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Anaerobic co-digestion of food waste and kraft pulp fibre to enhance digestate dewaterability

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Table of Contents

List of tables

List of figures

List of abbreviations

AD – Anaerobic digestion

AnCoD – Anaerobic co-digestion

BMP – Biochemical methane potential

BR – Biogas reactor

CH_4 – Methane

CO_2 – Carbon dioxide

C/N – Carbon to Nitrogen ratio

CST – Capillary suction time

CSTR – Continuous stirred-tank reactor

FW – Food waste

GHG – Greenhouse gas

H_2S – Hydrogen Sulphide

HW – Hardwood pulp fibre

HRT – Hydraulic retention time

LCFAs – Long Chain Fatty Acids

MD – Main digestion

MSW – Municipal Solid Waste

OLR – Organic loading rate

OM – Organic matter

PD – Post digestion

PF/PFD – Pulp fibre/Pulp fibre digesters

SD – Standard deviation

SW – Softwood pulp fibre

TS – Total solids

VFAs – Volatile fatty acids

VS – Volatile solids

ABSTRACT

Digestate produced during anaerobic digestion of food waste is recognised as a good alternative to mineral fertilizer which could also be used to amend soil properties. This has conventionally been applied directly and unprocessed to nearby farms or processed and transported elsewhere. The latter option has gained recognition due to environmental restrictions coupled with soil nutrient management objectives but is an expensive venture. With increasing biogas production and AD plants across Europe, production of digestate has however exceeded its demand. Improving the dewaterability of the digestate has the benefit of reducing the cost and time of processing and handling. The principal aim of this experiment was to enhance the dewaterability of food waste digestate by the addition of pulp fibre to the AD process. In doing so, the study also investigated the effect of co-digestion of food waste and pulp fibre on the performance and stability of the digestion.

Source separated food waste was digested at OLR of 3.5 ±0.1 g VS/L*d^{-1} for 163 days in 3 CSTRs with a working volume of 6L at HRT OF 23-26 days. Soft- and hardwood pulp fibres were added to 2 designated digesters for 104 days and increased stepwise at OLR of 0.5 ±0.1 g VS/L*d^{-1} PF until 1.5 ±0.1 g VS/L*d^{-1} PF with the 3rd digester serving as a control. 3 other post-digesters, each with a working volume of 1.41L were operated for 104 days with sludge from the 3 main digesters serving as inoculum and substrate. This was run at HRT of 7 days.

Pulp fibre addition of 1.5 ±0.1 g VS/L*d^{-1} OLR to 3.5 ±0.1 g VS/L*d^{-1} food waste increased the total biogas and methane production to 35-40% and 21-32% respectively. Though recording a higher biogas production, the corresponding specific methane production from the fibre addition was 12-8% lower than food waste digestion. Analysis of the digestate from post digestion showed that CST increased linearly from 595 ±13s for food waste digestate to 962 ±19s for pulp fibre addition. The experiment established a positive correlation between CST and organic matter content. Suspended solids increased from 128 ±10 mg/l for FW digestate to 177 ±12 – 237 ±10 mg/l for fibre addition. Addition of kraft pulp fibre types did not enhance the dewaterability of the digestate. However, the total methane production was enhanced by the addition of pulp fibre.

KEYWORDS: Anaerobic digestion, Digestate, Dewaterability, Food waste, Pulp fibre

1. INTRODUCTION

Energy is a vital resource for economic growth and development. However, over dependence on fossil energy is a great environmental concern owing to the pollution associated with it. Stakeholders including local authorities, business and researchers advocate for a transition towards sustainable energy alternatives to protect the environment and forestall the depletion of the planet's finite resources. Renewable energy sources have emerged prominently in the 21st century as an alternative approach to limiting the over-exploitation of carbon polluting fossil energy. One notable renewable energy source is bioenergy which is harnessed from biomass. Biomass refers to all biodegradable organic matter from living things which is readily available and accessible (Abbasi and Abbasi, 2010). The production of biofuels from biomass is on the ascendancy resulting to massive interest and investments from both industry and policy makers (Liu *et al.*, 2017). Bioenergy could be extracted from sources including municipal waste, agricultural waste, crop residues, wood processing residues, food waste, sewage etc (Yuanchun, 2013; Pagés, Jhosané and Díaz, 2015).

The growing demand for renewable energy streams to supplement humanity's current energy needs brings to light the role of bioenergy in reducing the dominance of fossil fuel in the global energy mix. Amongst the existing bioenergy technologies, biogas produced during anaerobic digestion of organic material has proved instrumental particularly in substituting fossil fuel in automobile use, heating (CHP) and electricity production (Mao *et al.*, 2015; Sawatdeenarunat *et al.*, 2016). There is also the added benefit of digestate production that could supplement mineral fertilisers (Vázquez-Rowe *et al.*, 2015). The gas production is however eclipsed by a myriad of challenges including its profitability and marketability (Ammenberg and Feiz, 2017). Feedstock characterisation and available technologies constitutes some the major setbacks to unearthing the true potential of biogas production (Sawatdeenarunat *et al.*, 2016). The energy potential of biogas is dependent on the feedstock choice, making it a key determinant of gas quality which equally affects its profitability. Anaerobic digestion (AD) of cheap and readily available materials such as organic waste is considered a cost-effective approach in addressing this dilemma. Many are the advantages in promoting AD as a sustainable energy pathway for a sustainable society including waste management, greenhouse gas (GHG) mitigation, energy and bio-fertilizer production as well as improving the environment and public health (Ahlberg-Eliasson *et al.*, 2017).

Waste management however, is one of the greatest environmental challenges in the Anthropocene (Shane, 2009). The rise in municipal solid/sewage waste (MSW) generation has been attributed to rapid urbanisation and economic growth (Kaza *et al.*, 2018). These phenomena are expected to increase with increasing world population size (Hertel and Baldos, 2016). According to the Kaza *et al.* (2018) about 2 billion tonnes of municipal waste is generated annually. Landfilling, incineration and composting have been the familiar waste treatment strategies often resulting to pollution and degradation of the biosphere (Xu *et al.*, 2018).

1.1 Problem statement

Increasing food waste (FW) generation is gradually becoming a global menace according to Svanes and Johnsen (2019). The problem is partly due to the burgeoning global population which is expected to reach 9 billion by 2050 (Berners-Lee et al., 2018). The Food and Agricultural Organization (FAO) estimates that about 20-30% of food produced globally is lost or wasted along the commodity supply chain (FAO et al., 2019). In the EU, an estimated 88 million tonnes of food waste is generated annually across its 28 members states (Eurostat, 2018). Until recently, a predominant section of the waste generated was indiscriminately disposed of in landfills. Though this approach to municipal waste management (MSW) is totally prohibited in Sweden and Germany (Nghiem et al., 2017), Sauve and Van Acker (2020) reported that about 23% of the 252 million metric tonnes of municipal waste generated in the rest of Europe during 2018 was landfilled. This is problematic for the environment as the unabated degradation of organic matter (OM) in the open contributes to GHG emissions globally resulting to an estimated 8% anthropogenic GHG contribution. Biodegradation of OM for example, in landfills and other exposed environments produces methane (CH_4) and carbon dioxide (CO_2) (Stan et al., 2018). CH_4 however, is considered a powerful GHG according to Loução, Ribau and Ferreira (2019) and is 28-36 times more effective than CO_2 in trapping heat in the atmosphere and contributing immensely to global warming as reported by the IPCC (2014).

Several remedies have been proposed to help mitigate FW generation, disposal and its harmful effect on the environment. As such, the UNs sustainable development goals (SDG) target 12.3 advocates for 60% reduction in food loss and waste as a drive to eliminate hunger by 2030 (UN General Assembly, 2015). By far, the EUs directive on landfilling (1999/31/EC) targeted at reducing 35% biodegradable waste disposal by 2016 was perhaps the most incisive decision towards reducing landfilling of FW. However, the OM content of FW streams is a good resource for energy production (Labatut and Pronto, 2018). AD of OM in the absence of oxygen to produce biogas and nutrient rich digestate presents an alternative approach to dealing with the burgeoning GHG emissions from the disposable of organic waste (Ware and Power, 2016a). With AD, the harmful GHGs can be harvested to produce energy. This presents a win-win situation for both society and the environment as the bioenergy could be used to mitigate society's dependence on fossil energy and reduce GHG pollution (Ahlberg-Eliasson *et al.*, 2017). AD of FW however faces several challenges including ammonia inhibition and volatile fatty acids accumulation which limits its energy potential.

The residual digestate produced during AD contains macro and micronutrients necessary to support plant growth and improve soil properties (Alburquerque *et al.*, 2012; Barzee *et al.*, 2019). This by-product however often has a lower solids concentration and a high moisture content that limits its application, storage and transportation (Eriksson and Runevad, 2016). Though a good alternative to conventional agriculture mineral fertilizer, the added cost in handling and treating the digestate is rather a disincentive for prospective farmers. Other challenges include nutrient variability, presence of heavy metals and pathogen levels. These issues collectively impact negatively on its marketability, usefulness and patronage (Eriksson

and Runevad, 2016). There is the need to therefore reduce the moisture content of the digestate to make it easily transferable. This would improve its marketability as well culminating to lesser logistics and transport cost. One way to improve the digestates versatility would be to enhance the dewaterability, which in effect boosts the liquid-solid separation process. Existing techniques used to improve digestate dewaterability including conditioning (i.e., adding polymers, coagulants, etc.) are rather costly and energy intensive. The use of lignocellulosic materials such wood and agricultural waste to increase solids content in sludge treatment is an established procedure in literature. This is also a common waste treatment practice within the pulping and papermaking industry. Fibre sludge is usually mixed with waste activated sludge (WAS) to improve dewaterability (Ekstrand *et al.*, 2016).

The pulp and paper industry is one of the largest business establishments in Sweden accounting for about 20-30% of gross annual total investments (Larsson, 2015). Due to its vast forest reserves, the country is the third biggest producer of pulp and paper products in the world (Abbas et al., 2011; CEPI, 2020). The pulping process consumes a lot of resources including water, energy and forest biomass whilst producing a lot of waste in the process. Residue fibre sludge (primary sludge) is one of the numerous waste materials generated during the paper making process. This is made up of pulp fibre (PF), waste waters and inorganic materials. Ekstrand *et al.* (2020) reports an estimated annual 80,000 MT of total solids of fibre sludge production in pulp and paper mills across Sweden in 2017. Ekstrand *et al.* (2013) amongst other researchers have investigated the significance of using various waste streams from kraft pulp and paper industry for anaerobic digestion in terms of its methane potential. However, the effect of PF (dewatered fibre sludge) on digestates dewaterability is yet to be studied. The focus of this experimental study is to investigate whether kraft pulp fibre addition during AD of FW would enhance the digestates dewaterability. This also presents the opportunity to assess the methane potential of co-digesting FW with PF.

A plethora of conduits exist for improving the dewatering of AD digestates. However, the idea to improve dewaterability of digestate through the co-digestion of FW and PF constitutes a grey area in research. This would likely be a novel attempt to improve the dewaterability of digestate using this approach and thus presents an opportunity to advance scientific knowledge. This embodies the researcher's overall aim.

1.2 Scientific hypothesis

AD of FW and its characteristics is well documented in literature (Ammenberg and Feiz, 2017; Paritosh *et al.*, 2017; Meegoda *et al.*, 2018). Challenges with FW degradation includes the high levels of ammonia produced (owing to protein degradation) and accumulation of volatile fatty acids (VFAs), often inhibiting the process (Wang *et al.*, 2014; Meegoda *et al.*, 2018; Xu *et al.*, 2018). However, depending on the pH and temperature of the digestion system, ammonium could develop instead of free ammonia. Ammonium nitrogen has been researched to be an important nitrogen source for plants, thus making it a good characteristic of bio-fertilizer (Alburquerque *et al.*, 2012). Nonetheless, the challenges with food waste digestion could be resolved by co-digesting with other substrates (Zhang, Lee and Jahng, 2011; Hegde and

Trabold, 2019). Lignocellulosic co-substrates with high C/N ratio for example, have been investigated to limit the acidification from FW digestion thus reducing inhibitions and foaming (Xu *et al.*, 2018). Though studies on the use of kraft pulp fibre as co-substrate in FW digestion is scarce, Ekstrand *et al.* (2013) acknowledged its superiority to other lignocellulosic substrates. This is potentially due to the treatment of the fibre (with chemicals and bleach), which reduces the lignin composition and degrades the hemicellulose (Ibid.). Co-digesting the nitrogen-rich FW with carbohydrate rich PF could overcome the nutritional and C/N imbalances aching to the mono-digestion of both substrates (Alburquerque, de la Fuente and Bernal, 2012; Fisgativa, Tremier and Dabert, 2016; Lin and Li, 2017).

In previous studies on AD, several researchers have expressed the need to reduce the water content of digestate from the AD process (Ware and Power, 2016b; Ekstrand, 2019). Digestate enhancement and dewatering have the benefit of reducing the transportation, storage and handling cost, providing a better alternative to mineral fertilizers whilst enhancing the economic viability of the AD residue (Li *et al.*, 2020). Within the kraft pulping industry, primary sludge is usually mixed with waste activated sludge (WAS) to improve its dewaterability, i.e., solid-liquid separation. Pulp fibre with similar characteristics when co-digested with FW could tentatively enhance the dewaterability of the digestate. Dewaterability has been described differently in literature with reference to filterability or drainability of digestate with several indices including CST and TS of centrifuge dewatered caked (Houghton and Stephenson, 2002; Zhang *et al.*, 2018). However, the underlining mechanism involves the efficiency of the liquid-solid separation of digestate.

The arguments and exploits of these researchers point towards the use of pulp fibre to improve digestate dewaterability. Thus, it is on this basis that the study hypothesises that:

H1. The co-digestion of food waste and pulp fibre improves the volumetric biogas production and methane yield.
H2. The co-digestion of food waste and pulp fibre improves the dewaterability of digestate.

Creswell & Creswell (2018) describe the hypothesis as a testable prediction of a relationship between two or more variables. A good hypothesis must be concise, testable and allow for falsification.

1.3 Aim of the study

The overall aim of the study was to investigate the effect of AD co-digestion of FW and PF on digestates dewaterability. The goal was to improve digestate dewaterability thereby increasing its use and value on the market. Thus, the specific objective was to determine if and to what extent the PF types affect the digestates dewaterability.

The study also investigated the corresponding gas production from the addition of kraft mill PF and its effect on ammonium nitrogen concentration. To research these aims and objectives, the experiments were guided by the following research questions.

1. *What is the effect of kraft pulp fibre addition on the performance and stability of AD of food waste in terms biogas and methane production?*
2. *What is the effect of different kraft pulp fibre types on nitrogen mineralization of food waste digestion?*
3. *What is the impact of different fibre types on the dewaterability of the digestate in terms of filterability, total solids and solids suspension?*

1.4 Scope and Limitation of the study

This study is based on the idea that cellulose materials can be used to improve the dewaterability of digestate from AD. According to (Ekstrand, 2019) this is a familiar strategy within the pulping industry where fibre sludge is mixed with waste activated sludge to improve its dewaterability.

The fibres used in this experiment were bleached soft- and hardwood pulp fibre obtained from chemical kraft pulping process. The fibres were sourced directly from the product line of the pulping process and may have different characteristics than fibres from dewatered fibre/primary sludge.

The thesis project was based on laboratory analyses and due to the Covid-19 pandemic, access to the laboratory was limited. This was based on restrictions on the number of people that could be in the laboratory at the same time, thus restricting lab hours. This affected the number of experiments and observations which could have been carried out. Some laboratory tests could not be initiated until the anaerobic digestion process (AD) was operating at stable conditions, which occurred much later than expected. This was unfortunate but could not have been anticipated and is sometimes the case when dealing with biological processes.

2. THEORETICAL BACKGROUND

2.1 The Anaerobic Digestion.

Anaerobic digestion occurs naturally in oxygen depleted environments. It is also biological energy recovery pathway that involves the degradation of organic compounds in the absence of oxygen to produce biogas. When applied industrially, AD also generates a nutrient rich digestate which is a potential biofertilizer to enhance agriculture production. AD thus is described as a sustainable technology due to the wide range of biomass that could be treated including organic waste (Sawatdeenarunat *et al.*, 2016). The controlled degradation of organic waste is a viable renewable energy source with immense environmental and agricultural benefits whilst contributing to responsible waste management practices (Auer *et al.*, 2017; Paritosh *et al.*, 2017). The increasing number of AD plants operating in Sweden is a testament to the importance of the technology to fulfilling the country's renewable energy targets (Swedish Energy Agency, 2017). In 2016, an estimated 279 operating plants existed in Sweden producing 2018 GWh of biogas (Ibid).

