Evaluation of Biological Hydrolysis Pre-treatment and the Biogas Potential of Sludge from Compact Waste Water Treatment

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Master Thesis 2015

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Master Thesis number: 2015-12

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Lund University

June 2015

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Picture on front page: The set-up used for biological hydrolysis of sludge. Photo by Lina Falk

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Acknowledgments

First of all, I would like to express my deep gratitude to my supervisor *Åsa Davidsson* for her valuable encouragement, patience and knowledge. Her willingness to give her time so generously has been very much appreciated. Further, I wish to thank my co-supervisor *Tobias Hey* for his professional guidance, expertise and useful critiques of this master's thesis. Without the support of my two supervisors, this process would have been so much harder.

My examiner *Michael Cimbritz* do I also thank for his optimism and proficiency. I am grateful to my supervisors and examiner for letting me write a master's thesis about such an interesting subject, for providing me with the necessary material and for always answering my questions. I would also like to thank *Janne Väänänen* in addition to the staff at Källby ARV for their help with providing me with sludge and the necessary data.

I would also like to express my gratitude to *Gertrud Persson* for not only teaching me how to perform and comply with the laboratory procedures and routines, but also for analysing the numerous VFA samples I managed to generate. Thank you for your patience with the not so helpful GC.

A special thanks to *Hamse Kjerstadius* and the rest of the Water and Environmental Engineering group at LTH likewise.

Thanks as well to *Nicolina Magnusson*, *Hjalmar Larsson*, *Jean Monhonval* and the other master's thesis students for their friendship and support in the lab.

This project would have been impossible without the support of *VINNOVA* and *Region Skåne*.

Finally, I would like to thank my beloved family and friends for their endless support and for believing in me.

Summary

Due to increasingly stringent outlet demands from waste water treatment plants, more resources in the form of electricity and chemicals will be needed if today's technology is to be used henceforth. Consequently, more efficient methods are being investigated, which allow not only for the waste water to be treated but also offer an opportunity to utilise the resources present within the waste water. A resource of particular interest for this study is the organic matter, which can be used for the production of renewable energy.

An innovative compact waste water treatment concept is currently being evaluated in the research project "Den varma och rena staden" (*The warm and clean city*). A pilot plant has been installed at the Källby waste water treatment plant in Lund consisting of a drum filter followed by a microfiltration membrane and a biomimetic membrane for forward osmosis. The idea of this new concept is to replace the primary clarifier and activated sludge step of a conventional waste water treatment plant. One of the advantages of this concept is an augmented extraction of organic matter from the waste water to the side streams generated. By the additional use of chemically enhanced primary treatment the separation efficiency of suspended solids and phosphorus can be further enhanced.

In the present study the side streams generated by the drum filter and the microfiltration membrane have been evaluated with regard to their suitability for anaerobic digestion. By means of biochemical methane potential tests, the methane potential of the drum filter sludge with and without chemically enhanced primary treatment was compared to that of the conventionally generated sludge at Källby waste water treatment plant.

Furthermore, in order to increase the solubilisation of organic matter and by that the biogas production the potential of using biological hydrolysis as a pre-treatment step before the anaerobic digestion has been evaluated as well.

The results from the biochemical methane potential tests using non-hydrolysed raw respectively chemically pre-treated drum filter sludge showed a higher methane potential than for the conventional mixed sludge currently fed to the anaerobic digesters at Källby waste water treatment plant. An even higher methane potential was achieved when using biological hydrolysis to pre-treat the raw respectively chemically pre-treated sludge. In addition to higher methane potentials for the drum filter sludge, a faster initial methane production was also seen for the drum filter sludge in the biochemical methane potential tests.

Furthermore, the application of biological hydrolysis as a pre-treatment step led to an increased solubilisation of the organic matter in the sludge from the compact waste water treatment process. Especially the measured amount of volatile fatty acids, a necessary component used for the production of methane, increased and conduced to a higher methane potential in the biochemical methane potential tests.

Moreover, both the drum filter sludge and the membrane retentate proved to be possible to thicken through measurements of the sludge volume index, although further analyses and tests are needed in order to determine the most suitable method. Further investigations are also required in order to evaluate the methane potential of the sludge generated for the other configurations of the compact waste water treatment pilot plant.

Sammanfattning

Med anledning av de ökande utsläppskraven för avloppsreningsverk kommer ökade resurser i form av elektricitet och kemikalier att krävas om dagens teknologi ska användas även i fortsättningen. Följaktligen undersöks för tillfället mer effektiva metoder, vilka tillåter inte bara rening av avloppsvattnet utan även erbjuder en möjlighet att ta tillvara på de resurser som finns i avloppsvattnet. En resurs av särskilt intresse för den här studien är det organiska materialet, vilket kan användas för produktion av förnyelsebar energi.

Ett innovativt kompakt avloppsreningskoncept utvärderas för närvarande i forskningsprojektet ”Den varma och rena staden”. En pilotanläggning har installerats på Källby avloppsreningsverk i Lund, bestående av ett trumfilter följt av ett membran för mikrofiltrering och ett biomimetiskt membran för osmos. Syftet med det nya konceptet är att det ska ersätta försedimenteringen och det aktiva slamsteget på ett konventionellt avloppsreningsverk. En av fördelarna med detta koncept är en ökad avskiljning av organiskt material från avloppsvattnet till sidoströmmarna som genereras. Genom ytterligare tillsats av fällningskemikalier, kan avskiljningsgraden för suspenderade partiklar och fosfor ökas ytterligare.

I denna studie har sidoströmmarna som genereras av trumfiltret och mikrofiltreringsmembranet utvärderats med hänsyn till deras lämplighet för anaerob rötning. Genom satsvisa rötförsök har metanpotentialen för trumfilterslammet med och utan kemiskt förbättrad förbehandling jämförts med den för det konventionellt genererade slammet på Källby avloppsreningsverk.

Vidare, för att öka lösligheten av organiskt material och genom detta även biogasproduktionen har dessutom möjligheten att använda biologisk hydrolys som förbehandlingssteg innan den anaeroba rötningen utvärderats.

Resultaten från de satsvisa rötförsöken med obehandlat respektive kemiskt förbehandlat trumfilterslam visade en högre metanpotential än det motsvarande konventionella blandslammet som för tillfället rötas i rötkamrarna på Källby avloppsreningsverk. En ännu högre metanpotential uppnåddes när biologisk hydrolys tillämpades som förbehandling av det råa respektive kemiskt förbehandlade trumfilterslammet. Förutom högre metanpotentialer för trumfilterslammet, påvisades även en snabbare metanproduktion för trumfilterslammet i början av de satsvisa rötförsöken.

Vidare ledde användningen av biologisk hydrolys som förbehandlingssteg även till en ökad löslighet av de organiska materialet i slammet från den kompakta avloppsreningsprocessen. Specifikt ökade den uppmätta mängden av lättflyktiga fettsyror, vilket är en viktig komponent i produktionen av metan, och bidrog till en ökad metanpotential i de satsvisa rötförsöken.

Dessutom visade sig, genom mätningar av slamvolymindexet, både trumfilterslammet och retentatet från membranet för mikrofiltrering möjligt att förtjocka även om ytterligare analyser och tester krävs för att fastställa vilken metod som är den mest lämpade. Ytterligare undersökningar krävs även för att utvärdera metanpotentialen för slammet som framställs från de olika konfigurationerna för den kompakta pilotanläggningen för rening av avloppsvatten.

Abbreviations

COD	Chemical oxygen demand
DF	Drum filter
FO	Forward osmosis
GC	Gas chromatograph
HRT	Hydraulic retention time
MF	Microfiltration
NF	Nanofiltration
PE	Population equivalents (based on 70 g BOD/pe·d)
RO	Reverse osmosis
SS	Suspended solids
SVI	Sludge volume index
TOC	Total organic carbon
TS	Total solids
UF	Ultrafiltration
VFA	Volatile fatty acids
VS	Volatile solids
VSS	Volatile suspended solids
WWTP	Waste water treatment plant

Contents

1 Introduction

1.1 Background

Waste water treatment is important with regard to several aspects. It is a crucial health measurement for the citizens of urbanized areas to efficiently remove pathogens as well as one of the vital measures in order to prevent further eutrophication of the recipients. Eutrophication due to an abundance of nutrients in the water is a severe problem leading to algal blooms and lack of oxygen at the bottom of e.g. the Baltic Sea as a result of the increased biological activity, which is largely caused by prolonged discharge of treated waste water into the recipients.

The Baltic Sea Action Plan (BSAP) however, adopted in 2007 by the European Union and the states surrounding the Baltic Sea, aim to improve the "ecological status of the Baltic marine environment by 2012", which in turn leads to increasingly stringent demands on the waste water treatment. As the demands on the effluent quality become more stringent, more resources in the form of electricity and chemicals will be needed if today's technology is to be used henceforth. With the increasing urbanization and as a consequence the increasing load to the waste water treatment plants taken into account, more efficient treatment methods will be needed.

The by-product when treating waste water is sludge, earlier regarded as a waste but now as a source of energy and nutrients. In order to benefit from the nutrients present in the sludge, it can be used as a fertilizer on farmland. The suitability of sludge for this application is however being debated due to its content of heavy metals, pathogens and pharmaceuticals, which renders it increasingly difficult to recirculate the nutrients to arable land. As a consequence it is of importance to decrease the sludge volumes and reduce the costs associated with the handling and treatment of the sludge. A possible approach would be to increase the efficacy of the anaerobic digestion, which is the common treatment process for sludge in Swedish municipal waste water treatment plants.

Concurrently with the increasing demands and load to the existing waste water treatment plants, the interest for new, more efficient treatment methods which allow not only for treatment of the water but also offer an opportunity to utilize the resources present in the water increases as well. One important resource, out of which a majority usually is being oxidized into carbon dioxide, is the organic material which can be used to produce biogas, which in turn can be used either internally at the plant or be upgraded and used as a biofuel to replace fossil fuels.

A possible future solution which is to be evaluated by the current research project "Den varma och rena staden" (The warm and clean city) is compact waste water treatment, for which a pilot has been constructed at Källby waste water treatment plant (WWTP), Lund. In order to decrease the required surface as well as the environmental impact, the conventional treatment steps, including the primary clarifier and the activated sludge tanks, are replaced by a drum filter and two types of membranes. The resulting side streams consisting of sludge from the drum filter and retentate from the membranes can then be hydrolysed in order to obtain easily degradable carbon, which subsequently is used for the biogas production.

The question is consequently if a more compact waste water treatment will lead to an increase in the biogas production, as the extraction efficiency of organic matter increases.

1.2 Aim

The aim of this project has been to investigate the methane production potential of sludge from compact waste water treatment in comparison to conventional waste water treatment. A complementary study of the effect on the methane potential when using biological hydrolysis as a pre-treatment method was also to be done.

Additionally, a simplified mass balance of the energy potential in the different side streams was to be put up in order to provide an overview of the differences between the two treatment concepts and what the compact waste water treatment concept might imply when used in full scale.

1.3 Delimitations

The focus of this study was on the sludge produced by the drum filter and the retentate from the microfiltration membrane, as this was where the major part of the organic matter was retained. Due to the high content of water in the retentate from the biomimetic membrane in relation to its low content of organic matter and substantial need of pre-treatment before being led to an anaerobic digester, it was not hydrolysed or anaerobically digested in this project.

2 Literature study

2.1 Conventional waste water treatment

Conventional waste water treatment usually consists of a combination of mechanical, biological and chemical treatment processes with the purpose of removing suspended solids, organic matter and nutrients. A categorisation of these processes into primary, secondary and tertiary or advanced treatment is generally made where the waste water initially is treated mechanically (primary treatment) after which follows some sort of biological treatment (secondary treatment) and more advanced treatment methods including e.g. chemical precipitation that can be combined with the preceding treatment steps.

Waste water treatment begins in general with various mechanical treatment steps including screens, grit removal and primary clarifiers, in order to remove e.g. rags and other large debris and prevent it from causing problems, such as clogging of pumps and abrasion on equipment, later in the process (Naturvårdsverket, 2009). After the coarsest material has been removed by the screens, heavy particles transported by the waste water are allowed to settle in sand traps where they can be removed and disposed of. The sand traps are often aerated in order to prevent lighter, suspended organic matter from settling before the primary clarifiers where it is removed as primary sludge.

When the waste water has passed through the initial mechanical treatment steps a substantial amount of organic matter still remains as does most of the nutrients and heavy metals. The subsequent treatment steps therefore normally comprise biological processes where dissolved biodegradable organic material is removed by aerobic microorganisms. The different processes utilized differ mostly by means of how the oxygen is supplied to the microorganisms e.g. by a natural air flow within trickling filters, which are filled with a support media upon which the microorganisms can grow, or in the activated sludge process where aerators are used not only to supply the microorganisms with oxygen but also to mix the suspension of waste water and microorganisms (mixed liquor) (Pescod, 1992).

Aeration is necessary in order to supply the microorganisms with oxygen for the metabolism, at the same time as it is one of the most energy consuming processes at a waste water treatment plant whose efficiency is sought to be improved (Tunberg, Sundin and Carlsson, 2009). In, as an example, an activated sludge process the microorganisms form flocs (aggregations of suspended particles) that are then separated in the following sedimentation tank leading to approximately 90% of the organic matter being removed as well as 20% of the nitrogen (Naturvårdsverket, 2009).

As a result of the biological processes only removing about 20% of the nitrogen further nitrogen removal may be required, which can be done biologically as well. However, due to it being a rather complex process biological nitrogen removal is in Sweden mostly found at larger plants dimensioned for over 10 000 population equivalents (Naturvårdsverket, 2009).

Biological nitrogen removal generally consists of several basins or zones within a basin where some are aerated in order to generate different conditions (anaerobic, aerobic and anoxic), which in turn will favour different microorganisms for the nitrification and denitrification processes. By incorporating a biological nitrogen removal step approximately 50-75% of the nitrogen can be removed (Naturvårdsverket, 2009).

Further, chemical treatment is regularly utilized for the removal of phosphorus. By adding chemicals consisting of iron or aluminium, the phosphorus can precipitate during flocculation and form large flocs which are then removed by the following sedimentation step resulting in a phosphorus removal efficiency of roughly 90% (Naturvårdsverket, 2009).

Lastly, waste water treatment plants with more stringent demands on the effluent might incorporate a filtration step where particles that have not been removed previously in the treatment process are separated and removed. A final disinfection step might also be required yet it is not very common in Sweden.

2.2 Sludge treatment

2.2.1 Types of sludge

Sludge resulting from waste water treatment is composed of solids (particles and cells) suspended in a liquid consisting of water and various dissolved substances, both organic and inorganic e.g. carbohydrates and ammonium as well as microorganisms.

A wide usage of notations exists for the sludge separated from each treatment step at a waste water treatment plant. Generally the notation depicts the origin of the sludge, whose composition might differ depending on where in the process it was withdrawn as the content of i.a. organic matter and microorganisms changes.

Primary sludge: Separated by the mechanical treatment, consisting of organic and inorganic particulates with a high content of fat and energy. The primary sludge is more inhomogeneous and compact than the following sludge as it contains coarse particles transported by the waste water such as food particles, fat, bacteria, cellulose fibres from toilet paper and so forth (Davidsson *et al.*, 2008).

Secondary sludge: Also known as *biological surplus sludge* as it comes from the biological treatment, containing microorganisms as well as particulates not degraded in the biological process (Kirk-Othmer Encyclopedia, 2006; Kemira Kemwater, 2003). If an activated sludge process is used it can also be denoted *waste activated sludge* (WAS). The secondary sludge can be further divided into *excess sludge,* the sludge removed from the biological process, and *return sludge,* the part of the sludge recirculated to the biological process with the purpose of avoiding a flush-out of, for the process necessary, microorganisms.

The secondary sludge is more homogenous and less dense than the primary sludge, containing flocs of relatively even size and composition. The biological flocs are however resistant to degradation as the cell walls prevent the enzymes from accessing the intercellular material and require pre-treatment in order to destroy the cell walls. Furthermore, the particles adhere to each other due to the presence of extracellular polymeric substances (EPS) which constitutes more than 50% of the biosludge (the rest is composed of 10-20% living bacteria and 10-30% of other organic material) making the sludge difficult to process (Davidsson *et al.*, 2008). A possible way of counteracting the formation of this gelatinous layer in sludge would however be to introduce hydrolysis or by adding chemicals (Kemira Kemwater, 2003).

Tertiary sludge: Otherwise known as *chemical sludge*. The chemical sludge is separated from the post-precipitation process if such a treatment step is used and contains mostly chemical precipitates in addition to some heavy metals and other contaminants (Kirk-Othmer Encyclopedia).

Furthermore, a mixture of primary and secondary sludge is occasionally called *mixed sludge* while *digested sludge* denotes the sludge resulting from the anaerobic digesters.

2.2.2 The treatment process

The treatment and handling of sludge is an expensive process at the waste water treatment plant, constituting between 40-60% of the total costs despite it composing only about 1% of the incoming volumes to the plant (Kemira Kemwater, 2003). Therefore, it is of importance that the volumes of sludge are reduced and that the sludge is ensured to be suitable for final disposal with regard to pathogens and pollutants present within.

The sludge separated in the different treatment steps is usually treated jointly in three steps consisting of thickening, stabilisation and dewatering. Since sludge consists of mostly water (approximately 93-98%) a considerable amount of it has to be removed in order for the volume to be reduced (Kemira Kemwater, 2003). The gelatinous structure of sludge however, poses problems when attempting to remove superfluous water. Depending on whether the water is found within cells or in between the particles of the sludge it is more or less difficult to remove. The water distribution within sludge can according to Kemira Kemwater (2003) be categorised as follows: surface bound water, trapped water, capillary water and cellular water, where the latter two are difficult to remove mechanically as the cell walls need to be destroyed first.

Common methods used for thickening of sludge are: gravity e.g. sedimentation, which is used especially for primary sludge and most chemical sludge, or drainage belts, dissolved air flotation (DAF) and centrifuges. Drainage belts and DAF, where the sludge is floating by means of air bubbles and then removed by scrapers, are most suitable for waste activated sludge.

Further, as the untreated sludge is composed of biodegradable compounds, which will remain biologically active until stabilised, anaerobic digestion, where biogas is produced, or equivalent treatment methods will be required (Kemira Kemwater, 2003). Once the sludge has been stabilised, the intercellular material will have been released resulting in the possibility of removing more water during the dewatering step. Polymers or conditioning agents are often added as well, in order to further improve the dewatering characteristics of the sludge. The most common method to dewater sludge is through centrifugation; other methods used are vacuum filters, belt filter presses and sludge drying beds, which are mainly applied to less significant sludge volumes in arid climates.

Once the sludge has been thickened, stabilised and dewatered it is to be disposed of or to be reused, if deemed suitable for the purpose considering the content of heavy metals and other contaminants. If the sludge is to be incorporated into the soil and used as a fertiliser on arable land it has to fulfil the demands of the Revaq certification used in Sweden on low concentrations of e.g. heavy metals as to avoid accumulation of toxic substances in crop used for food production (Svenskt vatten, 2015).

