

# Hydrodynamic cavitation applied to food waste anaerobic digestion

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# Abstract

Innovative pre-treatment methods applied to anaerobic digestion (AD) have developed to enhance the methane yields of food waste. This study investigates hydrodynamic cavitation, which induce disintegration of biomass through microbubble formations, impact on food waste solubilisation and methane production during following AD. Two different sub-streams of food waste (before and after the digestion) pre-treated by hydrodynamic cavitation were evaluated in lab scale for its potential for implementation in a full scale practise. First, the optimum condition for the hydrodynamic cavitation device was determined based on the solids and chemical changes in the food waste. The exposure time was referred to as the number of cycles that the sample was recirculated through the cavitation inducer's region. The optimal cycles were later tested as a pre-treatment step in a BMP test and semi-CSTR lab scale operation. The tests showed that sufficient impact from the hydrodynamic cavitation was achieved by 20 cavitation cycles. Due to the pre-treatment, food waste solubilisation increased, up to 400% and 48% in terms of turbidity and sCOD measurements, respectively. In the BMP test, the treated samples improved the methane yield by 9-13%, where the digested food waste increased its kinetic constant by 60%. Fresh food waste was then processed in the semi-CSTR operation and the methane yield was increased by up to 17% with hydrodynamic cavitation for two reference periods. These promising results suggest that the hydrodynamic cavitation can be implemented for full scale production with food waste.







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# Abbreviations

AD – Anaerobic Digestion

BMP – Biochemical Methane Potential

CHP – Combined Heat and Power

CSTR – Continuous Stirred-Tank Reactor

FW – Food Waste

GHGs – Green House Gases

HC – Hydrodynamic Cavitation

HRT – Hydraulic Retention Time

OLR – Organic Loading Rate

sCOD – soluble Chemical Oxygen Demand

TS - Total solids, which also refers as the dry mass

TSS – Total Suspended Solids

VFA – Volatile Fatty Acid

VS – Volatile solids

WAS – Waste activated sludge



# 1 Introduction

The rapid growth in population and consequent energy demand has created challenges to guarantee sustainability development in Sweden. In line with EU regulation and policy for sustainability, Sweden aims to reduce the GHGs emission level to at least 40% by 2020 compared to 1990, with the ambition to achieve zero net emission by year 2050 (Environmental and Agency, 2015; Naturvårdsverket, 2015a; European Council, 2014). In this context anaerobic digestion (AD) represent an optimal alternative way to generate energy from waste biomass otherwise disposed in landfills causing, in the long term, diffuse environmental pollution (Grizzetti et al., 2013). With more than 1 million tonnes of food waste (FW) produced every year in Sweden, this biomass can significantly contribute to achieve GHGs emission targets if utilised in the AD process. In order to increase the energy recovery and pollution reduction, Sweden targets to increase the recycle and digestion of FW to 40% in 2018, compared to 21% in 2013 (Naturvårdsverket, 2014; Naturvårdsverket, 2015b).

AD is a biological process where organic matters are degraded to biogas in absence of oxygen. The biogas, mainly a methane-carbon dioxide gas mixture, is a valuable and sustainable biofuel that can either be used as vehicle fuels or as combined heat and power (CHP) (Weiland, 2010; Reith et al., 2003). It has been estimated that the GHGs emission could potentially be reduced by 25% in Sweden, by using biofuels for national road transportation (Olsson and Falld, 2014). Conventional organic matter for AD include the utilisation of waste material such as food waste (FW) and municipal wastewater sludge (Deublein and Steinhauser, 2010). The utilisation of these waste materials contributes to the GHGs emission reduction. Additionally, it has been reported that technical and economical limitations has restricted theoretical biogas production potential on FW to 0.76 TWh. The current biogas production potential could be enhanced and improved by additionally 77% (SGC, 2012). The benefits from an enhanced AD process on FW would (i) use lesser amount of FW to produce the same amount of biogas, (ii) establish a more efficient biogas production with more final products, and lastly (iii) more biogas products would increase the renewable energy production.

In general, the AD process could be improved by several pre-treatment methods on the feedstock such as thermal, mechanical or chemical treatment (Hendriks and Zeeman, 2009). Ultrasonic pre-treatment is using the cavitation phenomena to ease the degradation of organic materials (Appels et al., 2008). However, due to the high energy demand, this method is inefficient for large scale applications. In comparison, hydrodynamic cavitation (HC) is using the same phenomena as ultrasonic treatment, but is considered to be more energy efficient for large scale applications (Deublein and Steinhauser, 2010; Gogate, 2011).

Currently, HC has mainly been studied in food and water sterilising applications with promising impact (Gogate, 2011). More recently, the interest has been shifted towards biofuels such as ethanol and biogas production. The HC treatments improved the digestion performances by primarily reduced the particles size and increased the solubilisation on the feedstock (Ramirez-cadavid et al., 2013) (Lee and Han, 2013). Mainly, HC has been introduced to AD on wastewater activated sludge (WAS), where limited studies have been performed on FW. Thus,



HC applied on FW as a pre-treatment step is a novel approach for the AD process. This thesis work investigates the HC impact on FW in lab scale and its potential to be introduced for full scale biogas production. The expectations are an enhanced process that increases the sustainable bio-methane and energy production in line with Sweden's GHGs emission and pollution reduction objectives.



# 2 Background

## 2.1 Principles of Anaerobic Digestion

Anaerobic digestion (AD) is a biological fermentation process where microorganisms convert organic materials into biogas in the absence of oxygen (Reith et al., 2003). The organic degradation process can be divided into four different phases (Figure 1): hydrolysis, acidogenesis, acetogenesis and methanogenesis (Deublein and Steinhauser, 2010).

During the first phase, the hydrolysis, organic substances such as complex proteins and polymeric organic compounds, are degraded into simple organic matters, such as carbohydrates, proteins and lipids (Deublein and Steinhauser, 2010). Subsequently, the suspended organic matters are further degraded to monosaccharides, amino acids, long-chain fatty acids and glycerol (Figure 1) (Angelidaki et al., 2011). Depending on the complexity of the substrate, the rate for the hydrolysis phase might vary from hours to days. For instance, carbohydrates are easier to be hydrolysed than proteins and lipids. The first, achieves a high degradation rate after only few hours, while days are required to process complex proteins and lipids (Angelidaki et al., 2011; Pind et al., 2011). For this reason, the hydrolysis phase is often the rate limiting step of the overall process preventing efficient functionality of the following steps. (Ariunbaatar et al., 2014; Dhamodharan and Kalamdhad, 2014; Pind et al., 2011).

During the acidogenesis phase the organic compounds decompose into smaller short-chain organic acids ( $C_1$ - $C_5$  molecules) and VFAs such as butyric acid, propionic acid and alcohols are formed (Deublein and Steinhauser, 2010; Angelidaki et al., 2011). The VFAs are then further degraded into acetic acid,  $CO_2$  and  $H_2$  by the acetogenic microorganism (the third phase). It is during this phase that ammonia formation from organic compounds that contain nitrogen and sulphur, e.g. proteins, takes place (Figure 1). The acidogenesis and acetogenesis phases are both considered as the fermentation step, and their products affects the AD process significantly (Massé et al., 2011). The accumulation of VFAs ( $> 3000$  mg/l) causes pH drops, while excess of ammonium ( $> 10000$  mg/l) result to pH increment and toxication for the process (Dhamodharan and Kalamdhad, 2014; Deublein and Steinhauser, 2010). In order to have a stable process and avoid inhibition, it is critical to maintain these substances at moderate level in consideration to the operating pH (Pind et al., 2003). The degradation rate for amino acids and sugar are rather fast and the time for the acido- and acetogenesis ranged in minutes. However, for more complex substrates it might require days to degrade (Figure 1).

Ultimately, the last phase in the AD process is the methanogenesis, where methane is produced by methanogenic microorganism that belongs to the archaea domain (Angelidaki et al., 2011). Approximately 30% of the methane production are formed by  $CO_2$  and  $H_2$  reduction, while the other 70% are from conversion of the acetate to methane (Deublein and Steinhauser, 2010) (Figure 1). The gaseous mixture of methane (55-75%), carbon dioxide (25-45%) and other minor gases such as  $H_2S$  forms the final product biogas (Reith et al., 2003). In



comparison to the previous phases, the rate for methanogenesis is considerable fast and ranges from seconds to minutes (Pind et al., 2003).

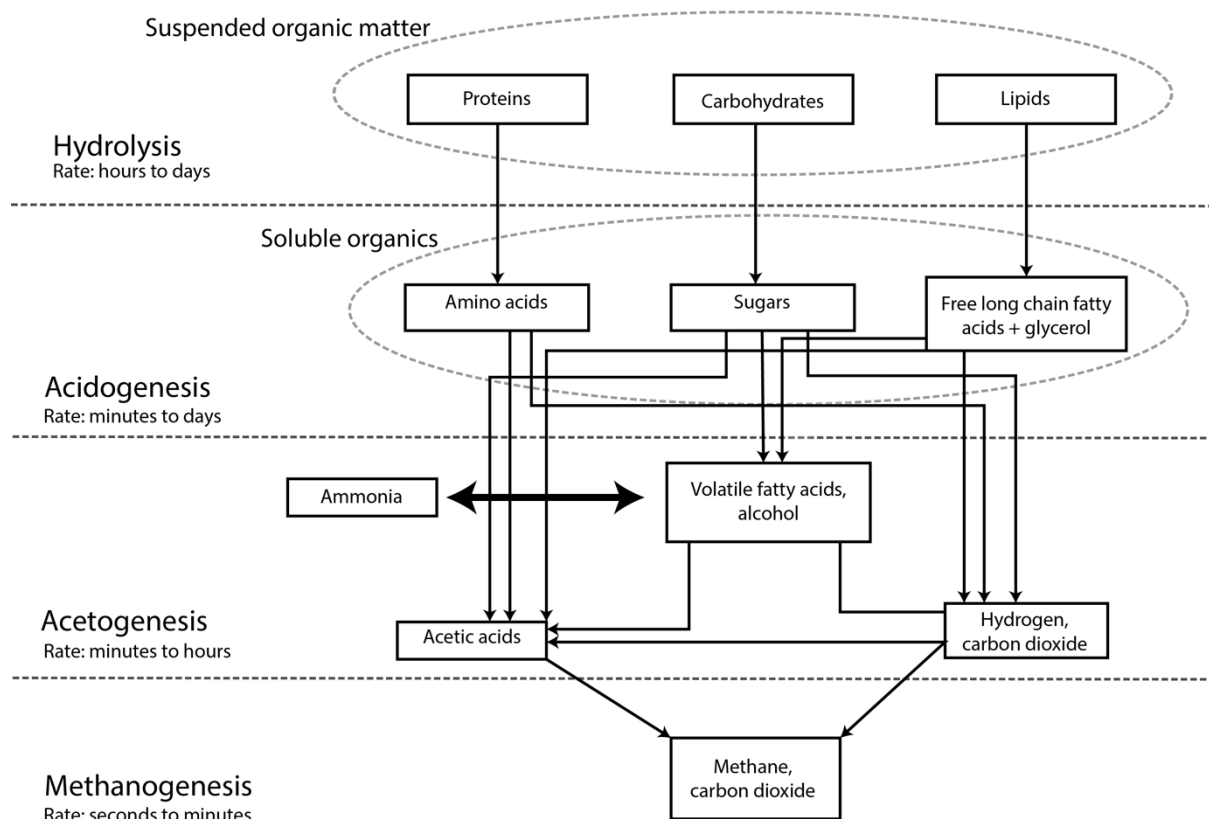


Figure 1. Scheme for the AD process. Organic materials are degraded into smaller substitutes by different groups of microorganisms in different steps, to form methane and carbon dioxide as the final products.

### 2.1.1 Monitoring of the AD process

The overall anaerobic digestion process is controlled by various parameters such as pH, temperature, organic loading rate (OLR) and hydraulic retention time (HRT). Optimum pH ranges between 6.5 and 8.5. However, different microorganisms operate efficiently even at lower or higher values. For instance, the acidogenic and acetogenic are not sensitive to pH changes, while the methanogenic community require a pH ranged between 6.5 and 7.2 for optimum functionality (Appels et al., 2008).