Biogas is the main intended end-product of the AD process. Biogas has numerous applications including heating (CHP) and electricity generation (Auer *et al.*, 2017). The gas produced is a composition of methane (CH_4; 50-70%) carbon dioxide (CO_2; 25-50%) and other gases (Dung *et al.*, 2014; Grimberg *et al.*, 2015). Alternatively, biogas could be purified to produce biomethane which could be harnessed as a transport fuel (O'Shea *et al.*, 2016; Ammenberg and Feiz, 2017).

2.2 Biogas Production

2.2.1 Microbiology

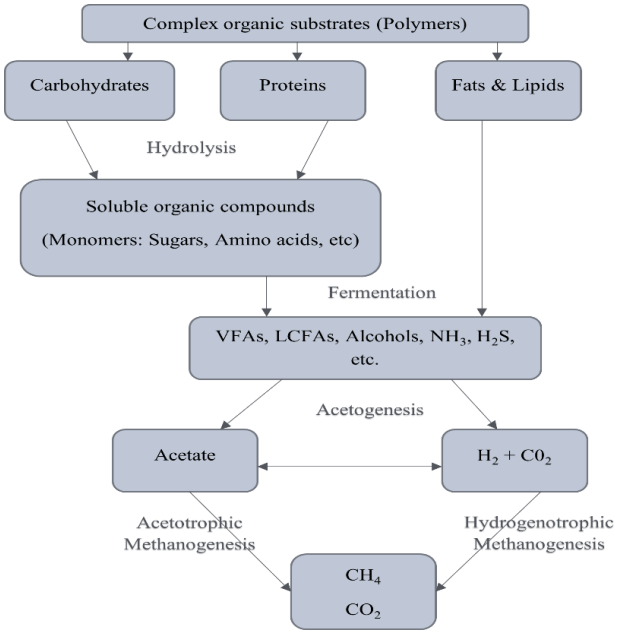
AD is a stagewise process comprised of four successive stages (figure 1) involving multiple stratified communities of microbes, thus is considered a complex process (Glass and Orphan, 2012). The microorganisms breakdown large and complex organic materials to produce the gas and nutrient rich digestate (Schnürer and Jarvis, 2018). The microbiology of the degradation describes a representation of bacteria and archaea comprising hydrolytic, acidogenic, acetogenic bacteria and methanogenic archaea (Ziels *et al.*, 2016). These group of microorganisms represent the four stages of the anaerobic digestion. The figure 1 below illustrates a simplified AD process.

Hydrolysis being the first stage of this process involves the breaking down of complex food compounds (polymers) into simpler soluble units (monomers). The complex organic compounds in the form of carbohydrates (polysaccharides), proteins and lipids come in insoluble forms which may be difficult for microbes in subsequent stages to digest. Monomers include simple sugars, peptides, amino acids, alcohol, etc. Hydrolysis is often considered a rate limiting step due to the time taken for the degradation of some polysaccharides (Lin and Li, 2017). Substrates containing polysaccharides like lignocellulose are difficult to digest and

require longer period to degrade (Ma *et al.*, 2015). This is primarily due to the lignin content which has been described by many as recalcitrant to microbial degradation (Sawatdeenarunat *et al.*, 2015; Hernández-Beltrán *et al.*, 2019). Acidogenesis also referred to as fermentation is the next stage in the biological process. Here several fermentative microorganisms use the soluble monomers as a substrate to produce organic acids (VFAs), alcohol, hydrogen sulphide, ammonia, carbon dioxide and hydrogen (Ma *et al.*, 2015). These are often referred to as intermediaries.

In the third step, acetogenic microbes degrade end products from the fermentation process to produce hydrogen gas, carbon dioxide and acetates. This could perhaps be considered the most significant step in the whole biogas process (Ma *et al.*, 2015). The balance between the amount of methane and carbon dioxide produced during the entire AD process is stringent on this process. As it depends on the close cooperation between the acetogens and the methanogens. Hydrogen gas is an important component in methane gas formation. However, its concentration in the system spurs or deters the anaerobic oxidation reactions, thus must be kept to a minimum. Aside the methane production, methanogens also consume the excess H_2 gas produced to maintain at a lower concentration. This syntrophic association between these group of organisms helps to maintain an equilibrium in the entire methane production system.

The final step involves the methanogens converting the hydrogen gas, carbon dioxide and acetate produced in the previous stage into methane and carbon dioxide. This stage is also considered rate limiting due to the slow growth of the methane producers. These organisms responsible for producing methane determine the fate of the entire anaerobic digestion. According to Schnürer & Jarvis (2018), the organisms are fragile and susceptible to system and environmental changes like pH, temperature, toxicity and inhibitions.



Modified from Appels *et al.* (2008) and (Schnürer and Jarvis, 2018)

Figure 1. A schematic representation of anaerobic digestion process

2.2.2 Parameters for AD operation

2.2.2.1 Reactor Design

Different reactor designs exist for the efficient operation of the AD process depending on certain factors. Factors such as substrate characteristics, scale of operation, processing temperature, digestion stages etc could be considered when choosing a suitable medium to house the digestion process (Logan and Visvanathan, 2019). Continuous stirred tank reactors (CSTR) is a preferred configuration in Europe (Schnürer, Bohn and Moestedt, 2016) when dealing with wet digestion of organic materials (e.g. food waste, animal manure, waste activated sludge, etc.) with low total solids content (TS ≤ 15%) (Eriksson and Runevad, 2016).

2.2.2.2 Stability and Performance Efficiency

Due to the complexity of the AD process several factors may collectively or singly limit or destabilize the intended output. The microbial activity requires a stable and controlled environment to be able to process the feedstock to produce biogas. The environment and operational conditions influence the stability and performance of the process, requiring monitoring. Parameters like pH, alkalinity, temperature, etc can affect the stability of AD. For performance this is evidenced by the substrate selection, organic composition, organic loading and retention times. As indicated by Schnürer and Jarvis (2018) specific methane yield and degree of degradation (VS% reduction) cane be used to evaluate the efficiency of the digestion process.

2.2.2.3 Substrate Selection

Literatures have opined the numerous opportunities and challenges that arise from the exploration of biogas as a renewable energy stream (Zhang *et al.*, 2014; Rosillo-Calle, 2016; Sawatdeenarunat *et al.*, 2016; Auer *et al.*, 2017). Amongst the obvious difficulties, feedstock selection is of great significance to the entire biogas process influencing the quality and quantity of the outputs (Ammenberg and Feiz, 2017; Schnürer and Jarvis, 2018). The efficiency of operation is of importance to both business and researchers as the cost of production impacts the economic viability of the gas produced. Basic decisions like substrate type and composition, mixing, C/N ratio need to be considered when choosing a substrate as they impact the methane potential. Other factors including cost and availability of the material are also paramount to the substrate selection process. Biochemical methane potential assays (BMPs) have proved instrumental in assessing the methane potential of substrates. BMP is a standard procedure used to determine the methane potential and biodegradability of organic substrates prior to large scale operations (Appels *et al.*, 2008). In the case of digestate, the choice of substrate is necessary as nutrients originally present in the feedstock is retained (or enhanced) in the digestate after anaerobic degradation. FW is thus considered a suitable substrate as it contains the necessary micro and macro nutrients needed for plant growth. Digestates from FW degradation are a potential fertilizer source due to the high phosphorus and ammonium nitrogen (NH_4^+-N) content. The presence of heavy metals such as copper and zinc however mitigate

against these benefits. Cellulosic materials like wood fibre have been documented to improve the soil's structure and enhances its water holding capacity.

2.2.2.4 Organic Composition

The organic composition of a substrate with respect to the amount of carbohydrate, protein and lipids content reflects its methane potential. These organic compounds form the basic unit of food and basis for anaerobic digestion. A balanced diet describes a meal that is rich in these compounds in a determined proportion. As important as it is from a human nutrition perspective likewise is it significant for the success of biogas production. During the AD process these complex organic polymers are first hydrolysed to produce the less complex monomers which are utilized by the microorganisms to produce biogas. Theoretically, lipids are reported to have higher methane potential (1.01 m^3/kg VS) compared to carbohydrates and proteins (0.42 and 0.50 m^3/kg VS) (Buswell and Mueller, 1952). For example, Ammenberg and Feiz (2017) reported a theoretical methane yield of 536 mL CH_4/g VS for FW composition of 19% VS lipid, 19% protein and 61% carbohydrates. Wood waste with a high and different carbohydrates composition (cellulose, hemicellulose and lignin) would have a lower methane potential per kilogram of the substrate treated under AD conditions (Monlau *et al.*, 2013). Using the stoichiometric equation (Equation 1a) it is possible to estimate the theoretical methane potential for a particular substrate based on its carbon (C), hydrogen (H), and oxygen (O) composition (Buswell and Mueller, 1952).

$$C_nH_aO_b + \left(n - \frac{a}{4} - \frac{b}{2}\right)H_2O \rightarrow \left(\frac{n}{2} - \frac{a}{8} + \frac{b}{4}\right)CO_2 + \left(\frac{n}{2} + \frac{a}{8} - \frac{b}{4}\right)CH_4 \qquad \textit{-Equation 1a}$$

This equation however only accounts for the CH_4 and CO_2 production from substrates, neglecting additional gases like hydrogen sulphide (H_2S) and ammonia (NH_3) (Meegoda *et al.*, 2018). Boylés Equation (Equation 1b) according to Nielfa, Cano and Fdz-Polanco (2015) presents a rather better estimation of products resulting from the degradation of proteins.

$$C_nH_aO_bN_xS_y + \left(n - \frac{a}{4} - \frac{b}{2} + \frac{3x}{4} + \frac{y}{2}\right)H_2O$$

$$\rightarrow \left(n - \frac{a}{4} + \frac{b}{2} + \frac{3x}{4} + \frac{y}{2}\right)CO_2 + \left(n + \frac{a}{4} - \frac{b}{2} - \frac{3x}{4} - \frac{y}{2}\right)CH_4 + xNH_3 + y\,H_2S \qquad \textit{-Equation 1b}$$

2.2.2.5 Carbon/Nitrogen Ratio

Coupled to the organic composition, carbon (C) and nitrogen (N) are the two basic and most significant elements that support microbial growth and activity during AD (Zhang *et al.*, 2014). The C/N ratio dictates the amount of carbon and nitrogen elements present in the substrate. Too high or low C/N ratio would tilt the balance of degradation leading to nitrogen deficiency or ammonia inhibition respectively (Schnürer and Jarvis, 2018). Hills (1979) reported a decreasing methane content from 67% to 51.7% with increasing C/N ratio of 8 to 51.7 respectively. The researcher thus suggested a ratio of 25 as appropriate for efficient AD performance. Other literatures suggest an optimal ratio of 15-30 (Fisgativa, Tremier and Dabert, 2016; Romero-Güiza *et al.*, 2016) as conducive for a sustained microbial functioning.

Considerations should also be given to C/N ratios in soil nutrient composition and digestates. Alburquerque, de la Fuente and Bernal (2012) argues that disproportionate C/N ratios could lead to nitrogen deficiency in crops.

2.2.2.6 Substrate Co-Digestion

Substrates have different energy potential and characteristics (Nwokolo *et al.*, 2020). As a result, mono digestion of certain substrates is difficult and complex in nature (Xu *et al.*, 2018). AD of solely cellulose and hemicellulose substrates without nutrient supplementation could lead to foaming. Also, degradation of proteins and lipids results to amino acids, ammonia and fatty acids accumulation in the reactor which may be detrimental to the operation. This is rather inhibitory to methanogenic microbes that are responsible for methane production. To overcome this setback, anaerobic co-digestion (AnCoD) of different types of substrates is favoured over single substrate digestion (Rabii *et al.*, 2019). According to Schnürer & Jarvis (2018) substrate mixing is favourable as it caters for improved buffering capacity and nutrients deficiency allowing increased organic loading. Co-digestion has the potential to enhance the quantity and quality of the gas as well as improve the digestates nutrient content (Ahlberg-Eliasson *et al.*, 2017). Degradation of FW at higher OLRs is a familiar case and is usually co-digested with other substrates. Recent studies have suggested the need to adopt AnCoD to improve the gas performance and the stability of the digestion process (Nielfa, Cano and Fdz-Polanco, 2015; Sawatdeenarunat et al., 2016). This suggestion is enforced by the assertion that co-digesting different substrates can ensure a proper C/N ratio. The substrates complement each other and cater for nutrient deficiency in others. Co-digestion has the benefit of also improving the total solids content.

2.2.2.7 Addition of Nutrients and Trace Elements

Additional nutritional requirements are usually needed to boost microbial and enzymatic activity (Ariunbaatar *et al.*, 2016; Xu *et al.*, 2018). According to Banks *et al.* (2012) AD of FW would be impracticable without trace elements like nickel and selenium. Zhang, Ouyang and Lia's (2012) experiment on AD of FW at 3.6 g VS/L*d^{-1}, 20 days HRT and 37^{o}C without nutrient supplement proved futile due to mounting process instabilities. Sulphur for example is produced from the degradation of protein-rich substrates. Sulphide is produced through sulphate reduction or mineralization of organic sulphur. However, in higher concentrations, sulphide combines with hydrogen to form H_2S which becomes inhibitory to the digestion process. The presence of H_2S in the gas produced becomes corrosive when used as fuel for motor engines. Schnürer and Jarvis (2018) cites H_2S concentrations below 50 ppm as the acceptable level in biogas produced. This brings into focus the importance of Iron (Fe) addition to the degradation. Iron precipitates the sulphur.

2.2.2.8 Organic Loading Rate (OLR)

For daily gas production, the AD system is fed continuously with a substrate. OLR represents the amount of organic load per unit time and volume. It is an expression of the total organic

load per working volume. It could be determined by using Equation 2 below (Schnürer, Bohn and Moestedt, 2016).

$$\text{OLR} = \frac{Qs \times VSin \times TSin}{\text{VR}} \qquad \textit{-Equation 2}$$

Where Qs represents the substrate volume fed to the reactor, VSin represents volatile solids % content, TSin represents total solids % content, VR represents the volume of the reactor.

OLR is an important indicator of how much gas could be generated and the stability of the digestion process. Increasing OLRs ideally corresponds to elevated gas production however this is usually not the case for some substrates (Labatut and Pronto, 2018). According to Schnürer and Jarvis (2018) the microbial community may require time to adapt to process operating conditions and substrate composition. Thus rapid loading could lead excess undegraded material in the digester causing pH reduction. Hegde and Trabold (2019) reported process instabilities leading to reactor failure when OLR was increased from 2.8 to 3.5 g VS/L*d^{-1}. Higher OLRs of FW for example have been associated with increasing levels of inhibitions and disruptions including VFA accumulation, decreasing pH, etc (Zhang, Ouyang and Lia, 2012; Ariunbaatar *et al.*, 2016).

2.2.2.9 Hydraulic Retention Time (HRT)

HRT describes the average time required to replace the entire working volume of the digester. It is an expression of volume of feed added and the total working volume. The significance of HRT in the AD operation relates to microbial growth and substrate degradability. Methanogens are slow to mature or grow and thus a shorter HRT could contribute to washout of the microbes, thus affecting the entire methane production (citation). This likewise reduces the residence time for substrates in the digester further affecting degradability. By conversion, a longer HRT culminates to better microbial degradation. HRT varies with substrate types and operating temperature. Proposed HRT for FW is 20-30 days for mesophilic digestion and 15-20 days for thermophiles (Schnürer and Jarvis, 2018). Longer HRT could however increase the cost operation.

$$\text{HRT} = V_F/V_R \qquad \textit{-Equation 3}$$

Where V_F = Volume of feed; V_R = Volume of reactor

2.2.2.10 Temperature

The performance of AD is greatly influenced by the operating temperature, affecting both AD products (i.e., biogas and digestate). Temperatures ranging from 10°C – 100°C have been reported for microbial activities with varying characteristics (Schnürer and Jarvis, 2018). However, the optimum temperature for rapid growth and responsive microbial activity ranges between 30°C - 60°C. Schnürer and Jarvis (2018) postulates that microbial work becomes less productive for temperatures beyond 70°C. Two common temperature ranges widely discussed in literature for effective and efficient AD operations are mesophilic 35 °C - 42°C and

thermophilic temperatures 45 °C - 60 °C (Schnürer and Jarvis, 2018). The choice between these temperature borders on stability and performance. Thermophilic AD systems are characterised with increased performance in terms of higher methane production, pathogen inundation and higher OM degradation, etc. However, at higher temperature, the microbiome exhibits less diversity, which renders them susceptible or less resistant to changes in the system. Thermophilic AD operations thus require careful monitoring due to process instabilities.