Provided the sludge is further dried, another possible disposal method of sludge is incineration. Incineration might however lead to the emission of particulates, nitrous oxides, metals etc., in what concentrations depends on the incinerator type used as well as its operation (EPA, 1995). Yet another option, which since 2001 is no longer legal in Sweden (SFS 2001:512), is to use sludge as landfill.

2.3 Anaerobic digestion

Biogas is produced when microorganisms degrade organic matter in environments free from oxygen, during the so-called anaerobic digestion process. The anaerobic digestion is composed of a large number of different microorganisms interacting during the degradation process of particulate organic matter (e.g. carbohydrates and protein) into carbon dioxide and methane. The natural process which can be found in rice fields, marshes and the stomachs of ruminants, is in anaerobic digesters used to digest i.a. waste water sludge, food waste and farm crops in order to produce biogas. Raw biogas is a mixture of primarily methane and carbon dioxide but also contains certain amounts of nitrogen gas, ammonia, hydrogen sulphide and steam, which is the reason for why upgrading plants are required when the gas is to be used as a fuel in order to separate the methane from unwanted by-products. Another by-product from the anaerobic digestion is the digestate that can, depending on its characteristics, be used as a fertilizer (Biogasportalen, 2015).

Sludge from the waste water process is according to Jarvis and Schnürer (2009) the largest source of biogas production and even though a large volume of biogas is being produced, not all of the organic material is completely degraded and some remains in the digestate. In Figure 2.1, the different fractions of the total chemical oxygen demand (COD) are shown to illustrate the respective amounts of readily and slowly biodegradable respectively inert COD.

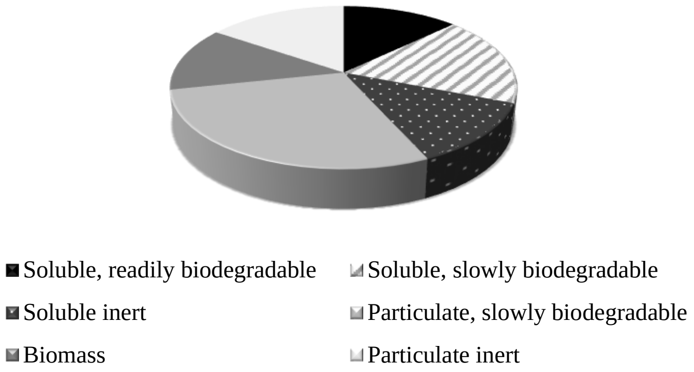

Figure 2.1. The fractionation of total COD in incoming raw waste water to large Scandinavian WWTPs (Barlindhaug and Ødegaard, 2005).

Further, the biogas and methane yields vary for the various substrates depending on the proportion of the different substrate components listed in Table 2.1.

Table 2.1. Biogas and methane yields for the different components of the substrate found in the sludge that is anaerobically digested (Carlsson and Uldal, 2009).

Substrate component	**Biogas yield (Nm^3 biogas/kg VS)**	**Methane yield**
Fat	1.37	70%
Carbohydrates	0.84	50%
Protein	0.64	80%

Anaerobic digestion or anaerobic microbial degradation of organic material comprises several stages, where organic polymers are being converted into smaller units (Davidsson, 2007, Gujner and Zehnder, 1983, Kemira Kemwater, 2003,). The central steps of the process are hydrolysis, acidogenesis, acetogenesis and methanogenesis, as shown in Figure 2.2 below. These four steps are performed by numerous microorganisms living in a syntrophic relation, as cooperation is necessary in order to degrade certain substances.

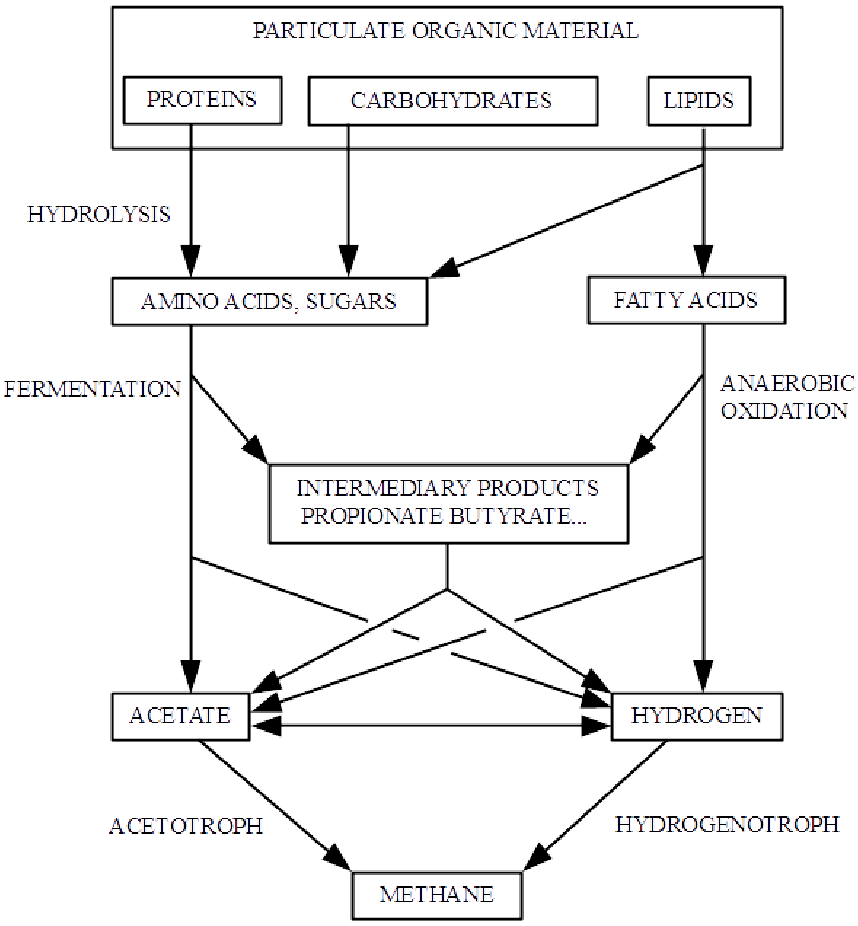

Figure 2.2. Pathways of the anaerobic digestion process (adapted from Gujer and Zehnder, 1983, with permission from IWA Publishing).

Hydrolysis

Hydrolysis is the process when larger particles, polymers are being decomposed into smaller components, monomers. The word describing this process originates from the Greek words *hydro*, meaning ‘water’ and *lysis*; ‘splitting’, implying how chemical bonds in long chains of molecules are broken through the addition of water molecules (Persson *et al.*, 2010). The uptake of and reaction with water is accelerated by the microorganisms’ production of extracellular enzymes e.g. cellulase, protease and lipase participating in decomposing i.a. carbohydrates into sugar, proteins into amino acids and fat into glycerol and long fatty acids respectively (Persson *et al.*, 2010).

The reaction speed does not only depend on the concentration of microorganisms but depends also on the amount of substrate available as well as the prevailing conditions regarding the pH and temperature. The hydrolysis is initiated once bacteria attach themselves to the surfaces of the particles and excrete enzymes decomposing the polymers whereupon the monomers made available are utilized in the formation of new bacteria (Persson *et al.*, 2010).

An important aspect with regard to the degradation process is the size of the particles as larger particles entail a smaller surface area onto which the enzymes can adhere, thereby decelerating the process (Davidsson *et al.*, 2008).

Acidogenesis

The process following the hydrolysis is called acidogenesis, where the products generated in the previous step are further fermented into short-chain volatile fatty acids such as formic, acetic, propionic, butyric, valeric and caproic acid including their isomers and a small amount of carbon dioxide and hydrogen gas (Persson *et al.*, 2010; Hey, 2013). The distribution of volatile fatty acids produced depends on i.a. the process conditions, the substrate available and the metabolic pathways of the bacteria.

The acidogenesis is considered to be the fastest and one of the most energy-generating steps in anaerobic digestion due to the fast-growing bacteria (Persson *et al.*, 2010). The production of fatty acids however consumes the alkalinity and lowers the pH, which in case of accumulation of VFAs in the reactors might inhibit the microorganisms involved in the following steps.

Acetogenesis

In the following step acetogenesis the acetogens, bacteria with a lower growth rate than the acidogens involved in the acidogenesis, further degrade the VFAs and long-chain fatty acids into acetate, hydrogen and carbon dioxide (Persson *et al.*, 2010; Hey, 2013). As it is a sensitive process that is easily inhibited by high concentrations of hydrogen, acetate etc. as well as low pH, the syntrophic relation to the methanogenic bacteria is of great importance for the continuous process as they contribute to maintaining a low partial pressure of hydrogen.

Methanogenesis

The final methane producing step can be divided into two different pathways; the acetoclastic and the hydroclastic methanogenesis, depending on the substrate used by the different types of methanogenic bacteria (Persson *et al.*, 2010). The majority of the bacteria belong to the former group utilizing acetate for the production of methane whereas hydroclastic bacteria use hydrogen and carbon dioxide. Further, the presence of ammonia and variations in pH might inhibit the acetoclastic bacteria, which is why a high content of proteins in the process may act inhibiting on the methane production.

2.4 Hydrolysis as pre-treatment

As a result of increasingly stringent demands on the waste water treatment the amount of sludge produced increases at the same time as the final deposition of the sludge poses a problem owing to discussions regarding the content of heavy metals and pathogens in the sludge.

A possible solution would be to increase the efficacy of the anaerobic digesters, which in turn will lead to a reduction in the amount of sludge. At present, the sludge supplied to the anaerobic digesters is only to some extent converted into methane due to approximately half of the sludge generated from waste water treatment consisting of stable biological flocs that are difficult to degrade (Persson *et al.*, 2010). Further, as the sludge is only partially digested and biodegradable material remains there is a risk of methane leaking from the processed sludge.

The anaerobic digestion process is limited by the hydrolysis step, which in part determines the necessary retention time in the anaerobic digesters. By introducing sludge hydrolysis as a pre-treatment method an increased degradation of the sludge in conjunction with an increase in the biogas production will be obtained according to Davidsson *et al.* (2008). Incorporating sludge hydrolysis in the sludge treatment process also adds to an improvement in the operation of the anaerobic digesters e.g. by shortening the required retention time, as well as an improvement of the possibility to efficiently dewater the digested sludge.

By separating the hydrolysis step from the remaining process the retention time can be regulated to diminish the risk of a direct flow through the digesters. The effect of the hydrolysis however, depends largely on the characteristics of the substrate (Persson *et al.*, 2010). As a consequence of the different microorganisms involved in the anaerobic digestion and their varying conceptions of what the optimal conditions are for growth, separating the process into different steps enables for a customisation of the conditions with regard to nutrients, pH etc.

Another possible field of application for sludge hydrolysis worth mentioning is the production of a carbon source to be used in the biological treatment for nutrient removal, which can diminish the dependence of the WWTP on externally produced carbon (Hey, 2013).

The main purpose of the sludge hydrolysis is to break down the cell-walls and disintegrate large organic compounds, which can be done by a number of different methods, such as mechanical, chemical, thermal and biological including several combinations. Most methods however, require an additional input of energy, chemicals and capital, rendering an optimisation of the microbiological steps increasingly interesting (Persson *et al.*, 2010).

During the hydrolysis process a large amount of soluble COD is produced. Consequently, in order to evaluate the efficiency of a hydrolysis process, the COD yield can be analysed. The fractionation of COD in waste water (see Figure 2.1 previously) is of importance and a high yield of filtered COD generally indicates a functioning hydrolysis process with favourable conditions for further applications (Barlindhaug and Ødegaard, 1996). The soluble COD of the hydrolysate originating from the biological sludge hydrolysis consists mostly of VFA, which constitutes roughly 60-80%, out of which 60-80% is acetate (Henze *et al.*, 2002).

The sludge obtained from the drum filter in the pilot has similar characteristics to primary sludge as the organic matter has not yet been degraded which is generally the case for waste activated sludge, making the sludge obtained especially interesting for both hydrolysis as well as for production of methane using anaerobic digestion. Further, the maximum COD yield (the soluble COD in relation to the total COD) differs for primary and activated sludge, where the former shows a yield of 10-20% whereas the latter is slightly lower with only 2-6% (Henze *et al.*, 2002).

Mechanical methods

By using e.g. garbage disposers, centrifuges, pressure homogenization and ultra sound, larger particles and cell-walls can be cut into smaller pieces in order for the content of the cells to be exposed to the enzymes, providing them with a large surface on which to attach (Davidsson *et al.*, 2008).

Thermal hydrolysis

Thermal hydrolysis e.g. by using electrical pulses conduces to destroying cell-walls, which also can be done chemically by adding acids or bases to dissolve the sludge.

Biological hydrolysis

Biological hydrolysis can be done in several ways either by utilizing the already existing biological decomposition, by facilitating the conditions for the microorganisms or by addition of bacteria or enzymes. The effect from adding enzymes however might vary as a specific enzyme is required for the degradation of each compound (Davidsson *et al.*, 2008).

A cost-effective biological hydrolysis method is anaerobic hydrolysis, where the process is divided into two separate steps; firstly, a separate tank in which the hydrolysis takes place with a short sludge retention time in order to prevent a premature production of methane, secondly, the tank used for production of methane (Persson *et al.*, 2010).

By utilising anaerobic biological hydrolysis, less chemicals and a reduced energy input will be needed. Further, several advantages are associated with the incorporation of a biological pre-hydrolysis step in the anaerobic digestion, according to Fox and Pohland (1994). It will, as an example, lead to an increase in the reaction kinetics and overall efficiency of the process as a result of there being less competition between the acidogenic bacteria and the bacteria involved in the methanogenic step. Also, the contact surface between the biomass and the substrate will increase as well as the concentration of enzymes. Moreover, a phase separation will result in a more stable process as the flow rate is evened out in the hydrolysis step and it will be easier to control and maintain an optimal value for the pH in the methanogenic step. Some other advantages are: reduced problems with foam in the anaerobic digesters in addition to an increased amount of pathogens that will be killed off (Persson *et al.*, 2010).

However, possible disadvantages might be associated with a phase separation such as the process being more difficult to construct and operate, the syntrophic relation between the bacteria might be rendered more difficult in addition to a lack of experience regarding the process and how the degradation of different substrates is affected by a separation of the process steps (Fox and Pohland, 1994).

3 Case study

3.1 Källby WWTP

Källby WWTP, built in 1933, is situated in southern Lund and dimensioned for 120 000 population equivalents (VA SYD, 2012). The process utilized is based on mechanical, biological and chemical treatment, beginning with 6 mm perforated screens followed by aerated sand traps and pre-sedimentation basins (see Figure 3.1). After the primary sludge has been separated, the water is led to the biological treatment, i.e. the activated sludge treatment step for removal of nitrogen, organic matter and to some extent phosphorus. In the activated sludge process pre-denitrification is utilized, enabled by the basins being divided into different zones providing aerobic, anoxic and anaerobic conditions alternately. The flocs formed in the activated sludge step are then separated in the following sedimentation basins as sludge. The sludge is to a large extent recirculated to the activated sludge step as return sludge (to maintain a constant concentration of suspended solids in the activated sludge process) whereas the rest, the so-called excess sludge, is taken to the sludge line for further treatment.

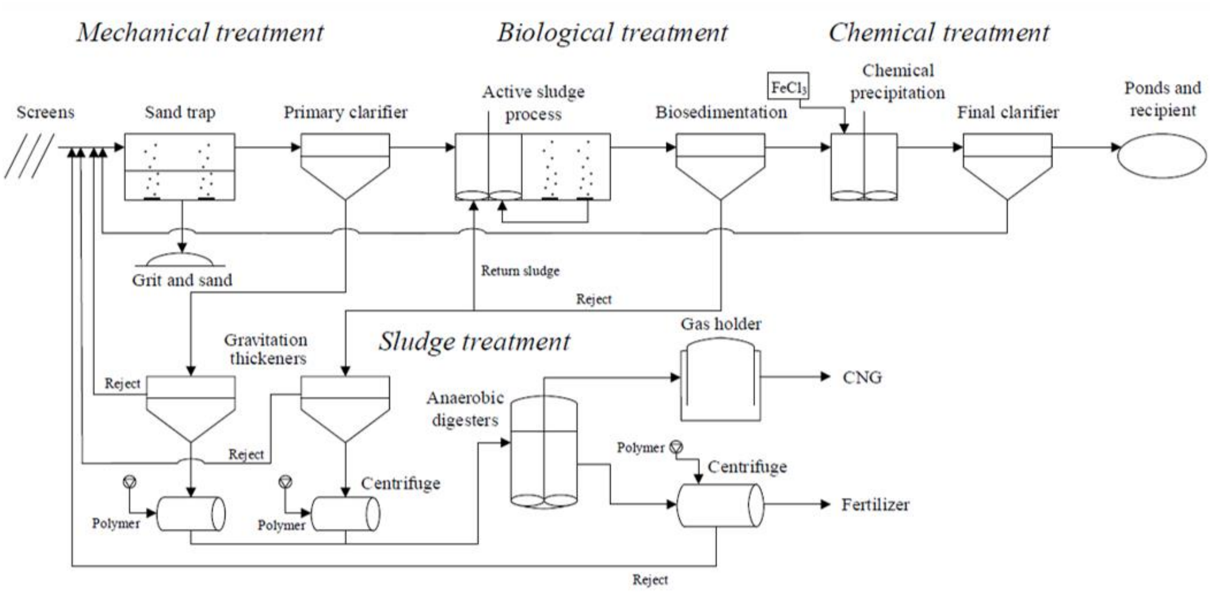

Figure 3.1. Process scheme of the Källby WWTP (adapted with permission from VA SYD).

After the activated sludge step follows phosphorus removal in the form of post-precipitation by means of iron chloride ($FeCl_3$) and additional sedimentation basins for separation of the chemical flocs formed. Eventually, the effluent is led into polishing ponds connected in series where the remaining nitrogen and phosphorus is eliminated by microorganisms before the treated water is discharged into the recipient Höje River (VA SYD, 2013).

The incoming waste water type is of medium strength with a total incoming flow of 11 347 000m^3 waste water/year and concentrations as specified in Table 3.1.

Table 3.1. Average incoming and outgoing concentrations in mg/l in relation to the outlet demands on commonly measured parameters in the waste water (reference Hey, 2015).

Parameter	Average incoming concentration (mg/l)	Average outgoing concentration (mg/l)	Outlet demands (mg/l)
BOD_7	203	< 3	10
P-tot	5.6	0.3	0.3
N-tot	40	8	10

Meanwhile, the chemical sludge from the final clarifiers is recirculated back to the pre-sedimentation basins where it is withdrawn with the primary sludge and pumped to a thickener as mixed sludge. Once the water content in the sludge has been partially reduced in the gravitation thickeners, the mixed sludge is mechanically thickened by a drum thickener (centrifuge) in combination with the addition of polymer. The reject water from the sludge treatment is led back to the inlet of the plant while the thickened sludge is further processed in the two anaerobic digesters operating under mesophilic conditions at 37ºC for the purpose of reducing the final sludge volumes and producing biogas, which is to be stored in a gasholder and upgraded to CNG (VA SYD, 2012). After having spent between 20 to 30 days in the anaerobic digesters, the sludge is further dewatered in a centrifuge for an increase in dry content from 5% to 25% before it is deposited while waiting to be e.g. spread on arable land (as it is certified according to Revaq) (VA SYD, 2012).