The OLR and HRT is substrate depended, which is why no general optimum range for AD has been reported in the literature. Often, high OLR combined with low HRT are responsible of overloading or bacteria washout, leading to process failure (Dhamodharan and Kalamdhad, 2014; Appels et al., 2008).

Conventionally, the AD systems are operated at mesophilic (20-40°C) or thermophilic (50-60°C) conditions (Reith et al., 2003). The organic degradation activity increases along with higher temperature with thermophilic conditions being more suitable for higher OLR and shorter HRT (Sánchez et al., 2001). Compared to mesophilic systems, thermophilic condition also have a positive impact on metabolic rates and specific growth rates enhancing biogas



production (Zhang et al., 2014). However, thermophilic systems are more sensitive to temperature changes. Therefore, these systems require higher heating maintenance and energy input, which limits their full scale applications (Sen et al., 2016; Zhang et al., 2014).

### 2.1.2 AD on food waste

The utilisation of FW as substrate in the AD, along with sewage sludge and manure treatment, is a well-established process for sustainable energy production (Sen et al., 2016; Zhang and Jahng, 2012). This substrate provides only in Sweden up to 0.8 TWh per year (SGC, 2012).

Depending on its primary source (e.g. household, industrial, country), its composition, energy content and bioavailability varies largely. On average, the moisture content of FW ranges between 74% and 90%, with a VS content equal to 80-97% of TS. The C:N ratio a key parameter to guarantee efficient digestion condition with optimum values between 20 and 40, is often between 14.7 and 36.4 (Zhang et al., 2007). These variations are responsible of methane yields fluctuation up to 22% depending on the specific FW processed. Under mesophilic condition, average values range between 400 and 500 ml/g VS (Table 1) with lower yield when processing households FW ( $419 \pm 45$  mL CH<sub>4</sub>/g VS) than commercial FW ( $535 \pm 20$  mL CH<sub>4</sub>/g VS) e.g. canteens, restaurants and hotels (Browne et al., 2013).

Stable AD on FW is usually achieved for OLR and HRT ranged between 1.68-2.8 g VS/L day and 12-20 days, respectively (Mata-Alvarez et al., 1992). Higher OLR (4 to 10 g VS/L day) has been reported to increase the risks for VFAs accumulation and inhibition of the methanogenic activity (Cho et al., 1995). Similar results were observed for Nagao et al (2012), when a HRT and OLR equal to 8 days and 5.5 g VS/L day, respectively, caused process failure.

It has been demonstrated that the mesophilic condition resulted to more solubilisation and higher biogas production on FW, than the thermophilic (Komemoto et al., 2009). However, other studies have reported a higher methane yield (518 mL CH<sub>4</sub>/g VS) with higher OLR (16 g VS/L) in thermophilic digester than the mesophilic (Table 1) (Liu et al., 2009). Kim et al. (2006) utilized a three stage anaerobic digestion and obtained 45-54% higher methane yield in the thermophilic digester compared to the mesophilic. Additionally, the performance did not reveal any inhibition of free ammonia, which confirms that the thermophilic microbial community tolerates significant more ammonia levels than the mesophilic one (Gallert et al., 1998)

Due to the complex FW characteristics, it is suggested to perform AD in single stage reactors rather than multistage (Zhang et al., 2014). Up to 38% more methane yield was obtained in single stage reactors compared to two-stage reactors (Nagao et al., 2012). In comparison to other conventional substrates for AD such as to sewage sludge, the FW lacks the heavy metal elements in order to support the microbial activities. For instance, selenium is an important trace element for increment in the OLR. Given this reason, the FW is required to be coped with addition or co-digested with substrates that are rich in trace elements (Zhang et al., 2014).



Table 1. Summary of other studies on AD with food waste. Adapted with some modification from (Dhamodharan & Kalamdhad, 2014).

Waste Type	Reactor configuration	Biogas yield	Conditions maintained	References
Food waste	Batch solid state AD	472 mL CH <sub>4</sub> /g VS added	Mesophilic condition	(Cho et al., 1995)
Food waste	Three stage anaerobic reactor	223 mL CH <sub>4</sub> /g soluble COD <sub>degraded</sub>	Thermophilic conditions and varies HRT	(Kim et al., 2006)
Food waste	Batch reactor	518 mL CH <sub>4</sub> /g VS	Thermophilic condition	(Liu et al., 2009)
Dairy manure and food waste	Hybrid anaerobic solid-liquid bioreactor	302 mL/g VS of fine materials	Mesophilic condition	(El-Mashad and Zhang, 2010)
Food waste	Single stage wet CSTR AD	455 CH <sub>4</sub> mL/g VS	Mesophilic condition and 9.2 kg VS OLR	(Nagao et al., 2012)
Food waste	Single stage wet CSTR AD	478 mL CH <sub>4</sub> /g VS	Mesophilic condition	(Mata-Alvarez et al., 1992)

### 2.1.3 Pre-treatment

Adoption of pre-treatments and process design optimisations, could increase the biogas/methane production between 10%-40% depending on the specific solution adopted (Table 2) (Dhamodharan and Kalamdhad, 2014). The benefits of pre-treatments are to increase the biodegradability through pre-degradation of the organic materials supporting efficient hydrolysis (Appels et al., 2008).

In respect to the pre-treatment's principle and working mechanism, pre-treatments can be classified into four separately categories: physical, chemical, biological and combined principles (Figure 2). The physical principle involves mechanical grinds, thermal heating or ultrasounds (cavitation) to induce particle size reductions and cell wall disruptions. As a result, it increases the surface area, which provides better contact between the substrate and the anaerobic microorganisms (Ariunbaatar et al., 2014). In contrast, the chemical principle involves strong alkali or acid to hydrolyse the cell walls for intracellular solubilisation (Hendriks and Zeeman, 2009). The biological principle, instead, utilize bacterial or enzymes addition to catalyse the hydrolysis reactions. The choice of pre-treatment depends mainly on the feedstock and overall process design. Modification on the feedstock may cause changes in the viscosity of the process liquid that affects the AD operation (Björn et al., 2012). Consequently, some AD operation require combined pre-treatment principles to achieve desirable results (Appels et al., 2008).



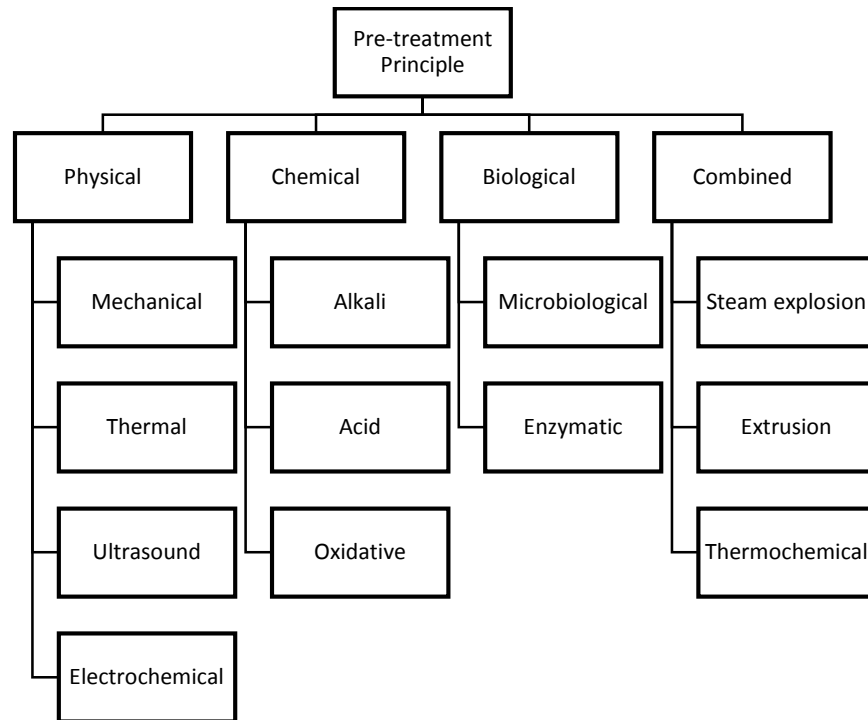


Figure 2. Illustration of different pre-treatment principles and techniques for AD. Adapted from (Appels et al., 2008)

A few examples on how different pre-treatments impacted FW digestion performances are reported in Table 2. Most of the organic content in FW exists in its solids form, i.e. the carbohydrates, proteins and lipids are enclosed in rice, vegetables and meat. The use of physical pre-treatment for particle size reduction has been reported to increased biomass solubilisation and final methane yields by different authors (Zhang et al., 2014; Dhamodharan and Kalamdhad, 2014). For instance, using bead mill, Izumi et al. (2010) reported a 28% methane production increment followed an average particle size reduction by 51%. Additionally, physical pre-treatment includes other objectives such as separation of non-wanted objects, hygienisation and mixing properties that supports the overall plant operation (Bernstad et al., 2013). Similar results were obtained with thermal, freezing-thaw, pressure-depressure and thermo acid pre-treatments (Table 2) (Ma et al., 2011).

However, pre-treatments do not always guarantee a positive impact of the biogas production. For instance, chemical acid treatment performed on FW decreased the biogas production by 66% (Ma et al., 2011). It has also been found that excessive of size reduction inhibits the AD process by VFAs accumulation (Izumi et al., 2010). Additionally, the biogas or biomethane increment are not always enough to cover the additional energy demand of the pre-treatment resulting in an undesirable cost (Zhang et al., 2014; Shahriari et al., 2013).



Table 2. Summary of different pre-treatment methods on food waste for AD. Adapted from (C. Zhang et al., 2014).

Pre-treatment principle	Technique		Result	Mechanism	Ref.
Physical	Microwave	145°C	Increased biogas production	Disrupted sludge and increased solubilisation	(Shahriari et al., 2013)
	Thermal	120°C + 30 min	Biogas production increased by 11%	Increasing the solubilisation	(Ma et al., 2011)
	Freezing-thaw	-80-55°C	Biogas production increased by 23%	Cell disruption	(Ma et al., 2011)
	Pressure-depressure	Pressure changed from 10 bar to 1 bar with CO <sub>2</sub> as pressurizing gas	Biogas production increased by 35%	Breaking up the microbial cell walls and increasing the solubilisation	(Ma et al., 2011)
	Mechanical	Bead milling at 1000 rpm.	Increased methane yield by 28%	Size reduction and increasing solubilisation.	(Izumi et al., 2010)
Chemical	Acid	With 10 mol/L HCl at room temperature (18±2°C) until pH 2 for 24 h	Biogas production decreased by 66%	Forming inhibitors	(Ma et al., 2011)
Biological	Biological solubilisation	FW + water	Decreased organic concentration in the effluent	Increasing the solubilisation	(Gonzales et al., 2005)
Physical-Chemical	Thermo-acid	With 10 mol/L HCl at room temperature (18±2°C) until pH 2 for 24 h, and then 120°C + 30 min	Biogas production increased by 18%	Increasing the solubilisation	(Ma et al., 2011)



## 2.2 Cavitation

Cavitation is a phenomena that occurs in liquid when pressure changes over time and distance (Ozonek and Lenik, 2011). The pressure changes create bubbles that collapse violently. As a result, this generates local high temperature and pressure, up to 10 000 K and 1000 bar respectively enabling physical and chemical transformations of the fluid (Iskalieva et al., 2012; Gogate, 2011; Gogate and Pandit, 2001; Badve et al., 2013). The physical transformation results in increased circulation and turbulence in the liquid, which affects the biomass structure. The chemical transformations increase the free radical formations such as OH, O, H radicals, H<sub>2</sub>O<sub>2</sub>, O<sub>3</sub> molecules inside the bubbles. These radicals diffuse later out from the bubbles to interact with the surrounding organic materials (Iskalieva et al., 2012). Thus, the features from the cavitation will enhance the biological degradation on FW, in line with the promising properties as a physical pre-treatment (Table 2, Figure 2).

Currently, cavitation can be performed in four different way based on their mode generation: acoustic, hydrodynamic, optic and particle cavitation. The optic and particle cavitation fails to induce desirable chemical changes in the fluid, which is why more focus have been on the acoustic and hydrodynamic cavitation (Gogate and Pandit 2001).