2.2.2.11 pH

The optimal pH for AD just like temperature is a trickery field to navigate due to the distinctiveness of the requirements of the different microbes in the digester. Certain groups of microorganisms have specific conducive pH ranges within which they most productive. Several literatures suggests a pH range of 6.5-8.0 as the pH range for biogas production (Appels *et al.*, 2008; Sawatdeenarunat *et al.*, 2016). Schnürer and Jarvis (2018) however explains that methanogens for instance are very sensitive and are most efficient within the pH range of 7-7.5. Hydrolytic, acetogenic and acidogenic bacteria nonetheless operate within a wider pH range of 6-8.5 (Schnürer and Jarvis, 2018). pH varies with substrate and digestion technique, thus affecting both methane production and VS reduction. The pH is also influenced by levels of CO_2, NH_3 and accumulation of organic acids like VFAs.

2.2.2.12 Ammonium/Ammonia

Organic nitrogen is an important nutrient for plant growth hence a significant component in predicting the quality and value of fertilisers (Alburquerque *et al.*, 2012). Sources of nitrogen in an AD system include from protein rich substrates, nutrient supplementation and nutrient recirculation from dewatered digestates (Vázquez-Rowe *et al.*, 2015; Peng, Nges and Liu, 2016; Nwokolo *et al.*, 2020). However during protein degradation, nitrogen is either converted to free ammonia (NH_3) or ammonium (NH_4^+) (Zhai *et al.*, 2015). As indicated by Schnürer and Jarvis (2018) both compounds are in equilibrium under stable conditions. However, changes in pH for examples causes a shift in equilibrium towards high concentration of NH_3 leading to disturbances in the AD process, culminating to instability. On the other hand, digestate rich in ammonium is of great commercial interest due to the readily available nitrogen to plants when used as biofertilizer (Westerholm, Moestedt and Schnürer, 2016). At higher concentrations in the soil, this could lead to eutrophication of surface water or leaching to pollute groundwater (Alburquerque *et al.*, 2012). Nkoa (2014) also highlights the GHG emission potential of anaerobic digestates due to the higher concentrations of NH_4^+-N which could easily lead to ammonia volatilisation (Alburquerque *et al.*, 2012).

2.3 Digestate

Digestate production during the AD process such as in CSTRs is inevitable and its disposal is quite challenging due to environmental concerns and regulations (Logan and Visvanathan, 2019). Increasing demand for biogas in effect has resulted to an increase in digestate generated (EBA, 2021). Digestate is the other by-product from AD in the form of sludge or slurry containing plant mineral nutrients (Alburquerque *et al.*, 2012). It has the potential to replace

conventional mineralized fertilizer whilst promoting nutrient recycling and recovery (Drosg *et al.*, 2015). Though biogas is the main intended output, Alburquerque *et al.* (2012) argue that the sustainability of the AD process is linked to the eventual management of the digestate produced, which is a costly procedure. This positions the digestate as equally important as the gas production. Regulatory issues and economic constraints have nevertheless made disposal and utilisation of digestate a topical issue. Public health safety and environmental concerns are amongst the major considerations of local governments and authorities, whereas high handling, transportation and storage cost of the digestate heralds as major economic hurdles (Eriksson and Runevad, 2016). Also, digestate processing constitute about 50% of the overall cost of plant operation, making it a costly venture (Ahlberg-Eliasson *et al.*, 2017). According to Logan and Visvanathan, (2019) challenges such as gaseous pollution from NH_3, CH_4 and N_2O as well as groundwater pollution constitute some of the environmental concerns. This requires proper and efficient treatment methods prior to disposal. Landfilling, composting, incineration and land application are the custom disposal streams for digestate (Eriksson and Runevad, 2016). Environmental concerns and nutrient recirculation however make utilization of processed digestate a preferred alternative to spreading of raw digestate especially for land application and composting (Sawatdeenarunat *et al.*, 2016). Mechanical separation techniques like belt, screw press and decanter centrifuge could be used to separate the solid and liquid components (Drosg *et al.*, 2015). The liquid fraction of the digestate could be further processed into a nutrient rich concentrate using membrane purification. Ammonia stripping and struvite precipitation could likewise be used to remove NH_3, NH_4^+ and potassium (Ibid). Techniques for solid digestate processing include compositing and drying (Westerman and Bicudo, 2005). However, efficient nutrient management are to be considered when applying digestate to prevent nutrient overload, grand water pollution and eutrophication (Logan and Visvanathan, 2019). Considering the obvious benefits, the application of digestate to soil is widely touted and discussed in literature (Alburquerque *et al.*, 2012; Nkoa, 2014; Barzee *et al.*, 2019). Digestate from AD have lower environmental risk than as compared to untreated waste like manure with respect to pathogen levels (Nkoa, 2014).

Digestate depending on its quality is mainly applied as a soil conditioner to improve soil properties or as a bio-fertilizer to enhance plant growth and production (Barzee *et al.*, 2019). The physiochemical characteristics of digestate however depends on certain factors including feedstock characterisation, AD process characteristics, digestion stage/process, storage, etc (Manser, 2015; Schnürer and Jarvis, 2018; Logan and Visvanathan, 2019). For example; AD under thermophilic conditions is known to neutralise pathogens present in the substrate (Auer *et al.*, 2017). Also, nutrients present in a substrate are retained and enriched rather than destroyed in the corresponding digestate during AD (Drosg *et al.*, 2015). As such, digestate from FW are considered safe for agriculture use due to their relatively low heavy metal concentration (Barzee *et al.*, 2019)(Lü *et al.*, 2015; Barzee *et al.*, 2019). That notwithstanding, digestate produced during AD usually contains 2-10% dry matter for liquid and above 10% for solid digestate (Li *et al.*, 2020). In which ever state the sludge may appear, it undoubtedly contains a lot of moisture. The high moisture content of the sludge renders it unwholesome for direct disposal and utilisation.

2.3.1 Dewatering

Dewatering is a digestate enhancement technique that separates the digestate into nutrient rich liquid and solid fractions (Wang *et al.*, 2020). During the process, most of the nitrogen and potassium contained in the digestate are concentrated in the liquid fraction whilst phosphorus is retained in the solid phase. The significance of this approach is for volume reduction and nutrient recovery (Drosg *et al.*, 2015). Dewatering is beneficial to the biogas plant as it allows for easiness in handling and reducing transportation cost of the processed digestate. It is also significant to nutrient recirculation back to the digester. Cost savings in nutrient supplementation can be realised from using liquid digestate (Drosg *et al.*, 2015). Dewatered digestate offers the plant better and longer storage options.

Centrifugation, drying beds and belt filter press are some of the known configurations for dewatering digestate/sludge. Prior to centrifugation and belt pressing, the digestate is usually pre-treated with conditioners (flocculants, coagulants) or thickened with dewatered sludge to improve its dewaterability. The polymers cause the solids portions to coagulate or flocculate making it easier for the onward application of the separation technique. Determining the optimal polymer dose is crucial as it influences the cost of digestate dewatering. Polymer use in agricultural digestate is however a contested according to Drosg *et al.* (2015). This is rather location dependent. However, this prohibited in Sweden due to the relatively low research on its impact on agricultural lands (Eriksson and Runevad, 2016). It can however be applied as a thickener for wastewater treatment sludges prior to digestion or post-digestion (Ibid).

Dewaterability or dewatering-ability refers to the efficiency of the liquid and solid separation. Capillary suction time (CST) and TS% content in cake (after centrifugation or belt pressing) are some of the commonly used procedures to evaluate dewaterability. Wang *et al.* (2020) demonstrated a negative correlation between CST and dewaterability of waste activated sludge. CST experiments measure the filterability of the sludge. This concerns the rate at which water is removed from the sludge. Dewaterability however is affected by factors such as the sludge particle size distribution, the bound water, Organic matter, extracellular polymeric substance and soluble microbial products (Arimieari and Ademiluyi, 2018). Nonetheless, solids concentration in cake rejects and suspended solids of supernatant resulting from centrifugation could also be used as assessment (Zhang *et al.*, 2018). This assessment is usually done after a dewatering step has been carried out.

2.3.2 Digestate Processing

As indicated, environmental regulations and restrictions make direct application of wet digestate challenging (Vázquez-Rowe *et al.*, 2015). Moisture removal techniques are applied as a primary step to reduce the moisture content and increase the total solids concentration in the digestate. These actions separate the digestate into liquid and solid portions. Conditioning and thickening are some of the recognised processes that could be employed to improve the digestate's dewaterability prior to liquid-solid separation (Zhang, Dong and Dai, 2019). AD is known to improve the stability of sludge and dewaterability (Wang *et al.*, 2020). Co-digesting

the digestate with lignocellulosic substrates have been investigated to offer some improvements as well (Hernández-Beltrán *et al.*, 2019).

2.4 Feedstock Analysis

2.4.1 Food Waste

Food is a basic requirement for human existence. However, the cultivation, distribution and utilization of food resources comes with some undesirable impacts. Loss of biodiversity, greenhouse gas emissions and waste could be considered as some of the negative externalities from the food supply chain. Globally, about one third of food produced is either lost or discarded along the food supply chain resulting to about 1.3 billion tonnes of FW generated annually (FAO, 2011, 2019). The reasons for the food losses and waste are many with varying characteristics (Fisgativa, Tremier and Dabert, 2016). However, consumer behaviour and management have widely been cited as some of the major causes of food wastage (Dung *et al.*, 2014; Abrahamsson, 2019). The situation in Sweden is not different from the global phenomenon. Household waste is the biggest contributor with about 70 percent of generation. Restaurants, grocery chains and other agricultural residues account for the remaining 30 percent (Naturvårdsverket, 2020). According to the Swedish EPA an estimated 133 kg of food per person is wasted with a national average of 1.3 million tonnes generated every year. With burgeoning population and rapid urbanization, this is expected to increase. Though landfilling of organic waste is prohibited in Sweden (Naturvårdsverket, 2012; cited in Arushanyan et al. (2017)), other traditional waste management approaches continue to impact the environment negatively. The incineration of high-moisture content organic matter produces air pollutants (Rabii *et al.*, 2019) and composting contributes to the release of greenhouse gases (GHG) into the atmosphere. Anaerobic digestion of FW to produce biogas presents a sustainable and environmentally friendly route to dealing with the high moisture and biodegradable organic content.

AD of FW is seen as suitable and innovative alternative for energy and nutrient recovery as well as treatment of the organic waste (Dung *et al.*, 2014; Xu *et al.*, 2018). Oldfield et al. (2016) suggested that AD of FW results to a lower environmental impact and the best carbon return on investment. Ammenberg and Feiz (2017) assessment of AD substrates reveals the significance of FW for biogas production as compared to other feedstocks. FW is widely available substrate with a high energy potential according to Zhang, Lee and Jahng (2011). This however is dependent on the organic composition and nutrient balance which are influenced by the type and source of FW. Paritosh *et al's.* (2017) review on the substrate thus reveals a high variability of the methane potential between 180 to 732 ml/g VS*d. The review cites factors such substrate composition, nutrient supplementation, operational conditions, etc. as accounting for the variations in methane production.

2.4.2 Pulp Fibre

The Pulp and papermaking industry (PMI) is a big enterprise both in Sweden and abroad. Sweden since 2018 exports about 80-90% of the 12M tonnes of pulp and paper produced

annually (CEPI, 2020). The industry is an integral part of the Swedish economy providing employment and contributing to the country's trade balance. Resource consumption is highlighted as the pulp and papermaking process consumes large amounts of energy, forest and water resources whilst generating substantial waste. To minimise the environmental impacts from waste generation, recycling and bioenergy production are amongst the several resource efficient and waste management strategies adopted by Swedish paper mills. According to Larsson (2015) AD of pulping waste streams could contribute to the sustainability of the sector. However as elucidated by the researcher the cost involved in the AD setup is rather a disincentive for paper mill operators. Assessment of the methane potential of different waste streams from various pulping processes suggest mixed and varying results. That notwithstanding, there exist a significant methane potential due to the vast amounts of waste generated (Ekstrand *et al.*, 2013). Larsson (2015) estimated the biomethane potential from the PMI as 700 GWh which is about 35% of current biogas production (Ahlberg-Eliasson *et al.*, 2017).

Pulp and papermaking depending on the preferred technology involves four stages engineered towards transforming cellulose from wood fibre into paper products. Primarily, the harvested wood goes through debarking, pulping, bleaching and finally papermaking. Recent technologies have been successful at recycling used paper into the production line. According to (Ekstrand *et al.*, 2013) the enormous water requirements during pulping and bleaching stages results to greater waste effluents demanding treatment and proper disposal. However, conventional disposal strategies including landfilling and incineration are costly with high energy consumption due to dewatering, aeration and nutrient addition. AD of the waste streams is seen a way out of this dilemma.

That notwithstanding, not all waste products from the pulping process are potential substrates for biogas production. This relies on the pulping process and wood species. Chemical pulping (specifically kraft pulping) accounts for about 80% of the global pulp production whilst 72.1% of softwood is used (CEPI, 2020). Ekstrand et al. (2013) sited three waste types as plausible substrates for AD to include, i.e., wastewaters, primary sludge and biological sludge (WAS). Characteristics and composition of these waste streams differ with respect to the organic matter content, pH, temperature, nutrient and chemicals present (Larsson, 2015). These characteristics collectively determine the suitability of the waste stream for efficient methane production. Notable features of pulp and paper effluents is their low trace metals, nitrogen, phosphorus constituents and low methane yields compared to other organic substrates like animal manure and slaughterhouse waste (Ware and Power, 2016a; Elnakar and Buchanan, 2018).

Fibre or primary sludge is a residue obtained after the primary clarification step of pulping process (Ekstrand *et al.*, 2013). This is characterised as having low buffering capacity due to the higher C/N ratio (111-943) and fibre than compared to other waste streams from pulping (Faubert *et al.*, 2016). This however makes AD treatment of fibre sludge challenging often culminating to process instabilities and accumulation of intermediary products like VFAs. According to Ekstrand, (2019) due to its low alkalinity, fibre sludge could be co-digested with WAS to overcome its AD operational difficulties. Other co-substrates with relatively low C/N

ratios could improve its AD operation. Dewatering the fibre sludge produces pulp fibre. Conventionally, pulp fibre could also be sampled directly from the product line during pulp processing as expressed by Ekstrand *et al.* (2020).

3. MATERIALS & METHODS

This chapter describes the research methodology employed in conducting the study. Specifically, the section presents the experimental set up, operation of the reactors as well as the analytical tools and measurements used in the experiment. The timeline of events beginning with the start of the rectors to the completion of the project is described here.

The research activities were categorised into two phases. Phase I represented the first part of the study which included the experimental start-up with only 2 reactors (digesters). This phase was designed to stabilise the reactors before addressing the research questions and to reduce the workload before the 2nd phase. Phase 2 embodied the main objectives of the study which was to investigate the effect of pulp fibre addition on the gas production and dewaterability of AD of FW.

2 digestion stages namely, main and post digestion, were designed to answer the research questions. The main digestion stage measured the biogas and methane production. pH, VFA and ammonium nitrogen concentration were monitored for the main digestion as well. These constructs were used in answering research questions 1 and 2. The post-digestion stage investigated the dewaterability of the effluents from the main digesters. The aim of this stage was to give the effluents from the main digesters extra 7 days retention period. CST and centrifugation were used to assess the dewaterability of the effluents from the post-digesters. This was used in answering research question 3.

The experiment and analyses were conducted with equipment from/at TEMA laboratory, Linköping University. Data collected was analysed in Excel and graphs presented. Results from the various experiments are presented in the next chapter.

3.1 Inoculum and Substrate

Inoculum for the start-up was sourced from an existing full-scale anaerobic digester (Södertörn, Sweden) and stored over-night at 2 degrees before inoculation. The Södertörn plant operates on FW as substrate at a maximum OLR of 5 g VS/L*d^{-1} with HRT of 26 days.

Two types of substrates were fed to the reactors during the experiment. These were source-separated FW and pulp fibre. The FW component was received from the Södertörn plant. This was already ground and hygenized by the plant. The pulp fibre (PF) used came in two forms, Softwood PF and Hardwood PF. Bleached and dewatered pulp fibres were obtained directly from the pipeline of a kraft pulp and paper mill in Norrköping, Sweden.

The FW was kept in 10L plastic containers and pulp fibres in big transparent plastic bags. Both substrates were stored in a -20^{o}C freezer container and subsequently transferred to room temperature fridge container and thawed for 20-24 hours prior to feed portion preparations.

