How the Choice of Primary Treatment Affects the Biogas Potential of Primary Sludge

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How the Choice of Primary Treatment Affects the Biogas Potential of Primary Sludge

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Picture on front page: Photo by Fanny Blom of AMPTS II (Bioprocess Control, Sweden) at Lund University.

Preface

This thesis was performed by me from a proposal by Sweden Water Research, in collaboration with the Department of Chemical Engineering at Lund University.

First, I would like to thank my supervisors Åsa Davidsson and David Gustavsson for their excellent advice, guidance and continuous support.

I would also like to thank Simon Gidstedt and Mirjam Victorin for showing me the AMPTS. The help they provided were valuable.

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Summary

The global heating has resulted in rising temperatures and more extreme weather conditions around the world. In combination with increasing energy demands, this has led to an increasing demand of renewable energy. One option is utilizing the wastewater to produce biogas. Therefore, wastewater can be seen as a resource rather than waste. Biogas production is based on anaerobic digestion where microorganisms transform organic matter into methane.

Primary treatment in wastewater treatment plants (WWTPs) remove mostly the particulate organic content, therefore the sludge gathered in this stage is rich in organic matter. The primary treatments available are the conventional primary settling treatment (PST) and filtration, e.g. rotating belt filter (RBF), where both can be combined with chemically enhanced primary treatment (CEPT). RBF requires less space in comparison to PST and might create a sludge with a higher energy output, due to a different composition of RBF sludge, e.g. it has a higher content of cellulose. RBF sludge has been found to have a higher biogas potential than PST sludge, when comparing primary treatment from different WWTPs.

The objective of the project was to determine if RBF has higher biogas potential than PST by using PST sludge and CEPT-RBF sludge from the same WWTP. Further, the biogas potential at mesophilic and thermophilic temperatures were tested. Settled sludge and settled CEPT sludge were produced in order to evaluate the potential of sludge without storage time in settlers.

Bio-methane potential (BMP) tests were used to evaluate the biogas potential. An automatic methane potential test system (AMPTS) was used. The AMPTS is a set up where reactors with substrate and inoculum is sealed and placed within a thermostatic water bath and connected through tubes to bottles that absorb water and carbon dioxide. In turn, these bottles are connected to a device that registers the gas that passes through it continuously.

The characterization of sludge from PST and RBF sludge indicated that different primary sludge have different compositions. The different types of RBF sludge (the CEPT-fermented RBF, the RBF and the CEPT-RBF) had the highest carbohydrate content, supporting the hypothesis that there is more cellulose in the RBF. That is reasonable since cellulose has a higher buoyancy and does not settle easily, it is more readily removed by filtration as RBF. The PST sludge had the lowest content of carbohydrates, while the protein and fat content could vary.

The results showed that thermophilic conditions improved the biogas potential. The BMP had a range from 100 to 250 NmL CH_4/g VS at mesophilic conditions for different sludge. In comparison, the thermophilic BMP test had a BMP range of 150-325 NmL CH_4/g VS. In all cases, the BMP of RBF sludge was larger than that of PST sludge. Further, CEPT increased the biogas potential when implemented, while the CEPT-fermented RBF sludge had a lower BMP than non-fermented CEPT-RBF sludge. The sludge produced at the laboratory that simulated different types of PST sludge with a short storage time had a higher biogas potential than both the CEPT-RBF and PST sludge. This data indicates that hydrolysis that occur during storage of primary sedimented sludge can decrease the biogas potential.

Therefore, the results indicate that the difference in composition has a significant impact on the biogas yield. Since the content of the different types of sludge have an effect on the biogas production, it can be concluded that there is a connection between the primary treatment chosen and the BMP.

Sammanfattning

Stigande energianvändning och global uppvärmning har lett till en ökad efterfrågan av förnyelsebara energikällor. Ett alternativ är att utnyttja avloppsvatten för att producera biogas. Avloppsvatten kan därav ses som en resurs snarare än avfall. I biogasproduktion används anaerob nedbrytning där mikroorganismer bryter ner organiskt material till metan.

Primär rening är första steget inom avloppsvattenrening, där partikulärt organiskt material avlägsnas. Slammet som samlas in från det steget är därav rikt på organisk material. Förutom den konventionella försedimenteringen finns även filter som t ex roterande bandfilter. Båda teknikerna kan kombineras med kemisk rening för att optimera reningen. Roterande filter har jämfört med försedimentering ett mindre ytbehov och producerar ett slam med en annan sammansättning än försedimenteringen, det har en högre andel energirik cellulosa. Filterslam har även en högre biogaspotential än försedimenterat slam när slam från olika avloppsvattenreningsverk jämförs.

Projektets mål var att undersöka om filterslam slam har en högre biogaspotential än försedimenterat slam när båda är producerade på samma avloppsreningsverk. Vidare, undersöktes biogaspotential vid mesofila och termofila förhållanden. Slam producerades i laboratorium med sedimentering och polymer för att undersöka påverkan av förvaringstiden i försedimenteringen

Biometanpotentialtester användes för att undersöka biogaspotentialen. Ett automatiskt metanpotentialtestsystem (AMPTS) användes för att utföra testerna. Systemet består av reaktorer som fylls med substratet och ymp när den anaeroba nedbrytningen sker. Reaktorerna är placerade i ett vattenbad och kopplade till behållare med natriumhydroxid som absorberar metan och koldioxid från gasen. Behållarna är kopplade till en enhet som registrerar gasen i volymenheter.

Karakteriseringen av slammen indikerar att det filterslammet (även det fermenterade filterslammet och det filterslammet utan polymer) har en högre mängd kolhydrater än slammet från försedimenteringen. Det stödjer hypotesen om att filterslammet består av mer cellulosa. Det är rimligt eftersom cellulosa har en högre flytförmåga och därför inte sedimenterar lätt och det avlägsnas enklare genom filtrering i roterande filter. Det försedimenterade slammet hade den lägsta mängden av kolhydrater, medan protein- och fett-innehållet varierade.

Resultaten visade att termofila förhållande var bättre för biogasproduktionen. För olika slam varierade biometanpotentialen under termofila förhållanden mellan 150 och 325 Nml CH_4/g VS. I jämförelse varierade biometanpotentialen mellan 100 och 250 Nml CH_4/g VS under mesofila förhållanden. Av det försedimenterade slammet och filterslammet, hade det roterande filterslammen en högre biogaspotential, vilket indikerar att sammansättningen av slammet påverkar biogaspotentialen. Det fermenterade filterslammet hade en lägre biogaspotential än det icke fermenterade filterslammet och ännu högre än det försedimenterade slammet. Bland de laboratorieproducerade slammen producerade det sedimenterade slammet med polymer mer biogas än slammet utan polymertillsats. Båda hade högre biogaspotential än det roterande filterslammet och det försedimenterade slammet. Eftersom hydrolys kan ske under slamlagringstiden tyder det på att förvaringstiden har en signifikant påverkan på biogaspotentialen.

Resultaten indikerar därför att skillnaden i sammansättning av slam har en signifikant påverkan på biogaspotentialen. Eftersom sammansättningen av slammen har en effekt på biogaspotentialen, kan slutsatsen dras att det finns en koppling mellan val av primär rening och biogaspotential.

Abbreviations

AMPTS = Automatic Methane Potential Test System

AD = anaerobic digestion

BMP = bio-methane potential

BOD = biological oxygen demand

CEPT = chemically enhanced primary treatment

COD = chemical oxygen demand

DAF =dissolved air filtration

DS = dry solids

I:S ratio = inoculum:substrate ratio

PST = primary settling treatment

RBF = rotating belt filter

SRT = sludge retention time

SS = suspended solids

TS = total solids

TSS = total suspended solids

VS = volatile solids

VFA = volatile fatty acids

WWTP = Wastewater Treatment Plant

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1 Introduction

1.1 Background

Global warming is one of modern societies great challenges. It has already lead to rising temperatures and extreme weather worldwide. One cause for the global warming is the ever increasing energy demand, where a substantial part is met by fossil fuels. It is therefore most important to limit energy use. In addition, it is essential that renewable sources of energy are investigated and utilized to their fullest extent. In wastewater treatment, there is an interest in biogas production from the sludge produced at wastewater treatment plants (WWTPs). The sludge is rich in organic material that can be transformed into biogas. Due to the potential of biogas production in wastewater treatment, there has been a shift in perception of wastewater – it is seen as a resource rather than waste (Ghasimi et al. 2016b).

By implementing biogas production at the WWTP, energy can be recovered and used to power the treatment of wastewater. Even though most WWTPs are not energy neutral at present, studies have shown that there are more potential energy contained in the wastewater than the treatment process demands. It could therefore be possible to design energy neutral WWTPs or even WWTPs with an excess in energy by optimizing the biogas production (Ghasimi et al. 2016b; Taboada-Santos, Lema & Carballa 2019).

The purpose of primary treatments at WWTPs is to reduce the organic load to the biological treatment downstream. The most common primary treatment is the primary settling treatment (PST). Due to spatial limitations, filters as rotating belt filters (RBF) and drum filters are good alternatives to the PST. It has been stated by Paulsrud, Rusten & Aas (2014) that RBF remove a larger amount of the organic load compared to PST. However, the article by Taboada-Santos, Lema & Carballa (2019) suggest that both PST and filters have similar removal of suspended solids (50%), indicating that the literature is contradictory on this subject (Paulsrud, Rusten & Aas 2014; Taboada-Santos, Lema & Carballa 2019).

Overall, there is a shortage of literature that has investigated the difference of the characteristics between PST and RBF sludge. It is however known that RBF removes a larger portion of certain organics, i.e. cellulose (Paulsrud, Rusten & Aas 2014). Since there is an extensive use of toilet paper in Sweden, the cellulose is a substantial part of the biological oxygen demand (BOD) in the wastewater. Cellulose has a substantial biogas potential, hence by implementing RBF, it is possible that a larger portion of the cellulose can be recovered and utilized for energy production. The part of the cellulose that is not removed from the wastewater continues to the secondary treatment, the nutrient removal step, and is partly oxidized in the aeration treatment. Thereby, there is a hypothesis that RBF as a primary treatment results in a higher biogas production compared to when PST is used due to its higher cellulose removal (Ghasimi et al. 2016b).

The study by Paulsrud, Rusten & Aas (2014) also states that RBF sludge has a higher biogas potential compared to PST. However, the study is based on primary sludge from different full scale WWTPs that is not necessarily comparable since they have a different inlet water. There is no study comparing different types of primary sludge from the same WWTP, thereby originating from the same inlet wastewater. The other study by Taboada-Santos, Lema & Carballa (2019) suggests that RBF in combination with chemically enhanced primary treatment (CEPT) can increase the removal of organics and thereby increase the biogas production. In CEPT, flocculants or/and coagulants are used to maximise removal of organics. Further, the possibility of pre-fermentation adds residence time for the sludge. In case of a short fermentation, this could result in a more accessible substrate for the methanogenesis and thereby in a higher yield and more complete production (Chen, Steen & Green 2004).