### 2.2.1 Hydrodynamic Cavitation

The hydrodynamic cavitation (HC) process can be induced by circulating fluid through an orifice. The orifice forms constriction that increases the liquid velocity at expense of the pressure drop (Figure 3). When pressure drops below the liquid's vapour pressure, cavities forms and collapse violently at the downstream, as the pressure degenerates back to its former condition (Gogate and Pandit, 2001). A dimensionless parameter, cavitation number ( $C_v$ ), relates the flow conditions with the cavitation intensity. Ideally, cavitation is generated when  $C_v$  is between 0.1-1, which can be obtained by adjusting the flow condition and reactor geometry (Bagal and Gogate, 2014).

Additionally, the cavitation effectiveness is divided into three groups of parameters that affects the functionality of cavitation (Ozonek and Lenik, 2011). The first group consist of parameters regarding the device construction such as the size and shape of the cavitation inducer and the flow chamber. The next group involves parameters that characterise the quality of the liquid medium. The last and third group of parameters involves the technological process, i.e. the number of times the medium are passing through the cavitation region. This thesis work, investigated the second and third groups of parameters to find the most optimum condition to perform the AD of FW with HC.



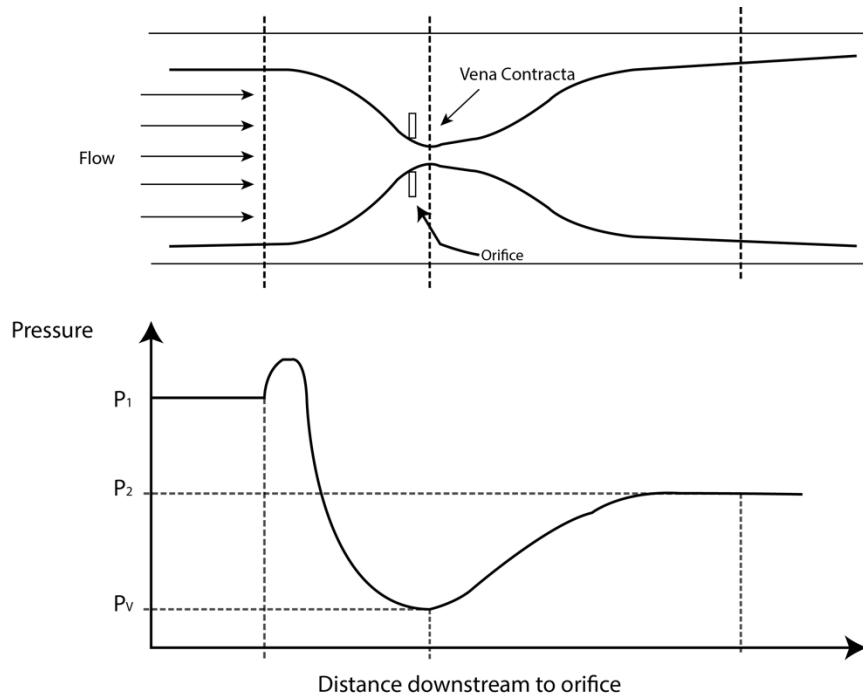


Figure 3. Illustration of the formation of cavitation. The applied orifice in a liquid flow will establish a pressure drop in the system. If the pressure drops below the vapour pressure of the liquid, usually around vena contracta or  $p_v$ , bubbles and cavitation will form. Ultimately, the bubbles will collapse when the pressure recovers from the expanding jet at  $p_2$ . Modified from Gogate and Pandit (2001).

### 2.2.2 Cavitation applications

Acoustic cavitation has already been established in full scale for waste water treatment plants (WWTP) for sludge disintegration (Appels et al., 2008; Pilli et al., 2011). Acoustic cavitation employs cavitation through sound waves, usually ultrasound (16KHz-100KHz) (Gogate and Pandit, 2001). It has been demonstrated that the acoustic cavitation on WAS has successfully increased the solubilisation ratio by 80%, enhanced the digestion by 46% with 40 min treatment at 200 W, and enhanced AD by 42.4% at intensity 18 W/cm<sup>2</sup> (Neis et al., 2000; Shimizu et al., 1993; Wang et al., 1999). Acoustic cavitation is promising, but the high energy demand prevents its full scale application (Weemaes and Verstraete, 1998). It is mainly applied in a bypass system where 30% of the sludge is treated (Deublein and Steinhauser, 2010).

Therefore, interest has been shifted towards the HC applications for AD, where this approach has been founded to be more energy efficient and superior than acoustic cavitation (Gogate and Pandit, 2001). Other studies concluded that the HC enhanced the AD process by shortened retention time, lower operating cost, better digested sludge, and thus improved the wastewater management (Table 3) (Petkovšek et al., 2015). In similarity, Lee and Han (2013) showed that the HC was more physically effective and energy efficient in comparison to ultrasound. The energy consumption for the HC (60-1200 kJ/kg TS) was reported to consume three times less energy than the ultrasound treatment (180-3600 kJ/kg TS). HC treated sample improved the methane yield by 13%, when HC was combined with NaOH it resulted in 71.4% higher yield than the control.



A few other examples of HC impact on various applications are reported in Table 3. For instance, a shockwave power reactor (SPR), a type of HC reactor, was utilized with promising impact on food sterilization processing (Milly et al., 2007). The reactor consisted of a stationary outer cylinder, and a rotating inner cylinder with orifices to generate cavitation. The mass transfer in the liquid was increased, and elimination of common spoilage microorganism such as lactic acid bacteria and yeast was obtained at lower pasteurisation temperature. Consequently, the energy consumption were reduced significantly from 258 kJ/kg to 173 kJ/kg (Milly et al., 2008).

More recently, there is an increased interest on HC for biofuels production, such as ethanol and biogas production. It has been reported successfully effect with HC on delignification for bioethanol production (Kim et al., 2015). Ramirez-cadavid et al. (2013) investigated a combination with controlled flow cavitation (CFC) and enzymatic cellulose hydrolysis to improve a commercial scale bioethanol production. The cavitation alone increased the ethanol production by average 2.2%. In addition with enzymatic cellulose, the ethanol yield was increase by 4.3%. The extra produced ethanol was equal to 38 times more in value than the energy used for the CFC.



Table 3. Overview of Hydrodynamic Cavitation on various application fields.

Application	Treatment	Experimental settings	Result	References
<b>Food sterilization</b>	Apple juice and skim milk	UV-SPR HC, varying rotational speeds of inner cylinder and exit temperature below 45°C.	Increased the mass transfer in the liquid. Increased inactivation of E. coli 25922.	(Milly et al., 2007)
<b>Food sterilization</b>	Apple juice, tomato juice and skim milk	SPR HC with 2 rotor speeds and flow rates to achieve two designated exit temperatures.	Common spoilage microorganisms such as lactic acid bacteria and yeast could successfully be eliminated at lower pasteurisation temperature. No impact on lethality for Gram-positive bacteria was observed.	(Milly et al., 2007)
<b>Food sterilization</b>	Calcium-fortified apple juice	SPR HC with rotating pock-marked inner cylinder.	Enhanced the lethality of spoilage microorganisms at reduced processing temperature and energy input.	(Milly et al., 2008)
<b>Ethanol production</b>	Corn (starch and cellulose)	Controlled Flow Cavitation (CFC)	Reduced the particle size, increased the starch and cellulose conversion, and ethanol production. The extra produced ethanol was 38 times more than the cost of the electricity used for the CFC.	(Ramirez-cadavid et al., 2013)
<b>Ethanol production</b>	Reed (lignocellulosic material)	HC assisted alkaline (NaOH) pre-treatment	HC treated sample exhibited a rapid hydrolysis rate and digestibility compared to ultrasound. After fermentation, an ethanol yield by 90% was achieved.	(Kim et al., 2015)
<b>Anaerobic Digestion</b>	Waste-activated sludge	HC with orifice plate consist of 27 holes of 1 diameter. Pressure upstream and downstream were kept at around 0.7 and 0.07 MPa. Cavitation number was estimated to 2.79. Maximum capacity was 1.5 L.	HC was considered to be more effective as a pre-treatment method compared to ultrasonic and thermal methods. A significant synergy between HC and NaOH as a combined pre-treatment was observed in the methane yield.	(Lee and Han, 2013)
<b>Anaerobic Digestion</b>	Waste-activated sludge	Rotational generator of HC with different number of passes through the instrument was evaluated. The execution was in a pilot plant with a volume by 400 L.	The sCOD was increased from 45 mg/L to 602 mg/L and 12.7% more biogas was produced after 20 times of passes.	(Petkovšek et al., 2015)



## 2.3 Aim and objectives

Based on the information available in the literature and preliminary investigation performed at Scandinavian Biogas Fuels AB, it has been hypothesized that HC applied to the AD process will support digestion efficiency. The limited information available on HC applied to AD, highlights the need of further investigation at lab scale to determine optimum HC condition (Figure 4). Hence, the aim for this project was to examine and optimise the HC, as a pre-treatment for AD of FW. Consequently, the following research questions (RQ) have been formulated:

- RQ-A. What is the impact of HC on FW physical and chemical characteristics?
- RQ-B. What is the impact of HC on FW methane yield and bio-degradation rate?
- RQ-C. What is the expected impact of introducing HC on full scale FW AD plants?



# 3 Material and Methods

## 3.1 Experimental plan

To address the formulated research questions, the work was divided into three experiments with differently procedures (Figure 4). The objective was to successively narrow down a range with number of cavitation cycles until the optimal condition was found for the last experiment.

*Experiment 1* consisted of preliminary experimental studies to primary address the research question A, and identify optimal cavitation conditions. Optimal number of cavitation cycles were investigated, based on the HC impact on the physical (solids changes) and chemical (sCOD, turbidity, etc.) development on FW.

*Experiment 2*, involved batch BMP tests and partially answered research question B. This experiment investigated the biomethane potential of cavitated FW. The optimal number of cycles that resulted in the highest methane yield, progressed further for the last experiment.

*Experiment 3*, was performed in a semi-continuous stirred tank reactor (semi-CSTR) processing untreated and pre-treated FW, aiming to simulate full scale operation (research question C).

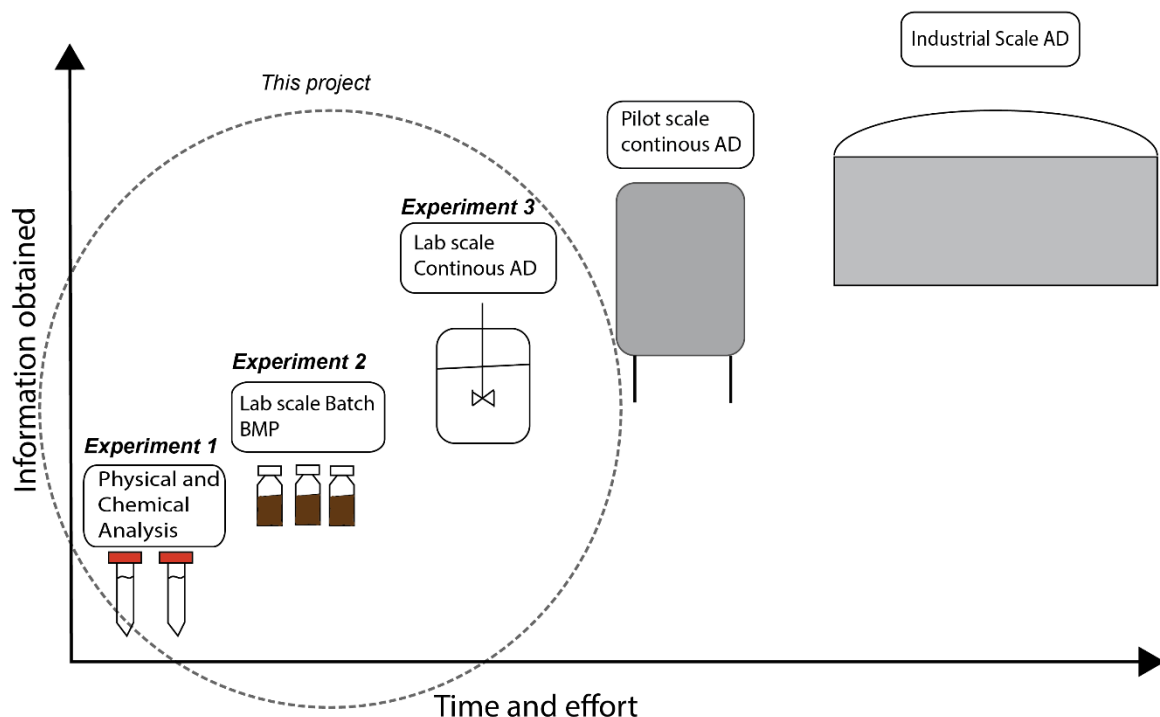


Figure 4. Overview of the experimental procedures used to study the impact of pre-treatment on the AD process. The project was divided into three experiments corresponding to the different procedures highlighted with a circle in the figure.

