right)=\frac{\text{Final VFAs (g COD)-Initial VFAs (g COD)}}{\text{Initial VS of FW (kg)}} \quad (3.2)$$

Specific hydrogen yield was computed with Equation (3.3).

$$\text{Specific hydrogen yield}\left(\frac{\text{L H}_2}{\text{kg VS}_{\text{added}}}\right)=\frac{\text{Total hydrogen produced (L)}}{\text{Initial VS of FW (kg)}} \quad (3.3)$$

COD balances for the LBR were built using Equation (3.4).

$$\text{FW}_{\text{initial}} = \text{FW}_{\text{remained}} + \text{Leachate} + \text{H}_2 + \text{cell growth} \quad (3.4)$$

where $FW_{initial}$ = COD of initial FW loaded to the LBR (g COD), $FW_{remained}$ = COD of residual FW in the food bucket, Leachate = leachate SCOD in the LBR (g COD), H_2 = COD of biohydrogen produced from the LBR (g COD), and cell growth = COD for cell synthesis (g COD). Volumes of H_2 were converted to moles of H_2 with ideal gas law at a temperature of 22°C, and then moles of H_2 were changed to g COD using a half reaction ($2H^+ +2e^- = H_2$, 1 mole e^- = 8 g COD). COD for cell synthesis was calculated with the difference between initial and final VSS concentration in leachate, given that the chemical formula of bacteria is $C_5H_7O_2N$ and 1 g VSS is equivalent to 1.42 g COD (Rittmann and McCarty, 2001). Remaining FW in the basket was calculated by multiplying the amount of VS remained in the basket with respective specific COD shown in Table 3.1.

3.3. Results and discussion

3.3.1. Effect of inoculum to substrate ratio (ISR)

To evaluate the effect of ISR on FW fermentation, the LBR was operated at ISR, gradually increasing to 15%. At the highest ISR of 15%, the food basket of the LBR was clogged completely on the first day, and hence the results at ISR 5% and 10% only are discussed here. Figure 3.3 compared solid reduction of FW in the LBR operated at different reaction times and leachate circulation rates for two ISR conditions of 5% and 10%. As shown in Figure 3.2, the trends of TSS

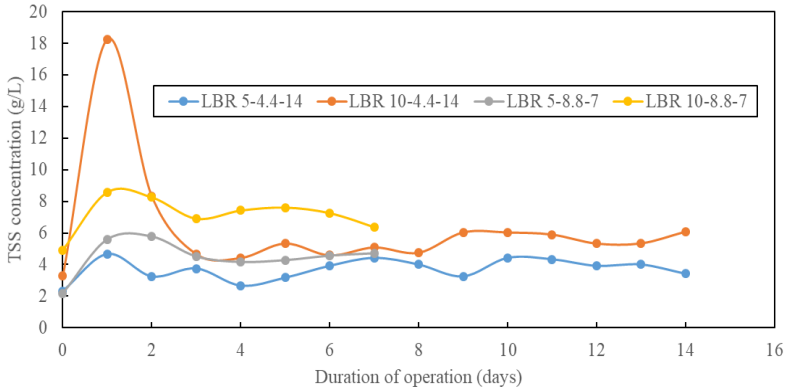
and VSS concentrations with time suggest that solid reduction of FW reached at steady state in 7 d at both ISR 5% and 10%.

Table 3.4 summarizes LBR performance at ISR 5% and 10% at different leachate circulation rates and reaction times. In a reaction time of 14 d, VS reduction in the FW basket, VFA yield and VFA to SCOD ratio improved at ISR of 10% over 5%. In comparison, the final SCOD concentrations and hydrolysis yield were close between the two ISR conditions. This result indicates that ISR can improve fermentation rate but not FW solubilization in the LBR for 14 d of reaction time. In a shorter reaction time of 7 d, solid reduction, VFA yield, solubilization were enhanced at ISR 10%, which implies that higher ISR but less than 15% can accelerate rates of FW hydrolysis and fermentation when the reaction time is limited to a week. Interestingly, the decline of VS reduction was observed at ISR 10% when leachate circulation rate was as fast as 8.8 L/hr. This suggests that increasing both leachate recirculation rate and ISR could have partially caused clogging on the food basket of the LBR, deteriorating FW hydrolysis. Hence, ISR should be optimized together with leachate circulation rate.

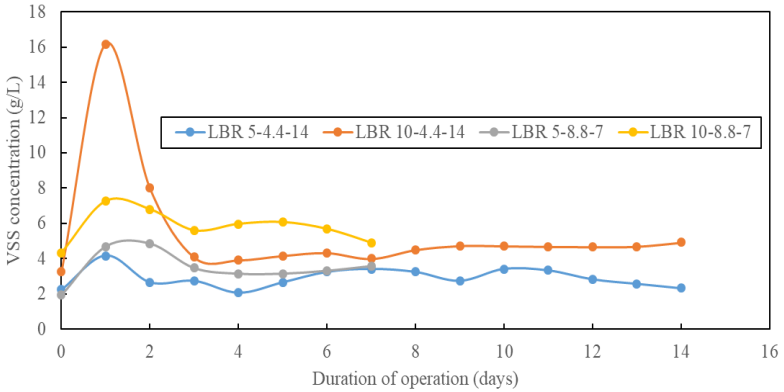
Table 3.4: Effect of ISR on reactor performance and leachate characteristics after fermentation.

Parameters	**Unit**	**$LBR_{5\text{-}4.4\text{-}14}$**	**$LBR_{10\text{-}4.4\text{-}14}$**	**$LBR_{5\text{-}8.8\text{-}7}$**	**$LBR_{10\text{-}8.8\text{-}7}$**
TCOD concentration	g/L	32.91±0.21	33.8±1.5	27.43±1.23	31.35±1.02
SCOD concentration	g/L	26.03±0.17	25.05±0.68	22.85±1.96	24.03±1.1
VFA concentration	g COD/L	19.7±0.98	22±1.64	17.45±1.18	17.95±0.59
Hydrolysis yield	g SCOD/kg VS_{added}	666	624	526	611.5
VFA yield	g SCOD/kg VS_{added}	495	571	419.5	467
VFA/SCOD	%	76±5	88±6	76±2	74.5±1.5
VS reduction	%	83.5±3.5	88±2	75±6	68.5±7

Figure 3.3 shows VFA distributions in leachate of the LBR. Regardless of ISR conditions, butyric acid was found to be the most dominant VFA in leachate (35-39% of the total VFA) followed by acetic acid (31-36% of the total VFA) and propionic acid (11-23% of the total VFA). Other VFAs including isobutyric, isovaleric, n-valeric, isocaproic, hexanoic acid and heptanoic acid account for the rest of the VFAs ranging from 11 to 19% of the total VFA. Similar trends were also reported by the literature (Xiong et al., 2019; Hussain et al., 2017), presenting butyric and acetic acids are key products in FW fermentation at neutral pH.



(a)



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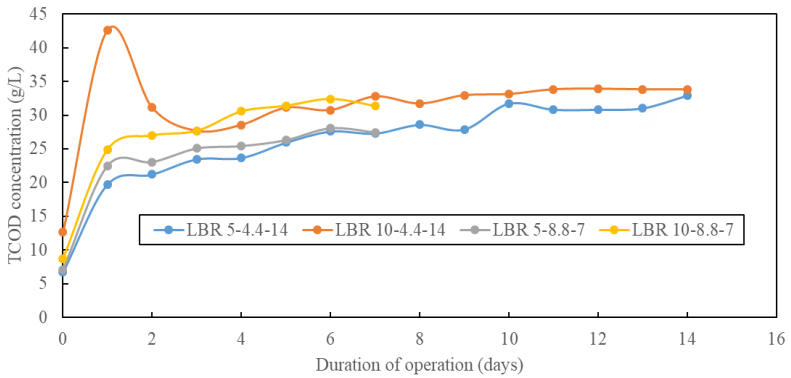

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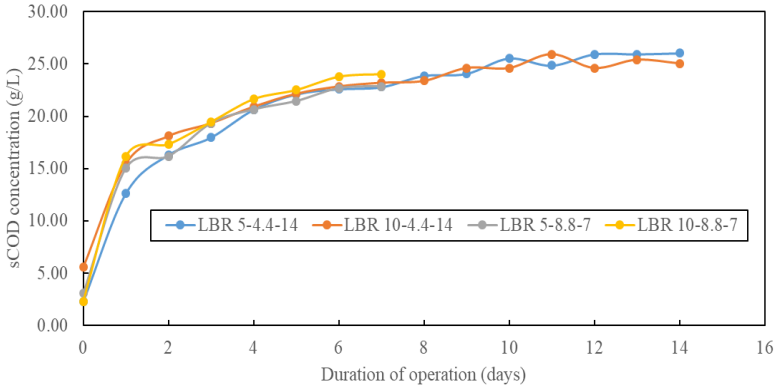


(d)

Figure 3.2: Variation of (a) TSS, (b) VSS, (c) TCOD and (d) SCOD in leachate with respect to different ISR.

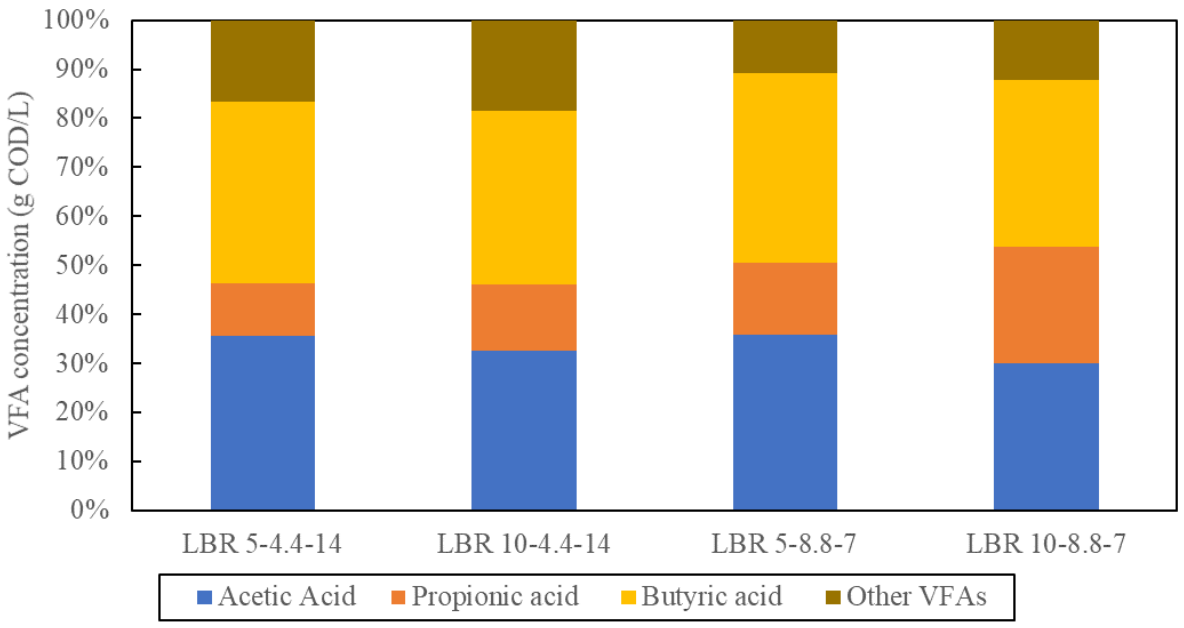


Figure 3.3: Effect of ISR on VFA distribution in leachate.