Källby WWTP in Lund, Sweden, has PST as a primary treatment in the main stream. Further, a RBF pilot with subsequent fermentation that treats a small part of the same inlet water that enters the PST. Källby also have a system for biogas production that utilizes thermophilic and subsequent mesophilic conditions in the anaerobic digestion (AD) in order to optimize the biogas yield.

1.2 Aim

The aim of the project was to determine if there is a difference in biogas potential of different primary sludge produced from the same WWTP. In addition to the sludges available at Källby, sludge was created in the laboratory from incoming wastewater to Källby WWTP in order to evaluate sludge not affected by hydrolysis. The biogas potential were determined at mesophilic and thermophilic temperatures. The report includes a comparison of:

- The characteristics of different types of primary sludge
- Bio-methane potential (BMP) at mesophilic and thermophilic temperature
- Laboratory produced sludge

1.3 Delimitations

The Automatic Methane Potential System (AMPTS) at the laboratory limits the number of sludge that can be evaluated. As a result, only sludge from Källby were used. The number of trials was limited to two due to limited availability of the AMPTS-units. Literature (Bioprocess Control 2021) states that 30-60 days are a common time period for BMP, therefore 30 days were used in consideration of the time constraint.

2 Literature Review

2.1 Primary Treatment

Wastewater treatment is divided into different steps depending on their purpose. These parts are often preliminary, primary, secondary, tertiary and advanced treatment. WWTPs can have all these steps or a combination of them. Preliminary treatment is placed at the start of the WWTPs and remove inert grit and material and equalize the incoming water. Thereafter, the primary treatment removes a significant portion of the organic material. The secondary treatment utilizes active microbial processes to reduce biodegradable material. Lastly, the tertiary and quaternary steps are often consisting of further phosphorous removal and pharmaceutical removal respectively (Davis 2010).

2.1.1 Primary Settling Treatment (PST)

The conventional and most implemented primary treatment is PST which reduces the flow in a basin and thereby allows particles to sediment on the bottom, see Figure 2.1. The settled sludge can then be removed. The design of PST tanks can be rectangular or circular, where the first optimizes the areal space and the resources available since a common wall can be used for parallel tanks, while the circular solution has less problems with sludge buildup in the corners. (Davis 2010) Although PST is cost effective and removes a significant portion of the organic material (approximately 50% of suspended solids (SS)), it has a large footprint. The large footprint of PST can limit the number of WWTPs that can expand the PST in order to treat more wastewater (Paulsrud, Rusten & Aas 2014; Taboada-Santos, Lema & Carballa 2019).

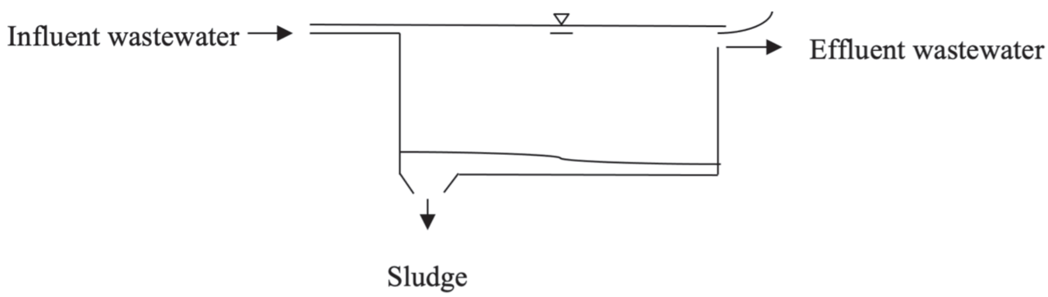

Figure 2.1 An example of a PST tank with sludge collection at the bottom.

2.1.2 Rotating Belt Filter (RBF)

RBF is a sieve technology for wastewater treatment that was launched in early 2000 (Nussbaum et al. 2006). The treatment has gained interest due to the benefits of replacing PST since it has a low footprint and a high removal of SS and cellulose (Paulsrud, Rusten & Aas 2014).

The technology utilizes a fine mesh sieve with a belt cleaning system and can be used either as sole treatment for wastewater or as primary treatment at a WWTP. A schematic, simplified picture of an RBF is found in Figure 2.2. At low water level, the wire mesh is immobile, allowing particles to accumulate on the wire mesh and creating a filter mat. As a filter mat is formed, the water level rises, and the wire mesh automatically moves after signals from a transmitter. The pressure transmitter measures and regulates the water level in order to control and set the optimal speed of the RBF. A less common alternative is a solution where the belt speed is constant and the water level varies. As the filter mat is formed, the removal capacity of the RBF increases. At first, the RBF removes the particles larger than the mesh size. These particles accumulate on the filter and form a filter mat. Thereafter, significantly smaller particles can be removed. The sludge is produced by cleaning the filter at the top of the belt and collecting the resulting sludge continuously as the belt rotates (Nussbaum et al. 2006; Taboada-Santos, Lema & Carballa 2019; Rusten et al. 2017). The sieves come in different mesh sizes, where a common mesh size in wastewater treatment is 0.35 mm (Rusten et al. 2017). A sludge dewatering system is included (Nussbaum et al. 2006).

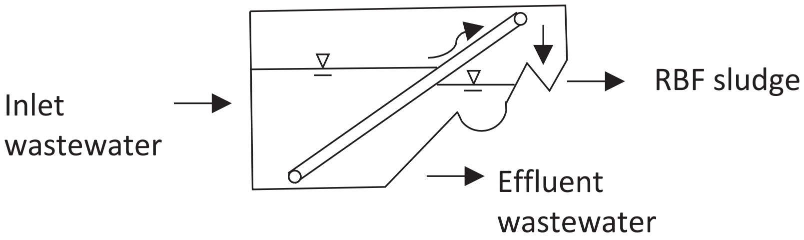

Figure 2.2 A schematic, simplified figure of an RBF illustrated in Nussbaum et al. (2006). The inlet wastewater is first treated with CEPT, before it enters the RBF. The filtered sludge is collected to be fermented.

The study by Nussbaum et al. (2006) state that the RBF treatment approximately removes 40 to 80 % of the total suspended solids (TSS). In other literature, it has been stated that RBF has a removal efficiency of up to 79% of TSS (Taboada-Santos, Lema & Carballa 2009). Paulsrud, Rusten & Aas (2014) stated that the removal is higher than for PST; at least 50%, while Taboada-Santos, Lema & Carballa (2009) and Franchi & Santoro (2015) state that both treatments remove about 50 %. This indicates some inconsistency in the literature on the perception of removal between PST and RBF. The explanation could therefore be that the range of TSS removal of the RBF can differ substantially, e.g. due to operation (Nussbaum et al. 2006).

Rusten & Ødegaard (2006) clarifies that the performance of the RBF technology depends on the operation and design of the specific implemented RBF unit in addition to characteristics of the inlet wastewater. E.g., at a water treatment plant at a brewery, different mesh sizes where tested and the more common 0.35 mm failed. The optimal mesh size was instead 0.25 mm (Nussbaum et al. 2006). Further, some secondary treatment processes need organics, in the form of biological oxygen demand (BOD), in order to function. Therefore, it is important to ensure that when an RBF is implemented, it is operated to ensure the supply to the subsequent process with enough BOD to function (Siegrist et al. 2008). Further, although the RBFs have many advantages, soluble chemical oxygen demand (COD) pass through the RBF (Taboada-Santos, Lema & Carballa 2019).

2.1.3 Chemically Enhanced Primary Treatment (CEPT)

CEPT is a treatment that utilizes coagulation and flocculation, two processes that have the objective to remove more organic material. This is done through adding chemicals to the wastewater. The most common chemicals used in CEPT is metal salts. Further, polymers are used to some extent. Polymers are more advantaged when there is a high turbidity or alkalinity, they are often used in combination with metal salts and in primary treatment. The chemicals turn the small particles into flocs (precipitates or suspended particles) that are larger and readily removed by settling, dissolved air flotation (DAF) or filtration. The chemicals added first condition the particles (coagulation) to simplify the process of flocculation, where the particles aggregate and precipitate to the larger flocs (Davis 2010).

By adding chemicals to the primary treatment, a higher removal efficiency can be achieved. CEPT can be used to optimize both PST and RBF (Taboada-Santos, Lema & Carballa 2019). The removal efficiency has been found to increase from 50% to 90% and 70% for TSS and COD (Taboada-Santos, Lema & Carballa 2019). According to Rusten et al. (2017), the TSS removal can increase with 20% when CEPT is added to the primary treatment.

There is a larger amount of chemicals needed for CEPT with PST compared to CEPT with RBF according to Taboada-Santos, Lema & Carballa (2019). Further, one disadvantage to RBF is that it has limited COD removal (Taboada-Santos, Lema & Carballa 2019). Therefore, a synergetic effect might be achieved when combining RBF with CEPT since the amount of chemicals required is minimized along with an efficient COD removal (Taboada-Santos, Lema & Carballa 2019). Thereby allowing high removal and biogas production while requiring less chemicals than when paired with PST.

The Taboada-Santos, Lema & Carballa (2019) study found that the removal efficiency was similar when sludge from CEPT-PST and RBF sludge were compared. Further, the study showed that CEPT with RBF had a higher removal than when paired with PST or only RBF. RBF and CEPT remove a larger portion of COD, 50% of the soluble fraction in addition to the removal of the particulate COD. When CEPT is paired with RBF, 85 % of the total COD is removed. Hence, strengthening the theory that pairing CEPT with RBF is more advantageous than with PST or only RBF.

2.2 Characteristics of Primary Sludge

When characterizing sludge, there are a number of different parameters that are important to include. These are the volatile solids (VS), the dry solids (DS), pH, protein, fat, carbohydrates, carbon/nitrogen ratio (C/N) and COD. The VS, COD and BOD are measures of the organic content (Carlsson & Schnürer 2011).

2.2.1 Content

Some sources of organic content in the sludge are carbohydrates, protein and fat. These are the major constituents that is transformed into biogas. When comparing the three, the fat and the protein are more readily degradable. Carbohydrates consist of e.g. cellulose and lignin, whereof lignin is more persistent to biodegradation by anaerobic conversion and is therefore not a good source of biogas (Ghasimi et al. 2016a; Angelidaki et al. 2009). Further, fat (1,014 NmL CH_4/g VS) and protein (496 NmL CH_4/g VS) have a higher theoretical BMP than carbohydrates (415 NmL CH_4/g VS) when an average chemical formula is used (Davidsson, 2007). When the composition fat, carbohydrate and protein content in the sludge are known, it is possible to calculate and compare the biogas potential theoretically through Buswell's formula (Angelidaki & Sanders 2004).