Experiment 1 – Physical and chemical characterization, experiment 2 – Lab Scale Batch BMP test, and experiment 3 – Lab scale continuous AD with semi-CSTR.



### 3.2 Food waste

FW samples were collected from the Scandinavian Biogas Fuels AB biogas plant, in Södertörn, Sweden. The process lay-out on site consists of six steps (Figure 5) involving receiving station, mill station, hygienisation, primary digester, secondary digester and downstream processes. At the receiving station, the FW is collected. The collected FW is further treated in the mill station for homogenisation and removal of non-organic materials, such as plastics and metals. Subsequently, the organic fraction is maintained at 70°C for a minimum 1 hour (hygienisation step) in respect to current EU regulation (Banks et al., 2011; Ariunbaatar et al., 2014). After hygienisation, trace elements and nutrients are added into the FW slurry to support high AD performance. The slurry is then pumped into the primary digestion with a HRT of about 20 days.

The effluent from the primary digester, i.e. digestate, proceed further into a secondary digester where the gas collection also occurs. Ultimately, the process proceeds into a downstream process for gas upgrade and fertilizer production performances.

Two different sampling points were identified for this thesis study: before the digestion (BeDi), between the mill station and the hygienisation step, and after the primary digester (AfDi) (Figure 5). The expected TS content of the samples were close to 12% for BeDi and 3% for AfDi, with a VS concentration equal to 92% and 68 % of the TS, respectively.

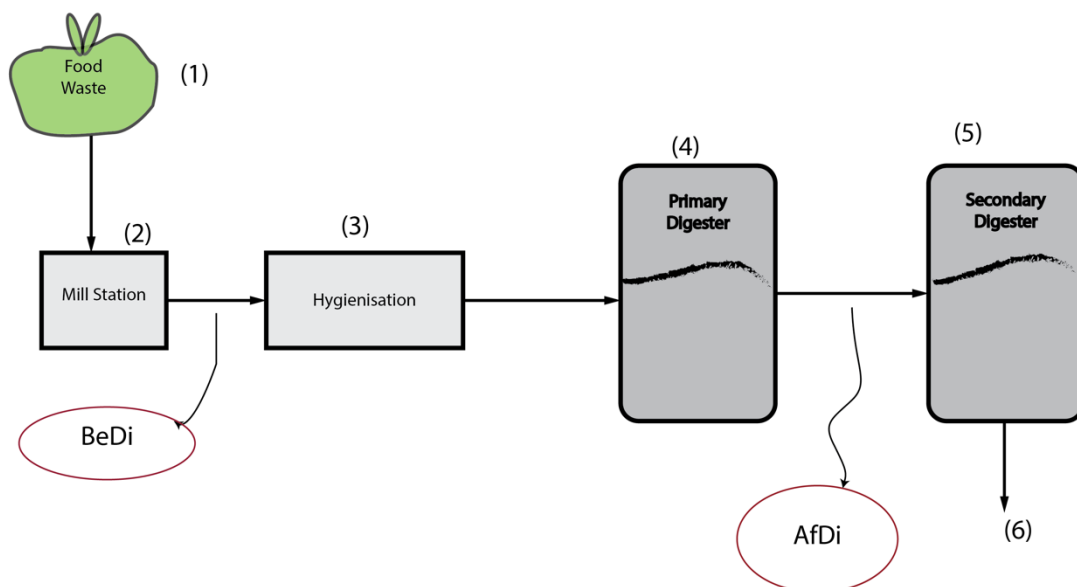


Figure 5. Overview of Scandinavian Biogas Fuels AB AD plant on food waste, Södertörn, Sweden. (1) Food waste collection; (2) Mill station; (3) Hygienisation of the food waste and addition of nutrients; (4) Primary digester; (5) Secondary digester; and (6) Downstream processes, respectively. The feedstock/substrate for this project were collected from Before Hygienisation (BeDi) and after the primary digester (AfDi).

Fresh FW samples were collected three times over the duration of the experiment. The first collection of BeDi and AfDi samples by 5 L each were utilised for experiment 1 and 2 and were stored at 4°C until utilization for maximum of 2 weeks. In contrast, the second (10 L) and third (5 L) collection were utilised with only the BeDi sample for semi-continuous experiment.



Subsequently, these FWs were stored in the freezer at -20°C in separate containers. One frozen sample per week was defrost and stored at 4°C until utilization.

### 3.3 Hydrodynamic Cavitation

The HC device was obtained from Efficiency Technologies Lmt, United Kingdom. The device consists of a PKm200 Perollo pump (0.19 L/s), two hydrodynamic cavitators (orifices), pipework and a plastic can-container (Figure 6). The diameter of the orifices is on the range of 1-3 mm.

The cavitation number ( $C_v$ ) was estimated 0.32 according to Equation 1 from Gogate and Pandit (2001):

$$C_v = \frac{P_2 - P_v}{\frac{1}{2}\rho v_{th}^2} \quad (1)$$

where  $P_2 = 101325$  Pa, equal to the atmospheric pressure in the present configuration,  $P_v = 2338.8$  Pa, vapour pressure of the liquid at 20°C,  $\rho = 999.89$  kg/m<sup>3</sup>, density of water, and  $v_{th} = 24.8$  m/s, the velocity of the liquid at the orifice constriction.

To support efficient operation at lab scale and reduce the risk of blockages in the system, undigested food waste samples (BeDi) was first filtrated with a 4 mm sieve. The fraction consisted of particle size above 4 mm was blended in a kitchen blender, and re-introduced back to the sample BeDi. All cavitation trials were performed on diluted samples with tap water and approximately 5% in TS, according to optimal operation practise identified by Abrahamsson (2015). Additionally, the author's findings encouraged this work to initially test the cavitation performance at the following number of cycles: 1, 5, 10, 20 and 40, respectively. Subsequently, the number of cycles' sampling points were progressively eliminated until optimal cavitation condition was identified for the experiment 3.

One cavitation cycle is defined as the time required for the all sample volume (2 - 2.5L) to pass through the system, in accordance to the flow rate. Before and after each cavitation run, temperature and power consumption were noted. In addition, each sample point were collected in duplicate for the chemical analyses: total solids, volatile solids, total suspended solids, soluble chemical oxygen demand, turbidity, and viscosity.



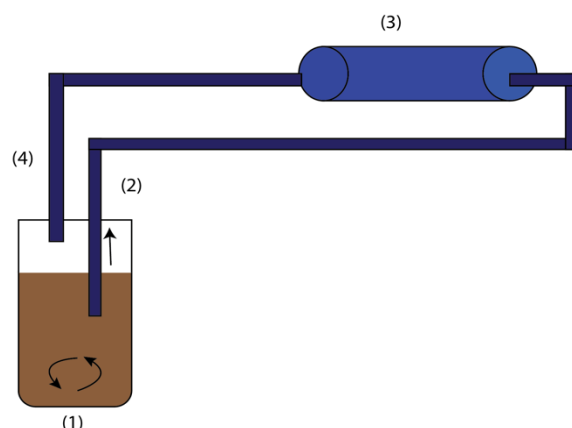


Figure 6. Schematic illustration of the hydrodynamic cavitation performance set up. (1) A container with substrate. (2) An inlet hose to the cavitation device (3). The cavitation device consists of a pump and cavitation inducer. (4) An outlet hose from the device that pump back the treated substrate to the container.

### 3.4 Batch Biochemical Methane Potential test

The biochemical methane potential (BMP) test was performed in accordance with the method described in Ekstrand et al. (2013). Briefly, serum glass bottles with a total volume of 250 mL were utilized, and a working volume was set to 100 mL. Fresh digested wastewater sludge was utilized as inoculum and collected at the same day as the start-up, from a local municipal wastewater treatment plant, Tekniska Verken, in Linköping, Sweden.

The OLR for BeDi and AfDi was close to 2.2 and 4.0 g VS/L, respectively, for untreated and treated (with 5, 10 and 20 cavitation cycles) substrate. The bottles, prepared with substrate, were flushed with  $N_2$  for 2 minutes before addition of 2 mL saline solution ( $NH_4Cl$ ,  $NaCl$ ,  $CaCl_2 \cdot 2H_2O$  and  $MgCl_2 \cdot 6H_2O$ ), 20 mL inoculum and milli-Q water.

Thereafter, the bottles were sealed with EPDM rubber stoppers and tightened with aluminium screw caps. In order to ensure an anaerobic condition, the headspace of the bottles was exchanged with  $N_2/CO_2$  gas mixture, and subsequently 0.3 mL of  $Na_2S \cdot 3H_2O$  (100mM) was added with syringe. Each sample was performed in triplicate and cultivated for total 62 days in a 37°C climate room. Three control samples in triplicates were prepared for the test: 20 mL inoculum without FW as control, 0.5 g Whatman paper as positive reference, and 50 mL  $CH_4$ . The biogas production and methane content were measured eight times during the incubation period: at day 1, day 3, day 7, day 12, day 20, day 30, day 40 and day 62. The biogas production was measured with a Testo 312-3 digital pressure gauge, and was performed before any gas sampling. 1 mL gas was withdrawn from the bottles for the determination of the methane content by Gas Chromatography Flame Ionization detector (GC-FID), from HP5880A series. The GC-FID was performed on the following conditions: column, Poraplot T; carrier gas,  $N_2$ ; column flow, 130 mL/min; injector temperature, 150°C; detector temperature, 250°C; oven temperature, 80°C; FID, air 250 mL/min and  $H_2$  30 mL/min. All data are reported at standard atmospheric pressure and 0°C as average.



### 3.4.1 Kinetic analysis

In order to determine the rate of degradation for respectively samples in the batch BMP test, following Equation 2 was used:

$$Y(t) = Y_m * (1 - \exp^{-kt}) \quad (2)$$

Where  $Y(t)$  is the cumulative biomethane yield in mL CH<sub>4</sub>/g VS,  $Y_m$  is the maximum biomethane potential in mL CH<sub>4</sub>/g VS,  $t$  is the time in days, and  $k$  the degradation rate constant (Allen et al., 2015).

## 3.5 Semi-Continuous Stirred Tank Reactor Anaerobic Digestion

Semi-continuous digestion was operated under mesophilic condition (37°C), using a 5 L glass CSTR with a working volume of 4 L. The stirrer was a mechanical mixer in stainless steel, and operated automatic every three hours for 15 minutes. During feeding, the stirrer was manually started and operated approximately 10 minutes prior and after the feeding procedure. The reactor, previously maintained by SBF, was operated for 13 weeks on the BeDi sample. During the first 5 and the last 2 weeks, no cavitation treated FW was utilized as feedstock. The initial two weeks was performed to allow stable digestion condition under the predetermined working condition (OLR = 2.2 g VS/L, and HRT = 20 days). Consequently, the first control period was performed at week 3-5. Between week 6 and 11, cavitation pre-treated FW was introduced into the digester. Based on the previous performance in experiment 1 and 2, respectively, the optimal cavitation condition was determined to 20 cycles. Ultimately, no cavitation treated FW was reintroduced and utilized as feedstock between week 12 and 13. The cavitation and feedstock preparation was performed once a week on a 2L sample, as described before. In addition, nutrients such as trace elements and metals was provided from the SBF and added into the feedstock. Two FW samples was collected for this operation.

The reactor was maintained every day, and in addition, analyses were performed on some specific days (Table 4) along with the R&D staff. The withdrawn reactor sludge was cooled down to 25°C prior the analysis performances.