3.3.2. Optimization of reaction time and leachate circulation rate

LBR performance was assessed at two reaction times of 7 d and 14 d. As shown in Table 3.5, FW was better stabilized at reaction time of 14 d than 7 d. At ISR 5%, hydrolysis increased by 31% when reaction time was increased from 7 to 14 d. SCOD and VFA concentrations in leachate also increased at 14d, but not as significantly as FW hydrolysis. Similar to ISR 5%, much higher increase of hydrolysis efficiency was found at 14d for ISR 10%, along with moderate increase of SCOD and VFA concentrations. These results indicate the significance of longer reaction time for FW hydrolysis, implying that FW hydrolysis is a rate-limiting step in FW fermentation (Hussain et al., 2017; Kim et al., 2014; Lee et al., 2010; Shen et al., 2013; Ventura et al., 2014; Voelklein et al., 2016; Wu, Kobayashi, Li, & Xu, 2015; Zhou et al., 2018).

Table 3.5: Effect of reaction time on reactor performance and leachate characteristics after the duration of operation.

Parameters	**Unit**	**ISR=5%, Leachate circulation rate = 4.4 L/hr**		**ISR=10%, Leachate circulation rate = 4.4 L/hr**	
		Day 7	**Day 14**	**Day 7[a]**	**Day 14**
TCOD concentration	g/L	27.31±0.91	32.91±0.21	29.75±2.65	33.8±1.5
SCOD concentration	g/L	22.79±0.35	26.03±0.17	22.32±1.67	25.05±0.68
VFA concentration	g COD/L	19.40±1.07	19.7±0.98	18.77±1.05	22±1.64
Hydrolysis yield	g SCOD/kg VS_{added}	510	666	487	624
VFA yield	g SCOD/kg VS_{added}	429	495	428.5	571
VFA/SCOD	%	85±5	76±5	84±2.5	88±6
VS reduction	%	-	83.5±3.5	79±6[b]	88±2

[a] Obtained by averaging the values of $LBR_{10\text{-}4.4\text{-}14}$ at Day 7 and $LBR_{10\text{-}4.4\text{-}7}$

[b] Obtained from $LBR_{10\text{-}4.4\text{-}7}$

In comparison, reaction time did not have significant impact on VFA distribution in leachate. As shown in Figure 3.4, the abundant VFAs in the leachate were consistently butyric acid, acetic acid and propionic acid. Their fractions were 30-35%, 26.5-30.5% and 9-12.5% of total VFAs in the order. Other VFAs account for 13-16% similar to those found in ISR experiments.

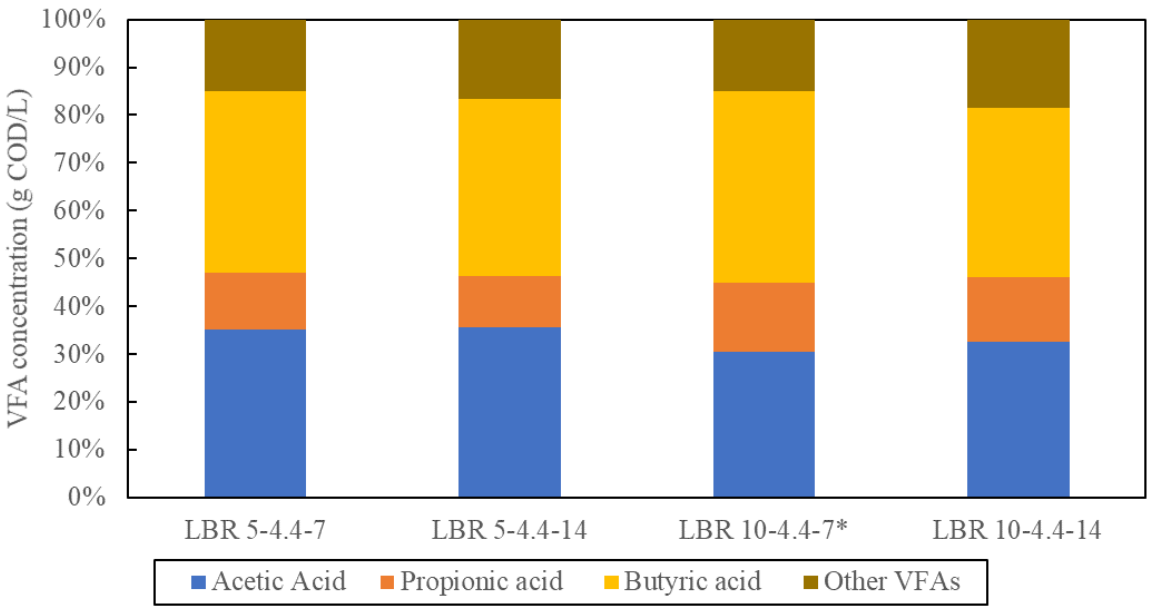


Figure 3.4: Effect of reaction time on VFA distribution in leachate.

Figure 3.5 shows LBR performance at different leachate recirculation rates from 4.4 to 13.2 L/hr; the LBR was operated at fixed ISR 10% and reaction time 7d. TSS and VSS concentrations were stable for $LBR_{10\text{-}4.4\text{-}7}$ (leachate circulation rate 4.4. L/hr), while they dynamically changed for $LBR_{10\text{-}8.8\text{-}7}$ and $LBR_{10\text{-}13.2\text{-}7}$ probably due to clogging events in the food basket caused by higher solid content in leachate and more frequent leachate recirculation. Decrease in VS reduction to increasing leachate recirculation rate, in Table 3.6, also suggests the clogging issue. Higher leachate circulation rates improved fermentation reaction rate in leachate. Hydrolysis yield increased by 34-45% at leachate circulation rates 8.8-13.2 L/hr. VFA yield also increased by 29-50% at the higher leachate circulation rates. These results indicate the significance of leachate recirculation rates that should be optimized together with ISR and food basket design of the LBR.

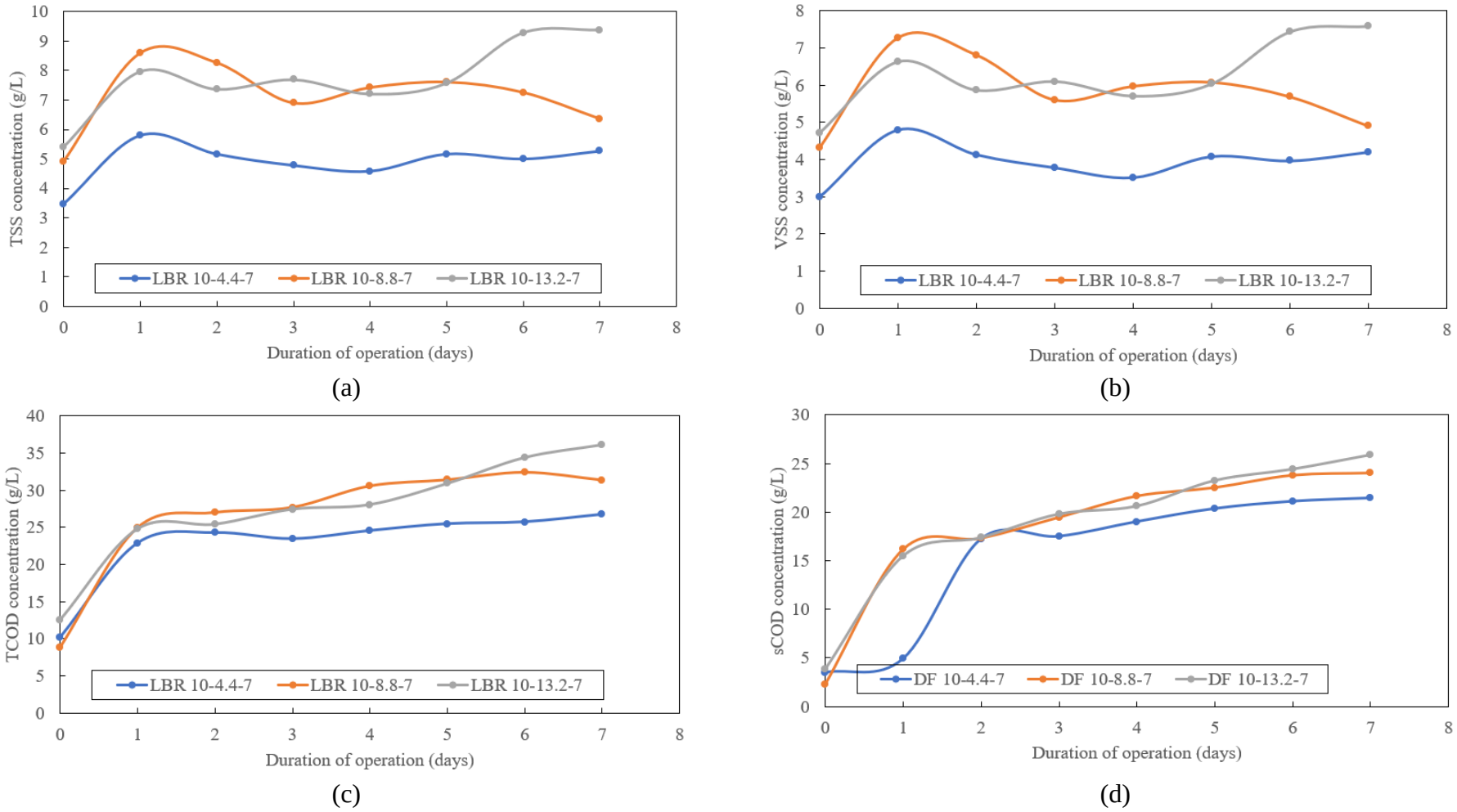


Figure 3.4: Variation of (a) TSS, (b) VSS, (c) TCOD and (d) SCOD in leachate with respect to different leachate recirculation rate.

Table 3.6: Effect of leachate circulation rate on reactor performance and leachate characteristics after the duration of operation.

Parameters	Unit	$LBR_{10\text{-}4.4\text{-}7}$	$LBR_{10\text{-}8.8\text{-}7}$	$LBR_{10\text{-}13.2\text{-}7}$
TCOD concentration	g/L	26.71±1.49	31.35±1.02	36.13±2.61
SCOD concentration	g/L	21.43±1.35	24.03±1.1	25.87±2.15
VFA concentration	g COD/L	16.62±0.78	17.95±0.59	20.3±1.28
Hydrolysis yield	g SCOD/kg VS_{added}	455.5	611.5	660.5
VFA yield	g SCOD/kg VS_{added}	363	467	544
VFA/SCOD	%	78±2	74.5±1.5	80.5±2
VS reduction	%	79±6	68.5±7	71±5

Consistent to ISR and reaction time tests, butyric acid was the largest VFA, followed by acetic acid and propionic acid, along with 12-14% other long chain fatty acids (see Figure 3.6). The VFA distributions show that leachate circulate rate does not influence fermentation pathway significantly in the LBR.

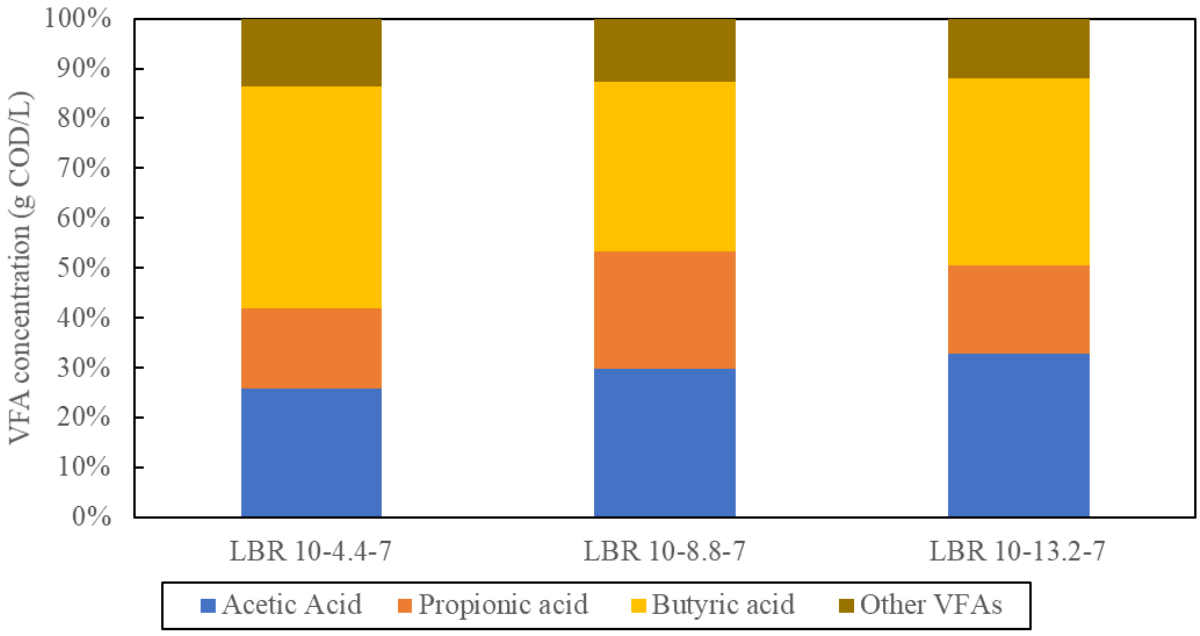


Figure 3.5: Effect of leachate circulation rate on VFA distribution in leachate.