3.2 Compact waste water treatment

Due to the conventional waste water treatment process being relatively energy-consuming, with regard to the aeration of the biological treatment, as well as resource demanding considering the need for chemicals, such as flocculants and possibly an additional need of carbon for the biological treatment, it is of interest to investigate alternative methods that might be more efficient. Furthermore, in places where the availability of land is scarce, new waste water treatment solutions with a smaller footprint will be necessary as an expansion of the conventional treatment process is not be possible.

The alternative waste water treatment method studied in this project consists of a micro-sieve followed by two different membrane types. The influent waste water is treated initially by mechanical means, passing through the screens and subsequent sand trap at Källby WWTP. The following primary clarifier however is thought to be replaced by a drum filter (DF) and a microfiltration (MF) membrane. The permeate (i.e. what passes through the membrane) can then be further treated using a biomimetic membrane for forward osmosis (FO), which in theory only allows for water molecules to pass and is thought to replace the activated sludge tep. Moreover, the separation efficiency of phosphorus can be enhanced by applying chemicals to the mainstream before it enters the compact treatment process. Chemically enhanced primary treatment with various coagulants (aluminium or iron) and flocculants (cationic and anionic polymers) at different concentrations is tested before the drum filter (Hey, 2015). Various configurations including and/or excluding the different treatment steps are also possible in order to evaluate which alternatives might prove to be the most efficient.

The set-up of the pilot, including the preceding mechanical treatment steps, is illustrated in Figure 3.2, where the point for addition of precipitation chemicals is accentuated in grey.

Additional photos of the various components of the pilot plant can be found in Figures 3.3 and 3.4.

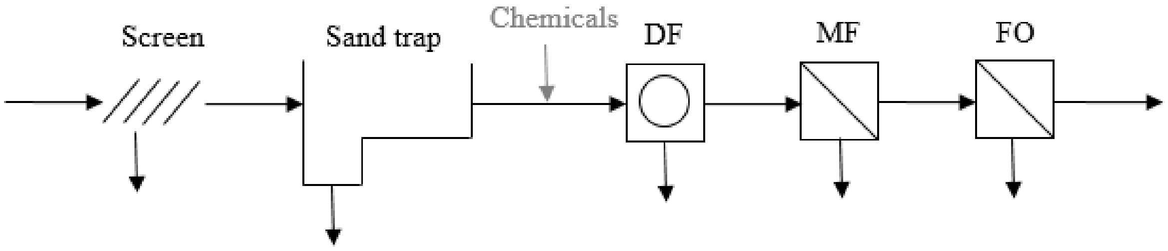

Figure 3.2. Set-up of the pilot for compact waste water treatment (as explained by Hey, 2015).

Figure 3.3. Photo of the pilot plant taken from the outside (left) and of the Hydrotech drum filter (right). Photos by: Hey, 2014.

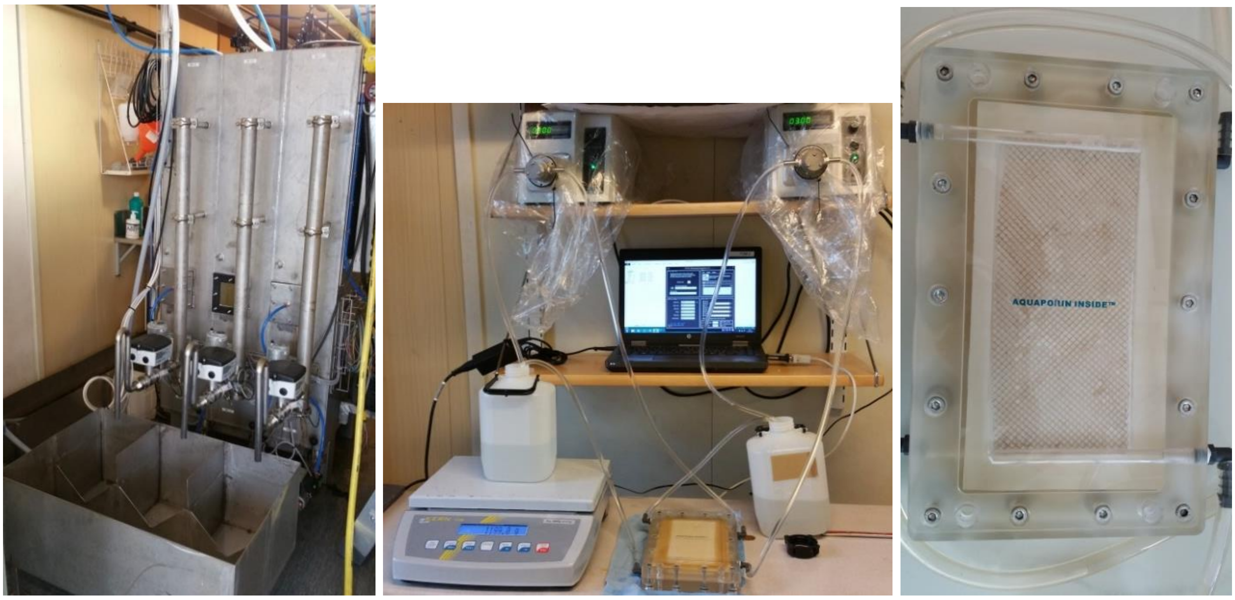

Figure 3.4. Alfa Laval MF membranes (left), biomimetic membrane FO filtration set-up (center) and the Aquaporin membrane itself (right) Photos by: Hey, 2015.

Some of the advantages of using compact waste water treatment methods are according to Paulsson (2014), Remy *et al*. (2014), Jenicek *et al*. (2013), Judd (2011) and Hai *et al*. (2014):

- No oxygen is needed leading to energy being saved, which instead can be used for the operation of the membranes as it can be costly to operate a vacuum or pressure-driven process (filtrate is “pushed”) or vacuum driven process (filtrate being “pulled”) in order to maintain the transmembrane pressure
- Less chemicals might be required and resource savings can be made
- A process consisting of membranes could be less sensitive to disturbances than a biological treatment process.
- Smaller footprint than conventional treatment methods
- High quality effluent
- The risk of causing emissions of nitrogen gas is reduced as no denitrification is taking place
- Allows for the nutrients within the separated sludge to be extracted
- Generates sludge of high quality similar to primary sludge that is easier to degrade biologically than e.g. waste activated sludge, which contains biological flocs and substances difficult to degrade

While some of the main disadvantages related to membrane processes might be:

- Fouling
- Maintenance
- Operation and capital costs, associated with e.g. the transmembrane pressure when higher pressures than the one used for the MF membrane in the pilot plant at Källby WWTP are required
- The concept has not yet been evaluated in full-scale

Furthermore, some of the possible variations of the process steps involved in this pilot plant have been illustrated in Figure 3.5. These variations include the possibility of applying chemical precipitation after the raw waste water has passed through the screens and subsequent sand trap.

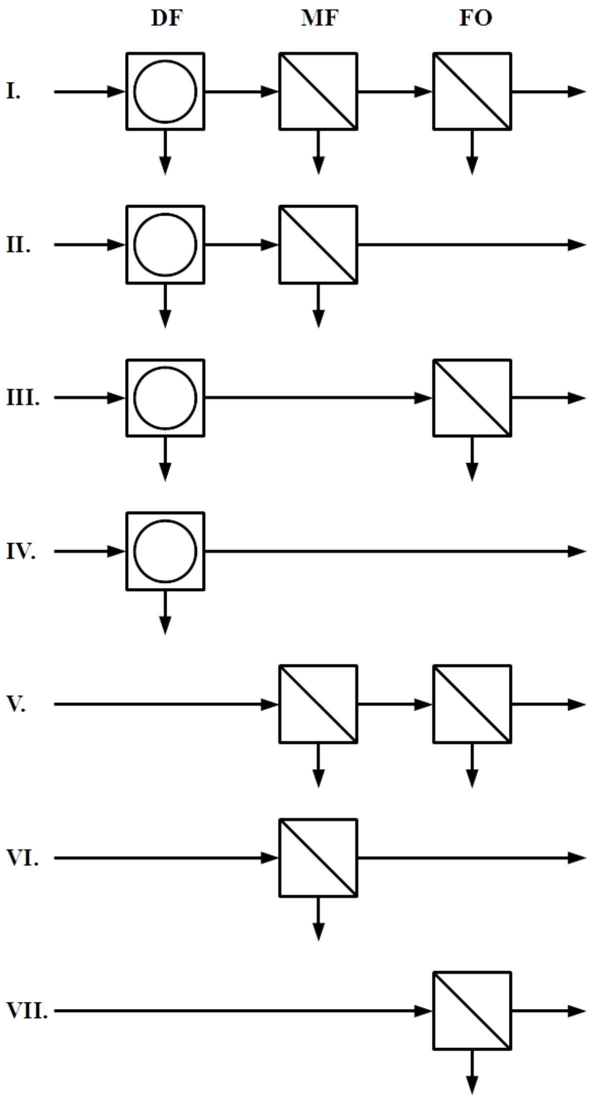

Figure 3.5. Summary of possible pilot configurations to be tested (as explained by Hey, 2015). DF = drum filter, MF = microfiltration membrane and FO = forward osmosis membrane where possible addition of precipitation chemicals can be done initially and with influent waste water having previously passed through the screens and sand trap in order to remove large material and coarse solids.

3.2.1 Drum filter

The drum filter supplied by Hydrotech AB functions as a microsieve with the purpose of reducing the load of suspended solids to the subsequent membrane and forward osmosis units.

The process begins by the influent entering the drum and passing through the filter, which prevents solids of a certain size from proceeding to the next treatment step. The filter consists of a cloth made out of polyester with a pore size of 100 µm (Hey *et al.*, 2014). A cloth with smaller pores (40 µm) has also been tested, resulting in a diminution of the capacity (Väänänen, 2015). The capacity of a drum filter is usually defined as the volume of waste water in cubic meters filtered per square meter filter and hour, also denoted the typical overflow rate, which for this set-up is 1.7 $m^3/m^2 \cdot h$ (Hey *et al.*, 2014). Further, the efficiency of the filter is evaluated by comparing the influent quality to that of the effluent as well as the doses of coagulants and flocculants used.

As the filter starts to clog with particles the water level within the drum rises and the drum rotates automatically enabling for the clogged part to be backwashed (Hydrotech, 2011). The solids removed from the filter cloth, i.e. the sludge formed, is then collected in a trough and may be further processed in the sludge treatment.

3.2.2 Microfiltration membrane

Background

The purpose of a membrane process is to separate physical or chemical components from a liquid by means of a membrane. The components of the feed that pass through the membrane are usually denoted permeate, meanwhile what is retained is called retentate, see Figure 3.6.

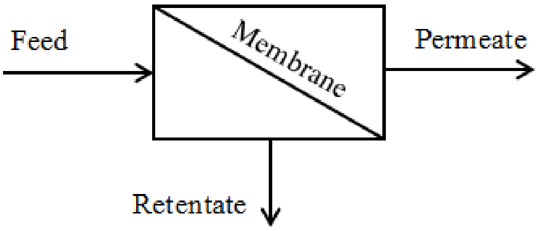

Figure 3.6. Definitions used in membrane technology.

A membrane process can be either pressure-driven or extractive, where the four most common separation processes microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) fall under the first category whereas forward osmosis is based on diffusion and falls under the second category (Judd, 2011). The pressure-driven membrane processes can be further divided into two categories, depending on whether they only retain suspended and colloidal particles as does MF and UF or if they are able to remove particles that are dissolved in the liquid as well, like NF and RO. The mechanism for micro- and ultrafiltration is therefore said to be based on size-exclusion according to Hai *et al.* (2014). Further, the membranes used for microfiltration constitute a physical barrier retaining not only particulates but also bacteria and protozoan cysts. Viruses however, are not retained by using only a MF membrane (Hai *et al.*, 2014).

Further, several aspects need to be taken into consideration when choosing a membrane separation process such as:

- Material (pore size, hydrophobicity, etc.)
- Permeability
- Flux
- Transmembrane pressure
- Required pre-treatment
- Fouling propensity
- Flow configuration

- Packing density
- Footprint
- Backflush possibilities
- Cleaning (physical, chemical)
- Replacement
- Costs

One of the main aspects, which needs to be considered, is fouling as it constitutes one of the principal restrictions to the process. As the filtration process proceeds, retained constituents accumulate on the membrane surface and cause fouling, which reduces the flux through the membrane (Judd, 2011).

By introducing membrane technology however, all bacteria and some of the viruses are removed according to Hai *et al.*, (2014), which implies that the required downstream disinfection and costs associated with the plant construction, operation and maintenance can be reduced.

Direct membrane filtration (DMF), where waste water is filtrated directly on a membrane, is an attractive process as it allows for water of high-quality to be retrieved in one step while valuable organics and nutrients remain in the water. Some of the main advantages of such a process apart from the high-quality permeate is the simplicity of the design and the automated maintenance (Ravazzini *et al.*, 2005). Further, direct membrane filtration also allows for recovery of organic matter, which is concentrated in the sludge and can be used for energy production.

However, filtration of raw waste water tends to lead to a strong flux decline and cause fouling problems. Yet, by utilising physico-chemical pre-treatment, such as coagulation, micro-sieving or sedimentation, prior to the membrane, the filtration rate and separation may increase. In the study made by Ravazzini *et al.* (2005), primary clarifier effluent and pre-filtered (0.56 mm sieve) raw sewage were used on UF membranes at a constant transmembrane pressure of 0.3, 0.5 respectively 1 bar in order to compare the filterability and fouling. It was found that the direct ultrafiltration experiments resulted in the same fouling mechanism but that the permeate production increased when using the primary clarifier effluent and that a TMP of 0.3 bar was to prefer during continuous operation.

Pilot set-up

The pilot consists of a stainless-steel tank with three compartments, in which five PVDF flat-sheet membranes (MFP2 from Alfa Laval in Denmark), with a nominal diameter of 0.2µm, are mounted together resulting in a total effective area of 1.024 m^2. The flux through the membranes is maintained by applying a transmembrane pressure (TMP) of 0.03 bar, which corresponds to the hydrostatic pressure over the membrane. Further, in order to reduce fouling of the membrane, air is used for scouring the membrane surface (Hey *et al.*, 2014).

3.2.3 Forward osmosis

Background

The cells found in living organisms are protected from the outer world by a cell membrane consisting of a lipid bilayer with integrated proteins. The cell membrane serves as a barrier by regulating the passage of different substances such as water molecules and amino acids (Nationalencyklopedin, 2015). Some small particles and ions are able to pass through the membrane by ways of diffusion while larger charged particles can only do so via transmembrane proteins.

Proteins with the purpose of transporting water are named aquaporins (see Figure 3.7), which can be found in nearly all organisms e.g. in the human kidneys concentrating the urine (Hill and Taylor, 2012). These aquaporins can nowadays also be found in so-called biomimetic membranes; membranes where designs found in nature are applied to man-made technology in order to solve engineering problems etc. (Mueller, 2008). The biomimetic membranes with integrated aquaporins enable a passive gradient driven diffusion of water which may lead to a high permeability in comparison to that of traditional membranes (Madsen *et al.*, 2014). The diffusion process of water molecules is called osmosis, which is the net movement of water across a semi-permeable membrane driven by a difference in osmotic pressure originating from different concentrations of solute molecules and/or ions on each side of the membrane (Cath, Childress and Elimelech, 2006).

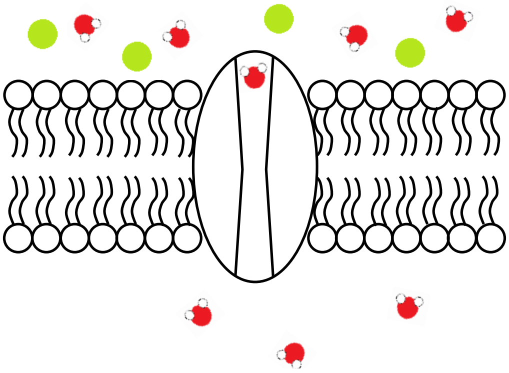

Figure 3.7. Illustrating a cell membrane consisting of a lipid bilayer with an integrated aquaporin transporting water molecules from a liquid containing pollutants (adapted from OpenStax College, 2011).

In order to distinguish the naturally occurring osmosis from the process called reverse osmosis (RO) used in the field of water treatment; it is generally referred to as forward osmosis (FO). In reverse osmosis, a hydraulic pressure is applied to the opposite side of that where the feed solution is kept in order to exceed the osmotic pressure, consequently reversing the naturally occurring process (Cath, Childress and Elimelech, 2006).

When using forward osmosis in waste water treatment an appropriate draw solution is necessary for the process to function. The draw solution used should be selected as to have a higher osmotic pressure than that of the feed solution as well as a low tendency of reverse leakage through the membrane (Cath, Childress and Elimelech, 2006; Lutchmiah *et al.*, 2014). Commonly used draw solutions for forward osmosis are e.g. sodium chloride (NaCl) and other compounds containing e.g. chloride or sugars. During operation, the concentration of the feed stream will increase as the concentrated draw solution is diluted. In order to extract the clean water from the draw solution a suitable recovery system such as reverse osmosis, thermal recovery or membrane distillation may be included as the final treatment step if the draw solution itself is of interest (Lutchmiah *et al.*, 2014). By using a suitable recovery system the draw solution can then be reconcentrated and reused. In the pilot plant however, the draw solution used is thought to originate from Öresund, which does not have to be recovered in a final treatment step.

Pilot set-up

The membrane (Aquaporin INSIDE™) used for the pilot is a biomimetic aquaporin membrane for forward osmosis with an active area of 0.014 m^2 supplied by Aquaporin A/S, Denmark (Hey, 2015).

3.3 Energy aspects related to waste water treatment processes

Waste water is a source of energy due to its high content of organic matter, which can be quantified as the chemical oxygen demand. The minimum internal chemical energy of this organic matter is, according to Heindrich *et al.* (2011), approximately 13-14 kJ/g COD i.e. roughly 4 kWh/kg COD. Remy *et al.* (2014) concludes thus, under the assumption that municipal raw sewage carries 120 g COD per PE and day, that the theoretical annual chemical energy potential of raw sewage is 175 kWh/PE. When putting this theoretical value in relation to the average power consumption of a WWTP (for over 10 000 PE) utilizing activated sludge processes with an annual energy consumption of 32-34 kWh/PE, it can be seen that the development of energy-positive WWTPs, with an energy recovery superior to the consumption, could be a possibility (Remy *et al.*, 2014). This view of self-sufficient WWTPs, without any need of external organic substrates, has previously been supported by Jenicek *et al.* (2013) as well.