2.2.2 Cellulose

In wastewater, a large contribution of the cellulose comes from toilet paper. Up to 30-50% of the SS in wastewater can consist of cellulose (Ghasimi et al. 2015; Ruiken et al. 2013).

The cellulose that enters the WWTP is removed in several steps. First, a portion of the cellulose that is larger in volume is removed through screens in preliminary treatment. In the subsequent primary treatment, the fact that cellulose have a tendency to float influence the removal of cellulose. I.e. a significant portion of cellulose does not settle in a PST. Most of it pass through to secondary treatment. Further, the floatation of cellulose could prevent the settlement of other

SS (Taboada-Santos, Lema & Carballa 2019; Ghasimi et al. 2016b; Ghasimi et al. 2015). Further, Sarathy et al. (2015) tested and found that RBF sludge did have a higher content of cellulose.

Cellulose is difficult to degrade aerobically, about 60% of the cellulose can be degraded through oxidation in the secondary treatment (Ruiken et al. 2013). Since the purpose of secondary treatment is to remove biodegradable organic material, it is beneficial if a larger portion can be removed before the secondary treatment to optimize the effectivity and the cost of the operation (Ghasimi et al. 2015; Ruiken et al. 2013). Further, the biodegradability and biogas potential of the sludge are dependent on if the cellulose is of a higher grade (from virgin pulp) or a lower grade (from recycled pulp). Higher grade cellulose was found to have a lower biodegradability and biogas potential. This indicates that the length and state of the fibers affect the biogas potential (Ghasimi et al. 2016a).

2.2.3 Organic Matter

Generally, the PST sludge have lower values of both DS and VS compared to the RBF sludge, indicating that the RBF removes a larger portion of organic material (see Table 2.1). In addition, the VS/DS is also higher for RBF sludge. Since the VS/DS ratio have been described as a measurement of the organic content, it indicates that RBF sludge has a higher organic content (Sarathy et al. 2015; Paulsrud, Rusten & Aas 2014).

Table 2.1 A compilation of characteristics of different types of primary sludge and some removal efficiencies of the different primary treatments.

Scale	Primary sludge	Characteristics	Removal	Source
Single units	Fine mesh sieve	DS 13.6-36.9 % VS 84.4-96.5 % DS COD/VS 1.3 g/g		Paulsrud, Rusten & Aas (2014)
WWTPs	PST	DS 0.5-6.6 % VS 73.7-90.1 % DS COD/VS 1.6 g/g		
	RBF - CEPT		COD_{tot} 84 % TSS 90 %	Taboada-Santos, Lema & Carballa. (2019)
	PST - CEPT		COD_{tot} 66% TSS 86%	
Pilot	CEPT -RBF	DS 5-7% VS 86-88% of DS	TSS 66%	Rusten et al. (2017)
	RBF		TSS 40-50%	
WWTPs	RBF	DS 3.13 % VS 2.83% VS/TS 0.91		Sarathy et al. (2015)
WWTPs	PST	DS 2.7% VS 2.38% VS/TS 0.88		

The COD content was substantially higher in RBF sludge than in PST sludge, since it has a strong correlation with the DS content (Paulsrud, Rusten & Aas 2014). COD/VS can indicate the biodegradability of a substrate. The biodegradability is important for anaerobic digestion (AD) in biogas production. Paulsrud, Rusten & Aas (2014) found that RBF sludge has a lower COD/VS than PST, see Table 2.1, i.e. that the PST sludge is more biodegradable. The low biodegradability in the RBF sludge could be a result of the fact that the different types of sludge are retrieved from different plants, where the RBF sludge in contrast to the PST sludge were often found at WWTPs without preliminary treatments. It is therefore not a conclusive result that sieves result in a sludge that have a lower biodegradability compared to PST (Paulsrud, Rusten & Aas 2014; Vollertsen & Hvitved-Jacobsen 2002; Hamilton 2016).

2.2.4 Bio-methane Potential of Primary Sludge

There are some studies that have investigated the BMP of primary sludge, see a compilation of some of the results from the literature in Table 2.2. The literature shows that BMP in the range of 287-500 NmL CH_4/VS have been reached.

Table 2.2 Biogas potential from different primary treatments at different scales. Some samples from implemented full-scale treatments and others from pilot plants. The residence times and substrate to inoculum ration (I:S) were included to increase the comparability. Some sources lacked information while some were allowed to be complete gas production (less than 1% of gas of the total production for 3 days or when gas production stopped).

Scale	**Primary sludge**	**BMP [NmL CH_4/g VS]**	**Mesophilic/ thermophilic**	**Residence time [day]**	**I:S**	**Source**
Single units	Fine mesh sieve	345	-	-	-	Paulsrud, Rusten & Aas (2014)
WWTPs	PST	287	-	-	-	
	Dewatered RBF	386	Mesophilic	Complete at 26 days	3:1	Taboada-Santos, Lema & Carballa (2019)
	CEPT-RBF	310	Mesophilic	Complete at 26 days		
	RBF-PST	327	Mesophilic	Complete at 26 days		
Pilot	CEPT-RBF	483	Mesophilic	-	-	Rusten et al. (2017)
	RBF	317	Mesophilic			
WWTPs	RBF	500	Mesophilic	Completed at 12 days	3:1	Sarathy et al. (2015)
WWTPs	PST	550	Mesophilic	Completed at 12 days		
WWTP	Fine sieve	309/ 338	Mesophilic/ thermophilic	Completed at 15 days	3:1	Ghasimi et al. (2016a)
WWTP	Fine sieve	291/ 338	Mesophilic/ thermophilic	Completed at 10 days	3:1	Ghasimi et al. (2016b)

The study Paulsrud, Rusten & Aas (2014) concluded that RBF had a higher BMP compared to PST, see Table 2.2. However, the lack of pre-treatment before RBF could affect the results, e.g. that there are more particles that degrade slower and have a higher BMP in the sieve sludge compared to the PST sludge (Paulsrud, Rusten & Aas 2014).

Contradictory, another study by Sarathy et al. (2015) found that the BMP is similar or even slightly higher in the PST sludge than in the RBF. Sarathy et al. (2015) commented that it could be due to the RBF not being operated with the right sieve fraction, thereby not capturing smaller fractions with a high biodegradability. Further, the characteristics of the inlet water could affect the sludge. Taboada-Santos, Lema & Carballa (2019) had a similar result. Generally, the study also found a higher BMP value from RBF sludge compared to BMP values from other literature of PST. Taboada-Santos, Lema & Carballa (2019) showed that sludge from CEPT-RBF had a lower BMP than sludge from RBF-PST. In this study, iron was used as a coagulant. The result could be a consequence of a low cellulose content in the incoming water as well as a too high dosage of the iron coagulant. Iron is thermodynamically favorable and might therefore limit the methanogenesis and the BMP obtained (Taboada-Santos, Lema & Carballa 2019). In contrast, Rusten et al. (2017) compared RBF and CEPT-RBF, obtaining the result that the BMP was significantly larger when CEPT and RBF was combined. This indicates that the treatment of CEPT might have been more optimized according to the specific conditions of the WWTP. Further, it implies that the CEPT process retrieved smaller particles with a higher degradability that would not end up in the RBF sludge without the addition of chemicals, see Table 2.2.

The I:S ratio and the residence time of a BMP test (Table 2.2) can affect the BMP and the comparability of the results. An insufficient residence time implies that the potential of the gas production in the AD has not been completed, resulting in a lower BMP. Further, a sufficient I:S ratio is significant to achieve accurate results. Since the BMP is presented in methane produced per VS, the results should be comparable with some uncertainty (Angelidaki et al. 2009).

One factor that can affect the biogas potential is the length of the storage time of the sludge before AD. The sludge is biologically active, therefore the sludge content and characteristics can be altered through hydrolysis and fermentation. As a result, recently produced sludge can have different characteristics when compared to sludge that has been stored. The degree of alteration is uncontrollable and dependent on the retention time. PST sludge is wasted periodically in contrast to RBF sludge that is removed continuously, resulting in different retention times (Sarathy et al. 2015).

2.3 Biogas Production

Biogas is produced when organic molecules are degraded in an anaerobic environment, in a process called anaerobic digestion (AD). Biogas mainly consists of methane and carbon dioxide. The production utilizes processes performed by microorganisms and can be divided into several steps; hydrolysis; acidogenesis; acetogenesis/dehydrogenation and methanogenesis. These steps occur nearly simultaneously through the process of biogas production (Weiland 2010; Schnürer & Jarvis 2017).

2.3.1 Hydrolysis

In the first steps, microorganisms hydrolyze the substrate (e.g. lipids, protein, carbohydrates, etc.), a process where the long polymers are degraded into oligomers and monomers (long chains of fatty acids, glycerol, amino acids, etc.) (Weiland 2010; Schnürer 2016). This step ensures degradation of substrate into organics that can be used by other microorganisms to produce biogas. The degradation rate depends on the characteristics of the substrate since some organics are more readily degradable. Further, some compounds might take days to transform into monomers while soluble carbohydrates are hydrolyzed in hours (Weiland 2010; Schnürer & Jarvis 2017). Since cellulose has a high biogas potential and degrades more slowly than other organic content, it is important that the hydrolysis is complete.

Hydrolysis is considered the rate limiting step when substrate originate from wastewater or is plant based due to the high content of cellulose, lignin and hemicellulose. The degradation of these compounds is complex since the compounds have a complicated structure (Schnürer 2016). Moreover, it is also considered rate-limiting in methane production when there is no inhibition or lack of nutrients in the other steps resulting in accumulating intermediates

(Ghasimi et al. 2016). Some hydrolysis might take place in the PST sludge prior to the AD in full scale or BMP test, i.e. in storage and during collection. If methanogenesis also occur, a lower BMP could be the result (Paulsrud, Rusten & Aas 2014).

2.3.2 Acidogenesis

The next step in the AD is the fermentation, also called acidogenesis, where microorganisms use the products from the hydrolysis as substrate, mainly creating organic acids (short fatty acids, alcohols, hydrogen and carbon dioxide). In this step, volatile fatty acids (VFA) are created, these are intermediates that might inhibit the BMP process since they create acidification if they accumulate (Schnürer & Jarvis 2017; Schnürer 2016).

2.3.3 Acetogenesis

In the acetogenesis, anaerobic oxidation processes utilize the acids and produce acetate, carbon dioxide and hydrogen gas from alcohols and VFA. The acetogenesis and the methanogenesis occur in close collaboration since acetogenesis can only be performed in absence of hydrogen gas and are therefore dependent on the methanogenesis to consume the hydrogen gas as it is produced (Schnürer & Jarvis 2017).