3.1.1 Feed Portions and Preparation

Food or feed portions were prepared in weekly batches and stored in a fridge (temperature 2-4 ^{o}C) in the laboratory. Transparent plastic bottles were labelled with names of reactors and a quantity of the FW was weighed into them. A known amount of water, Iron and trace elements were added to the prepared food portions. Water was added to regulate the hydraulic retention time (HRT). The trace elements included Nickel (Ni) and Selenium (Se). The reactors were solely fed with FW for the 1st part of the experiments.

For the 2nd phase of the study, pulp fibre was added to the FW and the mixture was fed to 2 of the reactors. Pulp fibres were added to designated feed bottles containing FW. Depending on the organic loading rate (OLR), known amounts of softwood and hardwood fibres were added to feed portions of the two reactors.

3.2 Experimental Set-Up and Reactor Operation

The set up consisted of six mesophilic 10L continuous stirred tank reactors (CSTR; Dolly ©, Belach, Bioteknik, Skogås, Stockholm, Sweden). The reactors were each equipped with a volumetric gas flow meter operating on the liquid displacement principle to monitor the gas production. Each reactor had an inlet where substrates were fed daily and outlets through which sludge could be withdrawn from the reactors. Gas sampling bags were connected to the gas outlet of the gas meter for regular gas collection and composition analysis. The volumetric gas production was recorded daily before feeding, onto a laboratory tablet and uploaded online. At the start-up of the experiment, 2 laboratory scale anaerobic digesters each with a working volume of 6L were labelled and each inoculated with 6L of sludge from a full-scale digester operating on source separated FW.

The digesters were initially fed at a lower organic loading rate (OLR) of 2.5 ± 0.1 g VS/L*d^{-1} with a hydraulic retention time (HRT) of 40 days and later increased stepwise to reach 3.5 ± 0.1 g VS/L*d^{-1} at HRT of 26 days. These were continuously stirred at 100 rpm and run at 37^{o}C. Protocol for reactor start-up and operation as described by Schnürer, Bohn, & Moestedt (2016) was followed. 0.6 mL of iron solution was added daily to the working volume through the feed portions to reduce the accumulation of H_2S in the gas produced. 40 days after the initial start-up, sludge from the 2 reactors were mixed up, and a 3rd reactor was borne out of them. This ended the 1st phase of the experiments.

Phase II of the experiment begun with the three newly created digesters. Starting on day 1, the digesters were run-on FW at OLR of 3.5 ±0.1 g VS/L*d^{-1}, 26-day HRT and subsequently designated as BR05 (control digester), BR07 (softwood digester) and BR09 (hardwood digester) as shown in Table 1. These reactors represented the main digesters for the experiment. The operation of the 3 reactors followed the same protocols as stated in phase 1 above. On day 35, 3 post digesters were started and labelled as BR06 (control sludge digester), BR08 (softwood sludge digester) and BR10 (hardwood sludge digester) as depicted in Table 1. Digestate from the main digesters served as inoculum for the start-up of the corresponding post digesters which was also added in steps, corresponding to 230ml of sludge a day up to working

volume of 1.41L (HRT = 7 days). Pulp fibre addition begun on day 59 (after 58 days of acclimatisation) to 2 of the main digesters (BR07 & BR09). 0.5 ±0.1 g VS/L*d^{-1} of pulp fibre (Softwood & Hardwood) was added to 181.4g of FW and fed directly to the 2 digesters. The OLR for the pulp fibre digesters were increased stepwise by 0.5 ±0.1 g VS/L*d^{-1} on day 74 and 100. An OLR of 1.5 ±0.1 g VS/L*d^{-1} of PF was maintained for the test digesters for the remaining periods of the experiment. The third reactor designated as a control experiment was only fed with 181.4g of FW and water was added to compensate/keep the same HRT as the test experiment.

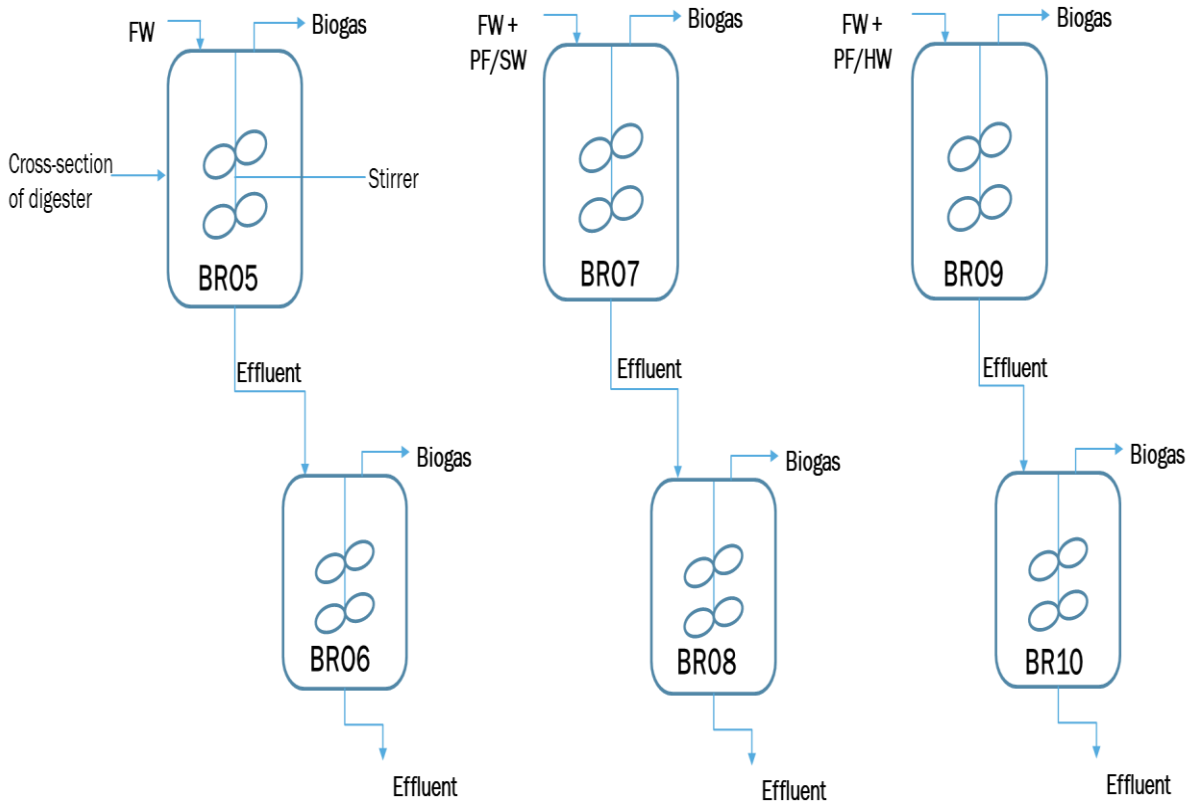


Figure 2. Experimental setup

Overall, the digesters were operated at an organic loading rate of 3.5 ±0.1 g VS/L*d^{-1} of FW and 0.5-1.5 ±0.1 g VS/L*d^{-1} of pulp fibres at an HRT of 23-26 days. All reactors were likewise monitored for their stability and performance.

Table 1. Summary of the reactors, the digestion stage and the substrates fed to the reactors during phase 2 of the experiment.

Reactor Name	Designation	Digestion	Substrate fed
BR05	Control	Main	FW
BR06	Control	Post	Sludge/Digestate
BR07	Softwood	Main	FW + SW
BR08	Softwood	Post	Sludge/Digestate
BR09	Hardwood	Main	FW + HW
BR10	Hardwood	Post	Sludge/Digestate

BR = Biogas reactor, FW = food waste, SW = softwood, HW = hardwood

3.3 Analytical measurements

The inoculum, substrates and reactor effluents were investigated for their chemical and physical composition including pH, volatile solids (VS), total solids (TS) and gas composition.

3.3.1 TS and VS

TS and VS of the substrate representing total dry solids and organic matter content respectively, were measured in accordance with standard procedures according to the Swedish Standard method (SS 028113). Tests were performed either in duplicates (digestates) or triplicates (substrates).

Empty crucibles were marked with pencils to indicate samples and weighed as W_0. The crucibles were then filled with 15-20g of the respective samples and the weight recorded as W_1. The samples were then dried in an oven at 105°C for at least 20h. After the period, the hot crucibles were removed from the oven, allowed to cool in a desiccator and weighed afterwards as W_2. This gave the total solids content. To determine the organic content i.e., VS, the samples were further heated in a muffled oven at 550°C for 2h and later allowed to cool in a desiccator. The weight was recorded as W_3. The TS and VS of the samples were computed as depicted in Equation 4a and 4bbelow. The VS% was estimated as a ratio of the total VS to total TS. Volatile solids destruction or reduction for the various setups were calculated as shown in Equation 4c below. VS% reduction represented the amount of organic matter degraded and converted biogas (Schnürer and Jarvis, 2018). This was only calculated on days when TS/VS was measured.

$$TS\ \% = \frac{dry\ weight\ after\ at\ 105℃}{Wet\ weight} \times 100$$

$$TS\ \% = \frac{W1-W0}{W2-W0} \text{ x } 100 \qquad \textit{-Equation 4a}$$

$$VS\%\ (\%\ of\ TS) = \frac{(dry\ weight\ after\ at\ 105℃) - (ash\ weight\ after\ 550℃)}{(dry\ weight\ after\ at\ 105℃)} \times 100$$

$$VS\%\ (\%\ of\ TS) = \frac{(W1-W0)-(W3-W0)}{(W1-W0)} \times 100 \qquad \textit{-Equation 4b}$$

$$\%\ VS\ Reduction = \frac{VSin-VSout}{VSin} \times 100 \qquad \textit{-Equation 4c}$$

Where VSin and VSout represented the VS of the influent substrate and effluent digestate respectively.

3.3.2 pH Analysis

The pH of the inoculum, substrates and effluents were determined according to the Swedish Standard 12176 (1998) using a pH meter (Inolab pH 7310, WTW, Germany). About 80mL of sample was measured into a glass bottle and placed on a magnetic hotplate stirrer (VMS-D S40, VWR, Belgium). The bulb of the pH electrode was lowered into the sample and the stirring regulated to about 100-150 rpm. The pH meter was calibrated weekly using buffer solutions (pH 4 & 7) to ensure accurate results. A third buffer/reference solution of pH 8.00 was used as a control measure.

3.3.3 Volatile Fatty Acids (VFA)

VFA concentrations were measured twice a week for the reactor effluents and once per substrate batch of FW as described by Jonsson & Borén (2002) using a gas chromatograph with a flame ionization detector (GC-FID; HP 6890 Series, Hewlett Packard, USA). The VFAs quantified included acetate, propionate, butyrate, isobutyrate, valerate, isovalerate, caproate, and isocaproate.

About 1-1.5mL of the samples (reactor fluid/substrate) were put in plastic 1.5ml Eppendorf tubes and centrifuged at 15,300 x g rcf for 10 minutes (5417C centrifuge, Eppendorf, Hamburg, Germany). 400 µl of the supernatant was pipetted into an injection vial and 40 µl of an internal standard (crotonic acid in a formic acid solution) was added to it. The prepared samples were loaded onto an auto sampler on a GC-FID (HP 6890 Series, Hewlett Packard, USA). for analyses. The detection and quantification limit of the instrument were 0.2 mM and 0.6 mM, respectively.

3.3.4 Ammonium nitrogen experiment

Ammonium tests were conducted using the LCK 302 ammonium cuvette test kit (47-130 mg/L NH_4-N, Hach Lange, Germany) and analysed using Hach DR2800 spectrophotometer (Hach Lange, Germany) based on the indophenol blue method. Each sample (digestate) was diluted with Milli Q H_2O using a dilution factor of 1:30 (digestate: Milli Q H_2O). Procedure for analysis was carried out following instructions as provided by the kit manufacturer. Test was conducted once every week with no replicates

3.3.5 Gas Measurements

Daily volumetric gas production was recorded using the gas flow meter (Belach, Bioteknik, Skogås, Stockholm, Sweden) and displayed on a real-time reactor monitor. The gas composition was analysed twice a week using the Biogas Check Analyser (Geotechnical Instruments, Chelmsford, United Kingdom). Gas was collected in 25L gas sample bags for 24 hours between feedings prior to analysis. The Biogas check was calibrated once every week to ensure accurate results. The gas meters were also calibrated twice during the experiment.

The specific methane yield was calculated from daily biogas produced. Specific methane yield describes the actual methane content of the gas produced normalized to VS content of the substrate. It is a function of the total gas production, the total VS fed, and the methane composition of the gas produced. This could be represented by the Equation 5 below.

$$Specific\ CH_4\ yield = \left(\frac{total\ gas\ prod.}{total\ VS\ fed}\right) x\ CH_4\ \% \qquad \text{-Equation 5}$$

3.3.6 Capillary Suction Timer (CST)

The CST apparatus (Type 304B CST, Triton Electronics Ltd., Essex, England) was employed in determining the filterability of the effluents from the post digesters. This was a measure to evaluate the dewaterability of the digestate produced from the reactors. The CST experiments were performed both on conditioned (with polymer) and unconditioned sludge (Eriksson and Runevad, 2016).

For the conditioning tests, 0.5 % polymer solution was prepared using polymer provided by the same full-scale plant that provided the inoculum and FW substrate. The dose of polymer varied between 7-8 g polymer/kg TS of sludge according to recommendations from the full-scale plant and was added to 50 mL of sludge in a 250 ml beaker. The mixture was turned between 2 beakers several times until flocs were formed.

The CST apparatus was started by plugging in the test-head assembly into the CST unit. A soft paper tissue was used to clean the two Perspex plates. A filter paper (70 x 90 mm dimension, grade 17 CHR) was placed in between the plates with the rough most side facing upwards and in contact with the electrodes (sensor). The 1.8 cm diameter funnel was selected due to the slow filtering nature of the sludge and inserted into the electrode block. The funnel was filled with sludge (sample) and the time until the liquid of the sludge had travelled between the two sets of electrodes was recorded. For each sample, the experiment was conducted in duplicates. The same procedure was followed for both conditioned (with polymer) and non-conditioned sludge.

3.3.7 Centrifugation

The specific objective of this section of the experiment was to measure the dewaterability of the post-digesters. The JA-10 rotor was selected and aligned into the centrifuge (Avanti J-E

Centrifuge, Beckman Coulter, USA). 2x160g of the effluent samples from each of the 3 post digesters were weighed into centrifuge tubes. A maximum difference of 0.3g for opposing samples was maintained before arranging the tubes in centrifuge. Operational parameters of rotor type, speed, time and temperature were input as JA-10, 4700 x g, 4 minutes and 20^{o}C, respectively. After centrifugation, the separated liquid reject was transferred into plastic bottles and the weight/volume recorded. Suspended solids analysis was performed on the liquid reject. The weight of the remaining cake was recorded, after which TS and VS analysis was performed for the solid fraction.

Suspended solids analysis was performed according to SS-EN 872:2005 using a vacuum filtration apparatus (Vacuum filtration unit, GV 100 series, Whatman) fitted with a suction pump. 50 ml of the liquid reject from centrifugation was filtered through a dried glass fibre filter (Munktell, MG A grade, Ø 47 mm, Ahlstrom-Munksjö, Sweden). The filter was removed and dried in an oven at 105^{o}C for at least 1h and the mass of the residue determined by weighing. The weight of the total suspended solids was calculated according to Equation 6 below.

$$mg\ of\ total\ SS = \frac{(weight\ of\ filter+residue)-(weight\ of\ filter)}{volume\ of\ sample,mL} \times 1000 \qquad \textit{-Equation 6}$$

3.4 Statistical Analysis

Statistical operations like mean and standard deviation were run on the collated data using Excel. Pearson correlation coefficient (R) was calculated and used for correlation analysis using SPSS 27. Graphs were presented in Excel and SPSS.

4. RESULTS AND DISCUSSION

This section of the report presents data and discusses the results from the various tests conducted on the substrates and residual sludges during phase 2 of the study. Results for the pre-experimental stage (i.e., phase 1) is presented in Appendix.

4.1 Substrate and digestate characteristics

Table 2 and 3 display the physiochemical properties of the substrates and digestates used during the experiment. The FW substrate had a TS and VS content of 12.7 ±0.3 % and 90 ±2 %. TS% was relatively low compared to what is reported in literature (Zhang, Lee and Jahng, 2011). According to Labatut and Pronto (2018) FW characteristics varies disproportionately with regards to the source, type and use. Fisgativa, Tremier and Dabert's (2016) study on 63 different FW samples revealed a significant variability in TS concentrations between 10-30%. However, for wet digestion of organic substrates in in continuous stirred tank reactors (CSTR), TS between 2-15% seemed appropriate (Schnürer, Bohn and Moestedt, 2016). The relatively low TS of the substrate could be traced to the pre-treatment of the substrate at the full-scale plant prior to digestion. Addition of water or other liquids to substrates like FW is a common practice in industry. A similar assessment by Ammenberg and Feiz (2017) on source-separated FW showed a TS and VS content of 11% and 82%. This has been noted to make the substrate pumpable and increase retention time especially for wet digestion in CSTR (Ammenberg and Feiz, 2017; Schnürer and Jarvis, 2018). Apart from water, liquid from dewatered digestate could be recycled for this purpose.