The biogas was measured daily by a gas meter. The gas composition was determined by the Biogas 5000 Geotech, from Scantec Nordic, to determine the level of CH<sub>4</sub>, CO<sub>2</sub>, O<sub>2</sub> and H<sub>2</sub>S in the produced gas. The produced gas was collected the day before the analysing day, which corresponds to a daily average gas composition. All data are reported at standard atmospheric pressure and 0°C as average.



Table 4. Scheme over the weekly analyses performed for the semi-CSTR experiment: biogas composition, pH, conductivity, ammonium, volatile fatty acids (VFAs) total solids (TS), and volatile solids (VS).

Week Day	Analyses
Monday	pH, conductivity, VFAs, TS and VS, ammonium
Tuesday	Cavitation and feedstock preparation
Wednesday	Biogas composition
Thursday	pH
Friday	
Saturday	
Sunday	

## 3.6 The analytical analyses

### 3.6.1 Food waste particle analysis

The particle analysis was performed by 500 mL of wet sample were poured into several layers of sieves of nominal diameters equal to 8 mm, 4 mm, 2 mm and 1 mm, respectively. Consequently, the particles that was larger than the diameter of the filters was collected, while the smaller fragments will proceed further into the next filter size. Each filter was weighted before and after the filtration. Thereafter, TS and VS analyses were performed on the collected samples in each filters. Subsequently, the wet weight and solids measurement determined the distribution of the particle sizes in the FW sample.

### 3.6.2 Solids analysis

The total solid (TS) content was determined by a sample of 12-15 grams were stored in a 105°C oven for at least 20 hours. Consequently, the sample were burned in 550°C for 2 hours in order to determine the volatile solid (VS) content. All the TS and VS analysis were performed according to standard American Public Health Association and in duplicates (APHA, 2012). The following Equation 3 and 4 were used:

$$TS (\%) = \frac{(\text{weight;dried at } 105^{\circ} C)}{(\text{weight;wet})} \times 100 \quad (3)$$

$$VS(\% \text{ of } TS) = \frac{(\text{weight;dried at } 105^{\circ} C) - (\text{weight;combusted at } 550^{\circ} C)}{(\text{weight;dried at } 105^{\circ} C)} \times 100 \quad (4)$$

For TSS determination, instead, diluted sample was filtrated using the filter equipment from Sartorius, Goettingen, Germany. The filters, Munktell Glass Microfibre discs with 47 mm in diameters, was utilized. Before the filtration, the filters was cleaned by heating for 30 minutes in 504°C. After the filtration, the samples in the filters was heated together with the crucibles



for at least 1 hour in 105°C to obtain the TSS-level. The total suspended solids (TSS) was performed in triplicate.

### 3.6.3 Soluble content analysis

Soluble chemical oxygen demand (sCOD) and turbidity analyses was performed on centrifuged samples (11 minutes in 8500 rpm with Heraeus Megafuge 8, from Thermo Scientific). Hach Lange DR2800 Laboratory Analysis Spectrophotometer and LCK 514 cuvettes, detection range 100-2000 mg/L O<sub>2</sub>, Hach Company, Germany, were utilized for sCOD determination according to provided instructions and standard methods (APHA, 2012). The same spectrophotometer was utilized for the turbidity measurements with their instructions according to EN ISO 7027 and detection range 40-400 FAU.

### 3.6.4 Viscosity

Viscosity measurements were performed in duplicate using a rotational rheometer from RheolabQC SN80609650, Anton Paar, Ostfildern, Germany. The instrument was equipped with a CC27-SN19237 measuring system and a C-LTD80/QC cell, connected with Rheoplus software. The measurements were performed at 37°C and at the same day as the cavitation treatment. The presented data are obtained with a 3-step-protocol according to Björn et al., (2012) and at a shear rate equal to 800 s<sup>-1</sup>.

### 3.6.5 pH, Conductivity and Ammonium

The pH was measured with inoLab 7310 pH meter from WTW, Wissenschaftlich-Technische Werkstätten, Germany. The pH electrode was HAM Polilyte Bridge Lab from Hamilton Company. Calibration was performed once a week with pH 7 and pH 4 at 20°C buffer solutions. Before and after any pH measurement, a control solution with pH 7.96 at 25°C was performed to validate the result. The conductivity was determined by Cond 3110 Conductivity meter including TetraCon 325 standard conductivity measuring cell, from WTW. The ammonium determination was performed with LCK 514 cuvettes with detection range 47-130 mg/L NH<sub>4</sub>-N, from Hack Company. All the measurements were performed with standard methods (APHA, 2012).

### 3.6.6 Volatile Fatty Acids

The VFAs analyses for acetic, propionic, butyric, isobutyric, capronic, isocaproic, valeric and isovaleric acid was performed once a week using standard methods (APHA, 2012). The instrument for VFA analyses was a GC System, HP 6890 Series from HEWLETT PACKARD. One 1 mL samples of reactor fluid were prepared and centrifuged (Centrifuge 5417C, eppendorf) at 12 000 rpm for 10 minutes. Thereafter, 400 µL of the supernatant transferred into an injection vials with 40 µL of internal standard. The internal standard was crotonic acid in a formic acid solution. References with known concentrations, 0.6 to 10 mM (millimolar), of the VFAs was used for calibration. The detection limit of the instrument is 0.2 mM and the quantification limit is 0.6 mM



# 4 Results and Discussions

## 4.1 Experiment 1 – Physical and Chemical Characterization

### 4.1.1 Food waste characteristics

The particle size distribution analysis revealed significant differences between the two collected FW samples. The before digestion (BeDi) sample, with a TS content of 12.4% (92.7% VS of TS) was characterised by a range of particle sizes (Figure 7). The distribution showed that 51% of the total TS content was associated with particles larger than 4 mm, 36% ranged between 4-8 mm, 8% between 1-2 mm and 20% below 1 mm. In contrast, the after digestion (AfDi) sample, having a TS concentration equal to 3% (68.3% VS of TS), contained significantly smaller particles. 77% of the TS had average size below 1 mm, no substances were larger than 2 mm and only 23% of the substances were ranged between 1-2 mm.

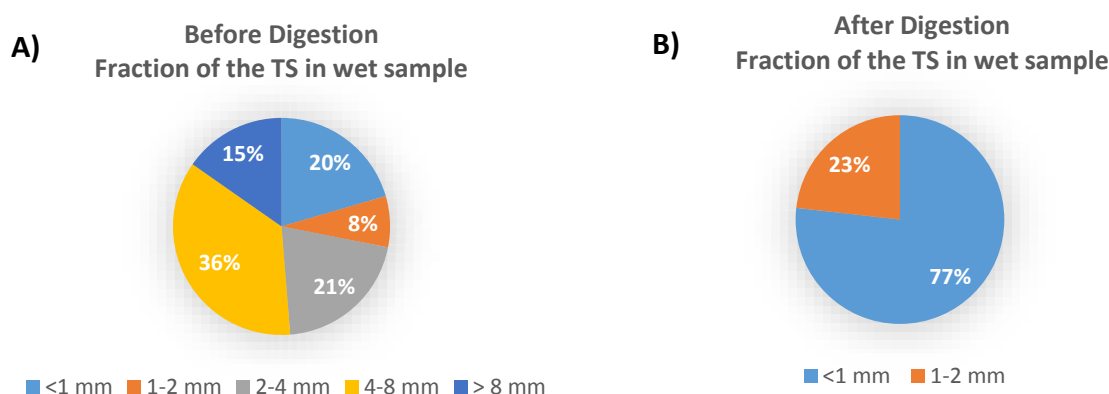


Figure 7. The distribution of particle size in the samples. A) The fraction of the TS in the wet BeDi sample: 20% (light blue) <1 mm, 8% (orange) 1-2 mm, 21% (grey) 2-4 mm, 36% (yellow) 4-8 mm, 15% (dark blue) >8mm. B) The fraction of the TS in wet AfDi sample: 77% (light blue) <1 mm, 23% (orange) 1-2 mm.

Observed differences between the two samples, are in line with the expected impact of the AD on the feedstock. The calculated VS reduction based on the collected sample was equal to 82%, in line with conventional efficiency at full scale mesophilic AD treating FW (Bernstad et al., 2013). From the analysis, differences between the two samples suggest different response of the biomass to the HC pre-treatment. For instance, HC applied to BeDi, will primarily reduce the size of the particles and promote homogenization. Consequently, it will ease the degradation and establish a more consistent gas production. In contrast, the expected impact from HC on the AfDi sample will primarily induce cell disruptions and increase solubilisation of the organic matter. This improves the processing of unsuccessfully digested materials from the previous digestion.



#### 4.1.2 Hydrodynamic cavitation impact

Impact of cavitation pre-treatment on samples were first evaluated based on temperature, sCOD, turbidity, and solids composition changes. The two FW samples were treated with 1, 5, 10, 20, and 40 cavitation cycles, followed by chemical analyses. In both samples, the temperature increased linearly with the number of cavitation cycles (Figure 8A). From an initial temperature close to 23°C (room-temperature), the value increased by 1.3°C/cycle, to reach up to 71°C after 40 cycles (equal to 7 min 16 sec of treatment).

Similar behaviours were observed for both samples on the amount of sCOD release with cavitation (Figure 8B). Both samples required at least 20 cycles to show sCOD increments, with maximal sCOD released after 40 cycles. To illustrate, before treatment, undigested FW (BeDi) had a sCOD content between 38000 to 40000 mg/L, typical of unprocessed FW. After an initial reduction the value increased to 38944 and 39804 mg/L at cycle 20 and 40, respectively. Similarly, the cavitation of AfDi samples showed no significant increment until 20 cycles where it reached 4414 mg/L and increased to 6536 mg/L at cycle 40. The differences in sCOD values between the two samples reflects the characterised of the two material as previous observations on particles size. Higher values correspond to the pre-digestion (BeDi) and low value to post digestion (AfDi).

In contrast, the turbidity measurements showed saturated and steady-state values for both FW samples, suggesting maximum effects of cavitation treatment (Figure 8C). The BeDi reached its maximum at cycle 20 and increased its turbidity by four times, from 148 to 613 FAU. The significant increment was observed at cycle 10, while no major impact was found for the BeDi sample after one and five cavitation cycles. In comparison, the AfDi sample reached its maximum after one cycle and no significant progression was observed afterwards (Figure 8C). Moreover, the AfDi sample started at a higher turbidity value (245 FAU) than the BeDi and was increased to 394 FAU (+61%).

There were no observed increases in solubilisation effect in the TSS/TS ratio measurements despite the observed sCOD and turbidity increments. The TSS/TS ratio remained constant around 0.65 in the AfDi samples, while it increased with the number of cavitation cycles from 0.4 to 0.5 in the BeDi samples (Appendix A.1). The TSS/TS ratio gives information of solid solubilisation where a reduction indicates increased biomass solubilisation (Ometto et al., 2014). This suggest fine flocs formation after cavitation treatment, supporting previous observations on extracellular polymeric substances (EPS) released during cavitation treatment inducing particle aggregations (Feng et al., 2009).

The viscosity measurement for the BeDi was only performed with 5, 10 and 20 cavitation cycles. The limit apparent viscosity value remained constant close to 5.7 mPa.s, suggesting that the cavitation treatment did not affect viscosity properties in FW (Appendix A.2).