At present, when using conventional waste water treatment processes, a substantial energy loss occurs as 40% of the total incoming energy in the raw waste water is lost by oxidation in the activated sludge step. Energy is further lost with the effluent discharge and the digested sludge, leading to only approximately 25% of the total energy in the raw waste water being transformed into biogas (Jenicek *et al.*, 2013). Consequently, there is an increasing interest in making use of the energy potential in the raw waste water and to recover it as sludge, with focus on anaerobic digestion (Paulsrud *et al.*, 2014; Jenicek *et al.*, 2012; 2013)

An optimisation of the anaerobic digestion technology as well as a development of the total energy consumption needed to treat waste water is however required in order to considerably increase the part of COD that is converted into biogas (Jenicek *et al.*, 2013). One of the essential factors which limits the part of COD converted to biogas is the efficiency of the primary clarifier, ergo the primary treatment step and its efficiency regarding the extraction of organic matter from the waste water into the sludge.

Several different possible solutions exist in order to render the primary treatment step more efficient. Paulsrud *et al.* (2014) performed a study on the use of fine mesh sieves as primary treatment, entailing advantages such as a small footprint and the possibility to use existing sedimentation tanks to increase the capacity of the biological step. Further, the effects of the removal of organic matter on the downstream biological phosphorus and nitrogen removal processes were studied as readily biodegradable organic material is needed in the subsequent treatment processes. Paulsrud *et al.* (2014) concluded that the effect on the denitrification rate of removing particulate COD was only minor.

Another innovative concept, similar to the one tested in this study, consisting of a drum microsieve has been tested previously in pilot scale by Remy *et al.*, (2014). The purpose of the study was to achieve maximum extraction of organic matter from waste water into sludge by use of chemically enhanced pre-treatment i.e. coagulation and flocculation, followed by microsieving and a post-treatment step, e.g. biofiltration. By using a 100 µm mesh and doses of 15-20 mg/l of aluminium coagulant in addition to 5-7 mg/l of cationic polymer, a total extraction of approximately 70-80% of the total chemical oxygen demand was obtained. Through anaerobic digestion of the produced sludge, a biogas yield of 600 NL/kg organic dry matter input (oDM_{in}) was achieved compared to 430 NL/kg oDM_{in} by anaerobic digestion of mixed sludge from a conventional activated sludge process. By putting up an energy balance for a waste water treatment plant dimensioned for 100 000 PE, the concept was shown to be energy-positive in comparison to conventional treatment processes. Further, the operating costs for the energy-positive concept related to chemicals and electricity turned out to be comparable with those for conventional waste water treatment.

As a result of only 20% of the total nitrogen being particulate and thus removed by the drum filter, additional treatment may be required. If conventional biological treatment methods are used, the denitrification potential of the organic matter in the effluent from the microsieve is important to determine. In the study performed by Remy *et al.*, (2014), the denitrification potential was evaluated by measuring the content of volatile fatty acids (VFA) as these are considered to be an easily accessible source of carbon for the denitrification. Additional kinetic denitrification tests further affirmed the denitrification potential of the drum filter effluent. It was noted that a content of 120 g/l of acetate-equivalents as VFA was found in the effluent of the microsieve, which consequently would be available for the following denitrification.

3.4 Mass balance for Källby WWTP

A total energy balance taking into consideration indirect factors, which conduce to the energy consumption e.g. the chemicals utilized, the transportation needed etc. can serve as an indication of what is needed for a WWTP in order to become energy self-sufficient.

However, in this study the focus lies on comparing the methane potential of the sludge from the compact waste water treatment pilot plant to the methane production achieved at present by the conventional waste water treatment process at Källby WWTP. Consequently, what is of interest in order to put up a simplified mass balance, incorporating the energy potential of the separated sludge, is firstly the amount of mixed sludge going into the anaerobic digesters and the resulting methane production, to serve as a reference. Secondly, the incoming and outgoing suspended solids concentrations and waste water flow for the drum filter and the microfiltration membrane, in order to estimate the removal efficiencies and the amount of sludge generated accordingly. Lastly, by utilising the methane potentials determined in this study, an estimated methane production can be presented.

4 Method

The project was executed with an initial literature study whereupon hydrolysis and biochemical methane potential tests were carried out in lab scale. The sludge used was collected from the pilot at Källby WWTP. A comparison of the methane production when using non-hydrolysed respectively hydrolysed sludge as well as chemically enhanced pre-treatment was made.

Further, in order to compare the obtained biogas yield from compact waste water treatment to the corresponding yield for conventional waste water treatment a mass balance, evaluating the generated energy potential, was set up once the results from the biochemical methane potential tests were obtained.

Moreover, with consideration to biochemical methane potential tests being time-consuming, several hydrolysis experiments were made in order to investigate whether any parallels could be drawn to the biochemical methane potential tests executed.

The performance of the hydrolysis was to be assessed by a quantification of the solubilisation degree of organic matter and the production of volatile fatty acids while the methane potential was to be evaluated by means of biochemical methane potential tests.

Another important aspect, which needs to be taken into consideration, is one of the main limitations to this study; the dependence on the equipment used. Due to the varying deliveries of sludge and due to it not always being possible to obtain sludge samples with the preferred characteristics, it was not possible at all time to take samples on a regular basis during the hydrolysis experiments e.g. during weekends and holidays.

4.1 Sludge characteristics and analyses

The characteristics of the sludge were determined and the following parameters were analysed: total solids (TS), volatile solids (VS), suspended solids (SS), volatile suspended solids (VSS), filtrated chemical oxygen demand (CODfil), total chemical oxygen demand (COD), volatile fatty acids (VFA), sludge volume index (SVI) and pH. See Appendix I for a more detailed description of the common procedures used.

The concentration and distribution of the different solids was analysed in order to determine the amount of organic material available in different forms for the hydrolysis and anaerobic digestion, while the change in particle size throughout the experiments was monitored as the chemical oxygen demand and amount of volatile fatty acids. The concentrations of solids were determined according to Swedish standard (SS 02 81 13 for TS and VS) and instructions described by Svenska vatten- och avloppsverksföreningen (1984).

Further, the sludge volume index was determined with the purpose of evaluating the settling characteristics of the sludge and indirectly its ability to be dewatered and thickened. The retentate obtained from the two membranes in the pilot plant only contained a small fraction of suspended material with small particle sizes, which is the reason for the sludge volume index being of interest.

4.1.1 VFA analyses

The samples were centrifuged in Eppendorf tubes or filtered on 0.45 µm Munktell micro-glass fibre filter papers (depending on what other analyses were made) after which 0.9 ml of the clear phase was pipetted into 2 ml clear glass vials with snap caps together with 0.1 ml phosphoric acid, 10% and stored in a refrigerator, in order to preserve the samples while waiting for the VFA to be measured.

The VFA content was analysed using a gas chromatograph (Agilent, 6850 Series GC System) with a flame ionization detector (FID) and HP-FFAP colon (30 m, 530 µm, and 1 µm). Specifically the volatile fatty acids acetate and propionate were measured, as these constitute the major part of the VFA, are the most easily measured and of interest when it comes to e.g. biological phosphorus (bio-P) treatment processes.

4.2 Biological hydrolysis

The biological hydrolysis was performed in lab scale using Kemira Flocculator 2000 equipment consisting of six beakers with lids, stirrers and a control unit. Sludge was withdrawn and transported from Källby WWTP the same day as the hydrolysis experiments were started.

As the purpose of the sludge hydrolysis was to increase the conversion rate of particulate organic matter into soluble, readily biodegradable matter to be used when determining the effect on the methane potential, the measurements during the hydrolysis experiments were chosen with focus on the rate of hydrolysis, by measuring the change in concentration of COD and VFA.

Firstly, the beakers were filled with 1 000 ml of sludge each, using replicates. Once the beakers were filled with sludge and the flocculation equipment was in place, tubes were connected to each beaker allowing for continuous flushing of the sludge surfaces with nitrogen gas, ensuring anaerobic conditions for the hydrolysis process (see Figures 4.1 to 4.3). The rotation speed of the stirrers was initially set to approximately 130 rpm for an infinite time interval using the control unit. Throughout the experiment, the beakers were kept at room temperature (approximately 20°C). Samples were withdrawn at the start-up from the remaining collected sludge, in order to analyse the content of TS, VS, SS, VSS, COD (total and soluble) and VFA in the sludge as well as the SVI.

Figure 4.1. The six beakers used in hydrolysis experiment 3 (day 2).

Figure 4.2. The six beakers used in hydrolysis experiment 4 at start-up.

Figure 4.3. The six beakers used in hydrolysis experiment 5 at start-up.

In order to prevent evaporation, the holes in the lids of the stirrers were taped over. Some evaporation however did occur, which was compensated for by addition of tap water. The levels of sludge in the beakers were marked using tape, which was moved each time samples were withdrawn.

During the experiment, daily measurements of dissolved substances, pH and temperature were made. The sampling (40-80 ml, depending on what measurements were needed) was done regularly after approximately 24, 48, 72 and 86 hours as well as after 6 days. The analyses performed on the samples taken were measurements of the VFA and the filtered COD in addition to monitoring of the temperature and pH, where the latter can be used as an indication of how the amount of VFA changes. When VFA are produced, the pH might decrease while an increase in pH might indicate that the VFA are being consumed.

By measuring the amount of dissolved organic substances, usually as COD or the total organic carbon (TOC) an estimation of the degree of hydrolysis can be made if compared to the total measured amount of carbon (Davidsson *et al.*, 2008).

After 6-7 days, the experiment was terminated and the final TS, VS, SS and VSS were measured as well as the COD and VFA.

4.3 Determination of methane potentials

When implementing a new substrate in an anaerobic digestion process, it is of importance to evaluate its suitability with regard to the biodegradability and methane potential (Paulsrud, 2014). This is most easily done by determining the maximum methane potential through biochemical methane potential (BMP) tests.

In order to determine the methane potential of the separated streams, the laboratory procedure presented by Hansen *et al.* (2004) was utilized. BMP tests are usually performed by mixing the substrate (the organic material to be examined) with an inoculum, containing the required microorganisms taken from an anaerobic digester in operation. The mixture is placed in bottles, which are flushed with nitrogen gas before start-up in order to ensure anaerobic conditions. The bottles are then incubated at the desired temperature while the methane production is monitored. Once the production of methane has ceased, the accumulated value of methane produced provides the maximum methane potential of the specific substrate. The unit commonly used for presenting the methane potential of a substrate denoting the volume of gas produced from a certain amount of organic material is Nml methane/g VS, where N stands for the normal volume at standard pressure and temperature (Carlsson and Schnürer, 2011).

4.3.1 Experimental set-up: BMP test

Firstly, 2-liter glass bottles were to be filled with degassed inoculum from the mesophilic anaerobic digesters at Källby WWTP, substrate and water to a total of 500 ml for the first BMP test respectively 600 ml for the second (for the values used, see Tables 4.2 and 4.3). Thereafter, the head space was flushed using nitrogen gas in order to ensure anaerobic conditions. Each bottle was then sealed with a thick rubber septum and an aluminium cap. The bottles were incubated at 37ºC during the test period (see Figure 4.4), which lasted for 42 respectively 41 days.

Figure 4.4. Bottles in the incubator.

Sampling was done using a 1 ml glass syringe equipped with a pressure lock and the sample taken was injected directly into the gas chromatograph (Varian 3800, equipped with thermal conductivity detector (TCD, see Figure 4.5), a HAYESEP mesh column with the dimensions: 2.0 m (length) × 1/8” (diameter) × 2.0 mm (film) and methane standards) with standardised pure methane gas used as a reference.

Figure 4.5. Injection into the gas chromatograph using a gas tight syringe.

4.3.2 Preparation

The inoculum was collected four days before each of the experiment start-ups and incubated at 37ºC (the same temperature as in the anaerobic digesters at Källby WWTP) in order to diminish the influence of the gas production resulting from the inoculum itself and degrade any remaining organic material. It is of importance to the process that the temperature during the incubation period is the same as in the anaerobic digesters from where it is taken as it is what the microorganisms are adapted to (Carlsson and Schnürer, 2011). Simultaneously, samples were taken for the determination of the TS and VS content (see Appendix I for the procedure used) that was needed for the calculations regarding the necessary amount of inoculum and substrate to be added.

The amount of inoculum and substrate required for the BMP tests was determined in order to fulfil the demand for a total volume of 500 ml respectively 600 ml of liquid within each bottle. The VS content provided for by the inoculum should constitute 60% whereas the substrate should constitute 40% of the total VS content, which should not exceed 4 g/bottle. If the correct volume was not attained, the remaining volume was filled up with tap water. The volumes of sludge, water etc. used were duly noted and bottles containing only inoculum respectively inoculum and a reference substrate were included in the experiment set-up. The substrate used as a reference was a cellulose mixture (Avicel® PH-101 and Cellulose powder microcrystalline [9004-34-6]). The three bottles containing only water and inoculum were used to estimate the methane production of the inoculum itself whereas the three cellulose samples were used to test the quality of the inoculum.

Moreover, no cellulose was included in the second BMP test since the inoculum was collected from the same anaerobic digester and of good quality judging by the previous BMP test.

4.3.3 Execution

The bottles were weighed individually with the caps off after which they were filled with water and weighed again. Once the bottles had been filled, the inoculum, substrate and water was added and the added amount of each component was noted. Thereafter, the bottles were flushed using nitrogen gas in order to remove the oxygen from the head space and to ensure anaerobic conditions. Further, the rubber septa and caps were screwed on (see Figure 4.6) and notes were made of the time and date of the start-up. As the content of TS and VS decreased during the degassing of the inoculum, additional measurements were performed in connection to the start-up.

Figure 4.6. The bottles after having been sealed.

The methane production in the bottles was measured by withdrawing triplicate samples of 0.2 ml from each bottle through the septum using a gas tight syringe and injecting the samples into the gas chromatograph (Hansen *et al.*, 2004).

In order to avoid overpressure and as a consequence leakage, the bottles were emptied of gas regularly using a needle and the concentrations of methane before and after the emptying were noted. The bottles were emptied once the rubber septa of the bottles were bulging.

The frequency of the measurements was more frequent in the beginning of the experiment (twice a week the first three weeks) when the largest part of the methane production occurred and subsequently less frequent (once a week). Before and after each measurement the air pressure and temperature in the room was noted as well as the time when the measurements begun and were completed. It was imperative to ensure that overpressure did not prevail within the bottle containing the methane gas and several samples were taken before measurements were made on the bottles containing the substrate in order to obtain accurate results. Further, to ensure satisfactory contact between the microorganisms and the substrate, the content was mixed after the sampling and left in an incubator until the next sampling.

Finally, the experiment was terminated once the methane production seemed to have levelled out, which normally takes approximately 35-50 days.

4.3.4 Calculations

The amount of substrate, inoculum respectively water needed in each bottles at the start-up was calculated according to the prerequisites mentioned previously.

In order to determine the amount of methane produced, the volume in the gas phase as well as the content of methane within the 0.2 ml samples was needed. The prevailing conditions were then carried forward to the standard conditions according to the formulas presented below (Lunds Tekniska Högskola, 2008) and the values of the previous measurement withdrawn after each emptying of the bottles.

$$X_{STT} = X_m \cdot \frac{T_{standard} \cdot P_m}{P_{standard} \cdot T_m} \qquad \textit{Eq. 1}$$

X_{STT} = the area under the chromatograph given during analysis using the GC at standard temperature and pressure, corresponding to the measured amount of methane

X_m = the measured value of the area for the methane reference at the time of the measurement

$T_{standard}$ = the standard temperature (273.15 K)

P_m = the pressure in the room at the time of the measurement [hPa]

T_m = the temperature in the room at the time of the measurement [°C] converted into [K]

$P_{standard}$ = the standard pressure (1013.25 hPa)

$$V_m = \frac{X_{STT} \cdot V_h}{X_{100\%}} \qquad \textit{Eq. 2}$$

X_{STT} = the area at standard temperature and pressure

V_m = the volume of methane in a specific bottle

V_h = the volume of the head space in a specific bottle

$X_{100\%}$ = the measured value of the area of a sample containing pure methane

4.4 Tested sludge

4.4.1 Sludge used in the hydrolysis experiments

The types of sludge used for the hydrolysis experiments are presented in Table 4.1 below.

Table 4.1. The duration of and the sludge types used in the hydrolysis experiments as well as the doses of coagulants and flocculants.

Hydrolysis experiment	**Beakers**	**Dates**	**Sludge type**	**Coagulation**	**Flocculation**
1	1 – 3	26/2 – 5/3	DF (100 µm)	5 mg/l (XL100, Al^{3+})	3 mg/l (Hytex 6161, anionic polymer)
2	1 – 3	7/3 – 23/3	DF (100 µm)	–	–
3	1 – 3	23/3 – 30/3	DF (100 µm)	–	–
	4 – 6	23/3 – 30/3	DF (100 µm)	6.5 mg/l (XL100, Al^{3+})	2.5 mg /l (6456, polymer)
4	1 – 2	13/3 – 20/3	MF (0.2 µm)	–	–
	3 – 4	15/3 – 21/3	DF (100 µm)	–	–
			MF (0.2 µm)*		
	5 – 6	15/3 – 21/3	DF (100 µm)	–	–
5	1 – 3	6/5 – 13/5	DF (100 µm)	15 mg/l PAX	2.7 mg/l anionic polymer
	4 – 6	6/5 – 13/5	DF (100 µm)	15 mg/l PAX	2.7 mg/l anionic polymer
			MF (0.2 µm)*		

* also denoted DF-MF retentate as the main stream has passed through the drum filter before sludge is withdrawn as MF retentate.

4.4.2 Sludge used in the biochemical methane potential tests

The content as well as the notations and the amount of each component in the bottles used for the first respectively the second biochemical methane potential test can be found in Tables 4.2 and 4.3.

Table 4.2. Content and notations for the bottles used in BMP-test 1.

Notation	Content	Inoculum (g)	Substrate (g)	Water (ml)
Y1	Inoculum	70.31	0.00	430.50
Y2	Inoculum	70.32	0.00	429.80
Y3	Inoculum	70.38	0.00	432.10
C1	Cellulose	70.89	1.73	428.80
C2	Cellulose	70.67	1.72	428.30
C3	Cellulose	70.32	1.70	430.20
R1	Raw DF sludge	74.00	400.30	30.59
R2	Raw DF sludge	70.38	404.40	30.07
R3	Raw DF sludge	73.77	405.80	29.85
K1	Chemical DF sludge	70.30	320.80	115.80
K2	Chemical DF sludge	70.67	320.55	110.20
K3	Chemical DF sludge	70.44	325.23	110.32
B1	Mixed sludge from Källby WWTP	70.40	36.08	394.74
B2	Mixed sludge from Källby WWTP	72.70	36.01	393.79
B3	Mixed sludge from Källby WWTP	70.64	37.35	395.35

Table 4.3. Content and notations for the bottles used in BMP-test 2.