2.3.4 Methanogenesis

The last step in producing biogas is the methanogenesis, where the mix of methane and carbon dioxide is generated. Methanogens (group of microorganisms) uses intermediates from acidogenesis and acetogenesis to produce methane (Schnürer 2016). If there is no significant concentration of the intermediate VFA in the end, the BMP process can be considered to be trustworthy and functional since accumulating VFA results in a loss of biogas, can inhibit some microorganisms and overall indicate that the process is unstable. It might be due to inhibited and incomplete acidogenesis and acetogenesis or methanogenesis, e.g. the growth rate of the microorganisms might differ. Since methanogenesis often consume hydrogen gas, it is important that the acids (VFA) do not accumulate and consume it. In that way, the methanogenesis can be inhibited (Schnürer & Jarvis 2017; Romero-Güiza et al. 2016).

2.3.5 Temperature Optimum

Each organism has an optimal temperature range in which it is most productive. The microorganisms can be divided into groups depending on their optimal temperature intervals: psychrophilic, mesophilic, thermophilic and extremophilic. A stable AD can be operated at psychrophilic (0-10°C), mesophilic (30-40°C) and thermophilic conditions (50-60°C), see Figure 2.3. The methane production rate is significantly lower at psychrophilic conditions, resulting in high demands on the design of the biogas and a longer incubation time and larger reactors (Schnürer & Jarvis 2017).

Studies have found that thermophilic conditions can produce more biogas at a lower retention time (Ghasimi et al 2016). The higher yield could be due to that thermophilic temperatures results in a more complete and faster hydrolysis. However, thermophilic conditions in anaerobic digesters have a higher energy demand and might be more unstable and sensitive to changes in the operation or substrate. Generally, the thermophilic process is more unstable due to that the range of microorganisms active at these conditions are less diverse. The advantages to operating under thermophilic conditions could however be considerable if the biogas yield is higher than the energy necessary to obtain thermophilic temperatures instead of mesophilic (Ghasimi et al. 2016b).

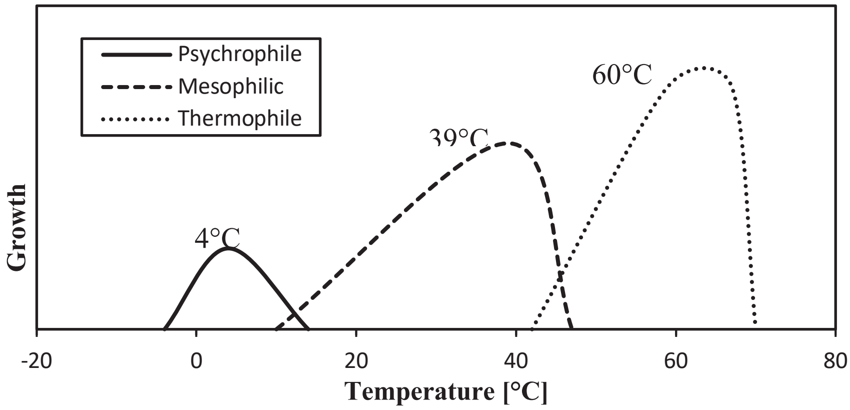

Figure 2.3 The methane production at psychrophilic, mesophilic and thermophilic temperatures with optimum temperatures. Modified from Schnürer & Jarvis (2017) and Madigan & Martinko (2006).

2.3.6 Fermentation of primary sludge before AD

The AD process at WWTPs can be extended with a fermentation process by adding hydrolysis reactors before the AD. In this step, the process is operated in certain conditions to ensure that the substrate is partly digested, i.e. it goes through hydrolysis and acidogenesis. The most important parameters for the fermentation are the solids retention time (SRT), temperature, characteristics and pH of the sludge. It is important to monitor these parameters to limit methanogenesis (Bouzas et al. 2002; Sukphun, Sittijunda & Reungsang 2021).

The advantage of this step is that there is material available for acetogenesis and methanogenesis in the AD (i.e. VFA), thereby optimizing the hydrolysis and the BMP. The disadvantage is that the process can be difficult to operate. Since the gas produced during this step is normally not gathered, there could be a loss of methane during the fermentation if the sludge retention time (SRT) is too long (Chen, Steen & Green 2004).

3 Materials and Methods

3.1 Källby WWTP

The sludge samples were collected from the primary treatment at Källby WWTP, see Figure 3.1. At the WWTP, the wastewater first passes through preliminary treatment before it enters the primary treatment. Most of it enters the mainstream treatment with PST. A side stream is lead to a pilot where the wastewater is first flocculated in the CEPT. In the CEPT, the water is treated with a cationic polymer from Kemira (Super floc C-6260). The effluent passes to the RBF where the filter separates organic material from water, creating an effluent stream of water and a collection of sludge. The RBF sludge collected is fermented in the following step.

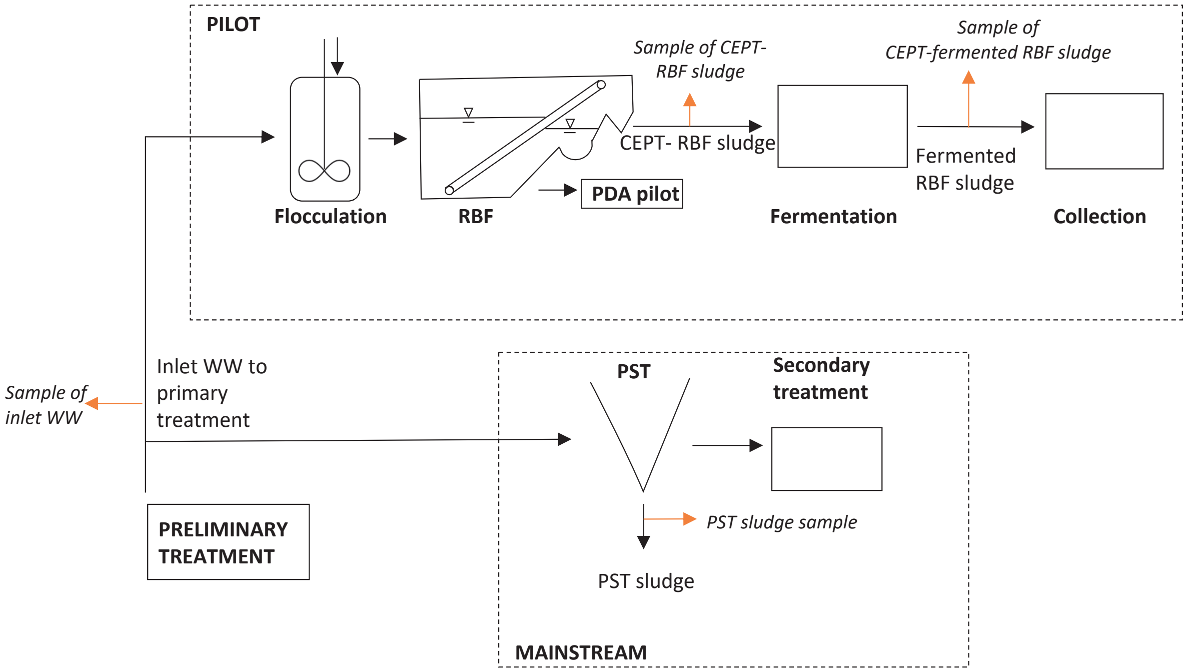

Figure 3.1 The mainstream and pilot primary treatment at Källby WWTP. The orange arrows represent the points at which sludge or wastewater was collected for the experiments.

3.2 BMP Test

The BMP tests were performed in the laboratory by mixing the substrate to be analyzed (the sludge) with inoculum, containing the microorganisms necessary since it is taken from anaerobic digesters that is in operation in full scale (a mesophilic inoculum from Källby WWTP and a thermophilic inoculum from Sjölunda WWTP). The mixtures are managed at anaerobic conditions, thereby generating a production of methane that is measured over time. The Angelidaki et al. (2009) protocol was used in the design of the experiment.

3.2.1 AMPTS Set Up

Two Automated Methane Potential Test system (AMPTS II, Bioprocess Control, Sweden) located at the Department of Chemical Engineering at Lund University were used for the BMP tests. One AMPTS has 15 reactors of 500 ml placed in a heated water bath, thereby five substrates can be tested in triplicates in each AMPTS unit, see Figure 3.2.

The AMPTS was kept in a fume hood. Each reactor is connected with a tube to one CO_2-fixating bottle each, containing 3 M NaOH and a pH indicator where acid gases as CO_2 and hydrogen sulfide (H_2S) are retained. Thereby, only methane is measured in the gas volume measuring device. In turn, each CO_2-fixating bottle are connected to the gas volume measuring device which measures and records the methane produced from each reactor. The volume of methane produced are measured by a certain amount of methane that flows through, creating a digital pulse. The data is recorded, analyzed and displayed by an embedded data system (Bioprocess Control 2021).

Figure 3.2 The AMPTS set up. To the left, the Gas Volume Measuring device above the CO2-fixating devices. To the right, the reactors with motors and stirrers are placed inside the heated water bath. The entire set-up is contained inside a fume hood.

Of the five substrates, one was a reference with cellulose as substrate and one a blank containing only inoculum. The reference is used to determine how well the inoculum performs with a standard homogeneous substrate as cellulose. The blank is included to evaluate the background methane production (Angelidaki et al. 2009).

In the first BMP test, the three substrates were PST, CEPT-RBF and fermented CEPT-RBF sludge from Källby WWTP and pilot plant, see Table 3.1. These were tested at mesophilic and thermophilic temperature. In the second BMP test, two different types of sludge were created in the laboratory and tested and compared to the PST sludge from Källby.

Table 3.1 The experimental plan of the two trials of BMP. It shows the substrate and inoculum used in each AMPTS, which date they were sampled from the WWTP and the date the AMPTS was started. An explanation of the simulated sludge can be found in Section 3.3.

Process	AMPTS	Substrate	Inoculum
BMP Test 1	1 (5/10-21)	PST, CEPT-RBF, CEPT-fermented RBF (4/10-21), cellulose	Mesophilic (1/10-21)
	2 (6/10-21)	PST, CEPT-RBF, CEPT-fermented RBF (4/10-21), cellulose	Thermophilic (1/10-21)
BMP Test 2	1 (18/11-21)	PST, CEPT-RBF, RBF (16/11-21), cellulose	Mesophilic (11/11-21)
	2 (18/11-21)	Simulated PST, simulated CEPT-PST (16/11-21), cellulose	Mesophilic (11/11-21)

3.2.2 Preparation

The inoculum needed were mesophilic and thermophilic. The thermophilic (52°C) was retrieved from Källby WWTP and the mesophilic (37°C) from Sjölunda WWTP, 5-6 days before the start of the BMP. The inoculum was stored at mesophilic (37°C) or thermophilic (55°C) temperature respectively. During this time period, the inoculum was pre-incubated, i.e. degassed, where the residual biodegradable material is depleted until there are no significant methane production. This stage is reached approximately 2 to 5 days after sampling and storage (Angelidaki et al. 2009).