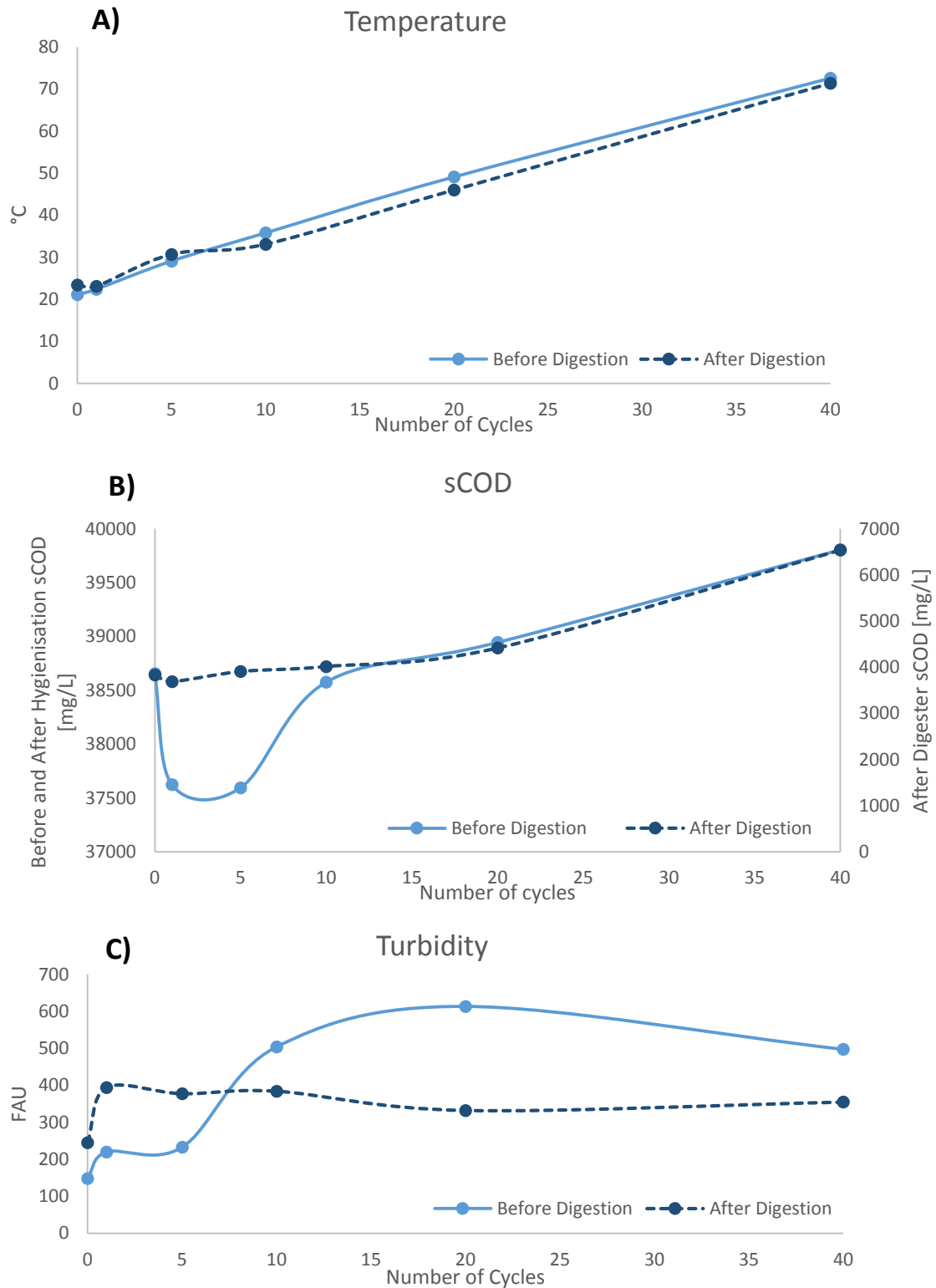


Figure 8. Hydrodynamic Cavitation impact at different cycles on the BeDi (light blue line) and AfDi (dashed dark blue line) on A) Temperature; B) sCOD and C) Turbidity.



The energy consumption for the cavitation performance was reduced with increasing number of cycles for both samples. After 40 cycles, the energy consumption decreased from 2.5 kWh to 2.4 and 2.3 kWh for BeDi and AfDi, respectively (Appendix A.3). This suggests that the HC reduced the size of the particles and assisted sample processing. The lower energy consumption required for AfDi indicates that the material was easier to process than the BeDi sample as a consequence to its characteristics of smaller particles (Figure 7). The larger the particle, the higher the energy is required to push the fluid through the cavitation orifice. A reduction in the energy consumption over time resulting from cavitation, confirms that particle size reduction and biomass solubilisation increases are responsible for changes in the turbidity measurements and sCOD.

In line with previous work with thermal pre-treatment, the temperature increment with cavitation did not reach sufficient temperature and exposure time to enhance biomethane production (Table 2) (Dhamodharan and Kalamdhad, 2014). Strong reduction in viscosity by temperature increment was observed in previous studies, this could not be confirmed in this study with limit apparent viscosity measurements (Bougrier et al., 2006). This suggests that the temperature development to 71°C with HC was insufficient to cause any influences on tested FW samples. However, the temperature development assists the hygienisation step in the full scale operation (Figure 5). The HC implementation reduces the heat-up procedure and hence the energy input to the hygienisation procedure.

The behaviour of both FW samples in this study is in agreement with previous work on WAS. Petkovšek et al. (2015) demonstrated a continuous sCOD increment within 20 recirculating cavitation, which was in line with the AfDi samples but not for the BeDi samples (Table 3, Figure 8B). Limited impact and disintegration was observed for one cavitation cycle, which is confirmed in this study with FW. Our results suggest that it requires more than five cavitation cycles to reveal any effects for more complex substrate, as observed in the BeDi sample. Limited data between cycle 20 and 40 restricts the solubilisation information within the intermediary region. However, the saturation observed in the turbidity measurements suggests limited effects after twenty cycles. Hence, phase 2 progressed with a maximum of 20 cavitation cycles in the batch BMP trials.

## 4.2 Experiment 2 – Batch Digestion Trials

Batch digestion BMP tests were performed on untreated and treated samples cavitated for 5, 10 and 20 cycles. No batch digestion tests were performed on samples below five cavitation cycles due to the low impact previously observed (low sCOD, low turbidity). The 40 cycles was not processed based on the turbidity measurements showing maximum impact after 20 cycles. Furthermore, the energy demand to perform a 40 cavitation cycles together with the temperature achieved (>70°C) prevent full scale application. It has been estimated that the performance requires an increment in methane yields by 49% to balance the energy input required (Appendix A.4). With expected increment being approximately 30% with pre-treatments according to previous studies, which suggests that 40 cavitation cycles is not feasible to proceed with (Table 2).



#### 4.2.1 Biochemical methane potential

Applied to both FW samples (BeDi and AfDi), the 20 cycle cavitation test showed the highest impact on methane yields (Figure 9). The lower cavitation cycles did not increase the biomethane potential, which confirms the limited HC impact previously observed (low sCOD released and low turbidity increment). When comparing the treated (20 cycles) and untreated BeDi sample, the biomethane potential increased from  $624 \pm 10$  to  $704 \pm 57$  mL CH<sub>4</sub>/g VS (+13%) (Table 5). Surprisingly, the kinetic degradation rate constant (K-value) for BeDi was reduced by 52%, starting from 0.31 days<sup>-1</sup> (untreated) to 0.15 days<sup>-1</sup>. This suggests the treated samples did not enhance the hydrolysis rate in the BeDi FW. However, at day 12, the untreated sample started to level off in methane production while the 20 cycle sample progressed further (Figure 9A). In line with the TSS of TS ratio increment observed for the BeDi, the additional release of EPS resulted in a higher methane yield for the treated sample. Ultimately, all the BeDi samples were depleted at day 20.

In contrast, the AfDi revealed a slower degradation rate than the BeDi and was depleted at day 30 (Figure 9B). The methane production for the AfDi samples were well reflected with the characteristic differences between the two tested FW samples observed in phase 1 (sCOD value, particle size distributions, solids contents). The already processed AfDi sample contained considerably smaller organic materials, VS content and lower sCOD value resulted in a 4-fold lower biomethane potential than the BeDi, in the range 150-170 mL CH<sub>4</sub>/g VS (Table 5). After twenty cavitation cycles the AfDi sample increased the methane yield from  $151 \pm 9$  to  $164 \pm 3$  mL CH<sub>4</sub>/g VS (+9%) (Table 5). The kinetic degradation rate constant was increased by 60%, in line with the progressive sCOD increment previously observed (Table 5, Figure 8B). This suggests that the cavitation improved the hydrolysis and increased the solubilisation of undigested materials with AfDi FW. By obtaining a faster hydrolysis the retention time for the AD process could potentially be reduced, hence a more efficient methane production.

In agreement with our results, Petkovšek et al. (2015) showed a biogas increment in pilot scale with WAS after 20 cavitation cycles, which was confirmed in the AfDi sample in the batch trials (Table 5). In similarity, Lee and Han (2013) improved the methane yield with HC treatment on WAS by 13%, the result was reflected for the BeDi sample. This suggests that the obtained results in the presented batch BMP trials are in line with previous work with HC, in both lab and larger pilot scale.

Cho et al. (1995) demonstrated a cumulative methane yield by 472 mL CH<sub>4</sub>/g VS with Korean FW (Table 1). In comparison, our methane yield with cavitation pre-treatment was 32-49% higher than their digestion performance. However, the degradation rate was significantly faster in their experiments. The digestion was already depleted at day 7 with the OLR equal to 2 g VS/L. In fact, faster methane production rate is reasonable, since they pre-treated all their FW with a mechanical mill and dry-freezing. Both these pre-treatments have been reported to have significant impact on AD and biogas production (Table 2). While only using mechanical bead mill on FW the methane yield increased by 28% (Izumi et al., 2010). This suggests that the synergy effect between HC and mechanical milling could be the reason for the fast



depletion and unclear kinetic rate reduction observed in the BeDi samples. Moreover, it was encouraging to find major increment in BeDi samples which occurred within 20 days' cultivation. This indicates that full digestion was achieved in the same range as the pre-determined HRT (20 days) for the semi-continuous digestion performance.

Despite the faster degradation rate constant achieved in the AfDi sample compared to the control, the sample treated with 10 cavitation cycles did not result in a higher methane yield. In this context, cavitation treatment below 20 cycles was insufficient for the AD process. Given this information, it was determined that the last semi-continuous digestion would be performed with 20 cavitation cycles.



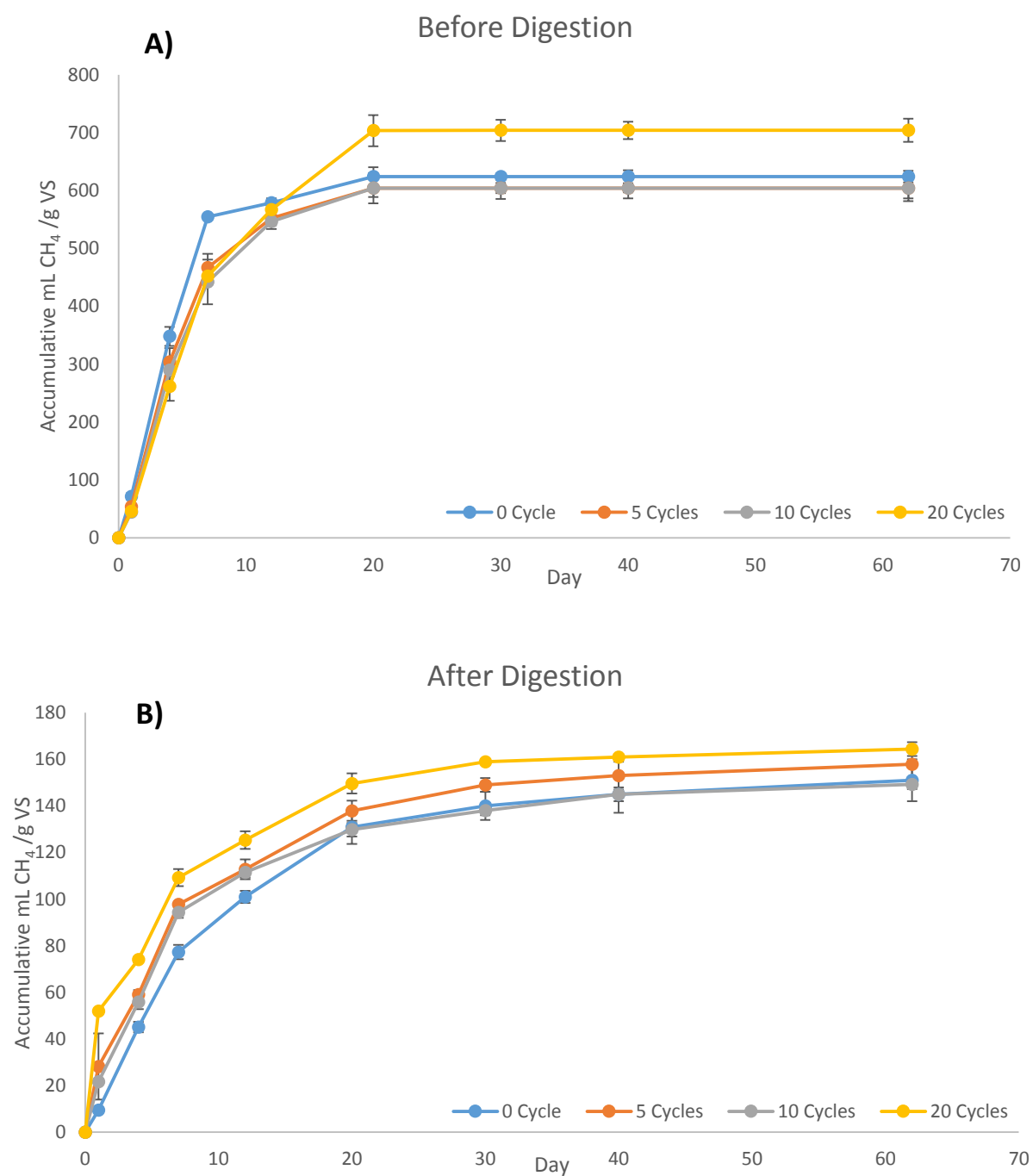


Figure 9. The accumulative methane production from the BMP test of different FW samples pre-treated with HC at different cycles before AD. A) Result for the BeDi sample. B) Result for the AfDi sample.