3.3.3. Biogas production

Biogas produced from fermentation of FW comprised mainly of H_2 and CO_2. Interestingly, biogas generation almost stopped after 2 d in all experiments, implying that fermentation reactions associated with H_2 evolution would be completed within 2 d. As shown in Table 3.7, ISR and leachate recirculation rate significantly influenced hydrogen production in the LBR. Biogas and hydrogen generation were improved up to 36% and 48%, respectively, at ISR 10% where hydrolysis yield and VFA yield enhanced by 5% and 13%. This indicates that better hydrolysis and fermentation led to higher biogas and hydrogen yield. Leachate circulation rate also improved hydrogen production rate in the LBR. Increasing leachate circulation rate from 4.4 L/hr to 13.2 L/hr enhanced biogas generation rate by 6 folds. This suggests mass transport limitation of hydrogen molecules from the leachate to the headspace due to slow mixing conditions in the LBR. Hence, mass transport of H_2 should also be considered in optimization of leachate circulation rate, along with hydrolysis and VFA yield.

Table 3.7: Hydrogen production in LBR in different operating conditions.

Operating conditions	**Total biogas produced (L)**	**Average percentage of hydrogen in biogas**	**Total hydrogen produced (L)**	**Specific hydrogen yield (L H_2/kg VS_{added})**
$LBR_{5\text{-}4.4\text{-}14}$	2.37	25	0.6	3.22
$LBR_{10\text{-}4.4\text{-}14}$	3.027	23	0.69	3.68
$LBR_{10\text{-}4.4\text{-}7}$	1.33	57	0.76	4.02
$LBR_{5\text{-}8.8\text{-}7}$	4.81	45	2.16	11.6
$LBR_{10\text{-}8.8\text{-}7}$	6.55	49	3.2	19.19
$LBR_{10\text{-}13.2\text{-}7}$	8.01	41	3.27	21.15

3.3.4. COD balance

COD balance was established for $LBR_{5\text{-}4.4\text{-}14}$ (5% ISR, 4.4 L/hr circulation rate, and 14 d reaction time) using an average COD of FW_{Feb} (see Figure 3.7). Because COD balances for other conditions showed similar trends to $LBR_{5\text{-}4.4\text{-}14}$, the COD balance for $LBR_{5\text{-}4.4\text{-}14}$ was only discussed here. The average COD of FW_{Feb} was 210±15 g and 123 g of the FW COD was converted to soluble forms in the leachate accounting for 58.8% of the initial FW COD. VFAs were 42.1% of the FW COD in which butyric acid, acetic acid, and propionic acid contribute to 14.9%, 4.1%, and 16.4% of the input COD, respectively. H_2 production only accounted for 0.2% of the initial FW COD, indicating the LBR mainly transformed FW COD into VFAs, not eliminating COD. The cell growth accounted for 2.6% of the initial FW COD which is an electron sink larger than H_2 production in the LBR. Particulate FW and organisms smaller than 1.5μm but larger than 0.45 μm in leachate constituted the rest of particulate COD, 6.5% of the initial FW COD. Remaining FW in the basket was 34.3 g COD accounting for 16.3% of the initial FW COD. The COD balance showed an unknown COD gap of 15.5% probably due to some clogged leachate present at the FW slurry in the basket that might not be quantified in COD measurements. The COD balance evidently presents that the main function of the LBR is to solubilize solid FW into VFAs, the primary electron sink.

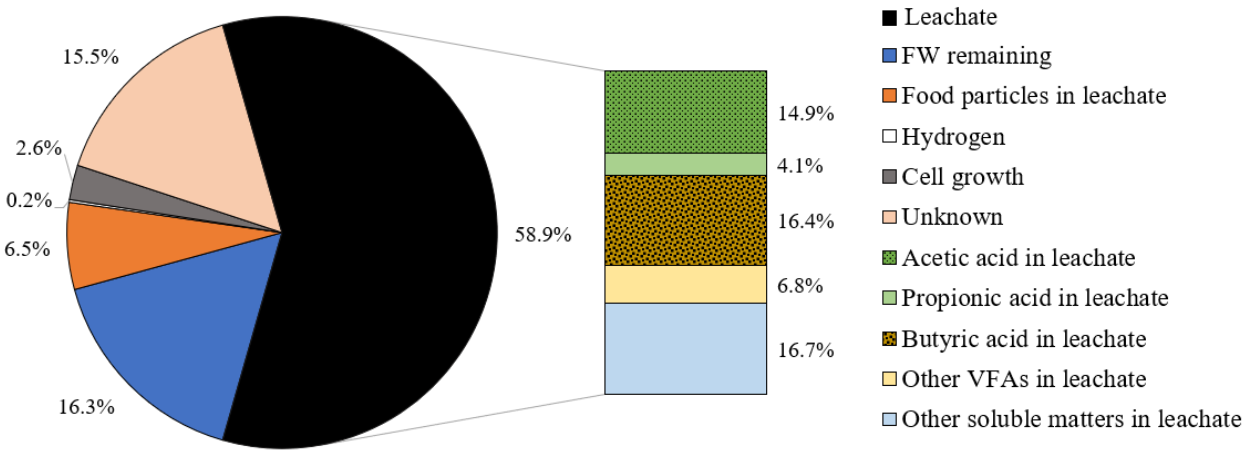


Figure 3.6: COD balance in $LBR_{5\text{-}4.4\text{-}14}$.

3.3.5. Characterization of nutrients in LBR leachate

Depending on the operating conditions, ammonium nitrogen and reactive phosphate were found to be 69-93% of TKN and 23-74% of total phosphate respectively in the LBR (see Table 3.8). Free ammonia was consistently low at 1.14-2.17 mg N/L, which means that free ammonia inhibition would not occur in the LBR (Chen et al., 2008). Leachate circulation rate had a significant impact on solubilization of particulate nutrients in FW. Solubilization of nitrogen (TKN and NH_4^+-N) and phosphorus (TP and RP) was improved up to 17 folds at a leachate circulation rate of 13.2 L/hr. ISR and reaction time also affected solubilization of solid nutrients in FW as such did in fermentation reactions. ISR 10% improved nutrient solubilization efficiency up to 13 folds, as compared to ISR 5%. Longer reaction time at 14d also doubled solubilization of solid nutrient in the LBR.

All the three parameters, ISR, leachate circulate rate, and reaction time have positive impacts on solubilization of solid nutrients. Higher ISR and leachate circulation rates shortened the reaction time needed for solubilization of particulate nutrients to a week, but at leachate circulation rate over 8.8 L/hr, hydrolysis and fermentation efficiency declined due to clogging events. It is therefore important to optimize these parameters considering both fermentation and solid nutrient solubilization factors.

Table 3.8: Concentrations of nutrients in the final LBR leachate (filtered with 0.45 μm filter).

Operating conditions	**TKN (mg/L)**	**TKN (g/kg FW)**	**NH_4^+-N (mg/L)**	**NH_4^+-N (g/kg FW)**	**TP (mg/L)**	**TP (g/kg FW)**	**RP (mg/L)**	**RP (g/kg FW)**
$LBR_{5\text{-}4.4\text{-}14}$	410±7	2068	355±15	1793	147±5	693	34±2	237
$LBR_{10\text{-}4.4\text{-}14}$	460±13	2481	360±6	2106	264±14	1427	132±6	713
$LBR_{10\text{-}4.4\text{-}7}$	245±12	1100	228±4	1026	87±4	392	22.5±1	101
$LBR_{5\text{-}8.8\text{-}7}$	334±24	1587	230±9	1093	48±2	228	11.2±0.6	53.2
$LBR_{10\text{-}8.8\text{-}7}$	480±14	2247	371±11	1736	218±8	1020	146±3	680
$LBR_{10\text{-}13.2\text{-}7}$	531±17	2938	433±18	2048	185±3	1903	137±7	1671

3.4. Conclusion

Based on the results obtained from this study, the following specific conclusions are drawn:

(a) At a short reaction time of 7d, ISR and leachate circulation rate played an important role in improving hydrolysis yield (16-45 % increase).

(b) At a reaction time of 14d, FW was well stabilized in the LBR showing high VS reduction, hydrolysis yield, VFA yield and nutrient solubilization regardless of ISR and leachate circulation rate.

(c) ISR and leachate circulation rate mainly influenced fermentation in 7d of reaction time.

(d) ISR, leachate circulation rate and reaction time did not change VFA distribution in FW leachate. The dominant VFAs were consistently butyric acid, acetic acid and propionic acid in all experiments.

(e) A high ISR of 15% clogged the FW basket completely, and high leachate circulation rate (>4.4 L/hr) at ISR 10% could cause partial clogging in the FW basket. Thus, ISR and leachate circulation rate should be optimized together to prevent clogging events.

(f) H_2 production only accounted for 0.2% of the initial FW COD, but the highest specific H_2 yield was as high as 21.15 L H_2/kg VS_{added}, indicating that energy recovery is 46 MJ per ton of FW (wet weight) added in the LBR.

(g) These results imply that not only the hydrolytic-acidogenic LBR has the potential of treating feedstocks with high solid content, like FW, but also is capable of extracting significant amounts of value-added products like hydrogen, butyric acid and acetic acid from FW with less energy consumption than conventional CSTRs.

Chapter 4 : Food waste treatment by two-stage anaerobic digestion using LBR and AnMBR

4.1. Introduction

In recent days, two-stage anaerobic digestion (AD) of food waste (FW) has gained popularity over single stage AD due to its high and stable treatability (Wu et al., 2015; Xu et al., 2012). In two stage AD, the first reactor acts as a hydrolytic-acidogenic reactor and the second one as a methanogenic reactor. Stage-separation in this process enables enrichment of target microorganisms in individual bioreactors (Kim et al., 2014; Lee et al., 2010; Shen et al., 2013; Ventura et al., 2014; Voelklein et al., 2016; Wu et al., 2015). Due to slower growth rates of acetogens and methanogens than acidogens (0.05-1.79 h^{-1} vs 0.008-0.173 h^{-1}), this staged bioprocess also increases treatment stability even at high OLRs and short HRTs than single stage anaerobic digestion (Santos et al., 2017; Xu et al., 2012; Ventura et al., 2014). Moreover, separate collection of hydrogen and methane gas in two-stage anaerobic digestion improves energy benefits from reuse of biogases (Shen et al., 2013; Voelklein et al., 2016; Wu et al., 2015).

Continuously stirred tank reactors (CSTRs) are commonly used for two-stage AD of FW but need a high capital and operating cost. In recent years, due to lower energy requirement, higher treatment capacity of solid feedstock, and solid-liquid separation capability, leach bed reactors (LBRs) have become very attractive as a hydrolytic-acidogenic reactor (Hussain et al., 2017; Li et al., 2017; Xiong et al., 2019). Though conventional CSTR-type of AD or upflow anaerobic sludge blanket can be combined with LBRs for FW treatment, anaerobic membrane bioreactors (AnMBRs) have better potential as they provide improved FW treatment, methane production and lower sludge disposal expenses than the former biotechnologies (Charfi, Ben Amar, & Harmand, 2012; Galib et al., 2016). However, there are no studies that attempt to integrate LBRs with AnMBRs for enhancing FW treatment to the author's knowledge.