Table 2. Substrate characteristics

Parameter	FW	PF	
		SW	HW
pH	3.7 ±0.0	N/A	N/A
TS %	12.7 ±0.3	28.2 ±0.5	28.0 ±0.2
VS % (% of TS)	90.0 ±2	99.7 ±0.3	99.6 ±0.1

4.1.1 pH and VFA

pH is by far the most significant parameter in AD as it indicates the stability of the process and is connected to VFA production (Fisgativa, Tremier and Dabert, 2016). These parameters exhibit an inverse proportionality relationship where increasing VFA and low alkalinity in an AD system usually leads to lower pH values and vice versa. From section 2.2.3.9, the effect of pH has reliably been explained as affecting both the stability of the degradation and the methane generation. Figure 16 (see appendix) shows the recorded pH and VFA trend during the experiment.

Table 3. Digestate characteristics

Parameter	Days	Main digestion			Post digestion		
		BR05 (Control)	BR07 (FW+SW)	BR09 (FW+HW)	BRO6 (Control)	BR08 (FW+SW)	BR10 (FW+HW)
pH	0-58	7.5 ±0.1	7.6 ±0.1	7.6 ±0.1	7.5±0.0	7.5±0.0	7.5±0.0
	59-74	7.5 ±0.1	7.4 ±0.1	7.4 ±0.1	7.6 ±0.1	7.6±0.0	7.6±0.0
	75-100	7.4 ±0.1	7.3 ±0.1	7.3 ±0.1	7.6 ±0.1	7.5±0.0	7.5±0.0
	101-163	7.6 ±0.1	7.3 ±0.1	7.3 ±0.1	7.6 ±0.1	7.5 ±0.1	7.5 ±0.1
TS%	0-58	3.5 ±0.2	3.4 ±0.2	3.3 ±0.2	2.8 ±0.1	2.9 ±0.2	2.8 ±0.1
	59-74	3.5 ±0.0	3.4 ±0.2	3.5 ±0.1	3.0 ±0.1	3.1 ±0.1	3.2 ±0.1
	75-100	3.5 ±0.3	3.8 ±0.0	4.0 ±0.4	2.9 ±0.0	3.0 ±0.1	3.1 ±0.1
	101-163	3.1 ±0.1	3.7 ±0.2	3.9 ±0.2	2.7 ±0.1	3.1 ±0.2	3.5 ±0.2
VS%	0-58	71 ±2	71 ±1	71 ±1	68 ±1	68 ±1	67 ±0
	59-74	72 ±1	72 ±0	72 ±0	69 ±1	69 ±0	69 ±1
	75-100	73 ±0	74 ±0	74 ±0	70 ±1	71 ±1	71 ±1
	101-163	73 ±1	75 ±1	75 ±1	71 ±0	72 ±1	73 ±1
CH_4 %	0-58	55 ±4	54 ±4	54 ±4	-	-	-
	59-74	59 ±1	57 ±1	57 ±1	-	-	-
	75-100	59 ±0	56 ±1	56 ±1	-	-	-
	101-163	59 ±1	56 ±1	56 ±1	43 ±1	46 ±1	48 ±1

Day 0-58: Start-up phase; day 59-74: Addition of Pulp fibre (95% FW + 5% PF) to pulp fibre digesters (PFD); day 75-100: increased PF loading (90% FW + 10% PF) to PFD; day 101-163; increased PF loading (85% FW + 15% PF) for PFD

From table 2, the pH 3.7 ±0.1 of FW was slightly acidic than the 5.1 ±0.7 as reported by Fisgativa, Tremier and Dabert (2016). The authors reviewed several literatures to compare pH from different substrates and found sewage sludge (7.8), cow manure (8.7) and green waste (7.3) to be higher than FW (5.1). However a study conducted by Ma *et al.* (2011) on kitchen waste revealed a similar pH of 3.8 ±0.2 as shown in table 2. This revelation further bolsters the discussions on the variability of food waste characteristics (Fisgativa, Tremier and Dabert, 2016; Labatut and Pronto, 2018). Low substrate pH is reported to contribute to rapid acidification of the AD process. This was further evidenced by a high acetic acid content of 51.8 ±4.8mM/L (also 1.6 ±0.2 mM/L for propionate and butyric acids) (see figure 15, in appendix) in the substrate prior to digestion. This observation suggested that hydrolysis and fermentation had been initiated presumably from pre-treatment of the substrate or during storage. Ariunbaatar *et al.* (2014) highlighted the significance of substrate pre-treatment as enhancing degradation and methane production. In addition to the pre-treatment effect on pH, regular freezing and thawing of the substrate before digestion, as was practiced during this study could have contributed to the pre-fermentation effect. Ma *et al.* (2011) investigated the effect of 5 different pre-treatment methods on FW and found that freeze-thaw enhanced the performance and solubilisation of digestion. A pH of 4 was reported for the freeze-thaw pre-treatment which was similar to this experiment. In summary, the low pH of the FW substrate depicted in Table 2 and the relatively high pH of the digestate as shown in Table 3 suggested a relatively high buffering capacity of the control reactor.

As reported in section 2.2.3, pH in the digester is an indicator of both stability and performance of the digestion process. According to Appels *et al.* (2008), methanogens most importantly are sensitive to changes in pH, resulting to fluctuations in substrate degradability and subsequently, gas performance. As expressed by Schnürer and Jarvis, (2018) most microorganisms thrive under neutral pH of 7-7.5 however, fermentative bacteria according to Appels *et al.* (2008) are least affected by low pH thus having a rather wide pH range of 4-8.5. In effect, a pH range of 6.5-8 has been suggested as the optimal pH range for a controlled AD of FW (Mao *et al.*, 2015). The mean pH for the main digesters varied significantly between the FW digester (pH 7.5 ±0.1) and the pulp fibre digesters (PF) (pH 7.4 ±0.1) (t-test, $p < 0.01$). The post digesters however maintained a relatively balanced pH of 7.5 ±0.1 as shown in table 3 and 4. In totality, the mean pH of the control reactor (BR05) was higher than the test reactors (BR05 & BR09) for most part of the experiment. Prior to fibre addition, the three main digesters maintained a relatively balanced pH of 7.5 ±0.1. Increasing OLR for pulp fibre reactors resulted to a drop in pH. The drop in pH however did not amount to VFA accumulation. From this observation, it could be inferred that the digestion process was stable. Hegde and Trabold (2019) likewise reported a steady digestion process at pH 6.8-7.5 when mixed cafeteria food waste at 2.8 g $VS/L*d^{-1}$ was co-digested with cow manure and paper napkins. That notwithstanding, Rabii *et al.* (2019) admonishes against such assertions, as pH alone is not a good measure of the stability of AD process. Rabii *et al.* (2019) argues that this wide pH range of 6.5 to 8 suggested in literature (Appels *et al.*, 2008) could be misleading as it is difficult to ascertain the veracity of the instabilities present in the digestion system. Several researchers further suggests the use of pH in conjunction with VFAs or alkalinity to evaluate the stability of an AD system (Appels *et al.*, 2008; Schnürer and Jarvis, 2018).

VFA analysis showed only traces of Acetic acid in the sludges. The analysis at the start-up of the experiment (between day 1-7) showed elevated acetic acid concentrations of ~0.8 ±0.1mM/L which was above the quantification limit of 0.6mM/L. Appels *et al.* (2008) cited VFA concentrations of 0.6mM/L– 0.9mM/L as being toxic to methanogens at relatively low pH. This was not the case for this experiment as the pH were relatively neutral. The VFA concentration during start-up could be explained as the microbial community adjusting to the new environment. For the remainder of the experiment, acetic acid concentration remained below 0.4 ±0.1mM/L. This suggested that much of the VFAs that were produced during digestion was subsequently consumed by the methanogens. The relatively low VFA concentration and stable pH was a good indication of the stable degradation process.

4.2 Effect of pulp fibre addition on gas performance

4.2.2 Biogas production

As can be observed in Figure 3, the overall volumetric biogas production was higher in the pulp fibre reactors (BR07 & BR09) as compared to the control FW only reactor (BR05). This could be attributed to the difference in the OLR for the 3 main digesters. This phase of the experiment started on day 0 with an OLR of 3.5 ±0.1 g VS/L*d^{-1} of 100% FW at 26 days hydraulic retention time (HRT) for the 3 digesters. Unstable gas production was observed from day 0-32. From day 33-58 the digesters recorded similar amount of gas (17.7 ±0.5 L/d) with BR05 demonstrating a marginally higher gas produced than other 2 digesters (t-test, $p < 0.0001$). This was further evidenced by a slightly higher TS% of 3.5 ±0.2 for BR05 compared to 3.3 ±0.2 for BR09 (t-test, $p < 0.05$) (mean TS% from day 0-58). There was no significant difference between TS% of BR05 and BR07 (t-test, $p > 0.05$).

The addition of 0.5 ±0.1 g VS/L*d^{-1} PF (95% FW + 5% PF) to BR07 and BR09 from day 59-74 however, resulted to about ~6% increase (t-test, $p < 0.0001$) in the gas production of the softwood fibre digesters at a degradation efficiency of 77 ±7% than the control reactor (table 4). There was no significant improvement in the hardwood fibre digester with regards to the control reactor (t-test, $p > 0.05$). However, the TS% and VS% of BR09 (3.5% ±0.1, 73% ±1 respectively) were observed to have slightly improved upon fibre addition (see table 3). The addition of PF however, contributed to a low alkalinity in the PF digesters as demonstrated by a pH drop from 7.6 ±0.1 to 7.4 ±0.1 (table 3). Mata-Alvarez *et al.* (2014) amongst other researchers have attributed this phenomenon to the low buffering capacity of lignocellulosic substrates due to the high C/N ratio. Though VFAs were noticed in all 3 digesters, their amounts were below the quantification unit of 0.6mM/L. HRT of BR05 was decreased to 25 days on days 59-74 to commensurate with the HRT of the PF digesters (BR07 & BR09) as suggested in Hegde and Trabold (2019). This together with an increased FW TS% corresponded to a relatively improved gas performance for BR05 of about 18.8 ± 0.5 L/d. A new batch of substrate at TS% 13.6 relatively higher than the reported meane was realized for this period. Marginal increases in biogas production were observed when PF loading was stepped up to 1.0 g VS/L*d^{-1} from day 75-100 (t-test, $p < 0.0001$).

A further stepwise increment to OLR/PF 1.5 g VS/L*d^{-1} from day 101-163 resulted to substantial a 40% and 35% rise in biogas production for BR07 and BR09 respectively as compared to 18.7 ±0.5 L/d for BR05 (see table 4). Concurrently the degradation efficiency of 79 ±3% and 78 ±2% for BR07 and BR09, were respectively higher than BR05 75 ±2% VS reduction (t-test, $p < 0.0001$). The VS reduction in these cases could be regarded as high signifying a better degradation of the co-digestion of the substrates. Labatut and Pronto (2018) suggest a degradation efficiency more than 75% as a good indication of a good microbial activity and digestion. A final HRT of 23 days (for BR05 at an OLR od FW 3.5 ±0.1 g VS/L*d^{-1}, and BR07 & BR09 at an OLR of 5 ±0.1 g VS/L*d^{-1} (85% FW + 15% PF) was maintained for the remainder of the study as depicted in figure 3. No inhibitions or process disturbances were realised during this phase of the experiment thus further bolstering the suitability of co-digesting pulp fibre with FW. Anaerobic co-digestion of FW with other substrates like sewage, manure and other agricultural waste have produced foaming and process instability (Zhang, Lee and Jahng, 2011). Mean and SD for all monitored parameters at the different OLR of fibres for BR05, BR07 and BR09 are presented in Table 4 in the Appendix.

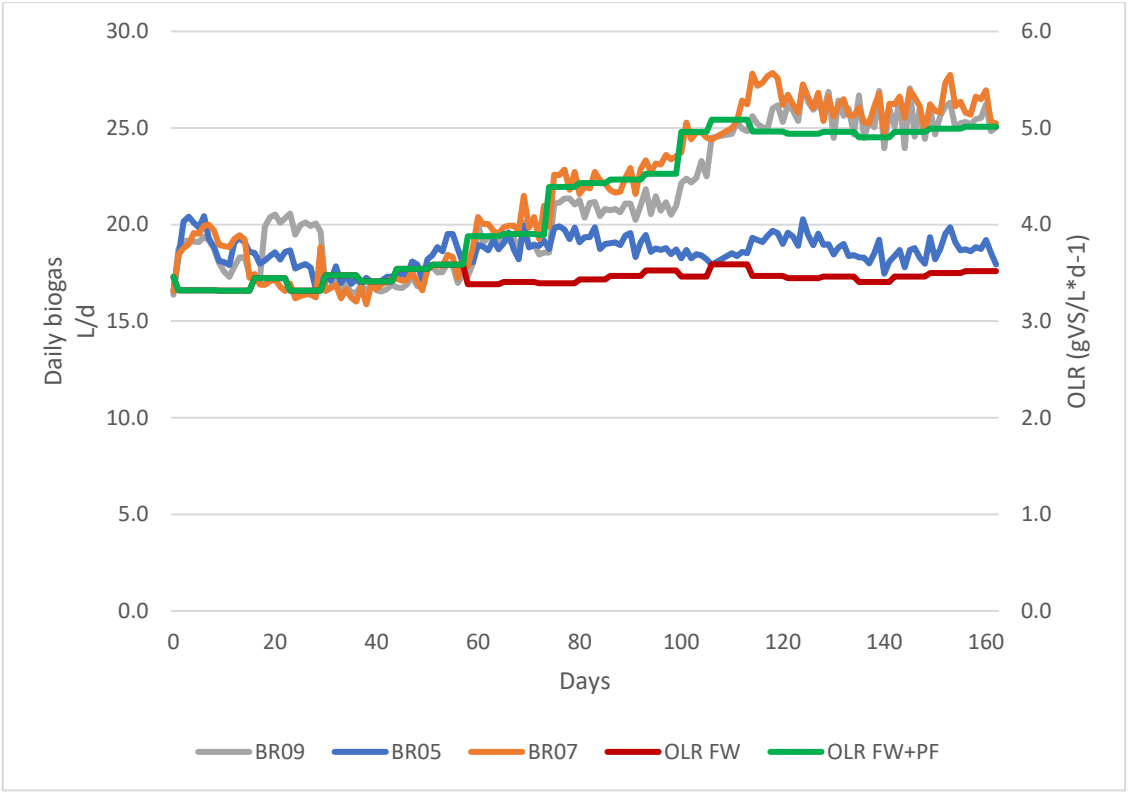


Figure 3. Daily biogas vs OLR

Post digestion begun on day 35 to day 163 of the main experiment. Due to the nature of substrate used (effluents from the main digesters), the OLR varied between 3.1 – 4.9 g VS/L*d^{-1} according to the TS% and VS% from the source as shown in Table 3. The post-digesters experienced variations in retention time for a greater part of the experiment due to uneven sludge withdrawals for digestate analysis. This was however regularised to HRT of 7 days from day 101 till the end of the experiment. Thus, the results presented in figure 14 (see appendix) for biogas production rather corresponded to days 101 – 163. Mean volumetric

biogas produced was relatively high in the fibre digesters, averaging 860 ±120 mL/d for BR10 (hardwood) and 810 ±100 mL/d for BR08 (softwood) than compared to BR06 with 480 ± 140 mL/d. The elevated gas produced in BR08 and BR10 reflected the high TS% and VS% in the effluents received from BR07 and BR09, respectively than as received by the control post-digester (table 3 and 4). Signalling a higher percentage of undigested OM present in digestates from BR07 and BR09. Hydrolysis is considered a rate limiting step during AD of lignocellulosic substrates due to their complex structure and composition, thus requiring a longer retention time for adequate degradation (Sawatdeenarunat *et al.*, 2015). This results to slower degradation of cellulose which is the major constituent of kraft pulped fibres (Ekstrand, 2019). The crystalline structure of cellulose makes it difficult for degradation partly due to their stronger bonding (Sun *et al.*, 2016; Gonzalez-Estrella *et al.*, 2017). This further suggested that the HRT of 23-26 days was unsuited to pulp fibre degradation. This was clearly manifested as the extra 7-day degradation resulted to improved gas production.