Notation	Content	Inoculum (g)	Substrate (g)	Water (ml)
I1	Inoculum	75.11	0.00	529.50
I2	Inoculum	75.33	0.00	525.40
I3	Inoculum	75.17	0.00	526.00
H1	Hydrolysed raw DF sludge	83.00	487.60	43.37
H2	Hydrolysed raw DF sludge	76.20	487.20	41.41
H3	Hydrolysed raw DF sludge	75.69	491.40	42.84
H4	Hydrolysed chemical DF sludge	76.96	524.10	9.01
H5	Hydrolysed chemical DF sludge	79.68	519.50	10.05
H6	Hydrolysed chemical DF sludge	75.76	529.80	9.01

5 Results and discussion

The results and discussion section of this report will mainly focus on the parameters related to the performance of the biological hydrolysis and the biochemical methane potential tests performed, irrespective of the effluent quality and results achieved by the compact waste water treatment pilot plant. However, what it comes down to in the end, when choosing a treatment method, would be the fulfilment of the outlet demands for the respective WWTP.

One of the main limitations to this study was the dependence on the pilot plant in operation, as it was not always possible to obtain sludge samples with the preferred characteristics whenever it was required.

Consequently, the varying deliveries of sludge resulted in it not being possible to take samples on a regular basis during the hydrolysis experiments, due to weekends and holidays.

5.1 Biological hydrolysis

Five hydrolysis experiments were performed using different sludge types. An overview of the sludge types and the corresponding chemical precipitation is provided in Table 5.1. The initial and final TS, VS, SS and VSS concentrations as well as the degradation expressed as a percentage can also be found in Table 5.1.

The hydrolysis experiments were performed at ambient temperature, which is roughly 5-10°C warmer than the incoming waste water temperature at Källby WWTP.

Solids content

The measurements of the solids concentrations were performed as triplicates thus the values presented in Table 5.1 are the calculated average values. During the hydrolysis experiment the organic matter is assumed to be solubilised and degraded, resulting in smaller, dissolved particles. It can be seen, in Table 5.1, that the solids content decreases for all of the sludge types except for the DF-MF sludge. Most of the suspended solids in the waste water has been removed by the drum filter, which results in a low concentration of suspended solids in the retentate from the MF membrane. The low content of organic matter and the non-homogeneity of the sludge might be a possible reason, especially concerning the SS-measurements, for why the final values of the solids-measurements in general are being higher than the initial values.

Moreover, due to a mistake made in the laboratory, filter papers of a different pore size were used for the first four hydrolysis experiments (0.45 µm) in comparison to the filter papers used for the fifth hydrolysis (1.6 µm). As a consequence, the SS and VSS measurements might not provide an unambiguous result. The resulting SS and VSS content for hydrolysis experiments 1-4 is slightly higher than the corresponding solids content for hydrolysis experiment 5, as filter papers with a smaller pore size retain a larger segment of small particles. The division of solids into suspended and dissolved solids however is not completely defined, which makes it difficult to characterise the suspended solids content. The most commonly used pore size for filter papers, according to Henze *et al*. (2002), is 1 µm or perchance 0.45 µm. Yet, in Denmark, filter papers of a pore size of 1.6 µm are being used. Furthermore, as the loads of water and pollutants varies on a daily, weekly and monthly basis

(Henze *et al.*, 2002), it is difficult to determine the exact disparity of the obtained values related to the use of different filter papers.

Moreover, the evaporation effect was not compensated for until after the first hydrolysis experiment. Approximately 60% of the content in the beakers had evaporated during the first hydrolysis experiment and is the reason for the increasing SS and VSS content in the hydrolysed sludge.

Table 5.1. The duration of and the sludge types used in the hydrolysis experiments as well as the doses of coagulants and flocculants in addition to the solids content, where the degradation in percent is accentuated.

<table>
<tr><th>Hydrolysis</th><th>1*</th><th>2</th><th colspan="2">3</th><th colspan="4">4</th><th colspan="3">5</th></tr>
<tr><td>Sludge type</td><td>DF</td><td>DF</td><td>DF</td><td>DF</td><td>MF</td><td>DF</td><td>MF</td><td>DF</td><td>DF</td><td>DF</td><td>MF</td></tr>
<tr><td>Coagulation</td><td>5 mg/l (XL100, Al^{3+})</td><td>–</td><td>–</td><td>6.5 mg/l (XL100, Al^{3+})</td><td>–</td><td colspan="2">–</td><td>–</td><td>15 mg/l PAX</td><td colspan="2">15 mg/l PAX</td></tr>
<tr><td>Flocculation</td><td>3 mg/l (Hytex 6161, anionic polymer)</td><td>–</td><td>–</td><td>2.5 mg /l (6456, polymer)</td><td>–</td><td colspan="2">–</td><td>–</td><td>2.7 mg/l anionic polymer</td><td colspan="2">2.7 mg/l anionic polymer</td></tr>
<tr><td>TS initial (g/l)</td><td>7.96</td><td>4.63</td><td>4.42</td><td>4.20</td><td>1.49</td><td colspan="2">0.84</td><td>2.26</td><td>4.26</td><td colspan="2">0.38</td></tr>
<tr><td>TS final (g/l)</td><td>10.68</td><td>4.26</td><td>2.67</td><td>3.42</td><td>1.34</td><td colspan="2">0.85</td><td>1.74</td><td>3.55</td><td colspan="2">0.45</td></tr>
<tr><td>TS degradation (%)</td><td>-34</td><td>8</td><td>40</td><td>19</td><td>10</td><td colspan="2">-1</td><td>23</td><td>17</td><td colspan="2">-18</td></tr>
<tr><td>VS initial (g/l)</td><td>5.64</td><td>3.84</td><td>3.31</td><td>3.08</td><td>0.87</td><td colspan="2">0.38</td><td>1.51</td><td>2.77</td><td colspan="2">0.09</td></tr>
<tr><td>VS final (g/l)</td><td>7.56</td><td>3.48</td><td>1.90</td><td>2.39</td><td>0.77</td><td colspan="2">0.32</td><td>1.05</td><td>2.21</td><td colspan="2">0.10</td></tr>
<tr><td>VS degradation (%)</td><td>-34</td><td>9</td><td>43</td><td>22</td><td>11</td><td colspan="2">16</td><td>30</td><td>20</td><td colspan="2">-11</td></tr>
<tr><td>SS initial (g/l)</td><td>6.66</td><td>4.61</td><td>3.85</td><td>3.86</td><td>1.08</td><td colspan="2">0.38</td><td>1.79</td><td>4.17</td><td colspan="2">0.01</td></tr>
<tr><td>SS final (g/l)</td><td>14.15</td><td>2.76</td><td>2.16</td><td>2.83</td><td>0.85</td><td colspan="2">0.42</td><td>1.20</td><td>3.24</td><td colspan="2">0.01</td></tr>
<tr><td>SS degradation (%)</td><td>-112</td><td>40</td><td>44</td><td>27</td><td>21</td><td colspan="2">-11</td><td>33</td><td>22</td><td colspan="2">0</td></tr>
<tr><td>VSS initial (g/l)</td><td>5.02</td><td>4.18</td><td>3.22</td><td>3.18</td><td>0.98</td><td colspan="2">0.42</td><td>1.54</td><td>3.07</td><td colspan="2">0.14</td></tr>
<tr><td>VSS final (g/l)</td><td>10.28</td><td>2.50</td><td>1.81</td><td>2.22</td><td>0.75</td><td colspan="2">0.42</td><td>1.02</td><td>2.33</td><td colspan="2">0.15</td></tr>
<tr><td>VSS degradation (%)</td><td>-105</td><td>40</td><td>44</td><td>30</td><td>23</td><td colspan="2">0</td><td>34</td><td>24</td><td colspan="2">-7</td></tr>
</table>

***** A different procedure was utilised for the first hydrolysis experiment after which the method was modified, in order to compensate for the evaporation. Consequently, the results from the first hydrolysis are not considered particularly reliable.

pH and temperature

The hydrolysis experiments where drum filter sludge was used showed the expected behaviour of changes in pH (see Figure 5.1), as the pH after initially having been relatively stable started to decreased. According to Ferreiro and Soto (2003), this may be attributed to the sludge buffer capacity, which is exceeded when the production of VFA increases. Once the VFA production diminished, the pH changed more slowly and remained relatively stable around pH 6.5 for the curves 2, 3.1 and 3.2 in Figure 5.1.

The corresponding pH measured for the hydrolysis of the retentate from the MF membrane (curves 4.1, 4.2, 4.4 and 5.2) however, was higher and showed an increasing trend. Some possible reasons for this increase could be an effect from the compensation of the evaporation as the tap water might have a buffering capacity that is more marked for low concentrations of organic matter. It could also be due to the mixing of the beakers, which might drive out carbon dioxide from the liquid thus increasing the pH (Moosbrugger *et al.*, 1993). Further, bacterial growth was observed in the pilot tank for the effluent from the drum filter when using chemical precipitation that might have an effect on the pH, e.g. in case of sulphate reducing bacteria (SRB) being present (Whittington-Jones *et al*., 2006).

Usually an increase in pH at the end of a hydrolysis experiment may be attributed to the beginning of the methanogenesis, where the VFA are consumed. In this case however, the increasingly high pH is probably not due to the beginning of the methanogenesis as the same increase is common to all of the MF retentate curves in the graph. Additionally, the methanogenic bacteria are rather slow-growing and when comparing the pH to the production of VFA, it can be seen that the VFA continues to increase throughout the hydrolysis (see Appendix III, where figures illustrating the concentration of VFA in mg/l COD during the different hydrolysis experiments are presented). If the methanogenesis would have started, the amount of VFA within the sludge would have decreased concurrently with it being consumed by the methanogenic bacteria.

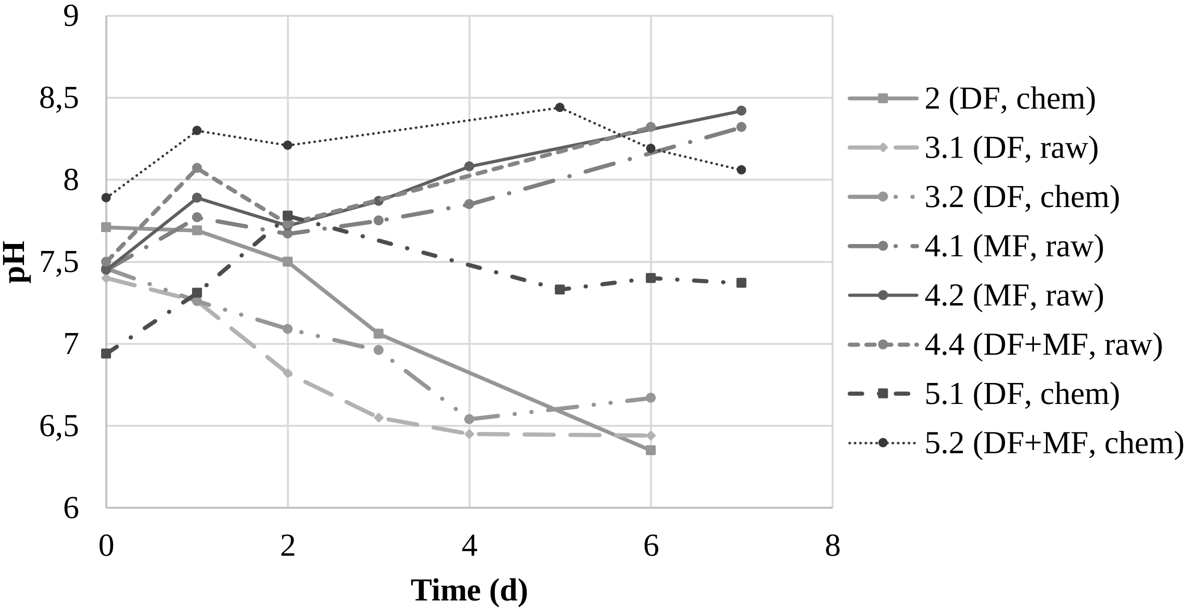

Figure 5.1. The pH as a function of time during the hydrolysis experiments.

The sludge had in general an initial temperature of around 15-16°C when it was taken to the laboratory, after which the temperature increased to the ambient temperature and remained relatively stable around 19°C, which can be seen in Table 5.4 where the average temperatures and the standard deviation (for calculations see Appendix VI) are presented for each hydrolysis experiment.

Table 5.4. The average temperatures (°C) during the hydrolysis experiments and the corresponding standard deviation in addition to the number of samples, N used for the calculations.

Hydrolysis	Average temperature (°C)	Standard deviation	Number of samples, N
1	–	–	–
2	19.0	1.75	5
3	19.8	0.84	12
4	19.3	1.76	24
5	19.4	1.34	12

Sludge volume index

Further, the sludge volumes index (SVI), see Table 5.5, was measured to serve as an indication of how easily the sludge can be thickened and dewatered as it is not tenable to digest sludge anaerobically at very low solids content without pre-treatment due to it requiring enormous volumes for the anaerobic digesters. The DF-MF retentate from the membrane where the waste water stream has passed through the drum filter is a good example of this, as it was not possible to determine a SVI for it.

However, a low value of the SVI implies that the sludge in question is possible to thicken, although the SVI does not account for the eventual content of solids in the clear phase. In general, the raw drum filter sludge with a SVI of 55 appears to be the easiest to thicken, according to the measurements. The assumption was however that by applying chemical precipitation, the thickening efficiency would increase e.g. resulting in the chemical DF sludge having a lower SVI than the raw DF sludge.

The relatively high SVI for the chemical drum filter sludge however might be explained by a larger amount of smaller particles that is precipitated and found in the thicker phase of the chemical DF sludge, in comparison to the raw sludge. In Figure 5.2, it can be seen that there seems to be small particles remaining in the liquid phase.

Table 5.5. Results from the determination of the sludge volume index.

Hydrolysis experiment	Sludge type	SVI (ml/g SS)
3	Raw DF	55
2	Raw DF	81
4	Raw DF	95
1	Chemical DF	100
3	Chemical DF	100
4	Raw MF	111
4	Raw DF – MF	132
5	Chemical DF	186
5	Chemical DF – MF	–

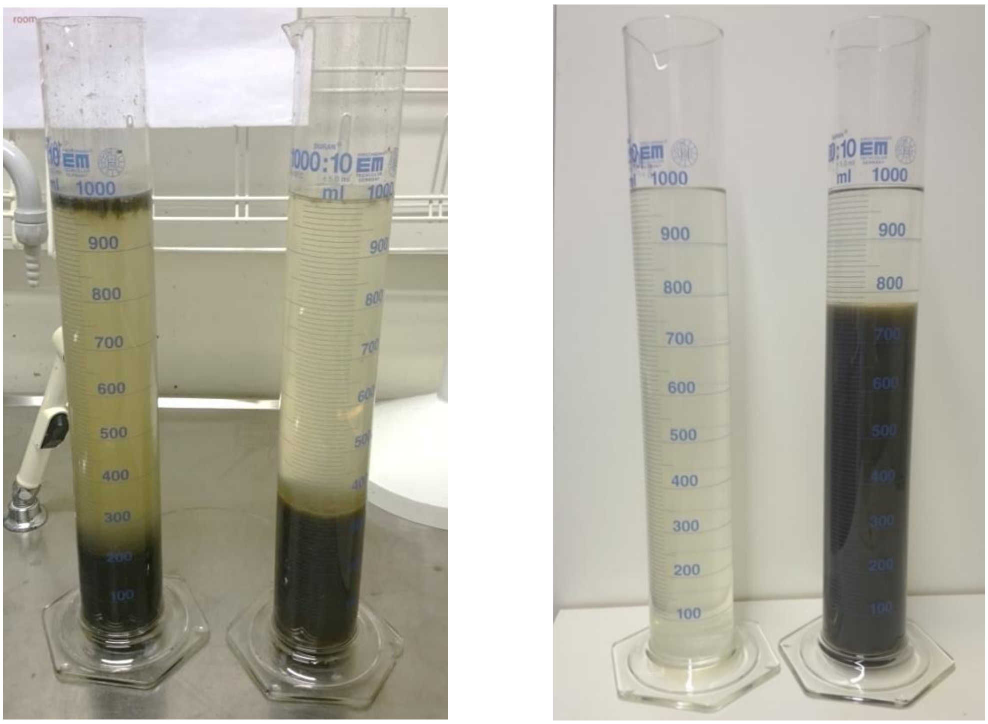

Figure 5.2. Sludge volume index for a) hydrolysis experiment 3 containing raw sludge (left) and chemical sludge (right) from the drum filter, respectively b) hydrolysis experiment 5 chemical DF-MF sludge from the membrane (left) as well as chemical sludge (right) from the drum filter.

To sum up, all of the tested sludge, apart from the DF-MF retentate that does not contain enough organic matter, seems to be possible to thicken. Consequently, the sludge is considered to have potential as a substrate for anaerobic digestion when it comes to it being possible to thicken. Although, it could be of interest e.g. to measure the SS content in the clear phase and to use other methods, in order to obtain a more comprehensive evaluation of which sludge is the easiest to thicken.

Organic content

Since the sludge had a high solids concentration it had to be diluted before measuring the total COD, which might be a possible source of error. Furthermore, as previously mentioned, filter papers of a different pore size were used for the measurements of SS as well as the COD during the last hydrolysis experiment, in comparison to the filter papers used for the earlier hydrolysis experiments. The resulting values from the filtered COD measurements are therefore lower than they would have been if the larger pore size would have been used for all of the hydrolysis experiments, as less of the particles are retained by the larger pore size leading to higher values for the COD.

The solubilisation of COD in relation to the amount of volatile suspended solids as a function of time throughout the hydrolysis is presented in Figures 5.3 to 5.7, where the number of beakers for which the mean values used in the graphs were calculated (see Appendix VI for the formulas used), can be found within parenthesis.

According to Norlander (2008) and Juncà (2010), the dissolved COD i.e. COD_f, should increase during the first days of the hydrolysis until it reaches a maximum value or starts to decrease when the methanogenesis process begins. The result from hydrolysis 1 (Figure 5.3) does not show such a trend, which is most likely due to the evaporation effect. The evolution of the second and third hydrolysis curves (Figure 5.4 and 5.5) shows a slight tendency to level out, although it is more distinct in Figure 5.6 for the fourth hydrolysis. Regarding the results from the fifth hydrolysis experiment (Figure 5.7), a slight decline is discernible the last day for both of the curves. Due to it being the last measurement, it is difficult to conclude whether the final values are unexpectedly low or if the previous measurement was higher than it should have been.

Moreover, the COD solubilisation in mg COD/g VSS is greater for the chemical DF-MF retentate than for the chemical drum filter sludge. This difference is related to the remarkably low VSS content of 0.14 g/l for the DF-MF retentate in comparison to 3.07 g/l for the DF sludge, as the COD values themselves (in mg/l, found in Appendix II) for the retentate are not even close to those for the DF sludge. A possible reason for the low COD values could be the inert fraction, which reprents approximately 50-80 mg COD/l and cannot be degraded during biological hydrolysis (Henze *et al.*, 2002).

Additionally, a decrease in the COD_f occurred in the second beaker of the fifth hydrolysis with chemical DF sludge, caused by the tubing for nitrogen, which fell out of the beaker during the night between the first and the second day of the hydrolysis, leading to lower COD values. The decrease in COD is most likely caused by oxidation of the organic matter within the beaker, when aerobic bacteria had access to oxygen. The results from the second beaker were therefore excluded from Figure 5.7.