Table 5. Summary of the BMP test for the before digestion and after digestion FW samples. All the gas result is cumulative production. The percentage within the parentheses are the gains compared to the control.

	Before Digestion			After Digestion		
	Biogas [mL/g VS]	Methane [mL/g VS]	K (days <sup>-1</sup> )	Biogas [mL/g VS]	Methane [mL/g VS]	K (days <sup>-1</sup> )
<b>0 cycles</b>	940±36	624±10	0.31	199±4	151±9	0.10
<b>5 cycles</b>	908±18	604±22	0.21	209±5	158±8	0.14
<b>10 cycles</b>	909±28	504±18	0.19	199±8	149±2	0.14
<b>20 cycles</b>	1145±15 (+22%)	704±20 (+13%)	0.15	223±10 (+12%)	164±3 (+9%)	0.16

### 4.3 Experiment 3 – Semi-continuous Digestion Experiment

Semi-continuous digestion was performed on a 5L digester for 76 days. FW was collected twice (sample I and II) during the entire duration of the experiment. The operation has been divided into three periods. During the first and third period no cavitation pre-treatment was performed and untreated FW was used as feedstock. In the second period, instead, FW was pre-treated with 20 cavitation cycles (optimal identified condition) and used as feedstock. The differences in the characteristics of the two samples were reflected in the biogas and methane yields. The measured sCOD for sample I was equal to 32000 mg/L, while sample II showed a sCOD of 24000 mg/L (Table 6).

#### 4.3.1 Digestion performance

Overall, fluctuations in the biogas production were observed for the untreated FW, while more constant daily biogas production was found for cavitated FW (Figure 10). The overall process was stable with no inhibition under the pre-determined operation conditions (OLR=2.2 g VS/L, and HRT=20 days). Untreated FW yielded between 510 and 530 mL CH<sub>4</sub>/g VS for FW I, and between 410 and 440 mL CH<sub>4</sub>/g VS for FW II (Figure 10), in the same range of data reported for semi-continuous digestion trials (Table 1). In contrast, the treated FW showed methane yields between 520 and 540 for FW I, and between 490 and 500 mL CH<sub>4</sub>/g VS for FW II. Giving an average methane increment of 3% and 17% for FW I and FW II, respectively. To illustrate, for the initial period 1, the specific biogas production was 824±35 ml/g VS with non-treated FW (NoCAV I) (Table 6). For the first 10 days, the biogas production declined from 860 to 760 mL/g VS. Eventually, the biogas production rose sharply to 880 mL/g VS at day 12, and fluctuated until the transition to treated FW (CAV I) at day 21. Consequently, the biogas production decreased from 890 to 770 mL/g VS between days 21-27, in period 2. Subsequently, the production was maintained steady (850-870 mL Biogas/g VS), between days 30-39.

In comparison to NoCAV I, the treated CAV I resulted in a small biogas increment by 2% (Table 6). However, despite the minor biogas increment, the summarising Table 6 revealed changes inside the reactor. The TS, pH, ammonium (NH<sub>4</sub>-H), and conductivity decreased from 1.9% to 1.8%, 7.5±0.1 to 7.3±0.1, 1217±159 to 729±159 mg/L, 14±2 to 10±1 ms/cm, respectively, while



the VS of TS increased from 67% to 70% and the acetic acids was maintained at 0.6 mM. The period 2 was pursued further with pre-treated FW, but the FW was replaced at day 53 to sample II. Consequently, CAV II was initiated and proceeded until day 61. In comparison to CAV I, a lower biogas production was observed for CAV II, in average by  $781 \pm 22$  mL/g VS. The ammonium and conductivity continued to decline, while the pH and VS of TS increased, to the average values of  $544 \pm 29$  mg/L,  $8.7 \pm 0.2$  ms/cm,  $7.3 \pm 0$  and 72%, respectively. While the TS content and acetic acid was maintained at the same level.

At day 62, the non-treated NoCAV II was introduced to the reactor. On the contrary to the treated CAV II, the non-treated NoCAV II demonstrated major fluctuations that lasted until termination at day 76. Thus, it is suggested that the pre-treatment increased the homogenization in the feed stock, which resulted in a more stable gas production than non-treated FW.

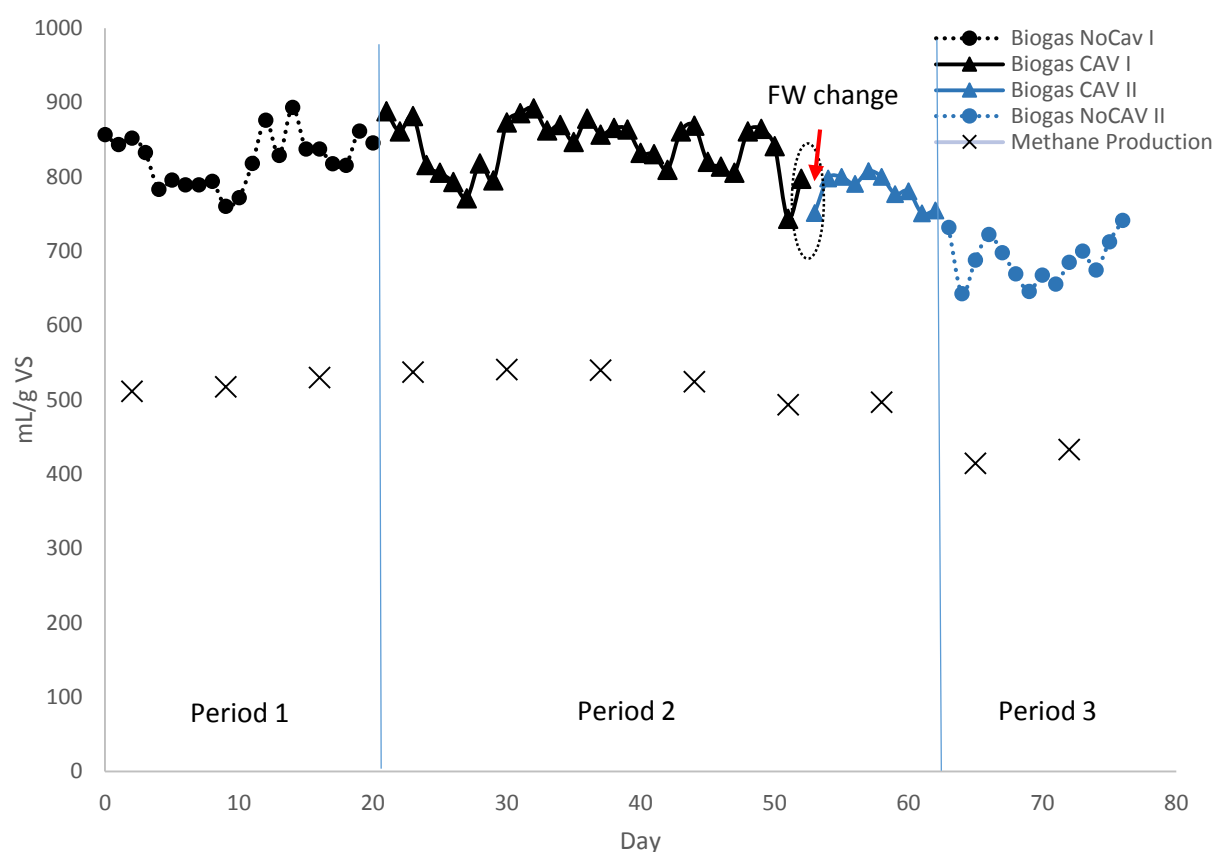


Figure 10. The biogas and methane production during the semi-CSTR AD. Additionally, the graph illustrate the main events during the performance. For period 1 and 3, no cavitation pre-treatment was applied on the food waste. Period 2, the food waste was pre-treated with 20 cycles' cavitation. At day 53, the first delivery of food waste was depleted and hence a new batch was utilized. Thereby, the comparisons will be evaluated only within the same batch delivery of food waste, i.e. NoCAV I with CAV I and NoCAV II with CAV II. The biogas production has been adjusted in consideration to the time interval in hours between feeding.



Table 6. Summary of the semi-CSTR operation. All the values are average for their corresponding course, where no cavitation pre-treatment on the FW was introduced for period 1 and 3, while cavitation treated FW was used for period 2. The percentage value within the parenthesis are the gain, in comparison to the control with no pre-treatment.

	Period 1	Period 2		Period 3
	NoCAV I	CAV I	CAV II	NoCAV II
<b>sCOD content [mg/L]</b>	32177*	37653	26382	24198
<b>Biogas [mL/g VS at 273 K]</b>	824±35	840±37 (+2%)	781±22 (+13%)	688±31
<b>Methane [mL/g VS at 273 K]</b>	520±9	536±7 (+3%)	495±2 (+17%)	424±13
<b>CH<sub>4</sub> content</b>	62%	62%	63%	60%
<b>TS [%]</b>	1.9±0.1	1.8±0	1.8±0	1.6±0
<b>VS of TS [%]</b>	67±1	70±1	72±0	72±1
<b>Acetic acid, VFA [mM]</b>	0.6	0.6	0.6	0.6
<b>pH</b>	7.5±0.1	7.3±0.1	7.3±0	7.2±0.1
<b>NH<sub>4</sub>-N [mg/L]</b>	1217±159	729±159	544±29	580±91
<b>Conductivity [ms/cm]</b>	14±2	10±1	8.7±0.2	8.7±0.2

\*single measurement.

The major changes inside the reactor between period 1 and 2 suggest that it was caused by influences from previous operations before the takeover. When looking into TS and VS content changes over the course it is implied that the reactor was diluted by the feedstock (Table 6). The VS of TS increased, on average, from 67% to 72% in CAV II (period 2) and no significant changes were observed during the transition to period 3. In this context, it is suggested that the reactor was stabilized in the CAV II procedure with no influences contributed from previous operations. As previously explained in the background, ammonia accumulation cause pH increment in the AD process. Significant ammonium (-55%) and conductivity (-39%) reductions was observed during the digestion performance, but the pH was maintained stable at around 7.3. In this context, the microbial environment was not affected by the ammonium reduction. While the proportional conductivity reduction to the ammonium loss confirms observations in previous work (Levlin, 2010).

The methane increment by 17% for CAV II confirms and correlates the biomethane yield obtained in the BMP test with 13% increment for the BeDi sample (Table 5 and 6). To illustrate, the BMP test on the BeDi sample was accomplished after 20 days digestion where a methane yield by 704±20 mL/g VS was obtained (Figure 9). When comparing this value in correlation to the semi-CSTR performance with a HRT equal to 20 days, it suggests that the methane production could potentially be increased by additionally 42%.