4.2.3 Methane Yield

According to Labatut and Pronto (2018) the efficiency of an AD system could be assessed by the amount of methane produced and the substrates degradability. Figure 4 depicts the daily specific methane yield for the BR05, BR07 and BR09 with regards to OLR. In general, the normalized methane produced for the pulp fibre digesters (BR07 & BR09) were lower than the control digester (BR05) solely running on FW. The normalized (specific) methane yield is a function of different parameters including VS (g), total biogas yield (mL) and gas composition (%) but differs from total methane which only represents the CH_4 composition in the total biogas generated. The reactors started off with an even specific methane production (SMY) of 490 ±30, 460 ±20 and 470 ±40 ml/g VS*d for BR05, BR07 and BR09 respectively when run on 3.5 ±0.1 g VS/L*d^{-1} of FW as shown in figure 4. Addition of 0.5 ±0.1 g VS/L*d^{-1} PF to BR07 and BR09 during days 59-74 did not significantly improve the SMY for the fibre digesters (t-test, $p > 0.05$). This gave a CH_4 yield of 480 ±20 and 460 ±20 CH_4 ml/g VS*d for BR07 and BR09. Contrary to the PF digesters, the methane yield for BR05 for the same period was 540 ±15 CH_4 ml/g VS*d. This reflected a decrease in the SMY in the PF digesters compared to FW digester.

This was surprising as the cumulative degradation efficiency of the fibre digesters measured on the same day TS/VS analysis were slightly higher than the control digester (data is presented in Table 4 in Appendix). A stepwise increase of OLR to 1.5 g VS/L*d^{-1} of PF resulted in methane yields of 490 ±20 and 470 ±25 CH_4 ml/g VS*d for BR07 and BR09. A corresponding decrease in the HRT to 23 days for BR05 showed a slight decline in methane yield to 530 ±20 CH_4 ml/g VS*d. That notwithstanding, the CH_4 produced from the FW digester could be regarded as high compared to that investigated by Ammenberg and Feiz (2017). The assessment was however based on a batch experiment but with similar FW characteristics as used this experiment. As witnessed, the normalized methane yields from the PF digesters were lower than the control reactor. Comparing to the total methane yield (fig 5), test digesters at 1.5 g VS/L*d^{-1} of PF produced ~32% (BR07) and ~20% (BR09) more methane than average total methane yield of the control digester (11 L/d). Thus, addition of pulp fibres increased the

volumetric methane production but reduced the specific methane yield. This implied that the total degradation rate decreased upon pulp fibre addition. This could only be attributed to the slow hydrolysis rate of cellulose material.

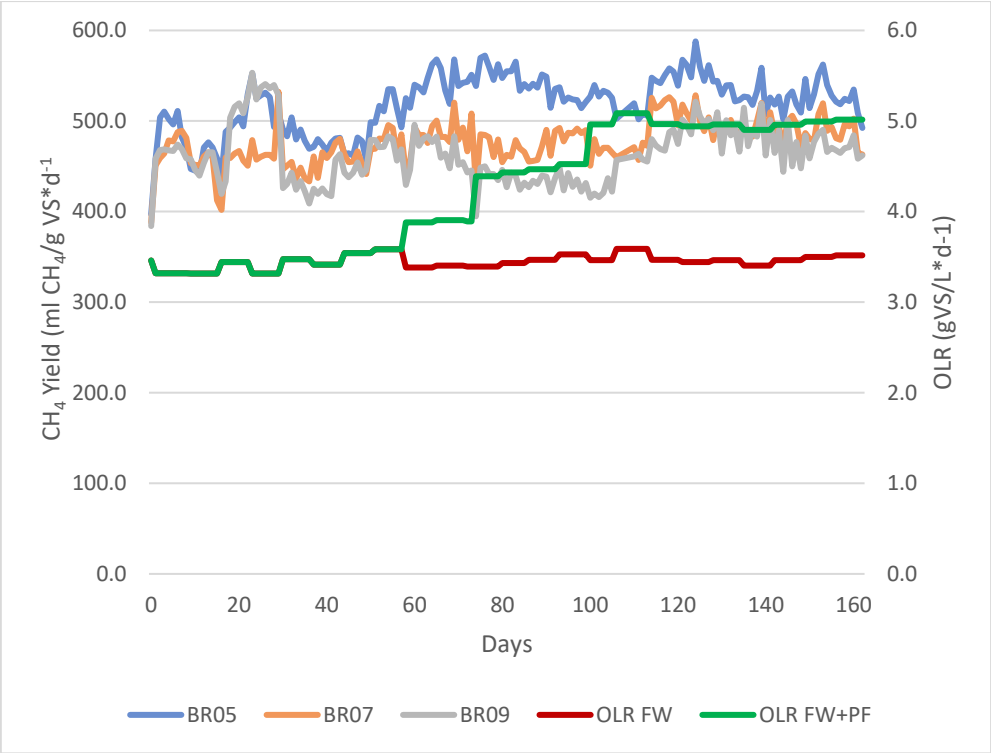


Figure 4. Specific CH_4 yield vs OLR

From literature, key determinants for methane potential are substrate composition and degradability (Labatut and Pronto, 2018). Though the volumetric biogas production was higher in the PF reactors than FW, this did not reflect in the methane generated. A plausible explanation lies with the gas composition. The gas composition presented in Figure 6 and 7, shows a slight percentage drop in methane content resulting to a percentage increase in the corresponding carbon dioxide composition in the fibre digesters. This is typical of carbohydrate-rich substrates as expressed by Schnürer and Jarvis (2018). Though the accumulation of other gases like H_2S, H_2 and N could also have explained the drop in CH_4 content, this was rather unlikely. The daily addition of iron (Fe) to the working volume reduced the H_2S content keeping it at the barest minimum. Other gases content registered less significant changes.

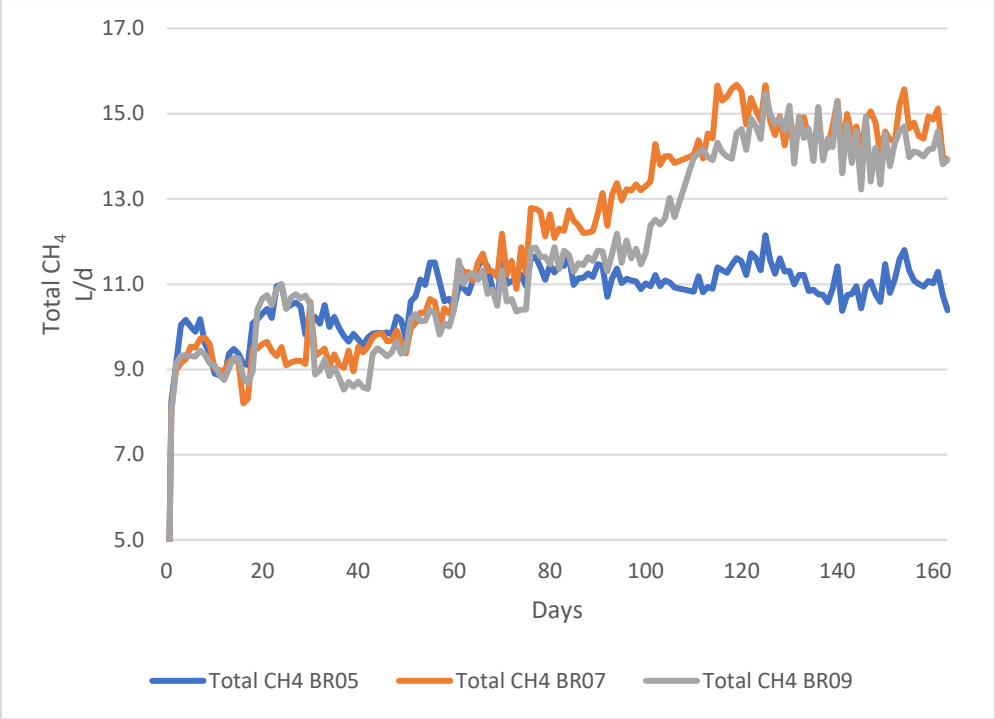


Figure 5. Total CH_4 production in main digesters

Methane production in the post digesters were monitored from day 101-163. This was due to the irregular HRT of the post digesters as explained in section 4.2.2. However, due to the relatively less amount of gas produced during post digestion, gas was collected over 4-day interval and analysed for its methane content. The results showed a high CH_4 % of 48 ±1% and 46 ±1% in BR10 and BR08, respectively than 43 ±1% in BR06. This observation supports the previous assertion that slow degradation during hydrolysis affected the gas performance and efficiency of the PF digesters. Another plausible explanation for the drop in methane yield could be the high C/N ratio of the pulp fibre. Gagnon, Lalande and Fahmy (2001) has reported a relatively high C/N ratio of 109 for pulp fibre considering the optimum ratio of 20-30 as suggested by Mata-Alvarez *et al.* (2014). As usually the case in anaerobic digestion, several characteristics combine to affect the process (Schnürer and Jarvis, 2018).

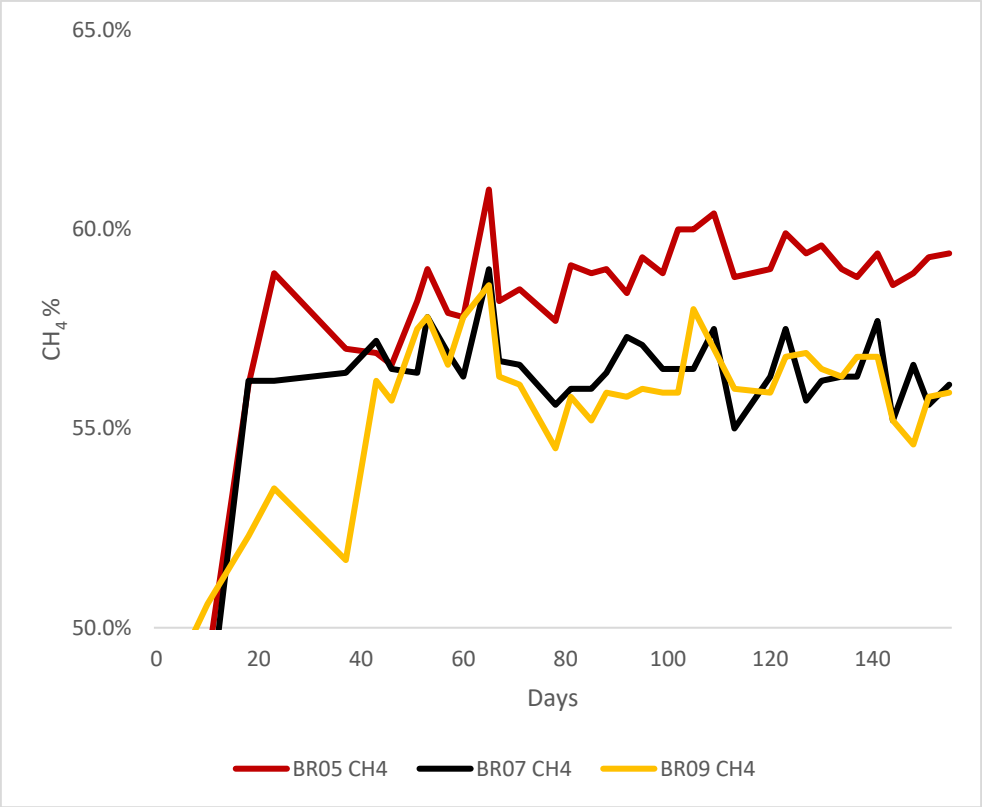


Figure 6. CH_4 content in produced biogas from main digesters

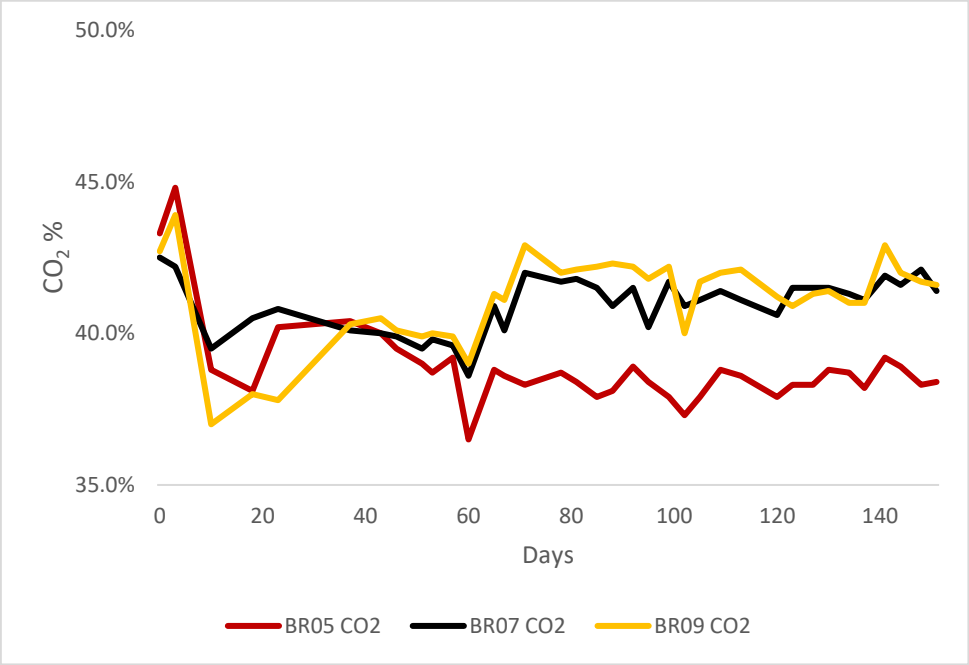


Figure 7. CO_2 content in produced biogas from main digesters

4.3 Effect of pulp fibre addition on nitrogen mineralisation

Nitrogen mineralisation was assessed based on the total ammonium nitrogen (TAN) (NH_4^+-N) concentration in effluents from both main digesters and post digesters (Schnürer and Jarvis, 2018). However, results presented here are for the main digesters whereas the data for post digesters is unreported. The results presented here reflected NH_4^+-N concentrations observed in the main digesters at organic loading of FW 3.5 ±0.1g VS/L*d^{-1} for BR05 and 5 ±0.1 g VS/L*d^{-1} (85% FW + 15% PF) for BR07 and BR09. Ammonium nitrogen concentration testing begun on day 112 of the experiment at OLR FW + PF of 5 ±0.1 g VS/L*d^{-1} and is shown in figure 8.

According to Chen, Cheng and Creamer (2008) inorganic nitrogen exist as ammonium ion (NH_4^+) and free ammonia (NH_3) during digestion. Amongst the two, NH_3 is considered toxic at high concentration and inhibitory to methane production. Inhibitory concentrations is sufficiently reported in literature as dependent on pH, temperature, etc., however Appels *et al.* (2008) suggested a concentration of 4-5.7 g NH_3/L was known to limit methane activity by 56%. BR05 recorded NH_4^+-N mean concentration of 4045 ±70 mg/L whilst BR07 and BR09 gave 2587 ±131 mg/L and 2079 ±205 mg/L respectively. The relatively high methane production from FW for this experiment suggested no inhibition. Nevertheless, the calculated ammonia concentration (196-201 mg/L using equation from Hansen, Angelidaki and Ahring (1998)) was within the range as suggested by Appels *et al.* (2008) to be beneficial for microbial degradation. However, from figure 8, it is evident that addition of PF rather culminated to a reduced total ammonium nitrogen (NH_4^+-N) content in the pulp fibre digesters. Lower NH_4^+-N content indicates that PF addition could have resulted in less degree of organic nitrogen mineralization. This however coincided with the discussed drop in pH in the AD system as experienced in the pulp fibre reactors. From table 3, pH of the pulp fibre digesters (BR07 & BR09) dropped from 7.6 ±0.1 to 7.3 ±0.1 following fibre addition. Ekstrand (2019) stated that the degradation of carbon rich organic substrates usually resulted to acidification of the digestion owing to low alkalinity. Schnürer and Jarvis (2018) further suggested that the degradation of protein rich substrates could explain the high buffering capacity of the control digester even at high TAN concentrations. This phenomenon could likely explain for the relatively lower ammonium nitrogen concentration in the pulp fibre digesters (BR07 & BR09) than present in the control reactor (BR05). Another plausible explanation for this effect could be due to the adsorption capacity of cellulose fibres as discussed in a study by Wrangbert (2021). However, the adsorption capacity for the pulp fibres were considered low. This couldn't have accounted for the large difference in TAN concentration between the control digester and the PF digesters.