To use AnMBRs in an efficient manner for treatment of FW leachate generated from LBRs, several key parameters should be studied and optimized. Firstly, hydraulic retention time (HRT) is one of the most important parameters of AnMBR operation and hence needs to be optimized (Lin et al., 2013). Although AnMBRs are known for their excellent COD removal efficiencies at short HRTs,

inadequate HRT doesn't allow microorganisms enough time to breakdown the organics. As a result, a major portion can go out of the reactor untreated. Moreover, since permeate flux is linked to HRT, too short HRT can lead to more frequent membrane fouling and reduction of membrane lifetime in the process. On the other hand, excessively long HRTs can lead to higher capital and operating cost due to larger reactors.

Secondly, solid retention time (SRT) also plays a vital role in AnMBR operation. Ideally infinite SRT (no sludge wastage) is desirable as this help microorganisms acclimatize in a particular condition without being washed out. But in reality, extensive SRT leads to higher biomass associated products, which are a part of soluble microbial products in the bioreactor. This will result in higher effluent COD and more membrane fouling (Fang, 2010). So, optimization of SRT in AnMBR is essential.

Finally, permeate flux is one of most significant factors that impacts capital and operating cost associated with an AnMBR (Bérubé, Hall, & Sutton, 2006). Maintenance cleaning of membrane is necessary to maintain desirable permeate flux due to membrane fouling events (Meng et al., 2017). Maintenance cleaning usually involves different chemical agents such as NaOCl and citric acid, which depends on type of wastewater, microorganisms, operating conditions, and so on (Wang et al., 2014; Meng et al., 2017). Therefore, optimization of maintenance cleaning to achieve higher flux is necessary to avoid extensive membrane fouling or damage to membrane and biomass (Meng et al., 2017). Although there is great significance of optimizing these three parameters in an AnMBR treating LBR leachate, there is no information on them in literature. This study aimed at systematically optimizing HRT, SRT and maintenance cleaning to achieve higher flux in an AnMBR treating LBR leachate which eventually led to optimization of the two-stage FW treatment process.

4.2. Materials and methods

4.2.1. Characterization of LBR leachate and inoculum

As discussed in Sections 3.2.1 and 3.2.3 in Chapter 3, FW leachate was collected from the LBR operated at ISR 10%, leachate circulation rate 8.8 L/hr and 13.2 L/hr, and reaction time of 7 days. The leachate was centrifuged at 9,500 rpm for 20 mins to retain the enriched microorganisms for

further LBR operations and to reduce solid loading in an AnMBR. The supernatant was stored in 4°C for around 2 weeks before using as feed for the AnMBR in this study.

AD sludge sampled from Kitchener wastewater treatment facility (Ontario, Canada) was used as inoculum to the AnMBR. The AnMBR sludge was enriched by operating the reactor in batch mode for two months with FW leachate to the sludge ratio of 1:1. Characteristics of LBR leachate and inoculum used for the AnMBR are shown in Table 4.1.

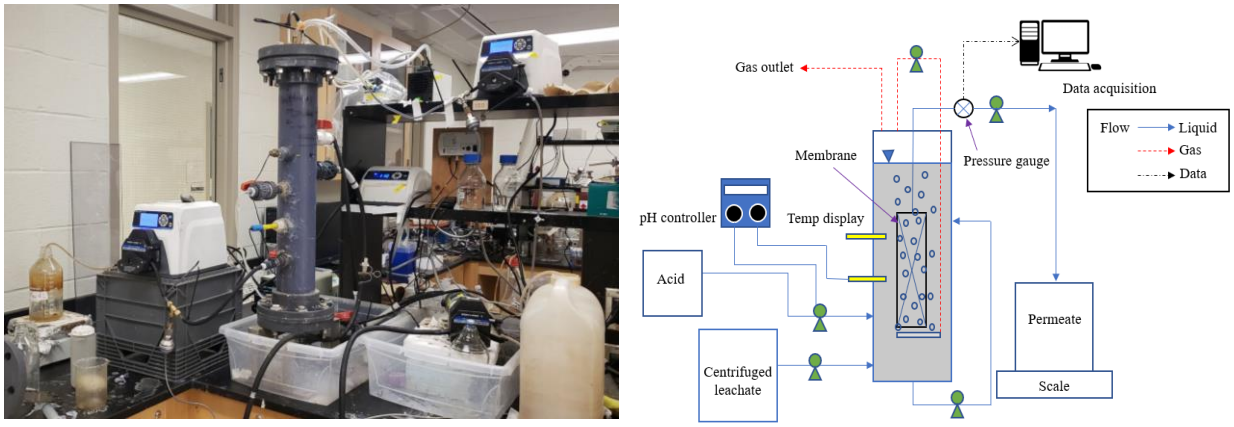
Table 4.1: Characteristics of feed and inoculum.

Parameter	Unit	Feed	Inoculum to the AnMBR
pH	-	7.91±0.33	7.11
TCOD	g/L	24.42±4.82	26.72±0.02
SCOD	g/L	21.23±4.22	0.29±0.01
VFA concentration (TVFA, HAc, HPr, HBu)	g COD/L	17.96±3.92, 6.86±1.2, 3.24±1.28, 5.82±2.12	Negligible
TSS	g/L	2.68±0.64	24.85±0.21
VSS	g/L	1.86±0.43	15.5±0.5
TKN	mg/L	502.75±142.51	-
TAN	mg/L	354.4±145.6	-
Total phosphate	mg/L	128.56±89.9	-
Reactive phosphate	mg/L	116±92.5	-

4.2.2. Reactor design

A lab-scale AnMBR was constructed with polyvinylchloride (PVC), and had an inner diameter of 10.3 cm and a height of 69 cm. The total volume of the AnMBR is 5.75 L with a working volume of 5 L. The AnMBR had five ports in sides for connections to the feed line, sludge recirculation line, and pH and temperature monitoring systems. The four openings on the top were used for biogas recirculation, and biogas and permeate production. Hollow-fibre ultrafiltration membrane

modules (BlueOcean Memtech Pte Ltd., Singapore) were immersed inside the reactor, as shown in Figure 4.1. Table 4.2 describes the characteristics of the membranes. A gas counter (MilliGas counter, Ritter Apparatus, Bochum, Germany) was attached to the top of the reactor to measure biogas production.



(a) (b)

Figure 4.1: (a) Picture and (b) Schematic diagram of the AnMBR setup.

Table 4.2: Characteristics of membranes used for the AnMBR.

Parameter	Specification
Material	PVDF
Pore size	0.04 µm
Hydrophobicity	Hydrophilic
Surface area (m^2)	0.0174 (membrane 1), 0.0024 (membrane 2)
Fibre diameter	1.2 mm (outer), 0.65 mm (inner)
Flow direction	Inside in
Fibre orientation	Vertical

4.2.3. Experimental setup

The AnMBR was operated under three phases, as shown in Table 4.3. An average SRT was maintained at 130 days in Phase 1 and 2, and it was decreased to 75 d in Phase 3 by daily withdrawing mixed liquor from the AnMBR. HRT was kept at 25 days in Phase 1, which was reduced to 13 days in Phase 2 and 3. Hence, the OLR to the AnMBR ranged from 0.75±0.11 to 2.22±0.25 kg COD/m^3/d. Feed and permeate were pumped using two peristaltic pumps (Masterflex L/S Digital Drive, 600 RPM, 115/230 VAC, Model no. 07523-80, USA). The permeate pump was programmed to operate for 4 mins for permeate production and stop operation for 1 min for membrane relaxation (no permeate production) in every 5 min. This intermittent permeate production and membrane relaxation helped to maintain the required permeate flux of 0.45 and 0.84 LMH in Phase 1 and 2 (with the first membrane module) and 6.07 LMH in Phase 3 (with the second membrane module). The permeate line was equipped with a pressure transducer (Model: 68075-32, Cole Parmer, Canada) connected to a data acquisition system (USB 6341, National Instruments, USA) to measure pressure drop and TMP in the lines using Labview SignalExpress 2017. Biogas and bulk liquid were recirculated at rates of 13 L/min and 0.45 L/min using a peristaltic pump (Model 07554-90, Cole-Parmer, USA) and an air pump (KNF N811 KVP vacuum/pressure pump, Cole-Parmer, USA), respectively, for mixing and creating a shear flow to mitigate cake formation on membranes. A pH controller (Milwaukee, MC-122 pH meter) was connected to the reactor through a pH probe to maintain a neutral pH of 7.3±0.2 by injecting 2 M HCl. The AnMBR was operated at room temperature (22°C) throughout the duration of this study, and temperature was monitored using a digital thermometer (VWR traceable thermometer, China).

Table 4.3: Operating conditions in the AnMBR.

Parameter	Unit	Phase 1	Phase 2	Phase 3
Period of operation	d	0-50	51-101	102-170
HRT	d	25	13	13
SRT	d	130	130	75
OLR	kg COD/m^3/d	0.75±0.11	2.22±0.25	1.97±0.17
Average flux	LMH	0.45±0.01	0.84±0.07	6.07±0.67
Membrane module	Number	22	22	3

4.2.4. Membrane maintenance cleaning

Membranes were chemically cleaned when the flux reduced by 20% or TMP increased over 40 kPa. Two cleaning methods were used in the study. In the first method (C1), the in-situ cleaning was conducted inside the AnMBR. This cleaning procedure consisted of four cycles: (1) citric acid 2,000 mg/L, (2) deionized water, (3) NaOCl 200 mg/L, and (4) deionized water. Cleaning agents (chemicals or water) were backflushed through the membrane modules for 40 s at 14.5 psig and the modules were relaxed for 3 min. Backwashing with deionized water was conducted to clean up the residues in the tubing and membranes. This cleaning method was used in Phase 1, 2 and first 48 days of Phase 3. The second method (C2) is an ex-situ cleaning process. First, the membrane module was removed from the reactor and cleaned in three different tanks having deionized water, 300 mg/L NaOCl and 18 M-ohm water, respectively (see Figure 4.2). Citric acid cleaning was skipped in the second method because NaOCl around 300 mg/L can effectively clean fouled membranes alone (Liang et al., 2015; Zhai et al., 2018). In this method, the fouled membrane was submerged in each tank for 10 mins and backflushed with the contents of each tank for 6 mins at a pressure of 14.5 psig.

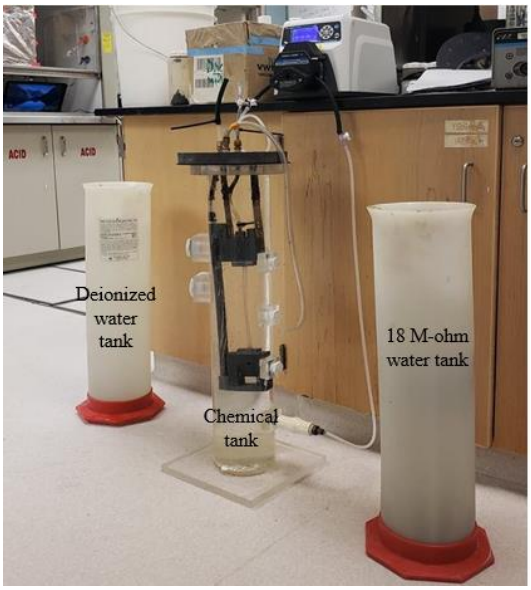


Figure 4.2: The second membrane cleaning method (C2) used in Phase 3.