Even though pre-precipitation was applied, the COD solubilisation for e.g. raw respectively chemical drum filter sludge followed the same pattern, which is due to the daily, weekly and monthly fluctuations in the waste water and pollutants load leading to the waste water characteristics changing drastically between each sampling (Henze *et al.*, 2002).

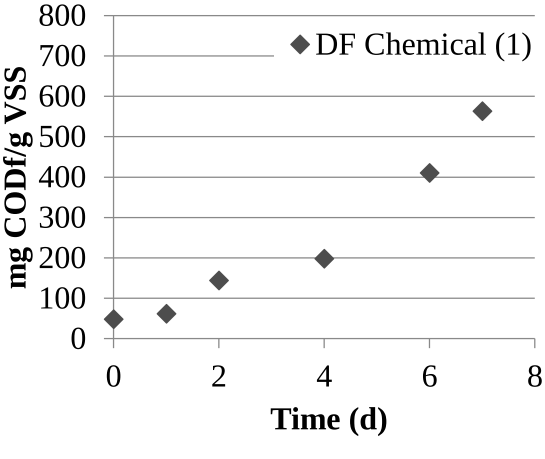

Figure 5.3. Hydrolysis 1: Average filtered COD in mg/g VSS as a function of time during the hydrolysis of drum filter sludge with chemical precipitation (N=1 for the calculations of the average).

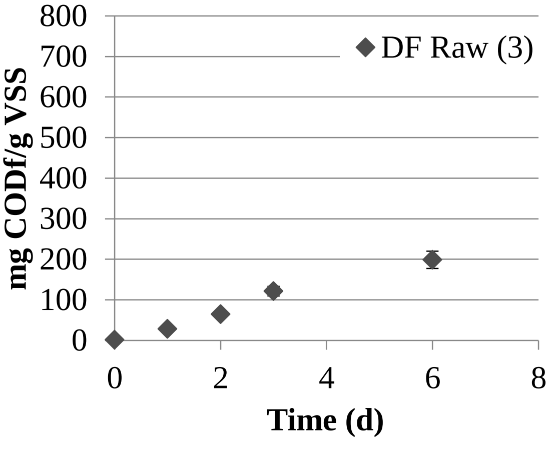

Figure 5.4. Hydrolysis 2: Average filtered COD in mg/g VSS as a function of time during the hydrolysis of drum filter sludge without chemical precipitation.

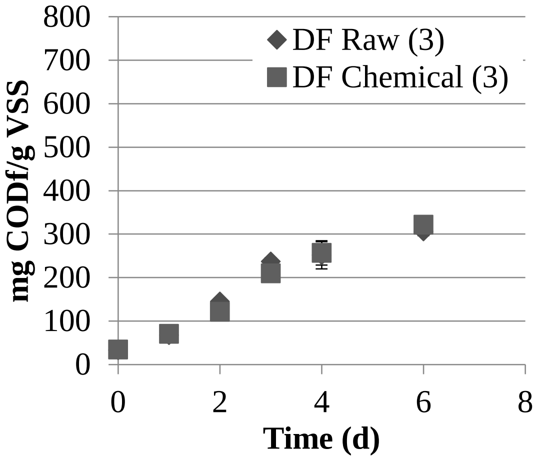

Figure 5.5. Hydrolysis 3: Average filtered COD in mg/g VSS as a function of time during the hydrolysis of drum filter sludge without respectively with chemical precipitation (N=3).

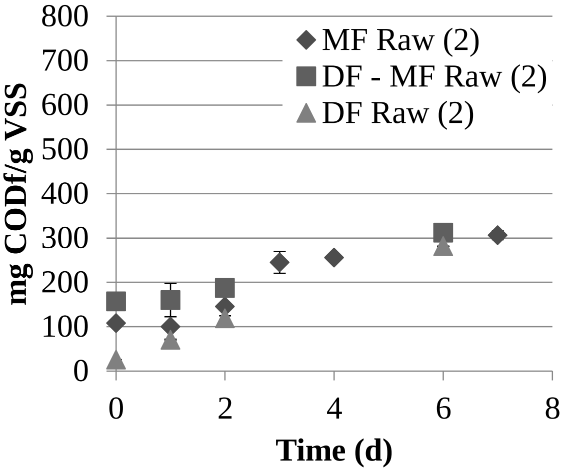

Figure 5.6. Hydrolysis 4: Average filtered COD in mg/g VSS as a function of time during the hydrolysis of drum filter sludge respectively DF-MF and MF retentate without chemical precipitation (N=2).

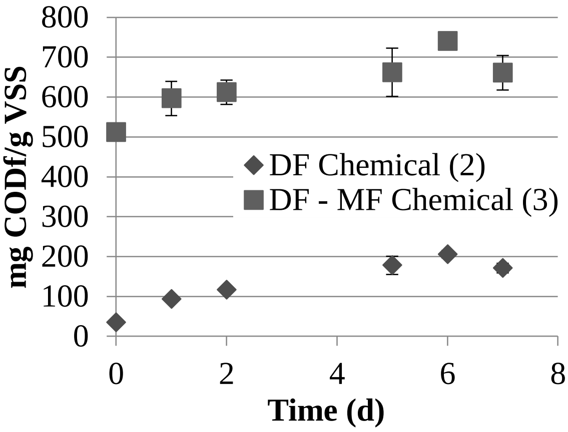

Figure 5.7. Hydrolysis 5: Average filtered COD in mg/g VSS as a function of time during the hydrolysis of drum filter sludge (N=2) and DF-MF retentate (N=3), without chemical precipitation.

A summary of the results from the hydrolysis experiments is provided in Figure 5.8, where it can be seen that the results from the hydrolysis experiments tend to follow the same pattern. The majority of the different hydrolysis experiments are also found in the same range of values, apart from those for the chemical DF-MF retentate and the chemical DF sludge in the first hydrolysis experiment as a result of the evaporation effect.

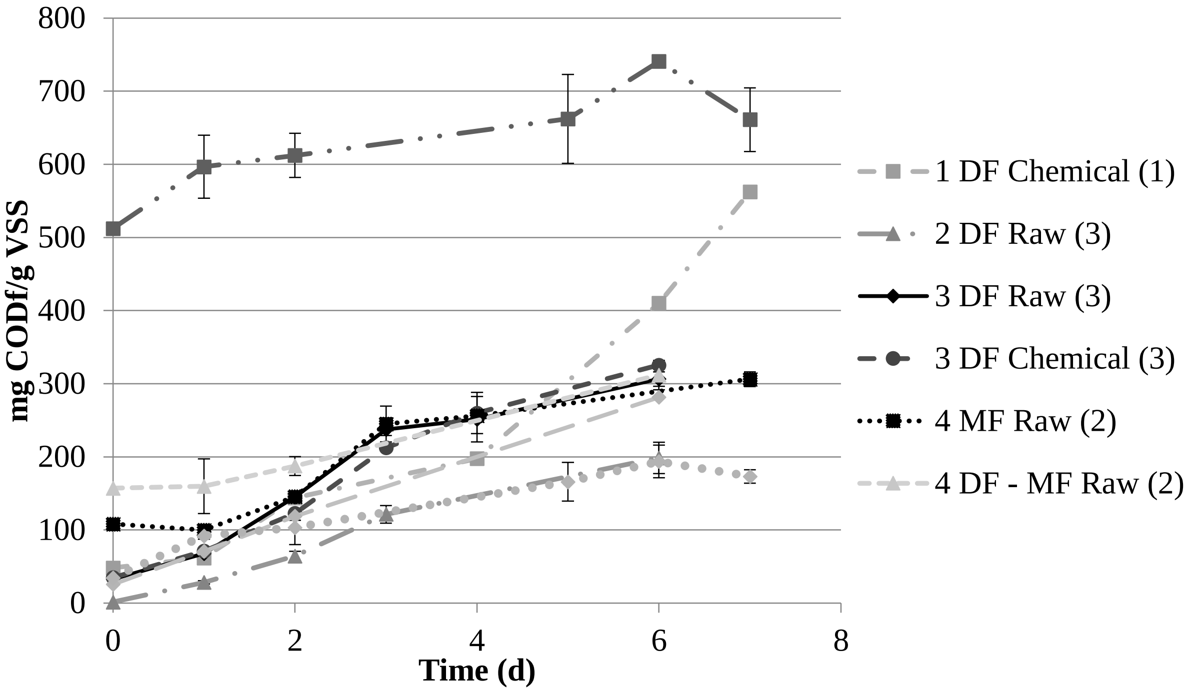

Figure 5.8. The amount mg CODf/g VSS as a function of the time in days.

Furthermore, in order to be able to compare the results obtained with results obtained in other studies, the COD yield (COD_f/COD_{tot}, see Table 5.6) was calculated, based on the filtered COD the last day (day 6 respectively 7) of each hydrolysis experiment. The maximum COD yields obtained are around 20% for the majority of the sludge, although no clear trend for the different sludge types can be distinguished. The large variation discerned for the values of each sludge type is due to a large variation in the characteristics of the incoming waste water, which can be seen when comparing the total COD for the different sludge types, as well as the varying dosage of the applied chemicals.

The COD yield for the DF-MF retentate in the fifth hydrolysis however is unlikely, as the total COD is lower than the filtered COD during the hydrolysis. This might be accountable to the filter paper used in the fifth hydrolysis experiment, which was made of cellulose and probably contributed to the content of organic matter. Due to the low content of organic matter in the retentate, the eventual contribution of COD from the filter paper might have a higher impact than it normally would have had.

Table 5.6. The COD solubilisation and standard deviation.

Hydrolysis	**Sludge type**	**$COD_{f,\ final}$ (mg/l)**	**COD_{tot} (mg/l)**	**$COD_{f,\ final}/COD_{tot}$ (%)**	**Standard deviation**
1	DF, chemical	3100	12000	26	–
2	DF, raw	935	6280	15	0.7
3	DF, raw	987	5500	18	0.6
	DF, chemical	1035	6040	17	0.3
4	MF, raw	300	1484	20	0.7
	DF – MF, raw	131	588	22	1.4
	DF, raw	434	1680	26	0.0
5	DF, chemical	532	9320	6	0.3
	DF – MF, chemical	96	74	129	8.5

When comparing the maximum COD yields of 6-26% obtained in this study to other studies having used biological hydrolysis as pre-treatment of primary sludge, it can be seen that the results correspond rather well. By way of example, maximum COD yields of 13-14% for hydrolysed mixed sludge from Källby WWTP respectively 21-22% and 17-20% for hydrolysed primary sludge from Klagshamn and Sjölunda WWTP, located in Malmö, were found in a study made by Juncà (2010). Similar values of 16% for the hydrolysis of precipitated primary sludge (respectively 19% for sludge without chemicals) at 20°C, have been found by Jönsson *et al.* (2008). Furthermore, a maximum COD yield of 20-24% for primary sludge hydrolysed at 20°C was found by Daton and Wallergård (2003, cited by Jönsson and Jansson, 2006).

Moreover, the COD yield for each sludge type was also calculated using the equation below and illustrated in Figure 5.9, in order to be able to relate the final values of the final COD to the initial filtered and the total COD thus obtaining the increase in COD yield. The COD yield obtained was then roughly 15%.

$$\frac{COD_{f,final} - COD_{f,initial}}{COD_{tot}} \ [\%] \qquad \textit{Eq. 3}$$

Another important aspect is to render the hydrolysis time more efficient and to determine if it is worthwhile to pre-treat sludge with e.g. a three day hydrolysis. Applying biological hydrolysis as a pre-treatment step for a time period of seven days or longer would imply a need for very large full-scale volumes. The degree of solubilisation is therefore included in Figure 5.9 for day 2-4 (related to when measurements were made), in order to evaluate if a shorter time period would be conceivable. In general, more than 50% of the total COD yield has been achieved after three days of hydrolysis making a shorter hydrolysis a possible solution in order to minimise the volumes needed.

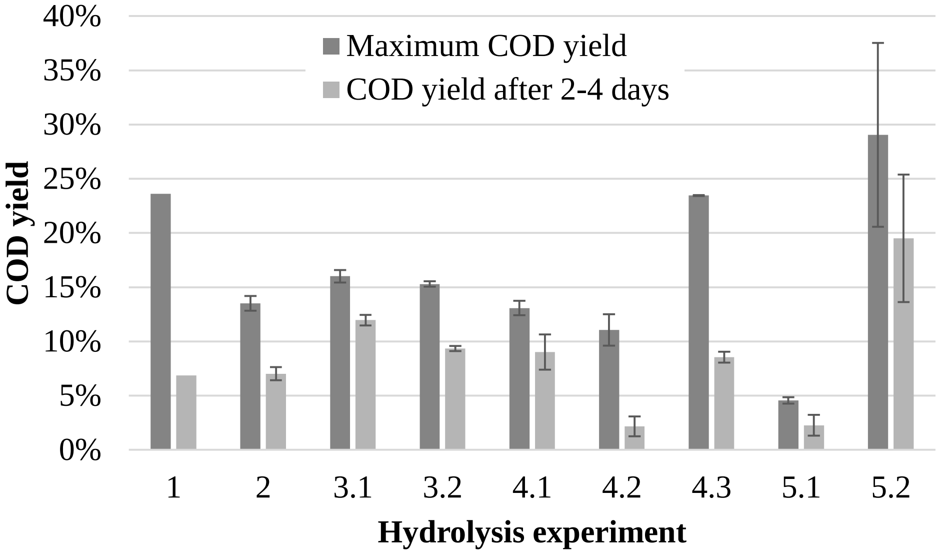

Figure 5.9.The maximum COD yield for each sludge type expressed as a percentage calculated using equation 3 after 6 days, respectively 7 days for hydrolysis experiment 4.1 and the COD yield after 2 days (hydrolysis 4.2-5.2), 3 days (hydrolysis 2-4.1) and 4 days (hydrolysis 1) together with the standard deviation.

As previously mentioned, the exorbitant results from hydrolysis 5.2 are possibly due to the filter papers used and the deviant results from hydrolysis 1 are probably due to the evaporation effect, which might have led to the higher COD yield. Furthermore, the very low COD yield for hydrolysis 5.1 could be a result of the sludge being very thick and difficult to hydrolyse.

VFA production

The volatile fatty acids and primarily acetate (as can be seen in Figures 5.10 to 5.18), are a part of the chemical oxygen demand (Henze *et al*., 2002), which is used for the production of methane on account of them being readily biodegradable.

In Figures 5.10 to 5.18, the ratios of mg COD_{VFA}/mg COD_f, expressed as percentages, are illustrated together with the specified fractions of acetate (the largest fraction) and propionate with the purpose of serving as an evaluation of the performance of the hydrolysis. It can be seen that the maximum VFA as a percentage of the dissolved COD is attained after a time period of approximately 3-4 days, after which the fractions remain relatively constant.

Further, it can also be suspected that the organic matter in the DF-MF retentate of hydrolysis 5.2 mainly consists of inert organic matter as the amount of VFA in relation to the dissolved COD barely changes.

Figures with the concentrations of VFA expressed in COD units (COD_{VFA} mg/l), corresponding to the total measured amount of acetate and propionate, can be found in Appendix III. It can be seen that the concentration of VFA increases similarly to the dissolved COD during the hydrolysis. What needs to be taken into account however, is that the actual

VFA production is slightly higher as other VFA are being produced as well and that further analyses would be required in order to provide the total content of VFA.

Further, if the methanogenesis would have started during the hydrolysis experiments, it would have been visible in these figures as the COD and VFA values decreases when part of it has been consumed for the production of methane.

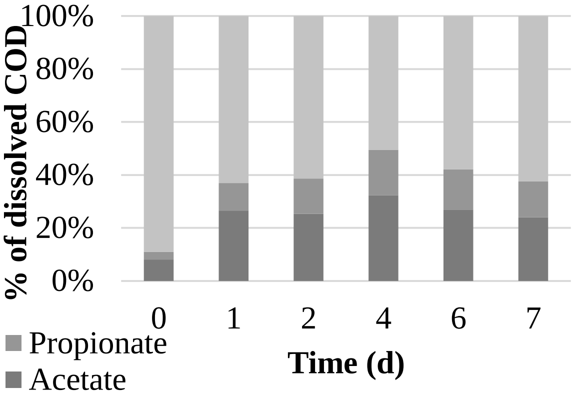

Figure 5.10. Hydrolysis 1: The average amount of acetate and propionate as the percentage of dissolved COD in the chemical DF sludge.

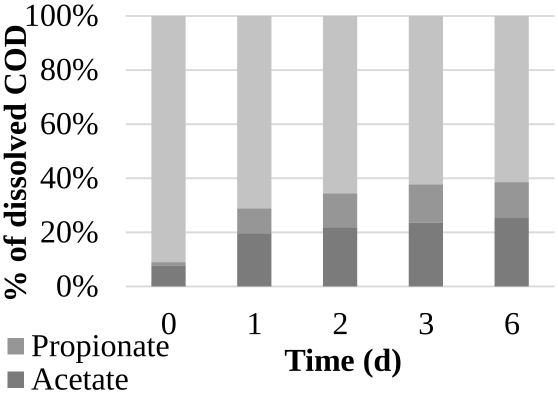

Figure 5.11. Hydrolysis 2: The average amount of acetate and propionate as the percentage of dissolved COD in the raw DF sludge.

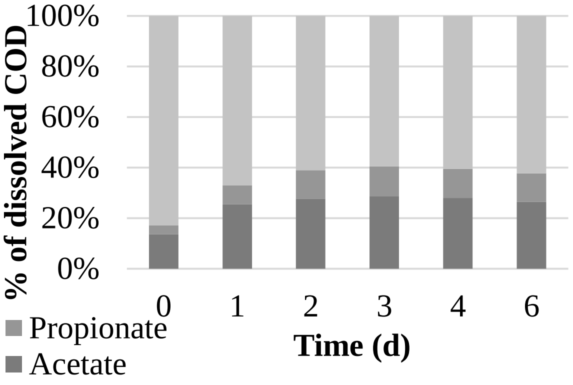

Figure 5.12. Hydrolysis 3: The average amount of acetate and propionate as the percentage of dissolved COD in the raw DF sludge.

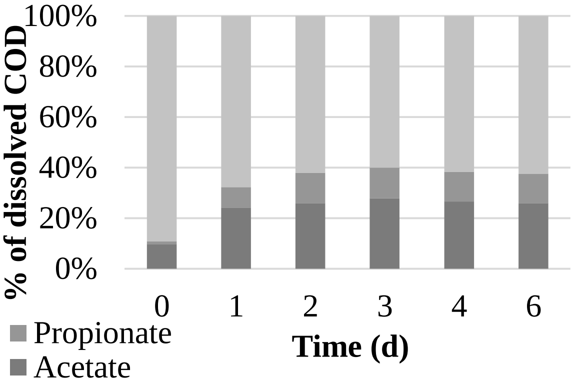

Figure 5.13. Hydrolysis 3: The average amount of acetate and propionate as the percentage of dissolved COD in the chemical DF sludge.