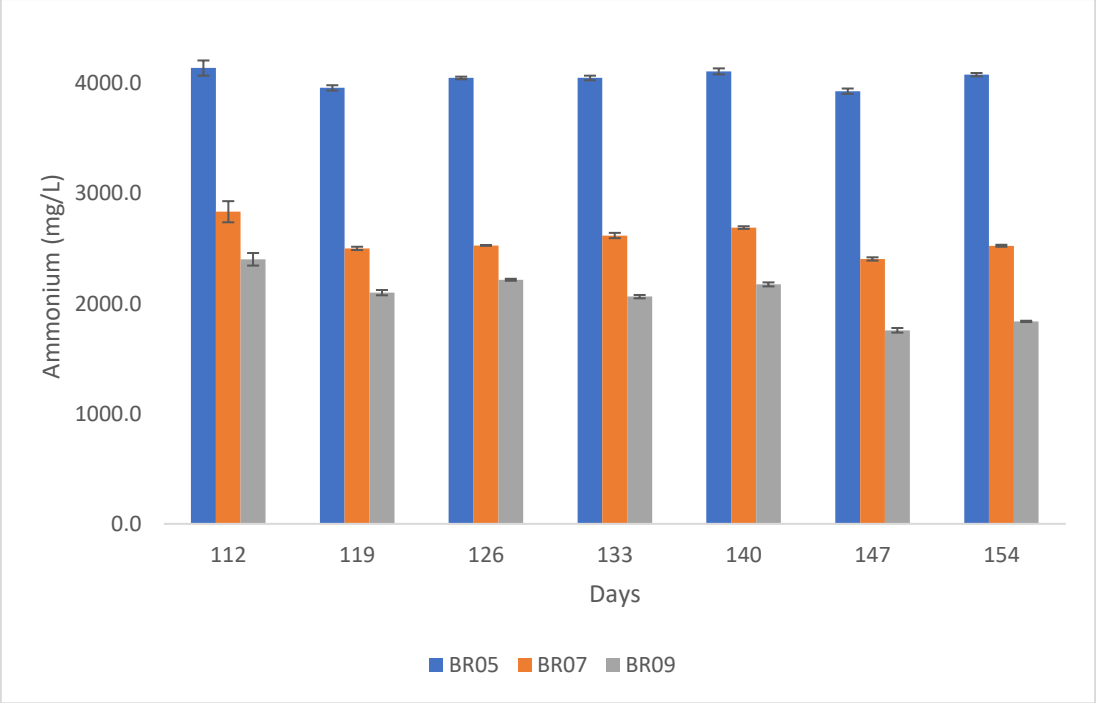


Figure 8. Ammonium nitrogen concentration in main digesters

4.4 Effect of pulp fibre addition on digestate dewaterability

This section of the experiment was structured to test the dewaterability of digestate. Effluents from BR06, BR08 and BR10 were tested. The capillary suction time (CST) measured the filterability of the digestate with respect to time. No conditioning or polymers were included in this experiment. The results suggested decreasing dewaterability according to CST. Wang *et al.* (2020) suggested a negative correlation between dewaterability and the time for filtration with shorter periods signifying better dewaterability. As depicted in figure 9, the CST for BR08 and BR10 increased over the duration of the experiment with increasing fibre addition. Prior to pulp fibre addition, the CST from post digestion of FW digestate averaged 520 ±30s. The control post digester experienced little changes in the CST over the duration of the experiment. From day 59-163, VS% of digestate from the post digesters were observed to be increasing with increasing CST. Wang *et al.* (2020) investigated a relationship between VS and dewaterability of waste activated sludges (WAS) and established a positive correlation between CST and VS. Increasing CST time corresponded to increasing VS% of the digestates from the post digester. This revelation supported the trend shown in figure 9 and 10. CST increased with increasing VS% for BR08 and BR09 thus indicating a reduced dewaterability according to Wang *et al.* (2020). This experiment also investigated this correlation between OM content and CST and found the assertion reliable. Result from the correlation analysis is presented in Table 5-7 and figure 17 (see Appendix). Results suggested a strong positive correlation between CST and VS% of the digestate for BR08 and BR10 with R-values of 0.824^{**} and 0.798^{**} respectively, significant at the 0.01 level. Correlation analysis for BR06 showed a positive BUT moderate correlation at R-value 0.558^{*} significant at the 0.05 level.

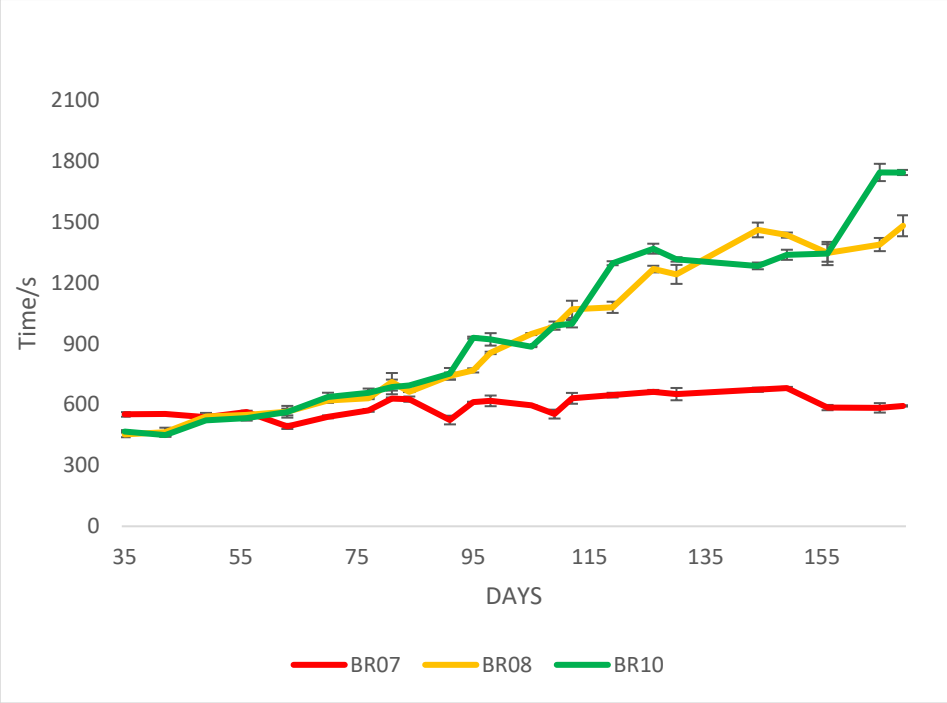


Figure 9. CST from sludge analysis for post-digesters

Figure 10. VS% from sludge analysis for post-digesters

Centrifugation is a popular sludge liquid-solid separation technique which could also be used in assessing dewaterability. For this experiment, the TS% of the cake and the suspended solids in the liquid reject were used to evaluate the dewaterability of the digestate. In all cases, a higher TS% content coupled with a lower weight of suspended solids reflected an improved dewaterability. As depicted in figure 11 and 12, there was a high variability in the TS% and SS dry weight for the centrifuged sludges. However, the mean values suggested a relatively high suspended solids in BR10 of 237 ±10 mg/L than 177 ±12 mg/L and 128 ±10 mg/L in BR08

and BR06 respectively (t-test, $p < 0.05$). Figure 11 suggest a relatively decreasing TS% over time for all the digesters. No significant difference was observed amongst the test samples.

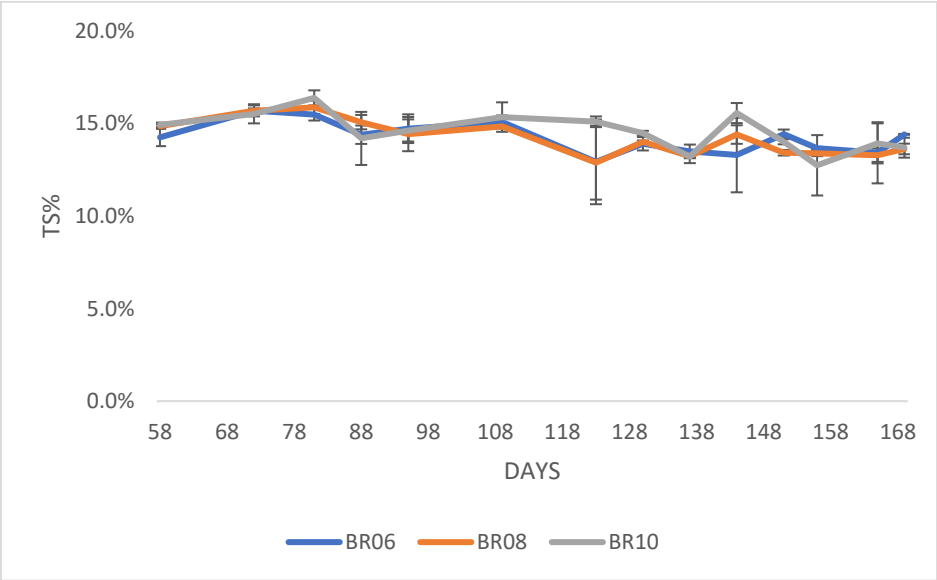


Figure 11. TS% from cake reject after centrifugation

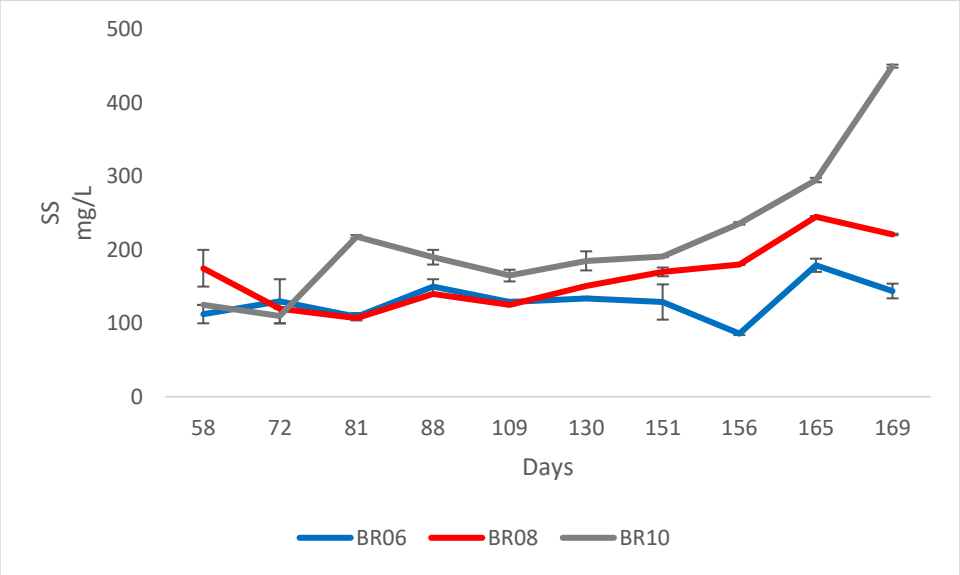


Figure 12. Suspended solids in liquid reject after centrifugation

5. CONCLUSION

The principal aim of this experiment was to enhance the dewaterability of food waste digestate by the addition of pulp fibre to the AD process. In doing so, the study also investigated the effect of pulp fibre as a co-substrate to food waste digestion on the gas performance and stability of the digestion. The study also assessed the effect of the fibres on nitrogen mineralization of the food waste digestion.

The study showed that the co-digestion of FW with pulp fibre improved the degradability of the co-substrates. However, the specific methane produced from the co-digested substrates was lower than methane from FW. Though both total biogas and total methane production were high with the addition of pulp fibre, the increased carbon dioxide production culminated to a reduced the specific methane production. This was because of the substrate composition with specific reference to the high C/N ratio and lignocellulose content of the pulp fibre. The digestion was nonetheless stable with a steady pH and no VFA accumulation.

Pulp fibre addition resulted to a reduction in nitrogen mineralisation of food waste digestion. This could be an alternative to reduce the ammonia content in food waste digesters. Though the addition of pulp fibre caused a reduction in total ammonium nitrogen content in digestate from food waste, the study only investigated this effect at a particular OLR (1.5 g VS/ L*d^{-1}). Further research would be beneficial to investigate this effect at varying organic loads.

The dewaterability was not improved by the co-digestion of FW and pulp fibre according to the results from the CST and centrifuge experiments. The filterability test conducted suggested a rather worsened dewaterability with addition of pulp fibre. The study established a strong positive corelation between CST and OM of the digestate.

The study demonstrated that contrary to previous studies on pulp fibre types, softwood pulp fibre had a marginally higher gas performance when co-digested with FW than as compared to hardwood pulp fibre. However, when given extra 7-day digestion, hardwood fibre produced higher methane than softwood. This suggested that hardwood pulp fibre digestion over a longer retention time could improve gas performance. From the results of this experiment, one could argue that adding fibre is a way to increase daily methane production without causing an inhibited system.

Future studies

There is the need to investigate the economic feasibility of using kraft pulp fibres as a co-substrate for anaerobic digestion.

There is also the need to perform further studies to ascertain or verify the true methane characteristics of the different kraft pulp fibre types when used as co-substrates for anaerobic digestion. Which suggest that the use pulp fibre as a co-substrate is good for AD co-digestion with FW as it did not cause any inhibition that could have resulted in VFA accumulation leading to a significant drop in pH.

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APPENDIX

Phase I

The experiment started with FW at OLR of 2.5 ±0.1 g VS/L*d^{-1} and HRT of 33 days and increased stepwise to 3.5 ±0.1 g VS/L*d^{-1}, HRT 26 days from day 1-6. This was run for 30 days and finally increased to OLR of 4 ±0.1 g VS/L*d^{-1} before setting up for phase 2 of the study. During the pre-experimental phase, Iron (Fe) and trace elements (Ni, Se) were added to BR1 and BR2 to boost microbial activity and curb the accumulation of H_2S when the OLR was increased to OLR of 3.5 ±0.1 g VS/L*d^{-1}. This observation was consistent with as stated in literature as FW digestion beyond OLR of 2.5 ±0.1 g VS/L*d^{-1} is fraught with a number of challenges including inhibitions, foaming, process instability and lower methane yield (Zhang, Ouyang and Lia, 2012; Hegde and Trabold, 2019). The effect of nutrient supplementation is well documented in literature with a little emphasis on FW degradation (Facchin *et al.*, 2013). Data from the experiment suggests or confirms the significance of supplementation of trace elements to the digestion of FW as the reactors started without any trace element supplements. Addition of Fe, Ni and Se manifested as an increase in gas production and later stable digestion due to stabilized hydrogen sulfides (H_2S) production. Iron (Fe) addition begun on the day 8 of the experiment and culminated to the precipitation of sulfides produced during anaerobic digestion. This resulted to a decrease in the H_2S concentration in the gas produced from about 130±10 ppm on day one to about 20±10 ppm on day 40 as displayed in Figure 13. According to Hegde and Trabold (2019), H_2S at low concentrations is significant in maintaining alkalinity. It becomes rather inhibitory to methanogens at higher concentration. However, the low but stable H_2S in the gas relative to the recommended levels of 50 ppm (Schnürer and Jarvis, 2018) continued throughout the rest of the experiment with apparently no effect on the process.