4.2.5. Analytical methods

Water quality parameters for the AnMBR were quantified with the same methods described at Section 3.2.4. TSS and VSS in the AnMBR (i.e., MLSS and MLVSS), COD and VFAs were measured at least twice a week. The daily biogas composition was analyzed by injecting 1 mL of biogas into the GC-TCD. The GC-TCD was installed with a packed column (PorapakQ, 6 ft x 1/8 inches, 80/100 mesh, Agilent Tech., USA) and helium (99.999%, PraxAir, Canada) was used as the carrier gas. The oven and detector temperature were set at 41°C and 200°C respectively during the analysis. Concentrations of the nutrients in feed and permeate were only measured after steady states on three consecutive days. All the parameters were measured in triplicates and the averages are reported with standard deviations.

4.2.6. Calculation

COD removal efficiency was determined using Equation (4.1).

$$\text{COD removal efficiency (\%)} = \frac{\text{Feed COD (g/L)-Permeate COD (g/L)}}{\text{Feed COD (g/L)}} \quad (4.1)$$

Membrane flux and transmembrane pressure (TMP) were calculated with Equations (4.2) and (4.3).

$$\text{Flux (LMH)} = \frac{\text{Permeate flowrate (L/hr)}}{\text{Membrane surface area (m}^2\text{)}} \quad (4.2)$$

$$\text{TMP (kPa)} = \left[\frac{\text{Pressure}_{\text{feed}}\text{(kPa)}+\text{Pressure}_{\text{concentrate}}\text{(kPa)}}{2}\right] - \text{Pressure}_{\text{permeate}}\text{(kPa)} \quad (4.3)$$

COD balances were established in the AnMBR using a cumulative approach to track the distributions of substrate electrons, according to Equation (4.4).

Influent (g COD/d) = Permeate (g COD/d) + Methane (g COD/d) + Wasted sludge (g COD/d)

$$\text{or, } \sum (Q_F . \text{COD}_{\text{in}}) = \sum (Q_P . \text{COD}_{\text{Permeate}}) + \sum Q_{\text{Methane gas}} + \sum (Q_w . \text{COD}_{\text{ML}}) \quad (4.4)$$

where, COD_{in}= COD concentration in the AnMBR feed (g COD/L), $\text{COD}_{\text{Permeate}}$= COD concentration in permeate (g COD/L), $Q_{\text{Methane gas}}$= Daily methane production (g COD/d). Volumes of CH_4 were converted to moles of CH_4 with ideal gas law at a temperature of 22°C, and then moles of CH_4 were changed to g COD using a half reaction ($CH_4+2H_2O=CO_2+8H^++8e^-$, 1 mole e^- = 8 g COD), COD_{ML}= COD concentration of the AnMBR mixed liquor (g COD/L), Q_F= Flowrate of the AnMBR feed (L/d), Q_P= Flowrate of permeate (L/d), and Q_w= Sludge wasting rate (L/d). Then, the overall COD balances were built including the LBR at the end.

Energy balances were established by calculating energy consumption and recovery from the LBR and AnMBR. Energy consumption for the LBR was calculated using Equations (4.5) and (4.6) (Xiong et al., 2019; Kim et al., 2011).

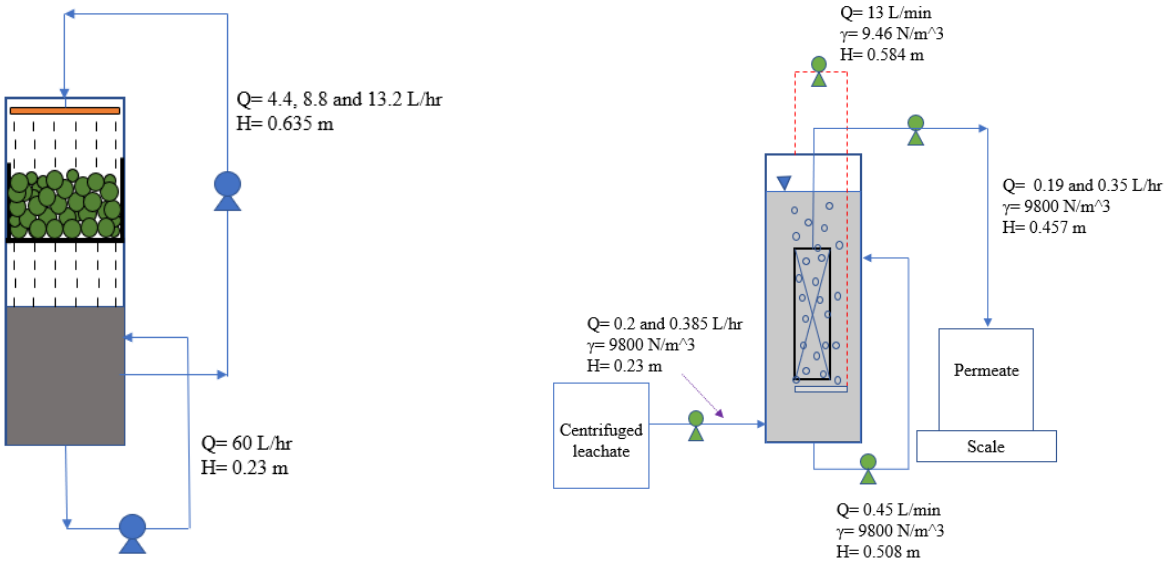
$$P = {Q\gamma H}/{1000} \quad (4.5)$$

$$E = P*t \quad (4.6)$$

where, P = Power consumption (kW), Q = Flowrate (m^3/s), γ = Specific weight (N/m^3), H = Hydraulic pressure head, E= Energy consumption (kWh) and t= Duration of operation (hr). In case of the LBR, hydraulic pressure head, H, was 0.23 m for mixing the leachate at a rate of 60 L/hr, and 0.635 m for leachate circulation at average rates of 4.4, 8.8 and 13.2 L/hr from the bottom to the food basket (see Figure 4.3a). Specific weight in this case was used 9800 N/m^3.

In case of the AnMBR, hydraulic pressure head was 0.508 m for sludge mixing at a rate of 0.45 L/min, 0.23 m for feeding at rates of 0.2 and 0.385 L/hr, 0.457 m for permeate at rates of 0.19 and 0.35 L/hr, and 0.584 m for biogas recirculation at a rate of 13.2 L/min (see Figure 4.3b). In this case, specific weight of sludge and biogas were used 9800 and 9.46 N/m^3 (73% CH_4 and 27% CO_2 at 22°C and 1 atm) respectively.

Energy recovery through hydrogen and methane generation in the LBR and AnMBR was calculated using the gross heating values (141.7 kJ/g of hydrogen and 55.5 kJ/g of methane) (Engineering Toolbox, 2017).



(a) LBR (b) AnMBR

Figure 4.3: Schematic diagram of the bioreactors with energy consumption components.

4.3. Results and discussion

4.3.1. Solid reduction and reactor stability

The AnMBR showed steady concentrations of MLSS and MLVSS, although VSS concentrations of the influent varied (see Figure 4.4). The steady state MLSS concentrations of the reactor were 2.39±0.11 g/L, 6.43±0.13 g/L and 4.9±0.18 g/L respectively, in Phase 1, 2, and 3. MLVSS concentrations were 1.64±0.06, 4.27±0.1 and 2.95±0.18 g/L giving MLVSS to MLSS ratio of

0.65±0.04 in three phases. HRT reduction in Phase 2 increased organic loading rate from 0.75±0.11 to 2.22±0.25 kg COD/m^3/d, and as a result MLSS and MLVSS concentrations amplified three folds approximately. In Phase 3, wasted sludge volumes were increased to meet an effective volume of 5 L in the AnMBR, while a high membrane flux of 6.07±0.67 LMH was kept in the bioreactor. Hence, MLSS and MLVSS concentrations decreased in Phase 3 as compared to Phase 2.

Membrane separation in the AnMBR completely removed particulate matter, which means that solid-free permeates are discharged from the AnMBR. In the two-stage process combining the LBR with the AnMBR, VS removal of food waste could be achieved as high as 79% in a short reaction time of 20 d at room temperature.

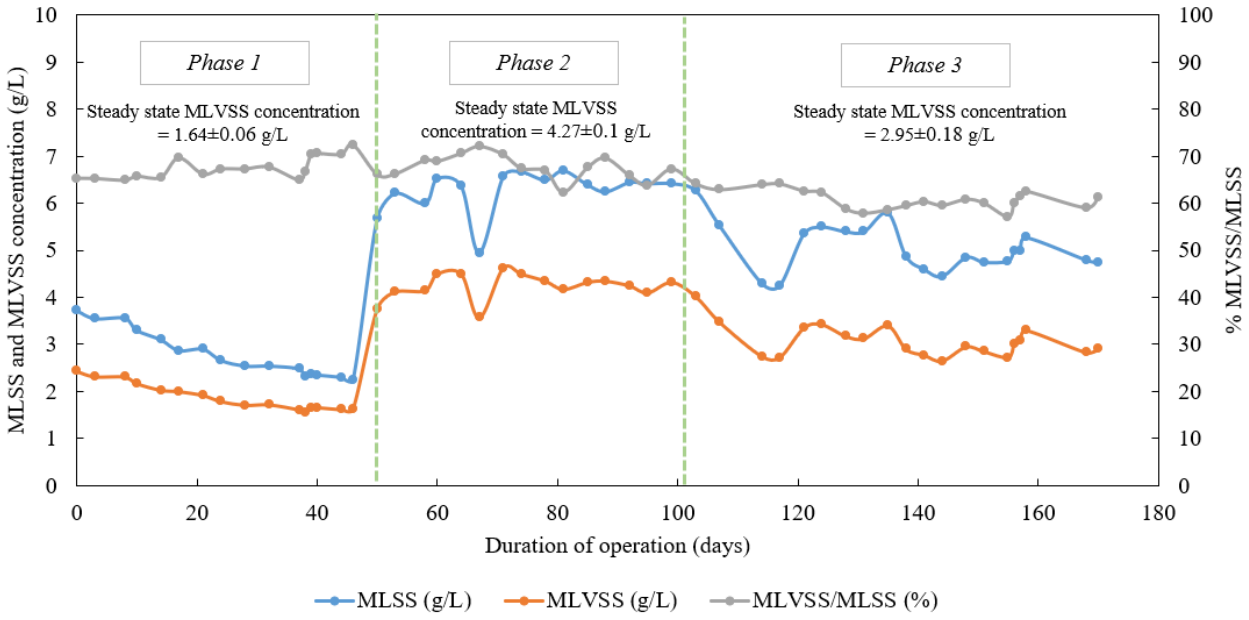


Figure 4.4: The evolution of MLSS and MLVSS concentration to time in the AnMBR.

4.3.2. COD concentration in membrane permeates

Figure 4.5 shows the COD profile of the influent and permeate. In spite of COD concentration change in the feed (LBR leachate), COD concentrations in membrane permeates were consistently low during the experiments. Average COD removal efficiencies were 90.56%, 84.29% and

86.41%, respectively. Decrease of HRT from 25d to 13d deteriorated permeate quality as shown in Figure 4.5, indicating that acetogenesis and methanogenesis would be incomplete in 13d of reaction time. However, decrease of SRT from 130 d to 75 d in Phase 3 did not affect COD removal that much as expected because SRT over 30 d is enough for acetogenesis and methanogenesis in anaerobic digestion (Trzcinski & Stuckey, 2010; Mutamim, Noor, Hassan, & Olsson, 2012; Galib et al., 2016; Halalsheh et al., 2005).