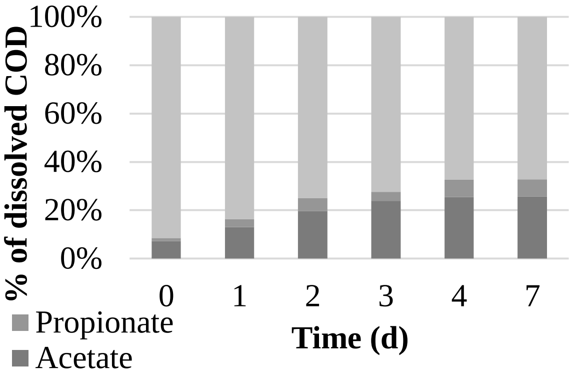

Figure 5.14. Hydrolysis 4: The average amount of acetate and propionate as the percentage of dissolved COD in the raw MF retentate.

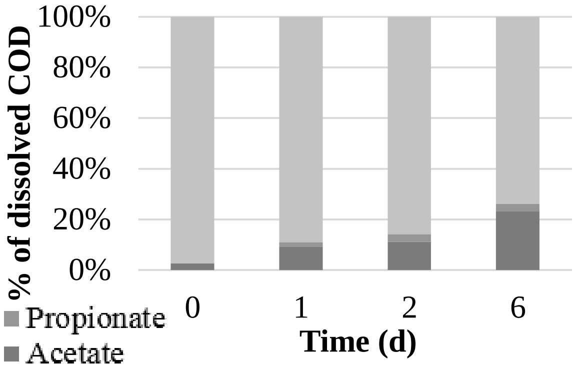

Figure 5.15. Hydrolysis 4: The average amount of acetate and propionate as the percentage of dissolved COD in the raw DF - MF retentate.

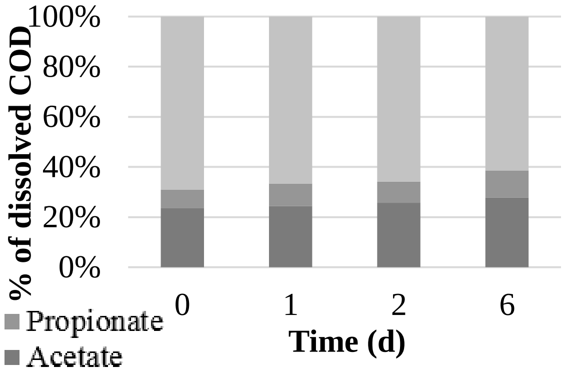

Figure 5.16. Hydrolysis 4: The average amount of acetate and propionate as the percentage of dissolved COD in the raw DF raw sludge.

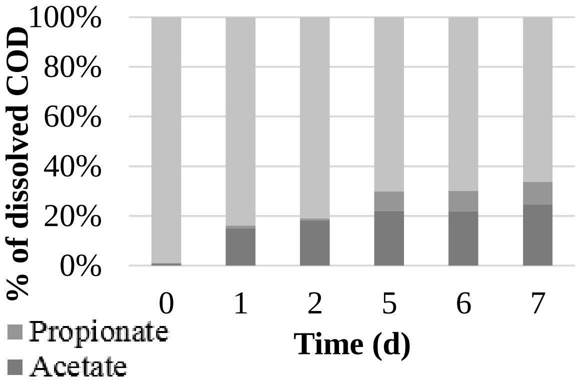

Figure 5.17. Hydrolysis 5: The average amount of acetate and propionate as the percentage of dissolved COD in the chemical DF sludge.

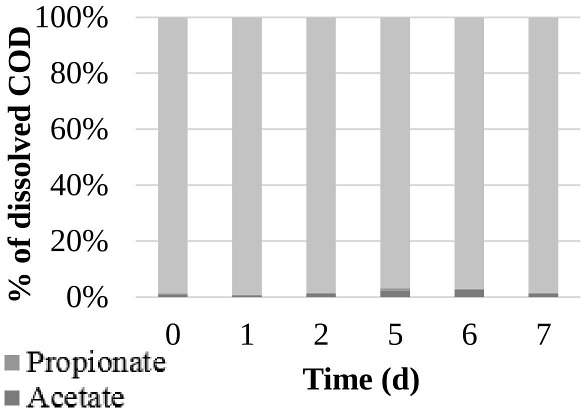

Figure 5.18. Hydrolysis 5: The average amount of acetate and propionate as the percentage of dissolved COD in the chemical DF - MF retentate.

Moreover, when calculating the initial and final concentration of VFA as mg COD_{VFA}/g $VSS_{initial}$ a large increase can also be seen from the start of the hydrolysis to the last day (see values presented in Table 5.7).

Table 5.7. The initial and final concentrations of VFA expressed as mg COD_{VFA}/g VSS for the different hydrolysis experiments.

Hydrolysis	Sludge type	mg CODVFA, initial/g VSS	mg CODVFA, final/g VSS
1	DF chemical	5.9	338.5
2	DF raw	2.0	140.7
3	DF raw	6.8	185.5
3	DF chemical	4.3	196.3
4	MF raw	12.3	149.0
4	DF-MF raw	4.3	93.5
4	DF raw	1.0	181.2
5	DF chemical	0.4	88.4
5	DF-MF chemical	6.9	10.2

5.2 Determination of methane potentials

Two biochemical methane potential tests were accomplished in order to determine the methane potential of the sludge from the compact waste water treatment concept in comparison to the conventionally generated sludge. Further, the potential of using biological sludge hydrolysis as a pre-treatment step for the sludge was also to be evaluated.

The first BMP test consisted of non-hydrolysed mixed sludge (a mixture of approximately 60% waste activated sludge, 30% raw sludge and 10% chemical sludge according to Hey (2015)) directly from the Källby WWTP as well as raw and chemical drum filter sludge from the pilot plant, while the second BMP test was composed of the raw respectively chemical drum filter sludge that had been hydrolysed in the third hydrolysis experiment for a time period of seven days.

The methane production of the sludge is expressed in Nml CH_4/g $VS_{initial}$, in order to be comparable to e.g. other studies. The production of methane was calculated as the average of the triplicate bottles that gave the maximum accumulated methane production, having excluded the accumulated production of methane coming from the inoculum. The VS content that was used to calculate the volumes of sludge used in the first BMP test was estimated, as it could not be measured before the start-up seeing that measuring the VS content of a sample takes at least 24 hours and that the start-up had to be done directly after the sludge had been collected. The values used for the calculations as well as the actual values, measured at the start-up, can be found in Appendix IV, where it can be seen that the estimated and measured values in general corresponds well with each other.

The tables containing the TS and VS content of the sludge (in Appendix IV) can also give an estimation of the amount of organic matter within the sludge that is inert. The TS content varies for the sludge as most of the sludge has not been thickened. Any differences in the thickness of the different sludge types were however compensated for through dilution, leading to the same conditions for all of the sludge types used in the BMP tests.

Furthermore, the effect of the inoculum might differ in the bottles containing only inoculum respectively the bottles containing substrate as well. When the average methane production of the inoculum is subtracted from each measurement it does not take into consideration that there might be a positive or negative synergetic effect when mixing the inoculum with substrate. It is difficult to determine this effect but important to keep in mind when performing BMP tests.

Another aspect when treating the results and interpreting the figures, is that normally a decrease in the accumulated production is not possible but as the production of the inoculum is subtracted this is not always the case and a slight decline might therefore be seen for certain parts of the curves.

BMP test 1

The results from the first biochemical methane potential test are to be found in Figure 5.19, where the corrected accumulated methane production is shown together with error bars illustrating the standard deviation.

In Figure 5.19, it can be seen that the chemical drum filter sludge had the highest methane potential (457 Nml CH_4/g VS) after 42 days followed by the raw drum filter sludge (366 Nml

CH_4/g VS). The lowest methane potential is encountered with the conventional mixed sludge (307 Nml CH_4/g VS) from Källby WWTP, which consists of roughly 60% waste activated sludge, 30% primary sludge and 10% chemical sludge. The use of waste activated sludge in the anaerobic digestion leads to a lower methane potential as some of the organic matter has already in part been degraded and the biomass may prove difficult to degrade. Primary and waste activated sludge have, according to Persson *et al*. (2010), different characteristics and rates of hydrolysis. Due to the high content of cells and the accumulation of cell parts difficult to degrade as well as extracellular polymers keeping the flocs together, the waste activated sludge poses certain difficulties to the anaerobic digestion process (Persson *et al*., 2010).

In addition to the difference in performance of the different substrates, it can be seen that the accumulated production of methane increases quickly for approximately 16 days (see Figure 5.19), after which the production starts to level out. Initially the methane production in the reference bottles containing cellulose show an acclimatisation phase of three days before the production increases.

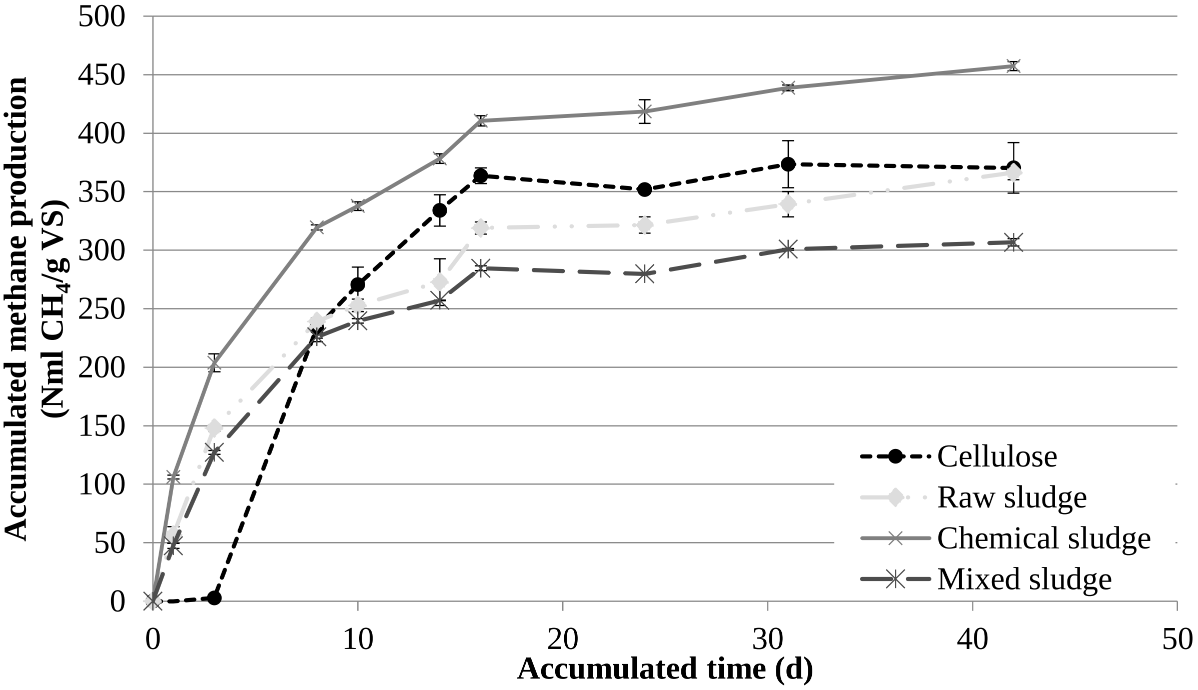

Figure 5.19. The accumulated methane production in Nml CH_4/g VS without the effect from the inoculum, as a function of the accumulated time in days for BMP test 1.

Additionally as the second bottle containing inoculum stopped producing methane after day 16; it was not included in the calculations. A possible reason for this reduction in the methane production might be leakage, as the other two bottles with inoculum exhibited no such behaviour.

Moreover, the bottles containing cellulose (with an accumulated methane production of 370 Nml CH_4/g VS) was only included as a reference to verify the quality and activity of the inoculum. It is comparable to values for cellulose found in literature: 379 Nml CH_4/g VS after 50 days (Hansen et al., 2004) and 353 Nml CH_4/g VS after 31 to 46 days (Davidsson, 2007), thus the quality of the inoculum can be concluded as satisfactory. Consequently, no cellulose was included in the second BMP test since the inoculum was collected from the same anaerobic digester and of good quality judging by the previous BMP test, a valid assumption, when considering the results from the second BMP test as well.

BMP test 2

The results from the second BMP test can be found in Figure 5.20, where it can be seen that the hydrolysed raw drum filter sludge (512 Nml CH_4/g VS) had a higher methane potential than the hydrolysed chemical drum filter sludge (462 Nml CH_4/g VS). The curves still show a slight increase at the end of the result but judging by the shape of the curves, this increase will not be substantial in comparison to the production during the forty-one days of anaerobic digestion.

Furthermore, the curves depicted in Figure 5.20, display the expected shape of a BMP test where the accumulated methane production levels out after the initial increase, similar to the curves presented in Hansen *et al*. (2004).

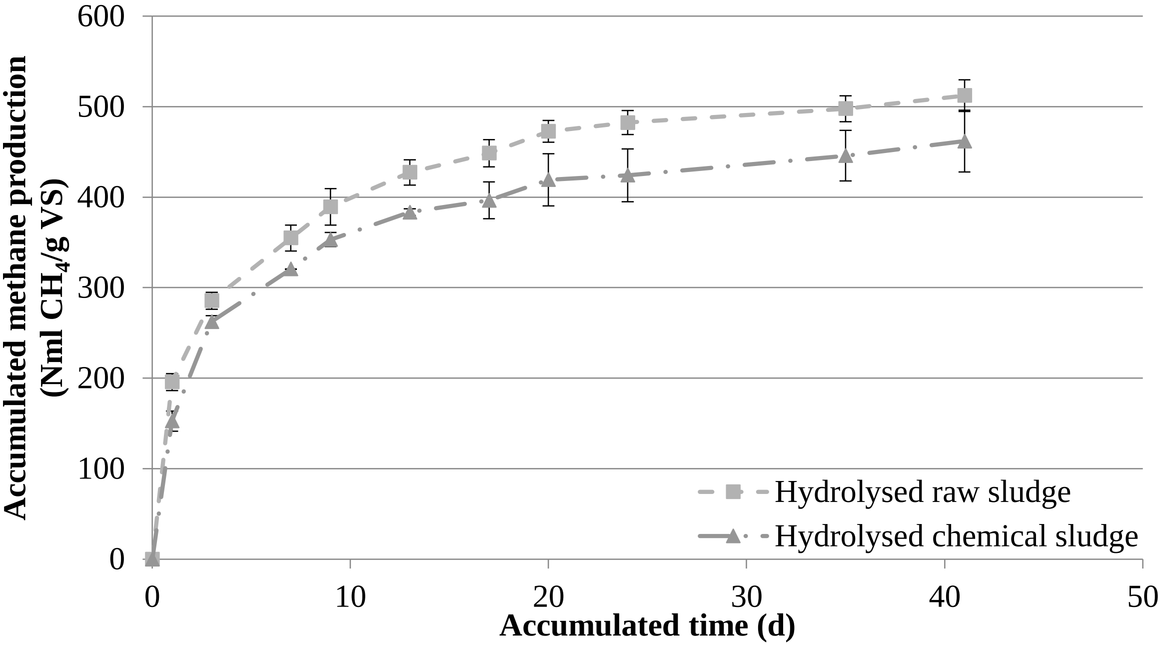

Figure 5.20. The accumulated methane production in Nml CH_4/g VS without the inoculum, as a function of the accumulated time in days for BMP test 2.

Comparison of BMP test 1 and 2

In order to facilitate the comparison of the results obtained in the two BMP tests, the accumulated methane production curves are presented together in Figure 5.21. The sludge with the largest accumulated methane production is the hydrolysed raw DF sludge followed by the hydrolysed chemical DF sludge and the non-hydrolysed chemical DF sludge. However, the hydrolysed chemical DF sludge showed initally a faster production, which in a continuous reactor with a short retention time might lead to a methane production similar to that of the hydrolysed raw DF sludge in reality.

Further, it can be seen that the methane production in the bottles containing sludge from the pilot plant (both hydrolysed and non-hydrolysed sludge) starts earlier than in the bottles with conventional mixed sludge from Källby WWTP. The accumulated methane production of hydrolysed raw sludge is at the beginning of the BMP tests roughly 44% higher than the corresponding methane production by the conventional mixed sludge from Källby WWTP.

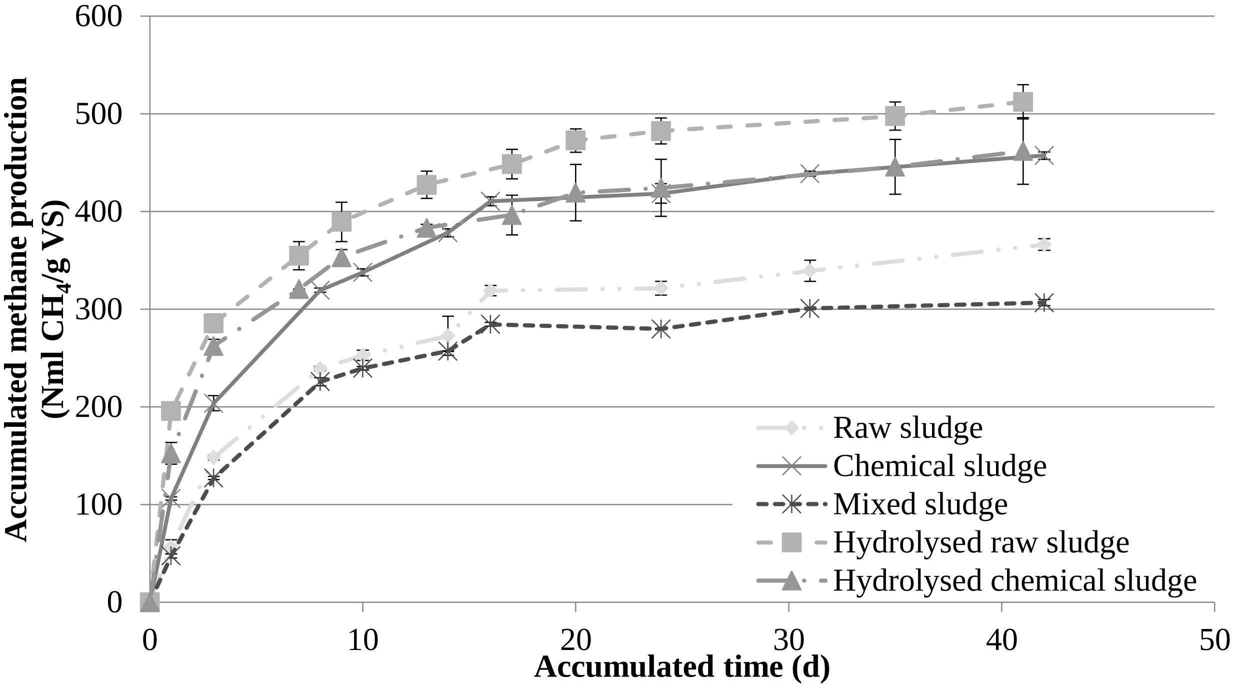

Figure 5.21. The accumulated methane production in Nml CH_4/g VS as a function of the accumulated time in days for BMP test 1 and 2.