In agreement with other reports on AD with FW, the presented semi-continuous digestion performance was similar to Mata-Alvarez et al. (1992) where the operation condition was as following: increasing OLR in the range of 1.68-2.8 g VS/L day and HRT 12-20 days, with diluted FW to 3.96% in TS content. The similar digestion condition showed neither process inhibitions nor bacteria washout, which are confirmed in this presented study. When comparing the methane yields, the cavitation treated FW in this study revealed 4-12% higher values than the reported untreated FW digestion. Using similar but higher digestion conditions (OLR = 9.2 g VS/L day and HRT = 16 days), Nagao et al. (2012) reported a methane yield by 455 mL/g VS in



lab scale. When comparing these reported results to the presented work with cavitated FW, it shows that greater methane yield (by 9-19%) was obtained at lower OLR (2.1 g VS/L day). 8.0 days HRT and OLR equal to 5.5 g VS/L day was reported to cause process failure in lab scale. While full scale operation on FW (900 m<sup>3</sup>) with OLR and HRT equal to 2.7 g VS/L day and 80 days, respectively, was observed to cause ammonia and VFAs accumulation (Banks et al., 2011). Further investigation with semi-continuous digestion at higher OLR with cavitation treatment could be interesting in order to identify the optimum conditions for full scale practise without causing inhibitions.

The highest yields in this study was found while comparing the CAV II and NoCAV II results, which was in line with previously reported data with pre-treatments on FW (Table 2). These two periods was significantly shorter than the former periods, subsequently no full retention time by 20 days was obtained. The interpretation for these two periods is evaluated with caution. However, this presented study confirms that the HC as a pre-treatment step improves the AD process in lab scale semi-continuous digestion. Hence, HC shows promising results for full scale AD production on FW.



# 5 Conclusions

The main conclusions from this thesis work can be summarised as following:

- Multiple cavitation cycles, equal or higher than 20, are required to affect the physical and chemical biomass characteristics of food waste samples (both before and after digestion).
- It appears that the turbidity was affected significantly by HC where increment up to 400% and 61% was observed for Before (BeDi) and After digestion (AfDi) samples, respectively.
- The sCOD contents were ranged between 25 000-40 000 mg/L and 4000-7000 mg/L for the Before digestion and After digestion, respectively, which reflected a four-fold methane yield difference between the tested two FW samples.
- Under batch digestion condition, the HC treatment improved the methane yield by 13% treating fresh food waste (BeDi) despite a kinetic degradation rate constant reduction by 52%. While treating digested FW (AfDi) HC improved the methane yield by 9% and increased the kinetic degradation rate constant by 60%.
- Successful simulation of full scale practise in lab scale semi-continuous digestion, without any inhibitions, was performed on two separately collected fresh FW samples (BeDi). HC as a pre-treatment step of the feedstock improved the methane yields up to 17% depending on the food waste characteristics



## 6 Future Work

The investigation has only been performed in lab scale using only one orifice size. Consequently, different orifice sizes and cavitation reactor geometry is interesting to investigate further. Additionally, the next step is to validate the HC pre-treatment in a larger scale such as pilot or even full scale reproducing lab scale cavitation condition. Also, more evaluation for the semi-continuous digestion is needed to find optimum condition for AD of the specific substrates. Promising results in the BMP trials for the AfDi suggest that further investigations on its digestion performance with cavitation is of interest for full scale implementation.



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# Appendix

## Appendix A

### A.1 TSS of TS ratio

The TSS of TS ratio development after 40 cycles with cavitation treatment during the preliminary experimental studies. When looking into the BeDi sample, no impact was observed with 1 cavitation cycle. Subsequently, the ratio increased with increasing cavitation cycles from 0.42 to 0.50. In contrast, the AfDi sample fluctuated with minor changes between 0.61 and 0.68 suggesting no impact from the cavitation and the ratio was constant.

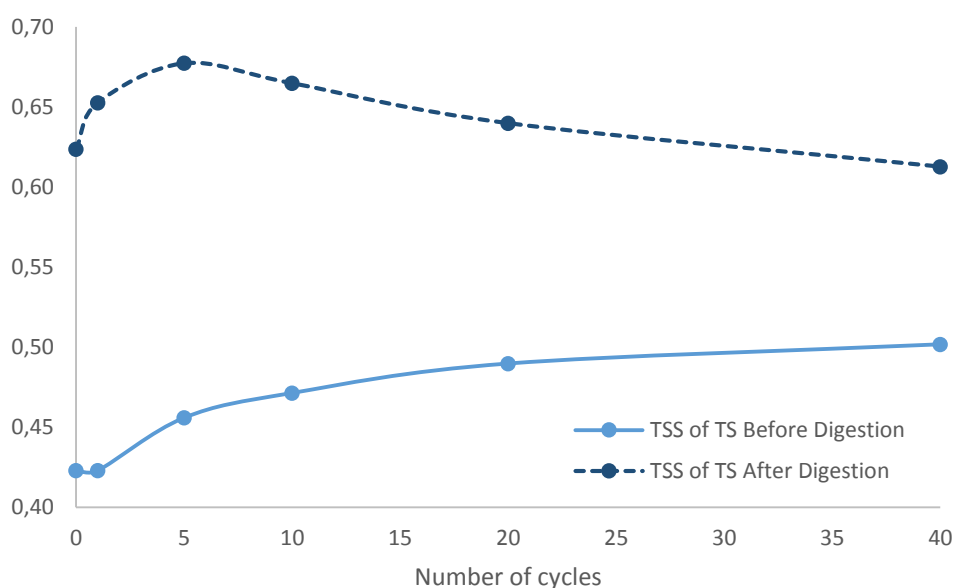


Figure 11. TSS of TS ratio for BeDi and AfDi samples. Increasing trend while treating BeDi FW with cavitation, while AfDi samples remained constant.

### A.2 Viscosity

The viscosity measurement for the BeDi sample showed limited impact from the HC treatment. The value was in average 5.71 with shear rate equal to  $800 \text{ s}^{-1}$  after 20 cavitation cycles.

Table 7. The limit apparent viscosity (at  $800 \text{ s}^{-1}$ ) measurement for BeDi sample. Minor changes with increasing cavitation cycles suggesting no major impact in viscosity from the cavitation treatment on FW.

Number of Cycles	Viscosity [mPa.s]
0	5.90
5	6.05
10	5.42
20	5.48



### A.3 Energy consumption

The power consumption was measured in connection with the cavitation performance, during the preliminary experimental studies. The energy consumption decreased for both FW samples with increasing cavitation cycles. This suggest the fluid was affected by particles size reduction and hence was easier to pump the fluid through the orifice.

*Table 8. The energy consumption and the processing time for the cavitation cycles. Both FW samples decreased its energy consumption with increasing cavitation cycles.*

Number of Cycles	BeDi		AfDi
	Time [s]	Energy consumption [kWh]	Energy consumption [kWh]
0	0	2.500	2.500
1	14	2.500	2.500
5	59	2.460	2.450
10	116	2.465	2.400
20	225	2.465	2.400
40	436	2.400	2.340

### A.4 Energy balance for 40 cavitation cycles

Based on the plant information provided from Scandinavian Biogas Fuels AB and the energy consumption during the cavitation performances, it is estimated that:

- 347 tonnes fresh FW are treated per day, approximately TS content by 12%
- 39 000 m<sup>3</sup> raw biogas is produced per day
- 146 kWh/tonnes wet FW is required to perform the 40 cavitation cycles on the feedstock

Further calculations on the cavitation performance provides the following information:

- Dilution of the fresh FW to 5% TS:  $\frac{347 \text{ tonnes fresh FW} \times 0.12 \text{ TS}}{0.05 \text{ TS}} = 833 \text{ tonnes wet FW}$
- The energy to perform 40 cavitation cycles on the daily FW:  
 $833 \text{ tonnes wet FW} \times 146 \text{ kWh/tonnes wet FW} = 121\,618 \text{ kWh}$
- 1 normal cubic meter methane equals to 10 kWh, and the methane content is approximately 63% in the biogas from AD on FW (SGC, 2012), consequently the energy content in the raw biogas equals to  
 $39\,000 \text{ m}^3 \text{ raw biogas} \times 0.63 \text{ methane content} \times 10 \text{ kWh} = 245\,700 \text{ kWh}$
- The required methane increment to perform 40 cycles cavitation is estimated to  
 $\frac{121\,618 \text{ kWh}}{245\,700 \text{ kWh}} = 49\%$
- Thus, it is required to improve the methane yield by 49% to balance the energy consumption for 40 cavitation cycles, which is not feasible.



## Appendix B

### B.1 Thesis work evaluation

Initially, this thesis work was planned for approximately 20 weeks, see figure. It was initiated with 2 weeks planning and literature research phase, phase 0. Consequently, phase 1 was performed for three weeks. The batch BMP test, i.e. phase 2, was started as planned at week 7. Subsequently, the phase 3 was initiated one week earlier as planned.

The early start of the semi-CSTR operation was appreciated. However, the overtaking of an existing reactor did not progress as planned. The stabilizing week was underestimated, which prolonged the whole phase 3 operation from 8 weeks to approximately 12. Additionally, other challenges rose up during the thesis work, such as unplanned holidays, writing issues, and appointment difficulties delayed this thesis work by 8 weeks.

Project week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
Calendar week	40	41	42	43	44	45	46	47	48	49	50	51	52	53	1	2	3	4	5	6	7	8
Activity																						
<b>Phase 0</b>																						
Planning the project																						
Introduction to the device																						
Briefing of the lab																						
Literature study																						
MS1-Planning report		✓																				
<b>Phase 1</b>																						
Experimental planning																						
Cavitation experiment																						
Evaluation of data																						
MS2-Screening					✓																	
<b>Phase 2</b>																						
Experimental planning																						
Biogas production																						
Evaluation of data																						
MS3-Start of batch						✓																
MS4-Screening prel.									✓													
MS5-Half time report															✓							
MS6-End of batch																✓						
<b>Phase 3</b>																						
Experimental planning																						
Biogas production																						
MS7-Start of CSTR									✓													
MS8-End of CSTR																	✓					
<b>Phase 4</b>																						
Writing the report																						
Evaluation of the result																						
MS9-Screening																		✓				
MS10-Final report draft																			✓			
MS11-Presentation def.																				✓		
MS12-Corrected report																					✓	
MS13-Reflection doc.																						✓

Figure 12. Gant scheme for the initial project planning.



Furthermore, the initial plan was to investigate three different food waste sub-streams of the plant, see figure. This report has only mentioned the Before Digestion and After Digestion, which was initially referred to as before hygienisation and after digester. In addition to these two, the third one was between those, i.e. after hygienisation (Figure 13). However, the preliminary experimental studies did not show any significant differences between the before and after hygienisation. Thus, in order to ease the reader, only two of these streams were presented.

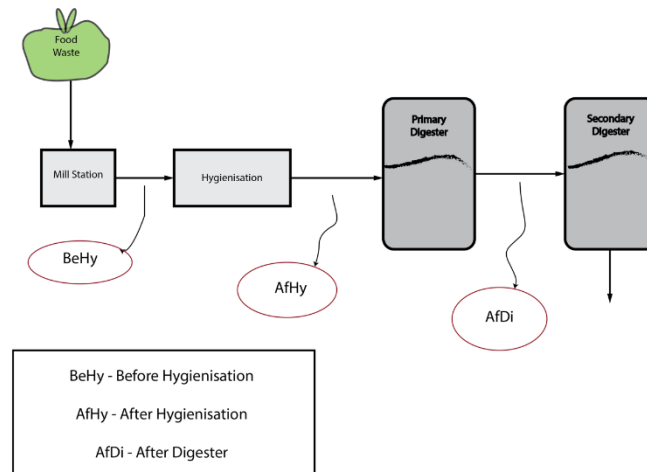


Figure 13. The initial project was processing with three food waste sub-streams.

Conclusively, this thesis has been an amusing journey including a lot of hard work, learning and joy. The most important personal lesson for the future involves the art of planning. The planning phase could probably be performed more accurately. However, as usual within world of science, there will always be unexpected incidents. These incidents will always be, if possible, challenging to solve. In the end, from my point of view, it was these unexpected incidents that enriched me with knowledge and personal development, both in work and personal life.