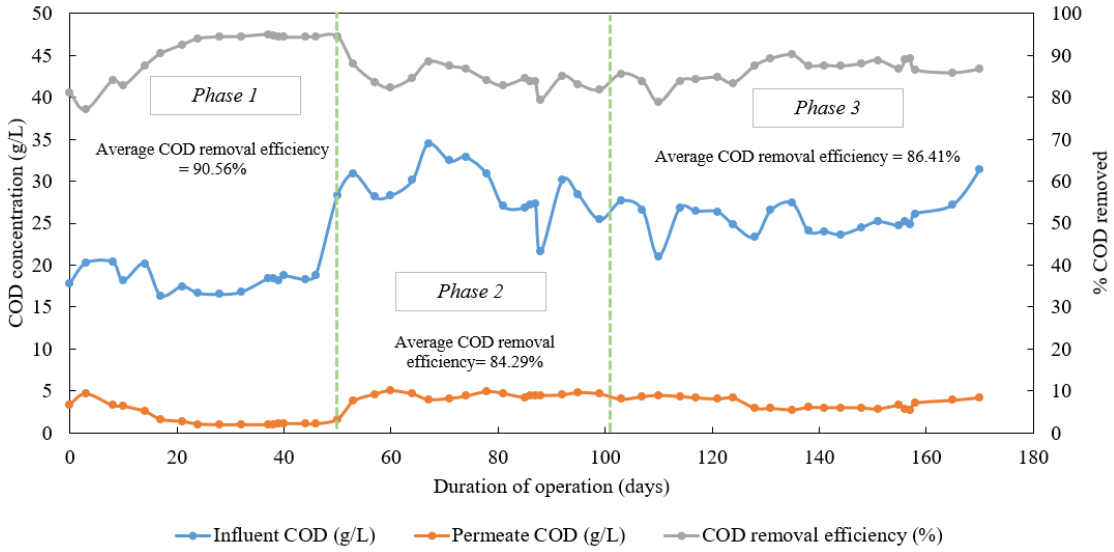


Figure 4.5: COD concentration in LBR leachates (feed) and AnMBR permeates. The leachates were centrifuged before being fed to the AnMBR (see Section 4.2.1).

4.3.3. Concentration of VFAs and nutrients in permeates

Unlike the feed, permeate had a different composition of VFAs in which acetic acid and propionic acid were dominant consistently throughout the experiments. Other volatile fatty acids such as isobutyric acid, butyric acid, isovaleric acid, valeric acid and isocaproic acids were also present but in very low concentrations in the permeates. Figure 4.6 shows the changes of VFAs to operating conditions. Acetic acid was the dominant VFA in Phase 1, but propionic acid increased and predominated in Phase 2 and Phase 3 where TVFA concentration also increased. This result suggested that acetogenesis converting propionate into acetate and H_2 would be limited in Phase

2 and 3 having a shorter HRT of 13 days. In 27 days of Phase 3 (Day 128), the concentrations of TVFA and propionic acid became steady, but they tended to increase again after day 160. Hence, it seems that more study is required to conclude AnMBR performance at Phase 3.

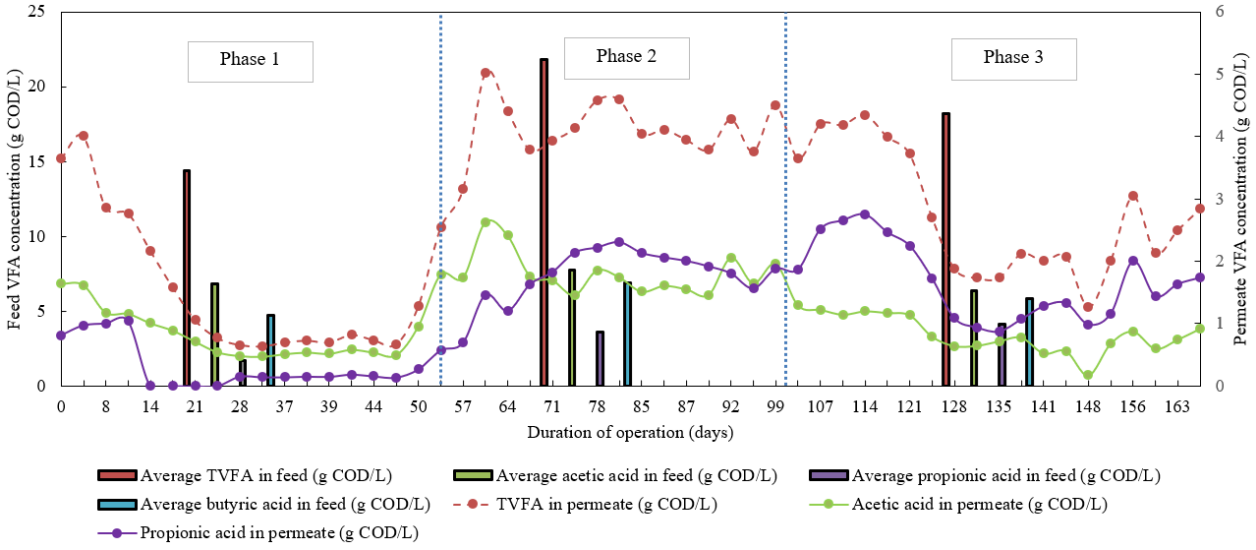


Figure 4.6: The profiles of TVFA, propionic acid, and acetic acid concentration in the feed and permeate from the AnMBR.

Concentrations of different nutrients in permeate such as NH_4^+-N, total phosphate and reactive phosphate were found to be very close to the feed concentrations which indicated that nutrient removal was small in the AnMBR (see Table 4.4). However, reduction in TKN concentration in membrane permeates would be related to nitrogen incorporation to bacteria cell after hydrolysis: synthesis of new bacteria cells (Rittmann and McCarty, 2001).

Table 4.4: Concentrations of nutrients in feed and permeate.

Nutrient	Phase 1		Phase 2		Phase 3	
	Feed	Permeate	Feed	Permeate	Feed	Permeate
TKN (mg/L)	549±6	384±12	279±14	160±5	605±43	473±9
NH_4^+-N (mg/L)	420±14	317±10	165±13	124±13	479±30	403±11
Total phosphate (mg/L)	18±3	23±6	143±10	111±6	224±5	213±2
Reactive phosphate (mg/L)	10±3	20±1	116±12	110.5±6	223±6	211±2

4.3.3. Biogas production

In the AnMBR, average methane productions in three phases were observed to be 0.8±0.12, 2.86±0.52 and 2.95±0.43 L/d, respectively, accounting for 68-78% of the biogas (see Figure 4.7). Average specific methane yields ranged from 0.24±0.05 to 0.3±0.05 L/g $COD_{removed}$ in three phases and showed higher specific methane yields in Phases 2 and 3. This result implies that more substrate was available at these phases. Influent concentrations of COD and TVFA in the Phases 2 and 3 were higher than in Phase 1 (see Figures 4.5 and 4.6). Change in SRT from 130 to 75 d did not affect the methane production rate and the specific methane yield, suggesting that methane production via endogenous decay was negligible at the AnMBR. Considering all the biogases (H_2 and CH_4), the combined process of LBR and AnMBR generated up to 7,500 L of H_2 and 87,840 L of CH_4 per ton of FW treated, approximately 907 kWh per ton of FW treated.

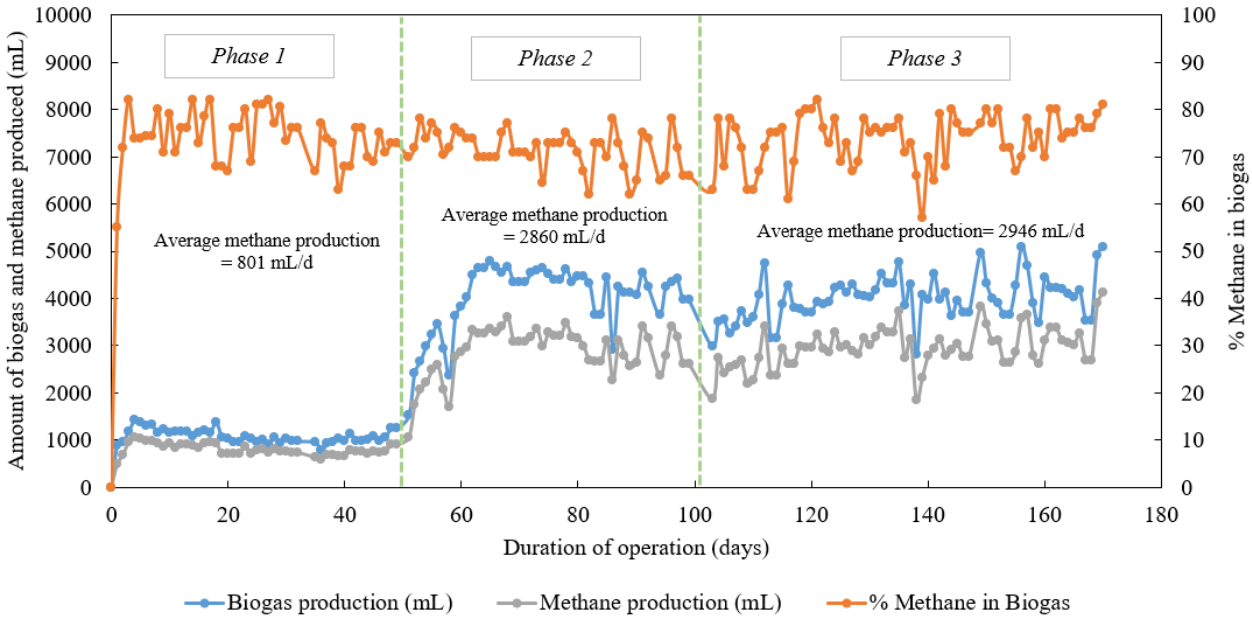
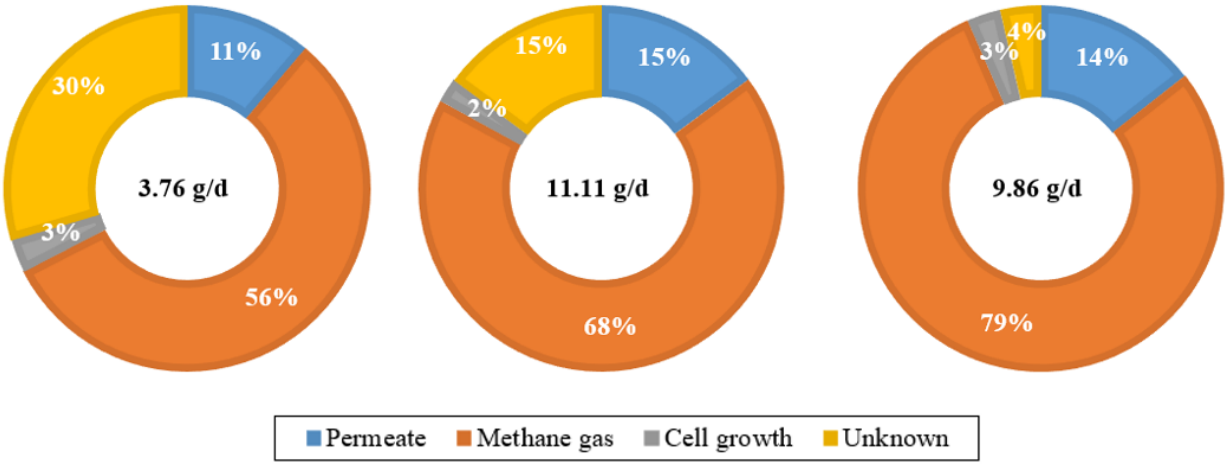


Figure 4.7: Biogas and methane generation in the AnMBR operated under three phases.

4.3.4. COD balances in the AnMBR and the overall process

Figure 4.8 illustrates the COD balances in the AnMBR for Phase 1, 2 and 3. The input CODs in the AnMBR were 3.76 g/d, 11.11 g/d and 9.86 g/d respectively in three phases among which gaseous methane production was found to be the largest electron sink (56%, 68% and 79% of input COD). Biomass or cell growth accounted for approximately 2-3% of input COD. Permeate contained around 0.42 g COD/d, 1.65 g COD/d and 1.42 g/d which were 11%, 15% and 14% of input COD respectively. However, unknown COD parameters were 4-30% of input COD, and could not be identified. Future research is required to identify the unknown electron sinks.