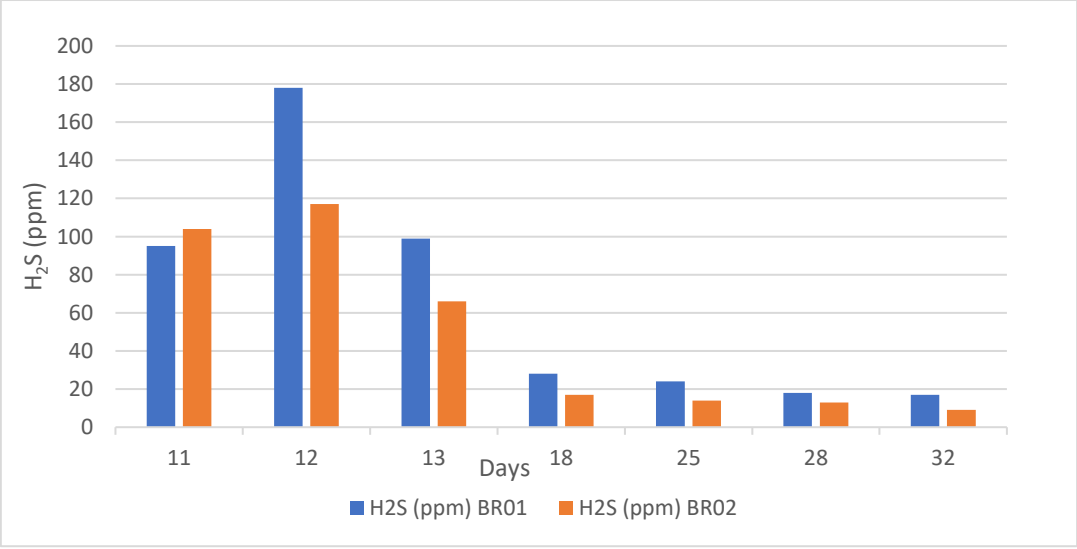

Figure 13. Hydrogen sulphide abatement

Monitored parameters

Table 4. Mean and SD for all monitored parameters at the different OLR for BR05, BR07 and BR09

PARAMETER	DAYS	BR05	BR07	BR09
pH	0-58	7.5 ±0.1	7.6 ±0.1	7.6 ±0.1
	59-74	7.5 ±0.1	7.4 ±0.1	7.4 ±0.1
	75-100	7.4 ±0.1	7.3 ±0.1	7.3 ±0.1
	101-163	7.6 ±0.1	7.3 ±0.1	7.3 ±0.1
TS %	0-58	3.5 ±0.2	3.4 ±0.2	3.3 ±0.2
	59-74	3.5 ±0.0	3.4 ±0.2	3.5 ±0.1
	75-100	3.5 ±0.3	3.8 ±0.0	4.0 ±0.4
	101-163	3.1 ±0.1	3.7 ±0.2	3.9 ±0.2
VS % (% OF TS)	0-58	71 ±2	71 ±1	70 ±1
	59-74	72 ±1	72 ±0	73 ±1
	75-100	73 ±0	74 ±0	75 ±1
	101-163	73 ±1	75 ±1	76 ±1
VS REDUCTION %	0-58	74 ±3	74 ±4	76 ±3
	59-74	74 ±5	77 ±7	76 ±8
	75-100	73 ±3	77 ±2	75 ±4
	101-163	75 ±2	79 ±3	78 ±2
OLR (G VS/L*D^{-1})	0-58	3.4 ±0.1	3.4 ±0.1	3.4 ±0.1
	59-74	3.4 ±0	3.9 ±0	3.9 ±0
	75-100	3.5 ±0	4.5 ±0	4.5 ±0
	101-163	3.5 ±0	5.0 ±0	5.0 ±0
HRT (DAYS)	0-58	26	26	26
	59-74	25	25	25
	75-100	24	24	24
	101-163	23	23	23
MEAN BIOGAS PER DAY (ML/D)	0-58	18.1 ±1	17.5 ±1	18.0 ±1
	59-74	18.8 ±0.5	19.9 ±1	19.0 ±0.6
	75-100	19.1 ±0.4	22.4 ±1	20.8 ±0.6
	101-163	18.7 ±0.5	26.1 ±1	25.2 ±1
CH_4 (% BIOGAS)	0-58	55 ±4	54 ±4	53 ±3
	59-74	59 ±1	57 ±1	57 ±1
	75-100	59 ±0	56 ±1	56 ±0
	101-163	59 ±1	56 ±1	56 ±1
MEAN CH_4 (CH_4 ML/G VS D^{-1})	0-58	490 ±30	460 ±20	465 ±40
	59-74	540 ±15	490 ±20	470 ±20
	75-100	540 ±15	470 ±15	430 ±15
	101-163	530 ±20	500 ±20	420 ±25

MEAN CH_4 (CH_4 ML/D)	0-58	10 ±0.6	9.5 ±0.5	9.5 ±0.7
	59-74	11 ±0.3	11.3 ±0.4	10.8 ±0.4
	75-100	11.2 ±0.2	12.7 ±0.5	11.6 ±0.3
	101-163	11.1 ±0.3	14.7 ±0.5	14.1 ±0.7

Day 0-58: Start-up phase; day 59: Addition of Pulp fibre (95% FW + 5% PF); day 75: increased PF loading (90% FW + 10% PF); day 101; increased PF loading (85% FW + 15%)

Graphs

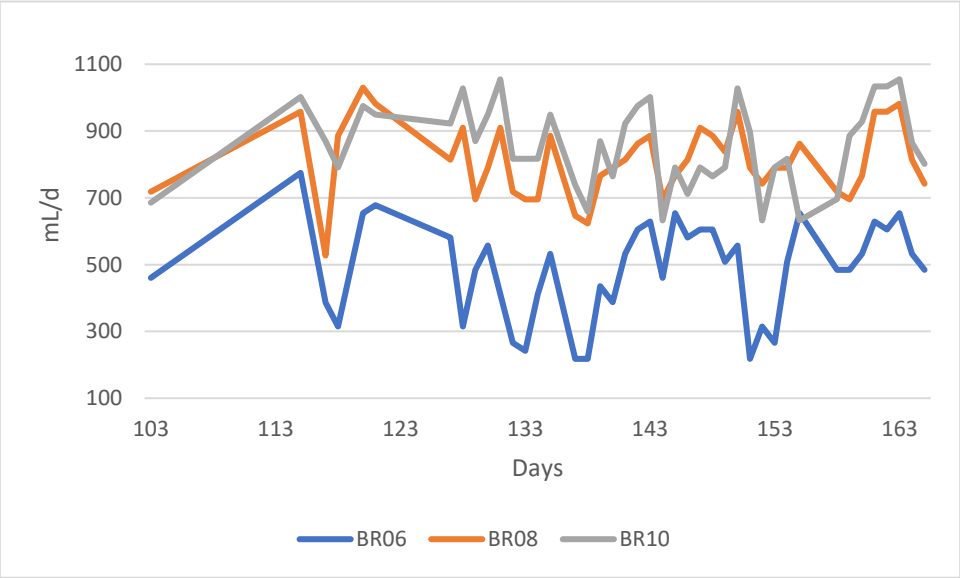

Figure 14. Volumetric biogas produced from post digestion from days 101-163

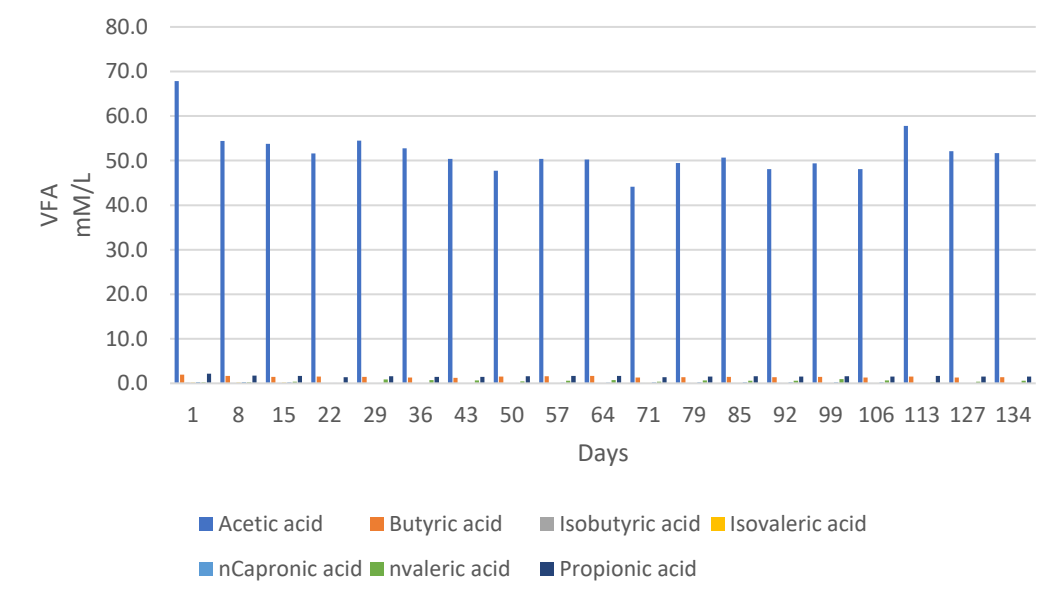

Figure 15. VFA concentration in food waste before digestion

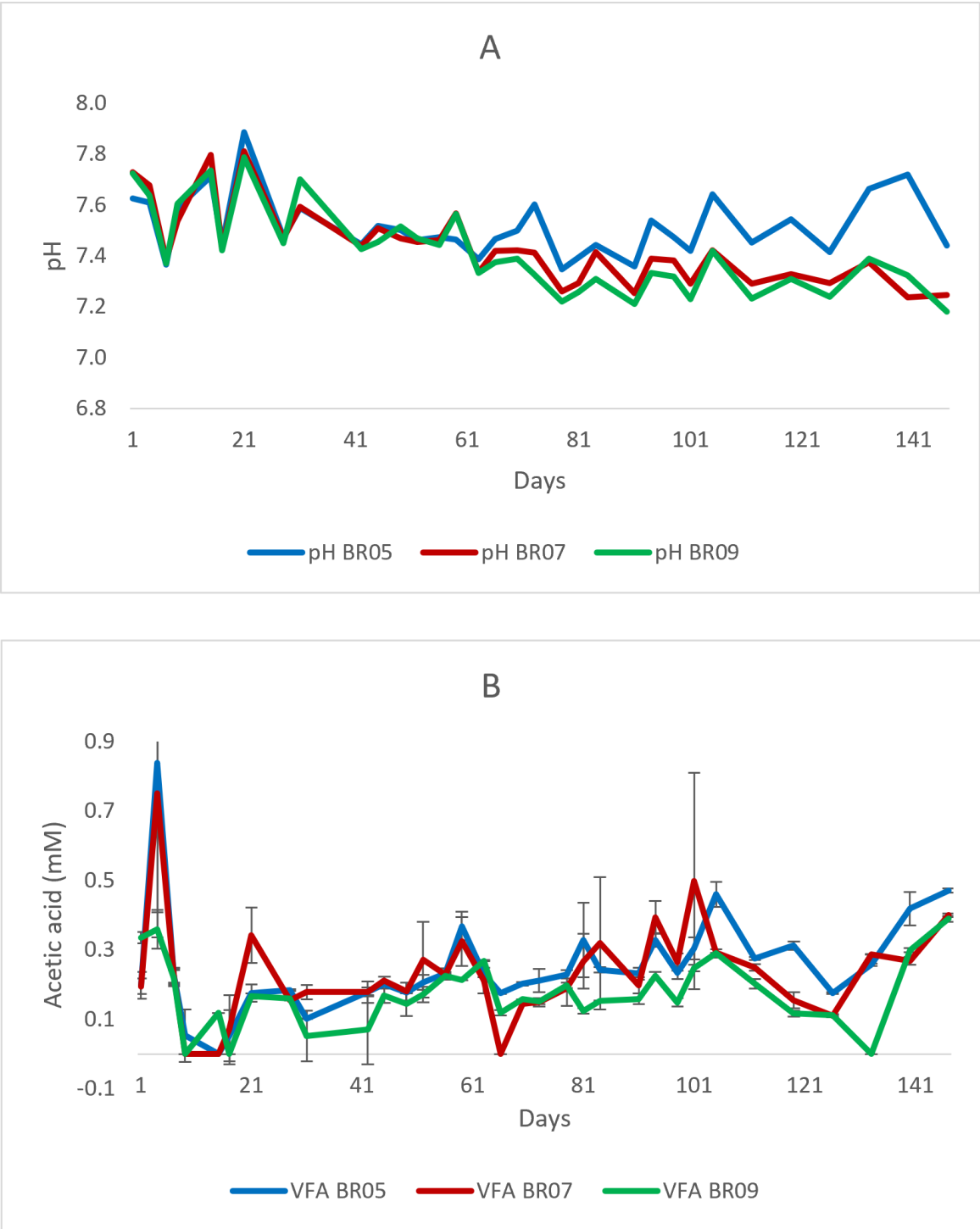

Figure 16. pH & VFA levels in main digesters

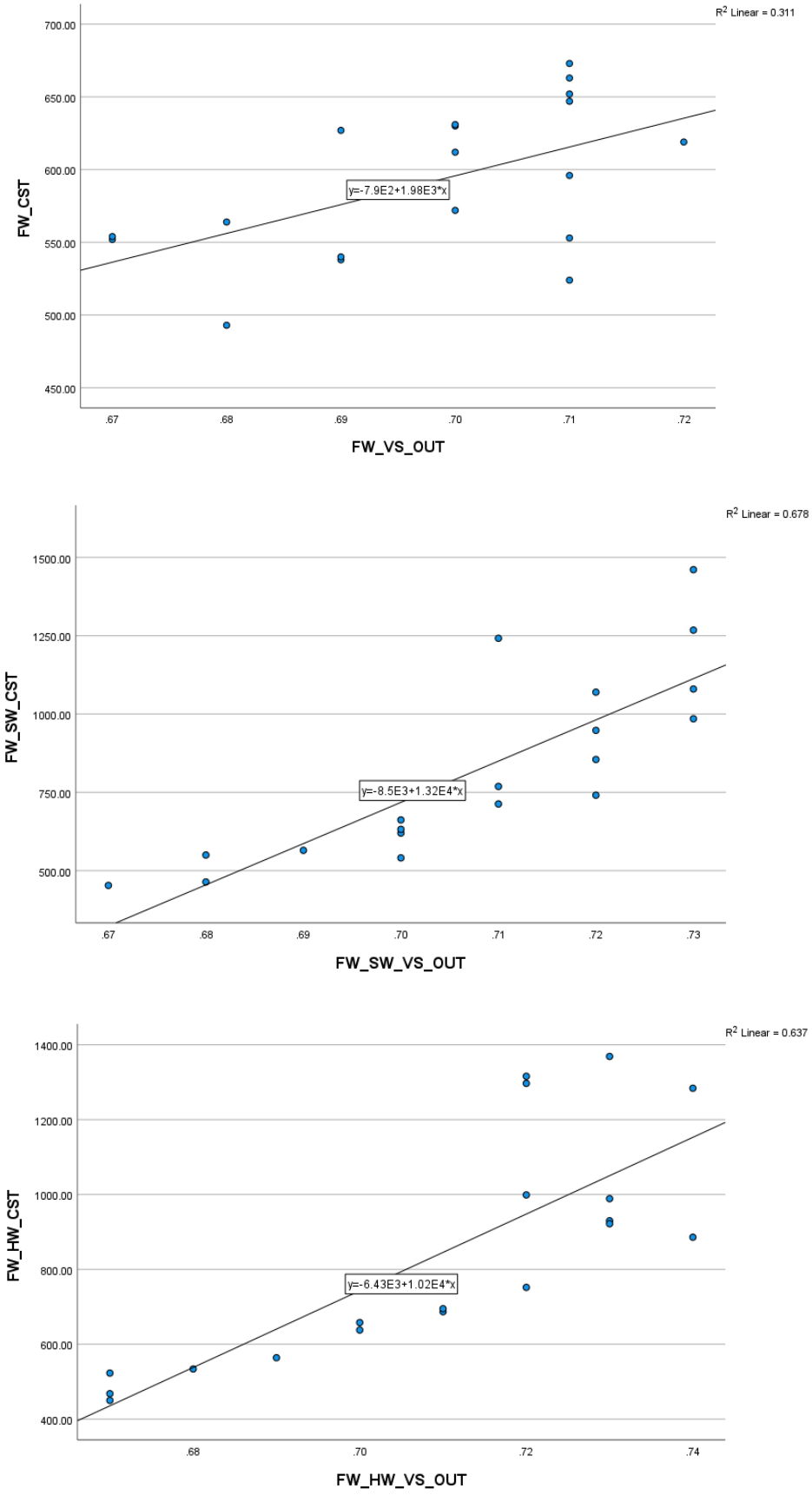

Figure 17. Scatter plots for BR06, BR08 & BR10; CST versus VS% of sludge from post digester

Table 5. Pearson correlation: BR06, CST & VS%

		VS_BR06	BR06_CST
VS_BR06	Pearson Correlation	1	.558*
	Sig. (2-tailed)		.013
	N	19	19
BR06_CST	Pearson Correlation	.558*	1
	Sig. (2-tailed)	.013	
	N	19	23

*. Correlation is significant at the 0.05 level (2-tailed).

Table 6. Pearson correlation: BR08, CST & VS%

		BR08_CST	VS_BR08
BR08_CST	Pearson Correlation	1	.824**
	Sig. (2-tailed)		.000
	N	23	19
VS_BR08	Pearson Correlation	.824**	1
	Sig. (2-tailed)	.000	
	N	19	19

**. Correlation is significant at the 0.01 level (2-tailed).

Table 7. Pearson correlation: BR10, CST & VS%

		BR10_CST	VS_BR10
BR10_CST	Pearson Correlation	1	.798**
	Sig. (2-tailed)		.000
	N	23	19
VS_BR10	Pearson Correlation	.798**	1
	Sig. (2-tailed)	.000	
	N	19	19

**. Correlation is significant at the 0.01 level (2-tailed).