The substrates originating from Källby WWTP were sampled 1-2 days before the start of the BMP tests and stored at a low temperature. The sludge that was produced in the laboratory were produced one day before start of the BMP test.

The content of VS in the substrates were used to calculate the amount of substrate and inoculum in the bottles. The ratio of inoculum and substrate was set to 2:1 of VS. Each bottle had a volume of 0.5 liter, where approximately half should be left as headspace for the production of gas. The substrate and inoculum solution had a volume of 300 ml and a concentration of 3 g substrate VS/l. (Carlsson & Schnürer 2011) Therefore, the amount of substrate VS per bottle was calculated as 0.9 g VS/bottle and the inoculum load as two times as high, 1.8 g VS/bottle. The weight of substrate and inoculum was calculated with the results of the characterization of DS and VS, see Table 3.3 of the inoculum and the sludge. Microcrystalline cellulose was used as cellulose source and it was assumed that this substrate had a VS concentration of 100%. To reach the wanted volume, water was added until the weight of the mixture reached 300 g (= 300 ml). In BMP Test 2, the volume was set to 250 ml with the same VS content to reduce the accumulation of water in the tubes, thereby the load of VS in the reactors were 3.6 g substrate VS/l.

3.2.3 Start up

Each reactor was placed on a scale, after which inoculum and substrate were added and weighted. The remaining weight was filled up with water to obtain a weight of 300 or 250 g. A stirrer and motor cap were inserted into each reactor before the reactors were flushed with nitrogen gas for 60 seconds to create anaerobic conditions and sealed with a tube. The reactors were placed in the heated water bath and connected to the CO_2-fixating units. The AMPTS was assembled according to the Bioprocess Control Manual (2021).

The BMP test was allowed to run for 30 days. The maintenance included filling up the heated water bath and the gas volume measuring device, checking that each bottle was sealed and that the CO_2-fixating bottles could fixate.

At the end of the experiment, the pH and the degree of degradation was tested. The pH in each bottle can indicate if acidification has occurred. The degree of degradation represents the amount of the organic material entering the process that has been degraded and transformed into biogas during a certain time. It is calculated as the ratio of VS entering the reactors and the VS left in the reactor after BMP. Equation 1 was used to calculate the degree of degradation (Schnürer & Jarvis 2017).

$$Degree\ of\ degradation\ (\%) = \left(1 - \frac{VS_{after}}{VS_{before}}\right) \cdot 100 \quad (1)$$

3.2.4 Theoretical BMP

The equation used to calculate the theoretical methane production is known as the Buswell equation and can be observed in Equation 2 (Angelidaki & Sanders 2004; Norberg 2004).

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

In Equation 2, $C_nH_aO_b$ represent the organic material with a different molecular form, e.g. proteins, fat and carbohydrates. To calculate the theoretical BMP, average chemical formulas were used for carbohydrates, fat and protein (Davidsson 2007).

3.3 Production of Sludge

The CEPT sludge was produced in the laboratory at the Department of Chemical Engineering at Lund University. One PST sludge and one CEPT-PST sludge were created, both to simulate primary sludge generated in large scale at Källby WWTP. Further, the laboratory created sludge were simulating sludge with short storage time.

Jar test vessels (Kemira Flocculator 2000) with a volume of 1 liter were used, with accompanying mixers connected to a control device, see Figure 3.2. Time was set aside to determine an effective method of creating sludge through jar tests in an iterative way. Pouring, centrifuging and syringes were techniques tested to separate the supernatant from the sedimented sludge. Concentrations of 0.5-4 g/l of the polymer were tested. The polymer used were a cationic flocculent from Kemira (Super floc C-6260) that is used at Källby WWTP, obtained as a mixture from VA SYD.

First, 1 liter of well mixed wastewater was measured and poured into each vessel. Thereafter, the polymer was added with a syringe below the surface of the filled vessel. The mixing was set to 10 seconds at rapid speed, 10 minutes at slow speed and 10 minutes of sedimentation. The optimal, time-effective method was determined as pouring, centrifuge for thickening, pouring and gathering of the sedimented sludge in a container. The DS and VS concentration of each sludge was measured at the laboratory. The test was performed at 20°C and the sludge were stored at a low temperature until the BMP could be performed.

Figure 3.2 Jar-Test set-up. Each jar has a mixer that is connected to a control device.

3.4 Characterization

The DS and VS analysis was done through heating a sample of a known weight in 105°C overnight and calculating the percentage of DS in the sample, see Equation 3 (Swedish Standards Institute (SIS) 2000a).

$$DS\ (\%) = 100 \cdot \frac{m_{105}}{m_{wet}} \tag{3}$$

m_{105} = weight of sample in dish after drying in 105°C

m_{wet} = weight of sample in dish

The VS percentage is obtained through heating the DS sample at 550°C for two hours and calculating it based on the DS content, see Equation 4. Standardized methods were used for the analyzes (SIS 2000b).

$$VS\ (\%) = 100 \cdot \frac{m_{105} - m_{550}}{m_{wet}} \tag{4}$$

m_{550} = weight of sample in dish after ignition in 550°C

Further characterization of sludge was outsourced to Eurofins Water Testing in order to investigate their composition of e.g. DS, VS, COD, carbohydrates, protein, fat and energy content, see Table I.3 in Appendix I for a list of all the parameters analyzed.

4 Results and Discussion

4.1 BMP Test 1

The presented results are the characterization, comparison of the different types of sludge and a comparison of mesophilic and thermophilic conditions.

4.1.1 Characterization of Sludge

The results of the characterization can be seen in Table 4.1. It shows that there is a larger concentration of DS and VS in PST and CEPT-fermented RBF sludge compared to CEPT-RBF. The large difference between the CEPT-fermented RBF and the CEPT-RBF sludge could be due to a sampling that was not representative of the sludge, since fermentation should not thicken the sludge. The VS/TS ratio was larger for the CEPT-RBF sludge than the PST sludge, indicating that there is a higher organic content in the filtered sludge. This is in line with the literature, e.g. Paulsrud, Rusten & Aas (2014).

Table 4.1 The DS and VS content of the substrates, the inoculum and the cellulose for BMP 1.

	DS (%)	VS (VS after degassing) (%)	VS/DS (%)
Mesophilic inoculum	3.00	1.76 (1.72)	58.6
Thermophilic inoculum	2.07	1.29 (1.15)	50.0
Cellulose	100	100	100
PST	3.33	2.56	76.9
CEPT-RBF	2.59	2.24	86.8
CEPT-fermented RBF	3.65	3.06	83.9

Due to the degassing of the inoculums, the VS changed between the day of retrieval and the day the BMP test was set up, see Table 4.1. Since both VS content decreased, it indicates activity in both inoculums. As a result, the actual total VS load in the reactors was calculated as approximately 2.6 g VS in the mesophilic and 2.5 g VS in the thermophilic, both slightly lower than the wanted 2.7 g VS/reactor.

A low VS content of the inoculum is favorable since it indicates low organic content. Therefore, the inoculum will have a smaller contribution to the total BMP production when the VS is low. Since the thermophilic inoculum has a lower VS content than the mesophilic inoculum, it could be a result of a more well adapted and active inoculum, see Table 4.1. However, this could also be a result of different settings of the operation of the process, e.g. parameters such as residence time and characteristics of sludge that is fermented. Operational issues at Källby WWTP had resulted in a thermophilic AD operated at 49°C instead of the usual 52°C. The VS/DS of the thermophilic inoculum show a lower value compared to the mesophilic inoculum, indicating a low organic content. However, the effective degassing implies that both inoculums are active and functioning and that the level of VS or the VS/DS is not the most important parameter.

The characterization of the inoculum and substrates obtained different result of the organic content (the DS and VS) when comparing the values from the laboratory at Lund University (Table 4.1) and the values from the external laboratory (Table 4.2). An explanation could be an insufficient mixing when sampling for the VS measurements, thereby obtaining a lower organic content. This could affect the credibility of the results. However, the mixing of the sampling was performed in the same way as the mixing when setting up the reactors. The result from the analysis at Lund University could therefore be considered reliable and representative. Further, the results from the outsourced characterization (Table 4.2) showed similar but slightly higher values of VS/DS than the characterization at Lund University (Table 4.1), indicating that the results are reliable.

Table 4.2 The results from the external characterization of the inoculums and substrates.

Sludge	Mesophilic inoculum	Thermophilic inoculum	PST	CEPT-RBF	CEPT-fermented RBF	Unit
DS	3.4	1.8	3.8	4.0	4.2	%
VS	2.2	1.1	3.1	3.7	3.8	%
VS/DS	65.6	62.9	80.3	91.4	90.7	%
pH	8.1	8.6	5.4	5.9	5.1	
Nitrogen Kjeldahl	3200	1500	2300	1300	1400	mg/kg
Nitrogen Kjeldahl	0.32	0.15	0.23	0.13	0.14	%
Ammonium nitrogen NH4-N	1600	940	260	280	420	mg/kg
Ammonium nitrogen NH4-N	0.16	0.094	0.026	0.028	0.042	%
Raw protein	1	0.35	1.28	0.64	0.61	%
Raw fat	0.56	0.49	0.57	0.72	0.56	%
Energy value (calculated)	0.46	0.27	0.63	0.76	0.75	MJ/kg
Carbohydrates (calculated)	0.51	0.2	1.2	2.3	2.6	%
COD-Cr	31 100	20 400	55 400	66 000	53 000	mg/L

As discussed in Section 2.2.1, the content of the sludge that is important for the biogas production is the carbohydrates, fats and proteins, where cellulose is a carbohydrate. The PST sludge had the lowest content of carbohydrate, while the protein content was significantly larger than in the CEPT-RBF and CEPT-fermented RBF sludge, see Table 4.3. In comparison to the PST sludge, the CEPT-RBF and CEPT-fermented RBF sludge have a significantly higher percentage of carbohydrate in the VS. This result is in line with the literature that suggests that PST has a different composition with a lower carbohydrate content, since a portion of the cellulose is not captured during sedimentation due to its buoyancy (Paulsrud, Rusten & Aas 2014).

Table 4.3 The percentage of protein, fat and carbohydrates of VS calculated from Table 4.2.