(a) Phase1 (b) Phase 2 (c) Phase 3

Figure 4.8: COD balances in the AnMBR.

COD balance of the overall process of the LBR and AnMBR was also established. COD balance was built for the LBR run at ISR 10%, leachate circulation rate 13.2 L/hr and reaction time 7 d. Regarding the AnMBR, COD balance was conducted for the conditions of HRT 13 d and SRT 75 d. As shown in Figure 4.9, input COD was 353±25 g, the initial COD of FW. It is evident that the largest known electron sink was methane, accounting for 28.7% of the input COD. Cell growth in the two bioreactors was 8.99% of input COD, making it the second largest electron sink. Generation of H_2 in the LBR was small at 0.612% of input COD. Remaining FW in the LBR and liquid in permeate and wasted sludge from the AnMBR contributed to 35.4% and 5.26% of initial FW COD, respectively. Centrifugation of the solids back to the LBR for further operation and loss during storage accounted for 8.03% of initial FW COD; this was calculated from the difference between average TCOD of the AnMBR feed and increased TCOD of LBR leachate after 7d of reaction time. However, the presence of dissolved methane in membrane permeate, accumulation of biomass on reactor wall or membrane surface in the AnMBR, and some of clogged FW slurry in the FW basket might accounted for the COD gap of 13%.

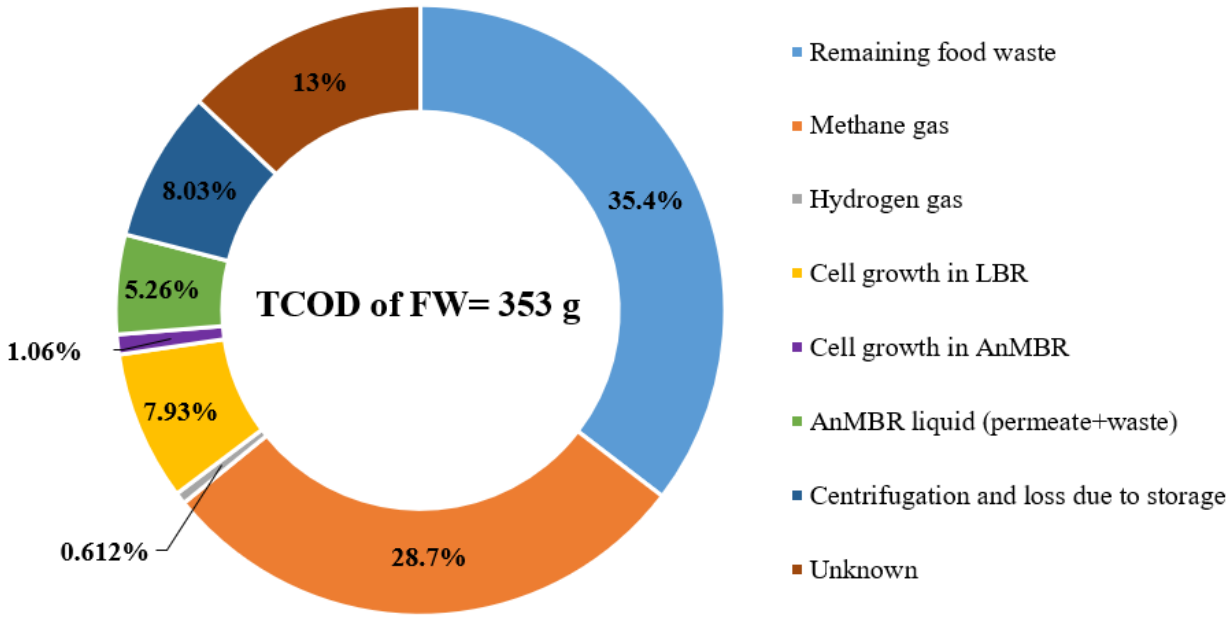


Figure 4.9: COD balance in the overall process of the LBR and AnMBR. The COD balance was established for the LBR operated at ISR 10%, leachate circulation rate 13.2 L/hr and reaction time 7d. The AnMBR operated at HRT 13d and SRT 75d (Phase 3).

4.3.5. Energy balances

Table 4.5 summarizes energy consumption and production in FW treatment using the LBR and AnMBR. Energy input to the combined process ranged from 64 to 95 kWh/ton $FW_{treated}$ depending on the operating conditions of the two reactors. This calculation also included the energy consumption of the centrifuge used to retain the solids in the LBR, which is assumed to be 1.3 kWh per m^3 of LBR leachate (Huber Technology, 2019). However, energy consumption can be compensated by the energy recovery from H_2 and CH_4 generated in the LBR and AnMBR (see Figure 4.10).

Highest energy benefit of 841 kWh/ton $FW_{treated}$ was computed when the LBR was operated with leachate circulation rate of 13.2 L/hr and reaction time of 7 d, and the AnMBR with HRT of 13 d. But partial clogging and lower permeate quality should be addressed along with the maximum energy recovery. Hence, it is important to optimize the energy benefit with the system stability and treatment efficiency of FW. Considering both the factors, the LBR with ISR 10%, leachate

circulation rate 4.4 L/hr and reaction time 7d, and the AnMBR with HRT of 13 d can be considered optimal. In this operating condition, COD and VS removal efficiencies were ~80%, resulting in a positive net energy benefit of 751 kWh/ton $FW_{treated}$.

Table 4.5: Net energy benefit from two-stage digestion of FW treatment.

Case	Operating condition of LBR (Leachate circulation rate, reaction time)	Operating condition of AnMBR (HRT)	Total energy consumption (kWh/ton $FW_{treated}$)	Total energy recovery (kWh/ton $FW_{treated}$)	Net energy benefit (kWh/ton $FW_{treated}$)
1	4.4 L/hr, 14 d	25 d	64	296	232
2	4.4 L/hr, 7 d	25 d	77	429	352
3	8.8 L/hr, 7 d	25 d	85	472	387
4	13.2 L/hr, 7 d	25 d	91	492	401
5	4.4 L/hr, 7 d	13 d	54	805	751
6	8.8 L/hr, 7 d	13 d	61	872	811
7	13.2 L/hr, 7 d	13 d	66	907	841

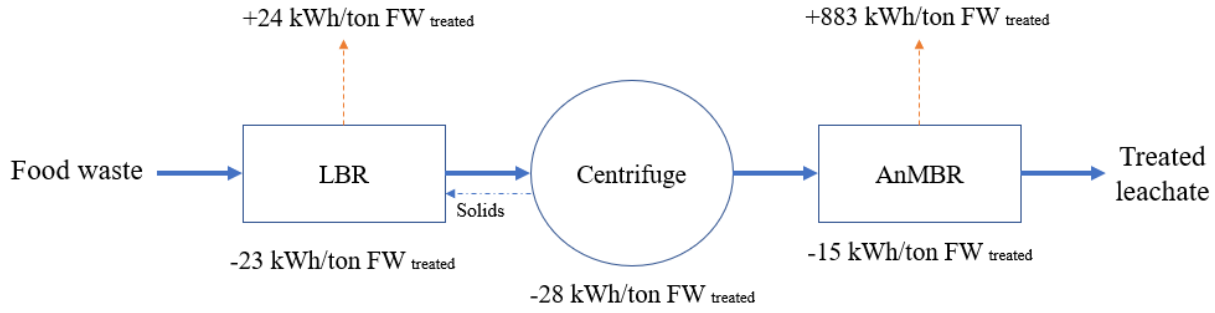


Figure 4.10: Schematics of the entire process with energy consumption and recovery components for LBR (Leachate circulation rate 13.2 L/d and reaction time 7 d) and AnMBR (Phase 3). The positive and negative values indicate energy recovery and energy consumption respectively.

4.3.6. Membrane flux and stability

The average fluxes were 0.45±0.01, 0.84±0.01 and 6.13±0.65 LMH, respectively, for Phases 1, 2, and 3. Figure 4.11 presents the trend of flux to operating time and phases. High shear flow of biogas and low permeation rate allowed no maintenance cleaning in Phase 1 having an average flux of 0.45±0.01 LMH. In Phase 1, the increase of TMP (dTMP/dt) was 0.27 kPa/d giving an average TMP of 4.9±1.62 kPa in Phase 1 (see Figure 4.12).

When HRT was decreased to 13 d in Phase 2, average flux and TMP increased by two folds, but at the same time, fouling of membrane was observed. Membrane flux decreased from 0.87 to 0.67 LMH in the first 16 days of this phase. TMP also increased from 9.89 kPa to 18.32 kPa during this period. However, maintenance cleaning recovered membrane flux and TMP in Phase 2, as shown in Figures 4.11 and 4.12. It is noticeable that in this phase TMP increased at a rate of 0.44 kPa/day before any maintenance cleaning. dTMP/dt rose to 1.07 kPa/day after the first cleaning in Phase 2 which suggests that maintenance cleaning would not completely remove foulants from the membrane.

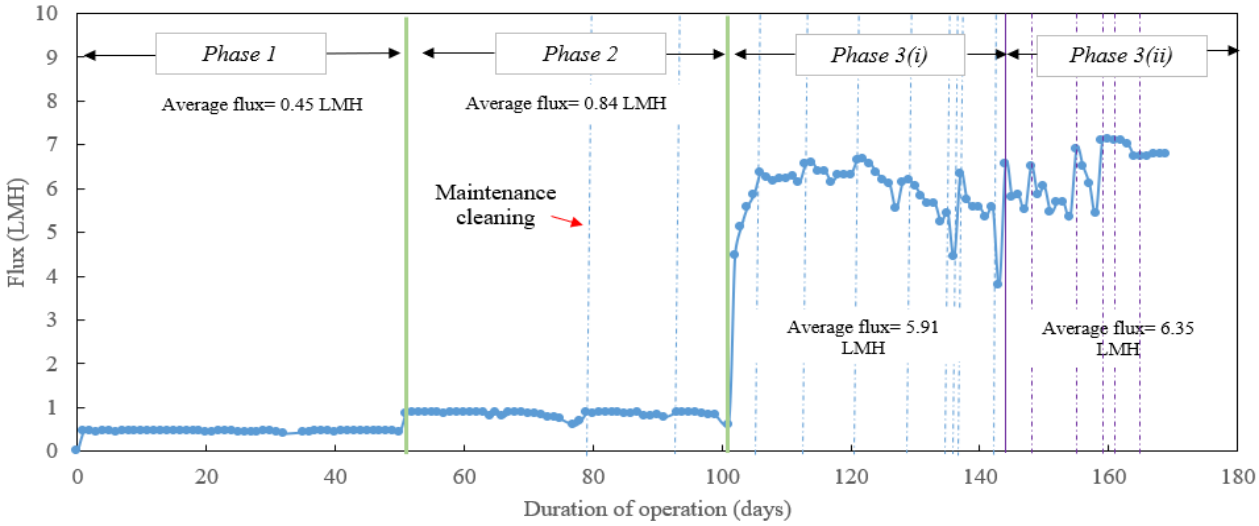


Figure 4.11: Profile of permeate flux during the AnMBR operation.

In Phase 3, the filtration unit was only switched from membrane 1 to membrane 2 keeping the HRT at 13 days. Extensive membrane fouling was observed as membrane surface area was

lessened in this phase (see Figure 4.11 and 4.12). As a result, SRT could not be maintained at 130 d, and hence was reduced to 75 days. Regular maintenance cleaning (once in every five days) was essential to maintain an average permeate flux of 6.13 LMH and TMP under the recommended value of 40 kPa. In Phase 3, two types of cleaning methods (C1 and C2) were used. Use of the second cleaning method (C2) increased membrane flux by 7% than the first one (C1). C2 was also found to be more effective in reducing the TMP to its initial value but the dTMP/dt values (7.29 kPa/d for C1 and 13.88 kPa/d for C2) suggests that it is more likely to foul faster when C2 is used. Future study is required to characterize membrane foulants in Phase 3. Figure 4.13 shows the comparative pictures of the fouled and cleaned condition of the membranes used in this study.