	PST	CEPT-RBF	Fermented CEPT-RBF	Unit
Raw protein	42	17	16	% of VS
Raw fat	19	20	14	
Carbohydrates	39	63	68	

In addition, the CEPT-fermented RBF sludge has a lower concentration of fat compared to PST and CEPT-RBF sludge. This is could be a result of the fact that fat is the most readily hydrolyzed component during exposition to hydrolysis in the pre-fermentation. It results in a degradation of fat that could be used in methanogenesis if the pre-fermentation is not operated correctly. Thereby, a portion of fat could have been hydrolyzed to smaller components as VFA and could even have been subject to a small methane production or it could be consumed by the microorganisms. Alternatively, the pre-fermentation could have a low efficiency, indicating that the material is not hydrolyzed. Hence, the lower fat concentration could be reasonable.

4.1.2 Mesophilic and Thermophilic BMP

The references with cellulose showed a sufficient production for thermophilic inoculum (325 NmL CH_4/g VS), see Table 4.4. The BMP test with cellulose and a mesophilic inoculum retrieved a significantly lower value (230 NmL CH_4/g VS). This indicates that the mesophilic inoculum did not have a high enough activity or that the mesophilic BMP test might have been disturbed. Further, the measured pH values of the reactors at the end (6.9-8.0) imply that no acidification has occurred in either BMP test, see Appendix II, minimizing the risk that there has been a buildup of VFA that inhibits the methanogenesis.

Table 4.4 The BMP of each substrate with the standard deviation at mesophilic and thermophilic temperature without background production from inoculum and per VS.

Substrate	**BMP [ml CH_4/g VS]**	
	Mesophilic	**Thermophilic**
Cellulose	230±16	325
PST	124±30	159±29
CEPT-RBF	225±41	267±30
CEPT-Fermented RBF	180±29	225±8

The BMP was higher when applying thermophilic conditions on the AD compared to during mesophilic conditions, coinciding with the results from Ghasimi et al. (2016a) that suggests that the biogas production should be equal or higher when using a thermophilic AD, see Table 4.4 and Table 2.2. Overall, the values of BMP were lower than the values presented in literature. For mesophilic test, the BMP varied between 100-250 NmL CH_4/g VS while the BMP for the thermophilic AD varied between 150-325 NmL CH_4/g VS. As a comparison, BMP found in literature varied between 280-500 NmL CH_4/g VS, see Table 2.2 for an overview.

The low values of the mesophilic test could be due to acidification, ammonium accumulation, temperature changes or difference in organic load (Schnürer & Jarvis 2017). Since the pH was neutral (see Appendix II), it implies that acidification was not a problem. In addition, ammonium accumulation is often connected to acidification, therefore it is not probable either. The low performance of the BMP could be due to temperature changes, the thermophilic inoculum were stored at 55°C instead of the temperature at the biogas plant and in the BMP (52°C). This change in temperature might have affected the microorganisms negatively. One possibility is that the measurements of organic content was wrong and impacted the results.

Further, the degree of degradation, see Appendix II, show significantly higher values for thermophilic conditions in the range of 45-50% in comparison to 23-51% for the mesophilic BMP, see Appendix II. There was, however, a large standard deviation (STD) of many substrates, indicating that the degree of degradation was not reliable. Due to the high uncertainty, it was difficult to draw conclusions from the degree of degradation.

4.1.3 BMP from Different Types of Primary Sludge

Table 4.5 show the theoretical BMP of each substrate used in BMP Test 1. It can be observed that the PST sludge has the highest theoretical BMP, closely followed by CEPT-RBF sludge and CEPT-fermented RBF sludge. The cellulose had the lowest theoretical BMP. The low variation between the different types of sludge indicate that the content of the sludge should have a limited effect on the BMP.

Table 4.5 The theoretical BMP of the substrates.

	PST	RBF	CEPT-Fermented RBF	Cellulose	Unit
Theoretical BMP	561	547	512	415	NmL CH_4/g VS

There is an observable difference in methane production when comparing CEPT-RBF, PST and CEPT-fermented RBF sludge, see Figure 4.1 and 4.2. The CEPT-RBF sludge had the largest BMP and the CEPT-fermented RBF sludge in turn had a larger BMP than the PST sludge. This is in line with earlier results from e.g. Paulsrud, Rusten & Aas (2014) and suggests that the content of filtered sludge can produce a more methane.

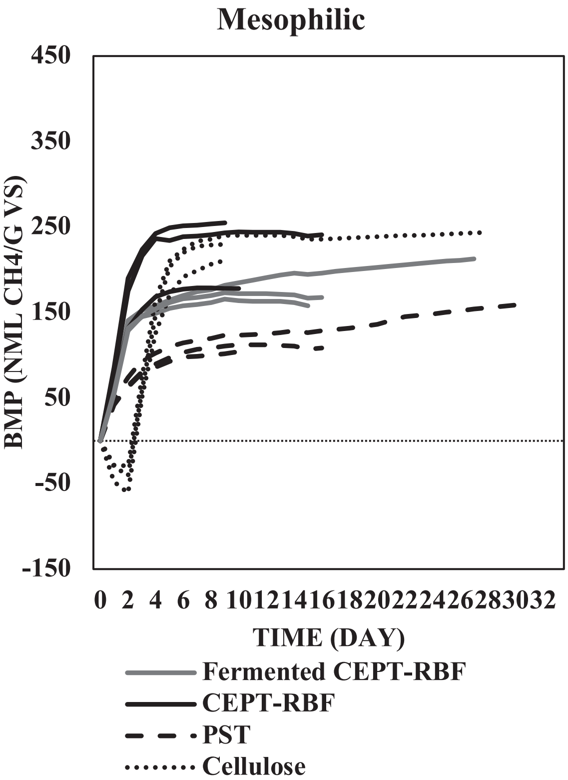

Figure 4.1 The BMP per VS for each substrate in the mesophilic AMPTS.

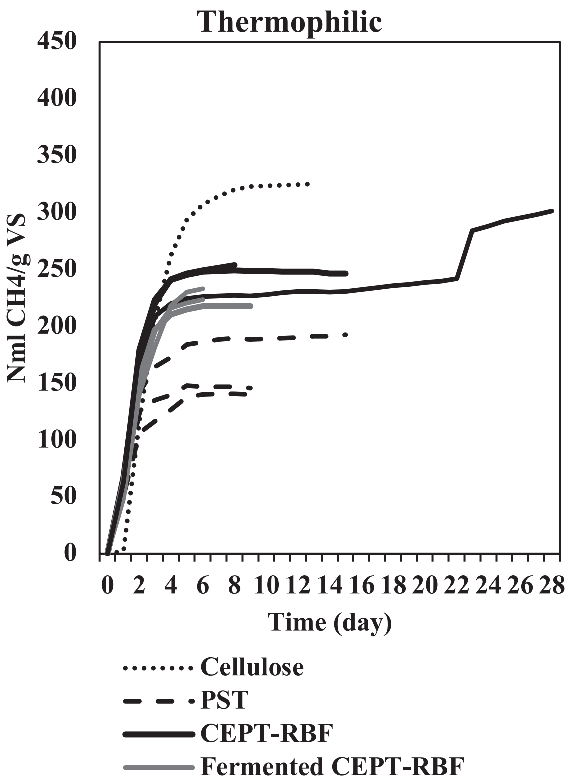

Figure 4.2 The BMP per VS for each substrate in the thermophilic AMPTS.

The CEPT-fermented RBF sludge did not have a larger BMP than the CEPT-RBF sludge. When observing the characteristics of the different sludge (Table 4.2), it shows that the CEPT-fermented RBF sludge had a high portion of carbohydrates, while the fat and protein content was lower than both the other substrates. Since fat and protein have a higher potential than carbohydrates, it is reasonable that CEPT-fermented RBF sludge has a lower BMP compared to CEPT-RBF. However, the theoretical BMP showed that the CEPT-fermented RBF should have had a lower BMP than PST also. Hence, the presence of carbohydrates might have a large influence on the result, producing a higher BMP from CEPT-RBF sludge than PST sludge. Alternatively, the theoretical potential of fat and protein have not been reached in the PST sludge.

The results show that many of the different reactors stopped producing earlier than the 30 days that the BMP test was set to, see Figure 4.1. One explanation could be collection of water in some of the tubes that potentially could block the gas passing to the detection unit. This was, however, not the case for all reactors. It is also possible that there were leakage in some of the reactors. Further, other studies have experienced a short BMP test as well, see Table 2.2. E.g. the study by Sarathy et al. (2015) had a BMP test that lasted 12 days. Ghasimi et al. (2016a), another example, suggest that a well-adapted inoculum probably was important for the rapid BMP tests. In BMP Test 1, a I:S ratio of 1:2 was used, instead of 1:3 as in other studies, which could have affected the results. According to Ghasimi et al. (2016b), both ratios are expected to achieve a complete AD if it has a sufficient residence time. Therefore, neither the residence time or the ratio should have disrupted the production of methane. Further, since the graphs of the BMP reached the characteristic flattening of the curve, there are no suggestions of inhibition, see Figure 4.2, indicating that the biogas production was complete.

4.2 BMP Test 2

In the second BMP test, laboratory produced sludge were analyzed and compared with sludge from Källby. In the following section, the results of the second BMP are presented and discussed. The results include characterization of the sludge and a comparison of the BMP.

4.2.1 Characterization

The characterization show that the inoculum had a similar organic content as in Test 1, see Table 4.1 and 4.6. Further, the organic content of both types of RBF sludge were higher when compared to the PST sludge, similar to other literature as Paulsrud, Rusten & Aas (2014). Among the two types of RBF sludge, the CEPT-RBF had a lower organic content than the RBF sludge. This might be a consequence of the difference in time that the RBF had been allowed to run after the operational stop, RBF for one day in comparison with a couple of hours for the CEPT-RBF.

Table 4.6 The DS and VS content of the substrates, the inoculum and the cellulose for BMP 2.

	DS (%)	VS (VS after degassing) (%)	VS/DS (%)
Mesophilic inoculum	2.83	2.01 (1.72)	60.8
Cellulose	100	100	100
Simulated PST	0.76	0.67	88.0
Simulated CEPT-PST	0.69	0.58	85.0
PST	2.60	2.04	78.2
CEPT-RBF	3.42	3.07	89.8
RBF	3.96	3.76	94.8

The laboratory generated sludge (simulated PST and CEPT-PST sludge) both had significantly lower organic content compared to the sludge from Källby. Most probable is that the low DS and VS content was a result of difficulty in separating the sedimented sludge from the water in the laboratory with a high accuracy. The VS/DS content of the laboratory sludge had comparable values to the other sludge, thereby confirming that the laboratory sludge are more diluted with a comparable organic content. The simulated PST sludge had a slightly higher organic concentration than the simulated CEPT-PST sludge. The dilution might be explained by a too low polymer concentration or the human factor – that the bottles with wastewater chosen for either simulated PST or CEPT-PST were not mixed completely and thereby altering the results.

Even though the concentration is lower, the organic content of the sludge could still be higher depending on the volume of the sludge retrieved from the wastewater.

The results of the external characterization of the sludge sampled at Källby WWTP show that the organic content (DS and VS) follow the same pattern as in the previous characterization, see Table 4.7 and 4.5) - the PST had the lowest VS content and the RBF had the highest. When comparing the values of DS and VS in Table 4.6 and 4.7, it can also be observed that the values of VS are similar, thereby supporting the measurements. This implies that the results are reliable and representative and should give sufficiently reliable BMP results.

Table 4.7 The results from the external characterization of the inoculums and substrates.

Sludge	**PST**	**CEPT-RBF**	**RBF**	**Unit**
DS	2.4	4.4	5.0	%
VS	1.88	3.96	4.6	%
pH	6.1	6.9	6.8	
Nitrogen Kjeldahl	1300	1300	1100	mg/kg
Nitrogen Kjeldahl	0.13	0.13	0.11	%
Ammoniumnitrogen NH4-N	240	210	170	mg/kg
Ammonium nitrogen NH4-N	0.024	0.021	0.017	%
Raw protein	0.66	0.68	0.58	%
Raw fat	0.44	0.54	0.82	%
Energy value (calculated)	0.4	0.78	0.95	MJ/kg
Carbohydrates (calculated)	0.75	2.8	3.2	%
COD-Cr	35 000	61 000	48 000	mg/l

Similar to the characterization for BMP Test 1, the results show a substantially higher carbohydrate content in both types of RBF sludge compared to the PST sludge, see Table 4.8. It might indicate that the cellulose content is higher in the RBF sludge due to a different primary treatment as indicated by other literature (Paulsrud, Rusten & Aas 2014). The content of raw protein and fat is larger in the PST sludge. Further, the content of raw fat is substantially higher in the RBF sludge than in the CEPT-RBF sludge. Therefore, it is not only carbohydrates that differentiate the different primary sludge, both protein and fat could have a significance.

Table 4.8 The content of protein, fat and carbohydrates as percentage of VS.

	PST	**CEPT-RBF**	**RBF**	**Unit**
Raw protein	35	17	13	% of VS
Raw fat	23	14	18	
Carbohydrates	40	71	70	

4.2.2 BMP

The theoretical BMP of the different types of sludge (Table 4.9) show that the PST sludge has the largest theoretical BMP while the CEPT-RBF has the lowest theoretical BMP, which is not in line with the BMP obtained from studies (see Table 2.2). It is important to notice that this does not consider the sludge production and the total BMP (ml CH_4/l sludge).

Table 4.9 The theoretical BMP for the PST, RBF and CEPT-RBF sludge calculated with the results of the characterization (Table 4.6).

	PST	CEPT-RBF	RBF	Cellulose	Unit
Theoretical BMP	577	517	531	415	NmL CH_4/g VS

The second BMP test had similar values of BMP for the cellulose (316 and 328 NmL CH_4/g VS) in both AMPTS, see Table 4.10, indicating relible results. Since the BMP of the cellulose also was in line with the BMP of cellulose in the thermophile BMP (see Figure 4.1), the results imply that the thermophilic BMP test and the BMP Test 2 were reliable and that the inoculum was active and well adapted to the substrate. Moreover, the pH values of the reactors at the end of the experiment (7.01-7.81) indicate that no acidification and accumulation of VFA have occurred in any of the reactors, see Appendix II. The results are therefore trustworthy.

Table 4.10 The BMP of each substrate with the standard deviation.

AMPTS 1		AMPTS 2	
Substrate	**BMP [NmL CH4/g VS]**	**Substrate**	**BMP [NmL CH4/g VS]**
Cellulose	328±31	**Cellulose**	316±10
PST	211±14	**Simulated PST**	351±20
CEPT-RBF	315±13	**Simulated CEPT-PST**	397±12
RBF	251±32		

The results show that the BMP for the sludge from Källby follow the same pattern as in the first BMP Test (see Table 4.4 and 4.10), the BMP of both types of RBF sludge are higher than that of the PST sludge. Regarding the two different RBF sludge, the CEPT-RBF had a significantly higher BMP than the RBF sludge. This migth be a result of the CEPT treatment, which causes flocculation and could retain organic material with different biogas potential that otherwise pass through the RBF. Hence, it supports results from Rusten et al. (2017) (see Table 2.2).

Further, the BMP obtained experimentally (for PST, RBF and CEPT-RBF sludge) from BMP Test 2 did not follow the same pattern as the values of the theoretical BMP (see Table 4.9 and Table 4.10). This might indicate that the difference in organic content of the sludge affect the BMP. Since the higher carbohydrate concentration is the parameter that differentiate RBF sludge from PST sludge, the carbohydrate content seems to be significant for the biogas production. However, the types of fat and protein available in the different types of sludge is unknown and it is therefore not certain that the carbohydrate content is the only significant content.

However, the BMP values were considerably lower than the theoretical BMP. Compared to the previous mesophilic test from BMP Test 1, the results were considerably higher in the second mesophilic BMP test (see Figure 4.1 and 4.4), indicating that the mesophilic BMP Test 1 was not well functioning. However, even if the results of the second BMP Test was higher than the previous mesophilic test, compared to some of the experimental data, see Table 2.2, the values achieved in this study were also quite low. The low experimental BMP compared to the theoretical shows that the maximal BMP have not have been reached.

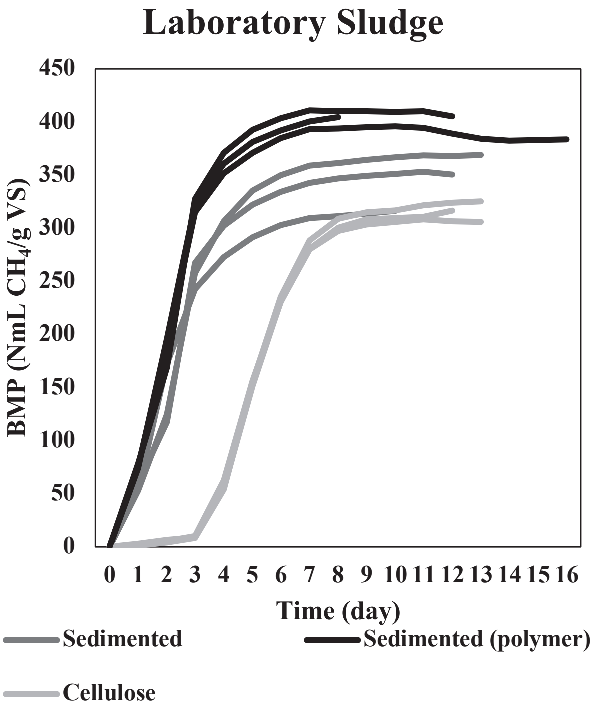

Figure 4.3 The BMP of the triplicate reactors for simulated PST and CEPT-PST sludge and cellulose.

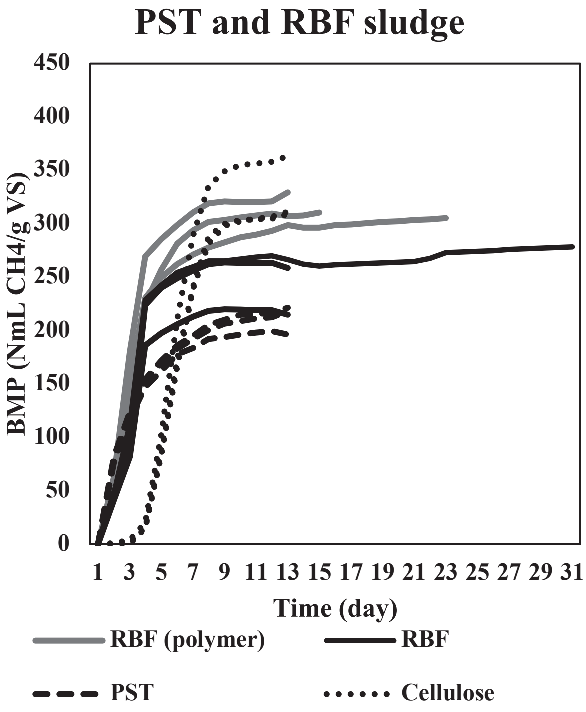

Figure 4.4 The BMP of the triplicate reactors for the PST, RBF and CEPT-RBF sludge and the cellulose.

Among the laboratory sludge, the simulated CEPT-PST had a larger BMP per VS than the simulated PST sludge, see Figure 4.3. Further, BMP of both types of simulated PST sludge had a larger BMP than the PST and RBF sludge from the primary treatment at Källby, see Figure 4.4. This implies that the residence time could have an impact on the content of the sludge due to hydrolysis and that could result in a lower BMP if the sludge have been stored. However, since the scale of the production of the sludge at the lab is low, there is uncertainties.

Similar to BMP Test 1, the reactors stopped producing methane as early as 13 days, see Figures 4.3 and 4.4. Since all the substrates have the characteristic flattening of the curve prior to the end of methane production, it can be assumed with the same arguments as presented earlier that the production stopped naturally. However, in this case, a significant number of reactors stopped production on the same day. It is therefore possible that the measurement of gas did not function accurately.

Since the degree of degradation of the inoculum and the cellulose have similar values in both AMPTS (see Table 4.11), the results of the calculated degree of degradation could be reliable. The degree of degradation of the PST sludge was lower than for both types of simulated PST sludge, implying that more of the organic material was degraded and converted into gas, which coincides with earlier results that PST and indicates that longer storage time affects the BMP. Further, the degree of degradation of PST sludge were also lower than for RBF and CEPT-RBF sludge. This indicates that the content of VS available was higher in RBF sludge. Further, this implies that the carbohydrates might be more readily degradable.

Table 4.11 The degree of degradation and the standard deviation between the triplicate reactors of the substrates from Källby (RBF with polymer, RBF and PST in AMPTS 1) and the substrates created in lab-scale (Sedimented and sedimented with polymer).

	AMPTS 1			**AMPTS 2**	
	Degree of degradation [%]	**Standard deviation [%]**		**Degree of degradation [%]**	**Standard deviation [%]**
Inoculum	15	0.7	**Inoculum**	15	1.3
CEPT-RBF	38	0.5	**Simulated PST**	39	5.5
RBF	39	1.4	**Simulated CEPT-PST**	35	0.9
PST	28	0.7			
Cellulose	42	2.0	**Cellulose**	43	0.9

Since a well-functioning BMP test should have a degree of degradation of 50-70%, the values for the test were too low. This indicates either that there is a larger portion of non-degradable organic material in the sludge or that the capacity of the BMP has not been reached. Further, as can be observed, the standard deviation was quite low for most of the different types of sludge, indicating a low uncertainty for the degree of degradation.