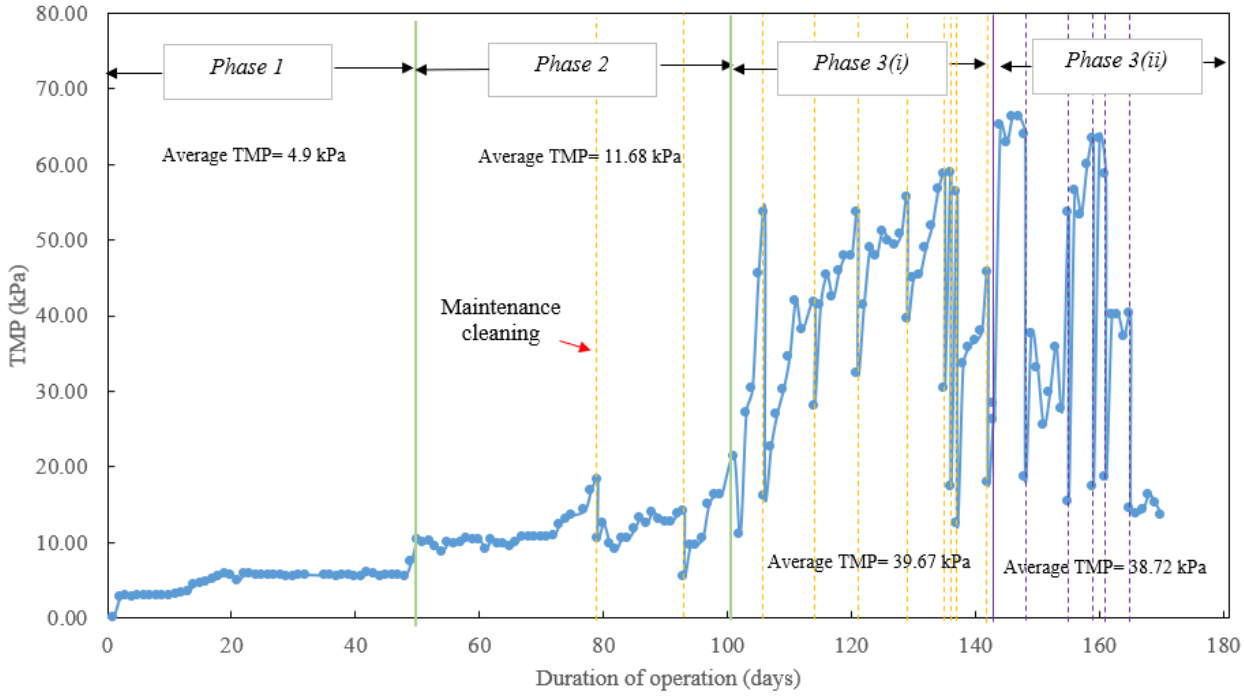


Figure 4.12: Profile of TMP during the AnMBR operation.

The cost analysis of operation and maintenance of the entire system was conducted by calculating the costs of the electricity required to operate the entire system, and the chemicals used for pH control and membrane cleaning. Table 4.6 describes operation and maintenance costs for the LBR and AnMBR; LBR operation at ISR 10%, 13.2 L/hr of leachate circulation rate and reaction time of 7d, and AnMBR operation at HRT 13d and SRT 75d (Phase 3), as an example. FW treatment

using the LBR and AnMBR was found $58 per ton of FW treated, which is $36-71 less than the cost in conventional AD facility ($94-129/ton $FW_{treated}$) (Arsova, 2010; G., 2008; Kelleher, 2007). Electricity and chemicals costs contributed to 15% and 85% of the total cost, respectively.

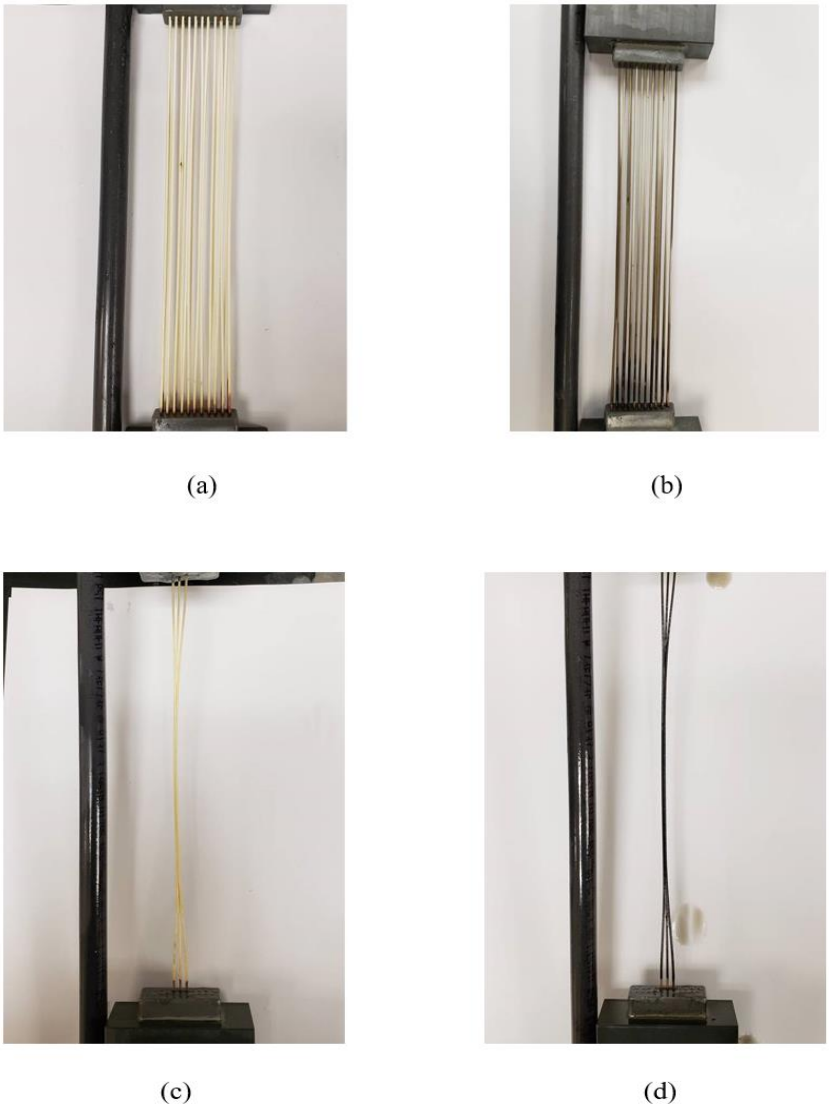


Figure 4.13: Comparative pictures of the membranes. (a) and (b) show cleaned and fouled conditions of membrane 1; (c) and (d) show cleaned and fouled conditions of membrane 2 respectively.

Table 4.6: Cost analysis for operation and maintenance of the entire process with LBR at ISR 10%, 13.2 L/hr of leachate circulation rate and reaction time of 7d, and AnMBR at HRT 13d and SRT 75d (Phase 3).

Reactor	Item	Units used	Unit cost	Total cost
LBR	Electricity	23 kWh	13.2 ¢/kWh[c]	$3.04
	$Ca(OH)_2$	82 kg	$197/ton[d]	$16.2
Centrifuge	Electricity	13.5 kWh	13.2 ¢/kWh[c]	$1. 8
AnMBR	Electricity	28 kWh	13.2 ¢/kWh[c]	$3.7
	35% HCl	126 kg	$264/ wet ton[e]	$33
	12.5% (w/v) NaOCl	0.22 L	$1.04 /gal[f]	$0.06
	Citric acid	278 g	$924/ton[g]	$0.26
Total cost per ton of FW treated				$58

c-g: The rates were obtained from Ontario Energy Board (2019), Ober (2018), Bowen (2017), Novak et al. (2011), Ciriminna et al. (2017) respectively. All the parameters are shown for per ton of FW treated. The prices are in Canadian dollars.

4.4. Conclusion

Based on the results discussed herein, the specific conclusions are as follows:

(a) VS reduction efficiency in the LBR and AnMBR was found up to 88%.

(b) COD removal was as high as 90.56% in the AnMBR, and the lowest permeate COD concentration was 990 mg/L in the AnMBR operated at HRT of 25 days and OLR of 0.75 kg COD/m^3/d.

(c) Acetic acid and propionic acid were dominant VFAs in membrane permeate. At longer HRT and SRT, higher acetic acid concentration was identified in membrane permeate, whereas shorter HRT tended to increase propionic acid.

(d) Biogas (H_2 and CH_4) produced from the LBR and AnMBR can provide net energy benefit up to 907 kWh/ton $FW_{treated}$. Considering both energy recovery and treatment efficiency, the optimum conditions are: ISR 10%, leachate circulation rate 4.4 L/hr and reaction time

7d for the LBR, and HRT of 13 d, OLR of 1.97±0.17 kg COD/m^3/d and SRT 75 d for the AnMBR.

(e) C2 method (membrane cleaning with deionized water, 300 mg/L NaOCl and 18 M-ohm water) was more effective for permeate flux recovery in the AnMBR run at ~6 LMH of membrane flux, but may cause faster membrane fouling than C1 method. Both methods should be carried out at least once in every five days to keep the flux.

Chapter 5 : Conclusions

This study aimed to assess the performance of two-stage food waste treatment using the leach bed reactor followed by the AnMBR operated at room temperature (22°C) and neutral pH. This study systematically evaluated the effects of ISR (5-10%), leachate circulation rate (4.4-13.2 L/hr), and reaction time (7 and 14d) on FW fermentation in the LBR and, optimized HRT (13-25d), SRT (75-130d) and maintenance cleaning in the AnMBR treating FW leachate. In all conditions, this innovative process improved treatment efficiencies and energy benefits through generation of hydrogen and methane.

The specific conclusions are as follows:

Effects of ISR, leachate circulation rate and reaction time on FW fermentation in the LBR:

FW was better stabilized at reaction time of 14 d than 7 d in the LBR showing high VS reduction, hydrolysis yield, VFA yield and nutrient solubilization regardless of ISR and leachate circulation rate. Though ISR and leachate circulation rate also influenced fermentation, ISR and leachate circulation rate should be optimized together to prevent clogging as a high ISR of 15% and leachate circulation rate over 4.4 L/hr at ISR 10% caused clogging events in the LBR. However, in no case, VFA distribution changed in FW leachate. The dominant VFAs were consistently butyric acid, acetic acid and propionic acid in all experiments.

Optimization of the two-stage FW process:

Highest COD removal was found to be 90.56% in the AnMBR with the lowest permeate COD concentration being 990 mg/L in the AnMBR operated at HRT of 25 days and OLR of 0.75 kg COD/m^3/d. Acetic acid and propionic acid were dominant VFAs in membrane permeate throughout the experiment. However, at longer HRT and SRT, higher acetic acid concentration was identified in membrane permeate, whereas shorter HRT tended to increase propionic acid. To maintain a membrane flux of ~6 LMH, maintenance cleaning was carried out at least once in every five days.

Highest VS reduction efficiency in both LBR and AnMBR was found to be 88±2%. Biogas (H_2 and CH_4) produced from the LBR and AnMBR can provide net energy benefit up to 907 kWh/ton

$FW_{treated}$. This implies that upto 1,088 kWh/year can be recovered from the amount of treated FW, an average Canadian household is disposing annually (Statista, 2019). However, energy recovery needs to be optimized with treatment efficiency. Considering both energy recovery and treatment efficiency, the optimum conditions were found to be ISR 10%, leachate circulation rate 4.4 L/hr and reaction time 7d for the LBR, and HRT of 13 d, organic loading rate 1.97±0.17 kg COD/m^3/d and SRT 75 d for the AnMBR.

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