5 Conclusion

In this thesis, the characteristics and biogas potential of different sludge from Källby WWTP produced from the same inlet water have been studied and analyzed by using a BMP test. The different types of sludge included were PST, CEPT-RBF CEPT-fermented RBF, RBF and laboratory simulated CEPT-PST sludge and simulated PST sludge. The following conclusions could be drawn from the characterization:

- The PST sludge had the lowest content of carbohydrates compared to RBF sludge, while the protein content could vary. This is reasonable since cellulose (a carbohydrate) has a higher buoyancy and does not settle easily, it is more readily removed by filtration as RBF.

- The content of fat was higher in CEPT-RBF sludge compared to the CEPT-fermented RBF sludge. This could be explained by the fact that fat is easily hydrolyzed and could have been used by microorganisms during the fermentation.

- The characterization of sludge proved that different primary sludge have different, characteristic compositions.

The conclusions drawn from the BMP tests:

- Thermophilic conditions might increases the biogas production compared to mesophilic conditions.

- For both mesophilic BMP tests and the thermophilic test, the different types of CEPT-RBF sludge had a higher BMP than PST sludge. Further, CEPT-fermented RBF sludge had a smaller BMP than CEPT-RBF.

- A longer storage time affects the organic material available for degradation and biogas production negatively, indicating that methanogenesis or consumption by microorganisms of organic material can occur on a significant level.

- Since the composition of the sludge have an effect on the biogas potential, it can be concluded that there is a connection between the primary treatment chosen and the BMP.

6 Future Work

Since this study uses BMP with the unit of methane per VS, a mass balance should be done to determine the total biogas production from the different types of sludge.

More research should be done on the CEPT-fermented RBF sludge to determine the reason that it has a lower performance of biogas production.

It has been concluded that the primary treatment affects the composition of the content in primary sludge. However, it could not be concluded that it was specifically cellulose. For future research, a BMP test with determination of cellulose content in addition to a characterization of carbohydrate, fat and protein content could be performed.

Further, the result that both types of simulated PST sludge had a higher BMP than the sludge that have been stored for a longer period, should be validated. To do that, a more extensive characterization on the sludge and BMP tests should be done.

In the production of simulated CEPT-PST sludge, a polymer was used as chemical treatment. For future work, simulated sludge with different chemicals, i.e. iron salts, could be tested and compared to CEPT-PST with polymer.

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Appendices

Appendix I

The design of each BMP test can be seen in Table I.1 and I.2

Table I.1 Design of the first test of BMP. One AMPTS was set to mesophilic conditions, one to thermophilic conditions.

PST	CEPT-RBF	CEPT-fermented RBF	Cellulose	Inoculum
PST	CEPT-RBF	CEPT-fermented RBF	Cellulose	Inoculum
PST	CEPT-RBF	CEPT-fermented RBF	Cellulose	Inoculum

PST	CEPT-RBF	CEPT-fermented RBF	Cellulose	Inoculum
PST	CEPT-RBF	CEPT-fermented RBF	Cellulose	Inoculum
PST	CEPT-RBF	CEPT-fermented RBF	Cellulose	Inoculum

Table I.2 Design of the second test of BMP using the sludge produced at the laboratory.

Simulated PST	Simulated CEPT-PST	Cellulose	Inoculum
Simulated PST	Simulated CEPT-PST	Cellulose	Inoculum
Simulated PST	Simulated CEPT-PST	Cellulose	Inoculum

CEPT-RBF	RBF	PST	Cellulose	Inoculum
CEPT-RBF	RBF	PST	Cellulose	Inoculum
CEPT-RBF	RBF	PST	Cellulose	Inoculum

Table I.3 List of all parameters analyzed, the method used to analyze all parameters and the laboratory which performed it.

Parameter	Method	Laboratory
DS	SS-EN 12880:2000	Eurofins Environment Testing Sweden
VS	SS-EN 12879:2000	Eurofins Environment Testing Sweden
pH	SS-EN 15933:2012	Eurofins Environment Testing Sweden
Nitrogen Kjeldahl	EN 13342	Eurofins Food & Feed Testing Sweden
Nitrogen Kjeldahl	Calculated from analyzed amount	Eurofins Environment Testing Sweden
Ammonium nitrogen (NH4-N)	Standard methods 1998, 4500 mod	Eurofins Food & Feed Testing Sweden
Ammonium nitrogen	Calculated from analyzed amount	Eurofins Environment Testing Sweden
Raw protein (Nx6.25)	Calculated from analyzed amount	Eurofins Environment Testing Sweden
Raw fat	Calculated from analyzed amount	Eurofins Environment Testing Sweden

Energy worth (calculated)	SLVFS 1993:21	Eurofins Environment Testing Sweden
Carbohydrates (calculated)	SLVFS 1993:21	Eurofins Environment Testing Sweden
Chemical oxygen consumption, COD-Cr	ISO 15705:2002	Eurofins Water teasting Sweden
Raw fat	NMKL 160 mod.	Eurofins Food & Feed Testing Sweden

Appendix II

The measured pH can be seen in Table I.4. It shows that all reactors at the end of both tests had a neutral pH (6.9-7.9), indicating that no acidification has occurred as a result of an accumulation of VFA. Since the accumulation of VFA suggests that the AD is not complete, the results of the BMP tests are reliable.

Table I.4 The pH of each bottle at the end of the experiment for BMP 1 and BMP 2.

	BMP Test 1	**BMP Test 2**
1	7.6	7,71
2	7.5	7.71
3	7.6	7.64
4	7.4	7.43
5	7.3	7.28
6	7.3	7.34
7	7.3	7.42
8	7.4	7.13
9	7.4	7.41
10	7.2	7.46
11	7.3	7.54
12	7.4	7.53
13	7.1	7.25
14	7.0	7.31
15	7.3	7.32
16	7.9	7.81
17	7.6	7.72
18	7.7	7.68
19	7.7	7.34
20	7.9	7.26
21	7.8	7.25
22	7.9	7.19
23	8.0	7.39
24	7.6	7.49
25	7.8	7.01
26	7.7	7.29
27	7.8	7.08
28	6.9	-

The degree of degradation, see Table I.5, show a significant difference in the result of the mesophilic and the thermophilic BMP Tests. In addition, the standard deviation between the triplicates are quite high, indicating a high uncertainty of the degree of degradation. The inconclusive result can be a result of the low accuracy of the analysis of VS. Carlsson & Schnürer (2011) comments that the degree of degradation is a parameter with a risk of high variance and do not recommend it as a method of analyzing the results of the BMP.

Table I.5 The degree of degradation and the standard deviation between the triplicate reactors of the substrates at mesophilic and thermophilic temperature.

	Thermophilic		Mesophilic	
	Degree of degradation [%]	**Standard deviation**	**Degree of degradation [%]**	**Standard deviation**
Inoculum	12	13	10	12
Cellulose	45	3	48	-
PST	48	13	43	8
CEPT-RBF	51	1.7	23	15
CEPT-Fermented RBF	44	10	36	8

Appendix III – Populärvetenskaplig Sammanfattning

Slam från vattenrening - miljövänlig energikälla istället för avfall

Föroreningar i tätbebyggda områden, extremväder och förhöjda temperaturer är problem som samhällen över hela jorden nu står inför. Först och främst beror dessa problem på en ökad energianvändning och utsläpp från fossila bränslen. Den viktigaste lösningen är att minska energiförbrukningen. I andra hand så måste de stora bovarna inom energianvändning som kolkraft och fossila drivmedlen ersättas med grön, förnyelsebar energi. Med grön energi kan konsekvenserna av fossila bränslen minska. Med i sämsta fall 10 år kvar tills 1,5 graders målet inte längre kan uppnås enligt den senaste klimatrapporten från IPCC är det viktigare än någonsin att ställa om energiproduktionen. Ett sätt att producera grön energi är att använda slam. Men vad har slam med energi att göra?

Slam är restprodukten från ett reningsverk. Slammet separeras från avloppsvattnet och liknar en sörja i konsistens och färg. Det är ett organiskt material likt gödsel, hushållsavfall och växter. Eftersom slam är organiskt kan det brytas ner till biogas av mikroorganismer i en syrefri miljö. Biogas är en blandning av olika gaser där största delen består av metan. Idag finns biogasproduktion på flera stora vattenreningsverk i Sverige och biogasen har flera användningsområden. Den kan användas lokalt för att driva vattenreningsverket eller kollektivtrafik. Idag använder 17 av 21 svenska regioner biogas i kollektivtrafiken. Det är ett framsteg eftersom det ersätter fossila drivmedlen och därför inte bidrar till utsläpp av växthusgaser.

I takt med att användningsområdet av biogas ökar, behöver även biogasproduktionen öka. Av den anledningen pågår idag forskning på vattenreningsverk för att öka kunskapen om biogasproduktion. En del fokus ligger på optimering av själva biogasprocessen, medan en del har flyttats till slammet som används för biogasproduktionen. Bland annat så har forskning visat att slam med olika produktionssätt och från olika reningsverk har olika förmåga att omvandlas till biogas.

Det finns primärt två olika sätt att avskilja organiskt material från avloppsvattnet och skapa slam. Den första är att leda avloppsvattnet in i stora bassänger där en del av det organiska materialet sjunker till botten, likt sand som blandats med vatten. Det material som samlas på botten är slam och kallas sedimenterat slam. Detta är den äldsta och vanligaste metoden att rena vatten. Sedan början av 2000-talet finns det en ny metod som kan användas istället för stora bassänger. Det är ett typ av filter som fångar upp organiskt material medan vattnet passerar igenom det. Filter som reningsmetod har visat sig ha flera fördelar. Det tar upp mindre yta än stora bassänger och skapar slam som innehåller mer cellulosa än konventionellt sedimenterat slam. Filterslam har även visat sig kunna generera mer biogas i förhållande till sedimenterat slam, något som potentiellt kan kopplas till den större andelen cellulosa i slammet.

På reningsverket Källby i Lund har en pilot byggts för att undersöka filterslam och jämföra det med slammet från de stora bassängerna som idag används för att rena vattnet som kommer till reningsverket. I det här examensarbetet har sedimenterat slam jämförts med filterslam, för att avgöra om den nya metoden kan öka biogasproduktionen. Resultaten från studien stödjer att biogaspotentialen kan öka genom att välja filter som reningsmetod istället för stora bassänger. Det här resultatet bidrar till ökad förståelse av biogasproduktion och till hur framtida vattenreningsverk kan designas utifrån perspektivet att slam är en energikälla.

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