The resulting values of the methane potential in Nml CH_4/g VS together with the standard deviation (for calculations see Appendix VI) are presented in Table 5.8.

Table 5.8.The methane potential in Nml CH4/g VS for the sludge types used in the first and the second BMP test together with the standard deviation.

Sludge	Methane potential (Nml CH_4/g VS)	Standard deviation
Raw drum filter sludge	366	6.0
Chemical drum filter sludge	457	3.7
Conventional mixed Källby WWTP sludge	307	3.2
Hydrolysed raw drum filter sludge	512	17.5
Hydrolysed chemical drum filter sludge	462	34.1

A large standard deviation in Table 5.8, which is likely due to the fact that only two samples (N=2) were used for each calculation (see Appendix VI).

Norlander (2008) performed a study on the methane potential of primary sludge respectively mixed sludge (consisting of 60% primary sludge and 40% return sludge) from Klagshamn WWTP that had been hydrolysed for 10 days and where the hydrolysate had been removed to be used as a carbon source for the denitrification process. It is commonly considered that removal of the hydrolysate might have a negative impact on the methane production although the results obtained by Norlander (2008) proved not have a sigificant impact on the methane potential.

A methane potential of 374 Nml CH_4/g VS was shown for the primary sludge respectively of 371 Nml CH_4/g VS for the mixed sludge after 40-43 days when the hydrolysate had not been removed while the corresponding values obtained when the hydrolysate was withdrawn were 385 Nml CH_4/g VS respectively 340 Nml CH_4/g VS (Norlander, 2008). The sludge hydrolysed by Norlander (2008) show a methane potential similar to that of the raw drum filter sludge in this study whereas the hydrolysed raw and chemical drum filter sludge present an even higher methane potential, which might be attributed to the increased extraction of organic matter into the sludge by the compact waste water treatment pilot plant.

The methane potentials in Table 5.8 are also considerably higher than those found by Juncà (2010), although they are expressed in the unit Nml CH_4/g $VSS_{initial}$, which might imply they are slighlty lower than the values expressed in Nml CH_4/g $VS_{initial}$. Juncà (2010) performed, similarly to Norlander (2008), a study where four days of biological hydrolysis was used for the production of an easily accessible carbon source including continuous withdrawal of the hydrolysate before BMP tests were carried out on the remaining sludge.

Primary sludge from Klagshamn and Källby WWTP in addition to mixed sludge from Sjölunda WWTP was used and a duration of 46 days was applied for the BMP tests using non-hydrolysed sludge respectively 38 days for the hydrolysed sludge. The BMP tests resulted in methane potentials of 303 Nml CH_4/g VSS and 246 Nml CH_4/g VSS for the hydrolysed sludge from Sjölunda and Källby WWTP respectively. The corresponding values for the non-hydrolysed sludge were 282 Nml CH_4/g VSS and 285 Nml CH_4/g VSS for Sjölunda and Källby WWTP.

Consequently, the hydrolysed as well as the non-hydrolysed sludge from the compact waste water treatment pilot plant presented higher methane potentials than those found for the non-hydrolysed and hydrolysed sludge generated by conventional waste water treatment methods.

5.3 Case study for Källby WWTP

However, it is important not to over-interpret the results obtained from a biochemical methane potential test. A possible solution is to create a mass balance for comparison of the results obtained to those achieved at present in reality. An illustration of the delimitations used for the calculations regarding the conventional waste water treatment process at Källby WWTP can be found in Figure 5.22. The calculations made in order to obtain the results in Table 5.8 can be found in Appendix V.

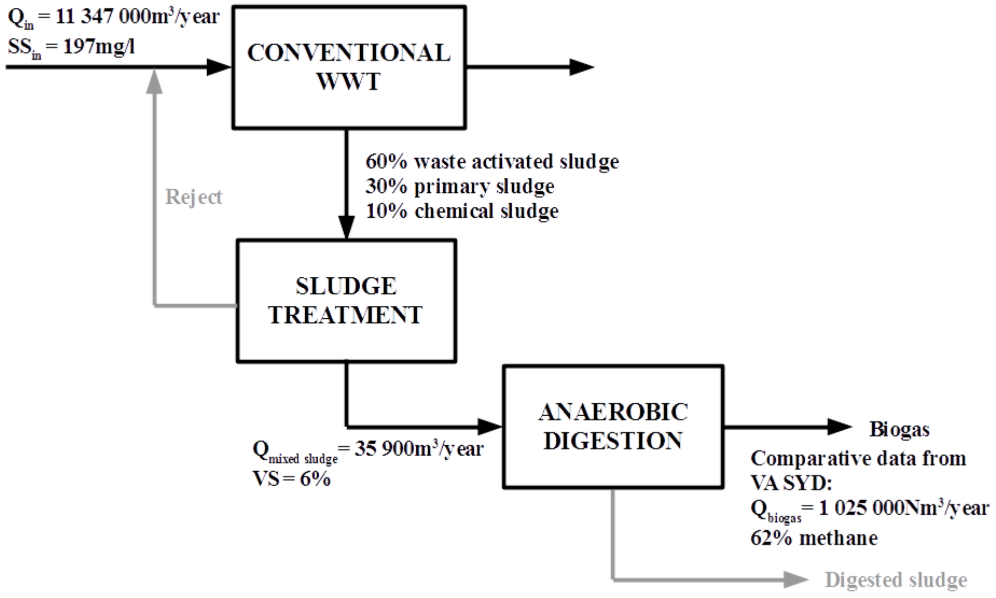

Figure 5.22. A simplified process scheme of the conventional waste water treatment process at Källby WWTP and the figures used for the calculations (Hey, 2015; Murto, 2015).

An annual methane production for Källby WWTP of 635 500 Nm^3/year was obtained based on calculations using data from Hey (2015) and Murto (2015), which is to be considered as the reference. The yearly methane potentials calculated based on the results from the BMP tests were multiplied by an assumed factor of 0.9 to compensate for the longer retention time and favourable process conditions during the biochemical methane potential tests compared to what is realistically achievable.

The results in Table 5.9 show higher yearly methane potentials for the sludge produced by the compact waste water treatment pilot plant, especially the sludge that had been pre-treated using biological hydrolysis.

Table 5.9. The yearly methane potential in Nm^3/year for the different sludge types used.

Sludge type	**Yearly methane potential (Nm^3/year)**
Mixed sludge from Källby WWTP	595 150
DF raw	620 577
DF chemical	720 812
DF chemical hydrolysed	765 133
DF raw hydrolysed	807 562

6 Conclusion

- The biochemical methane potential tests showed a higher methane potential for the sludge originating from the drum filter in the compact waste water treatment in pilot scale in comparison to the sludge currently fed to the anaerobic digester at Källby WWTP generated by conventional waste water treatment methods.

- The highest accumulated methane production was achieved by the hydrolysed raw drum filter sludge (512 Nml CH_4/g VS) followed by the hydrolysed precipitated drum filter sludge (462 Nml CH_4/g VS) and the non-hydrolysed chemical (457 Nml CH_4/g VS) respectively non-hydrolysed raw (366 Nml CH_4/g VS) drum filter sludge.

- The lowest accumulated methane production (307 Nml CH_4/g VS) was represented by the conventional mixed sludge from Källby WWTP, consisting of approximately 60% waste activated sludge, 30% primary sludge and 10% chemical sludge.

- The initial rate of the methane production was higher for the sludge generated by the compact waste water treatment, which in a continuous reactor with a short retention time would be more advantageous in comparison to the results of the conventionally generated sludge.

- The hydrolysed raw sludge showed initially a 44% higher production of methane than that of the conventional mixed sludge from Källby WWTP, which indicates that it would require a shorter retention time e.g. smaller reactor volumes if the same production is sought after or it would result in a larger production when using the same volumes.

- The results from the mass balance showed a positive trend of increased annual methane potentials by applying compact waste water treatment, similar to the results from the biochemical methane potential tests.

- Application of biological hydrolysis of sludge led to an increased solubilisation of the organic matter, especially the VFA that are necessary for the methanogenesis and an increased production of methane in the biochemical methane potential tests.

- The maximum VFA production as a percentage of the dissolved COD, with acetate and propionate constituting between 30-50% of the dissolved COD, was attained after a time period of 3-4 days indicating a shorter duration of biological hydrolysis might be sufficient in order to increase the methane potential of the sludge.

- A $COD_{f,\ final}/COD_{tot}$-yield of approximately 20%, corresponding to values found in the literature, was obtained after 6 days of biological hydrolysis.

- The calculated yield of $(COD_{f,\ final} – COD_{f,\ initial})/COD_{tot}$ was estimated at 15%, out of which 50% was achieved within 3-4 days indicating that an acceptable degree of hydrolysis can be attained during a shorter period of time with the purpose of pre-treating sludge and converting organic matter into easily degradable organic matter, which can be assimilated by the bacteria in the anaerobic digestion process.

- Further, it can be concluded, based on the results of the sludge volume index that it is possible to thicken the sludge generated by the compact waste water treatment in pilot scale before anaerobic digestion, although further analyses might be required.

7 Future work

Relatively few studies have been made related to the methane potential of sludge from compact waste water treatment concepts. It is therefore suggested that further investigations are made. To begin with, it could be of interest to perform a biochemical methane potential test using the retentate from the microfiltration membrane with respectively without chemical precipitation and to compare the effects when including biological hydrolysis as apre-treatment step for the retentate.

Another interesting aspect would be to perform biochemical methane potential tests after a duration of the hydrolysis of e.g. three to four days, in order to find the optimal pre-treatment time before the anaerobic digestion. Furthermore, tests in full-scale with continuous digestion could be carried out as a way of evaluating the potential of biological hydrolysis to be used as a pre-treatment method to increase the methane production.

Since the sludge volume index proved not to be a sufficient method of evaluating the possibility to thicken the different sludge types, it might be interesting as well to further investigate other methods which could be used as well as what methods would be the most efficient to use when dealing with the different sludge types in question.

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Appendix I

Determination of the content of total solids, volatile solids, suspended solids and volatile suspended solids

Waste water contains a number of various contaminants with a wide particle size distribution. The organic particles found in waste water are composed of approximately one-third each of dissolved (substances smaller than 0.1 µm), colloidal (0.1-1.0 µm) and suspended (1-100 µm) particles whereas the particle size distribution of inorganic particles is mostly concentrated to dissolved substances (Kemira Kemwater, 2003). Due to the fact that a large part of the contaminants are too small to be separated from the waste water by mechanical means e.g. sedimentation, increased separation might be required. When treating waste water it is of interest to measure the content of solids, which is generally done by measuring the content of total solids, volatile solids, suspended solids and volatile suspended solids.

The total solids (TS) content indicates the remaining amount of compounds in a material after the water within has been evaporated at 105ºC (Carlsson and Uldal, 2009). By further treating the sample taken, in an oven at 550ºC, the volatile solids (VS) content may be determined. The VS content is commonly used to determine the organic content of e.g. a substrate used in anaerobic digesters. A high content of VS indicates a high biogas yield, as the VS constitutes the organic part of the TS which can be degraded in an anaerobic digester and consequently contribute to the production of biogas (Carlsson and Uldal, 2009).

For the TS measurements aluminium weighing dishes were marked and weighed individually on high-precision scales (0.01 and 0.0001 g) each time before being filled with a certain amount of sludge, a minimum of 30 g. The baking tins were then left in a furnace for 20-24 hours at 105ºC after which they were weighed again. The TS was then calculated as:

$$TS = \frac{b-a}{V}\ [g/l] \qquad \textit{Eq. 4}$$

a = weight of the weighing dish [g]

b = weight of the weighing dish and dried sample [g]

V = volume of the sample [l]

$$TS = \frac{100 \cdot c}{d}\ [\%] \qquad \textit{Eq. 5}$$

c = dry weight of the sample [g]

d = wet weight of the sample [g]

After the samples had been weighed the VS content (also denoted the loss on ignition, LOI) was determined by putting the samples in a muffle furnace set to 550ºC for 2 hours after which they were weighed once more and the values were calculated from Equation 3 respectively Equation 4 in order to obtain the results in the unit %. The residues after ignition are so-called fixed solids whereas the weight loss represents the VS.

$$VS = \frac{b - a}{V}\ [g/l] \qquad \textit{Eq. 6}$$

a = weight after ignition [g]

b = dry weight of the sample [g]

V = volume of the sample [l]

$$VS = 100 - fixed\ solids\ [\%] \qquad \textit{Eq. 7}$$

$$Fixed\ solids = \frac{100 \cdot c}{d}[\%] \qquad \textit{Eq. 8}$$

c = weight after ignition [g]

d = dry weight of the sample [g]

Moreover, samples were taken for the determination of the suspended solids (SS) content. Suspended solids are the solids which can be removed mechanically from the waste water, e.g. through sedimentation or filtration, in comparison to the dissolved solids which are Firstly, the filter papers (Munktell 0.45 µm micro-glass fibre filter papers for hydrolysis experiment 1-4 respectively VWR 1.6 µm filter papers for hydrolysis experiment 5) were weighed after which 10ml of sample was filtered through each filter, using the vacuum filtration equipment consisting of a glass funnel, glass connector, clamp and vacuum filter flask. Secondly, the filters were left to dry in an oven set to 105ºC for 20 hours. By measuring and weighing the residues, the concentration of SS was determined as:

$$SS = \frac{b - a}{V}\ [g/l] \qquad \textit{Eq. 9}$$

a = weight of the weighing dish and filter [g]

b = weight of the weighing dish with filter and filtrate [g]

V = volume of the sample [l]

Similarly to the method used for determining the VS, the SS samples were, after having been weighed, left in the furnace set to 550ºC for 2 hours. The VSS content could then be determined using Equation 10.

$$VSS = \frac{b - c}{V}\ [g/l] \qquad \textit{Eq. 10}$$

b = weight of the weighing dish with filter and suspended solids [g]

c = weight of the weighing dish with filter and suspended solids after ignition [g]

V = volume of the sample [l]

The SS, VSS, TS and VS concentrations were only measured at the start-up and the finish according to the methodology previously described following SS 02 81 13 and Svenska vatten- och avloppsverksföreningen (1984).

Determination of the chemical oxygen demand

Another way of estimating the organic content in waste water is to determine the chemical oxygen demand (COD), which is a measure of the amount of oxygen needed to degrade a certain amount of organic material. A high value of the COD indicates similarly to a high VS concentration, a high biogas yield (Carlsson and Uldal, 2009).

The chemical oxygen demand was measured using Hach Lange tests (Test LCK 114 respectively LCK 314), following the instructions written on the packaging in addition to a thermostat (Hach Lange LCK LT200) and a spectrophotometer (Hach Lange DR2800). The sample used for determination of the total COD had to be diluted initially in order to ensure results within the limits of detection. The sample was diluted 20 times by adding tap water while the sample used for COD_f was filtered through a Munktell micro-glass fibre filter paper with a pore size of 0.45 µm (hydrolysis experiment 1-4) respectively a VWR micro-glass fibre filter paper with a pore size of 1.6 µm for (hydrolysis experiment 5) using the vacuum filtration equipment consisting of a glass funnel, glass connector, clamp and vacuum filter flask. As the concentration of COD_f increased during the hydrolysis, the filtrated samples occasionally had to be diluted similarly to the total COD. A volume of 2 ml was pipetted into the Hach Lange-vials after which they were heated at 148ºC for two hours in the thermostat LT200-equipment. Once the vials had cooled off, the spectrophotometer was used to determine the COD values.

Figure I.1. Test LCK 114 (Hach Lange) for measuring of COD.

Determination of the sludge volume index

In order to monitor the settling characteristics of activated sludge and other biological suspensions, the sludge volume index (SVI) is applied. The SVI is determined by filling a 1 000 ml measuring cylinder with sludge, which is left to settle for 30 minutes. The volume in millilitres occupied by 1 g of a suspension after 30 minutes of settling represents then the SVI, which is calculated as follows (APHA, AWWA and WPCF, 1989).

$$SVI = \frac{settled\ sludge\ volume\ [ml/g] \cdot 1000}{suspended\ solids\ [mg/l]} \qquad Eq.\ 11$$

Appendix II

Figures illustrating the average filtered COD in mg/l as a function of time in days, during the hydrolysis of the various sludge types with respectively without chemical precipitation and the number of beakers, from which samples were withdrawn and the average calculated, within parenthesis.

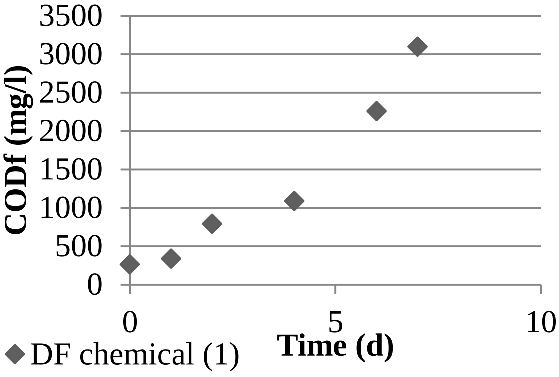

Figure II.1. Hydrolysis 1: Filtered COD in mg/l as a function of time during the hydrolysis of drum filter sludge with chemical precipitation (number of beakers within parenthesis).

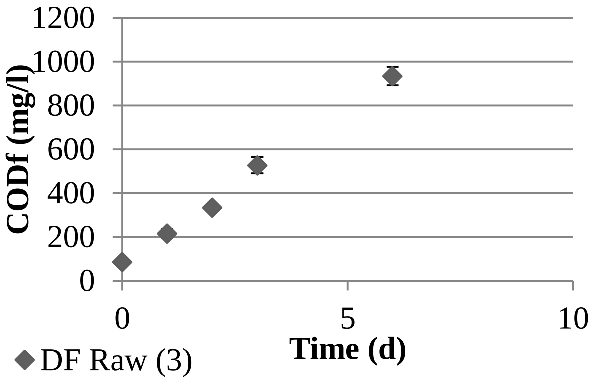

Figure II.2. Hydrolysis 2: Filtered COD in mg/l as a function of time during the hydrolysis of drum filter sludge without chemical precipitation (number of beakers within parenthesis).

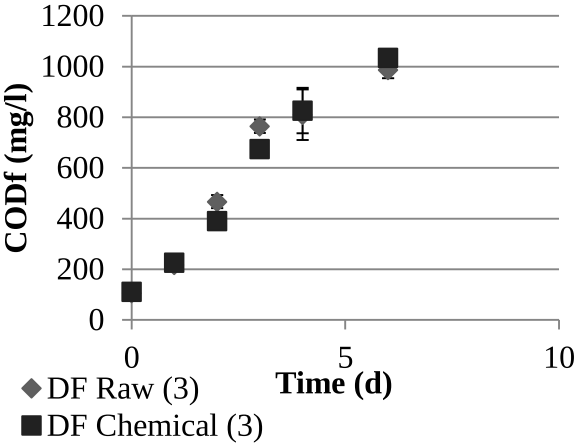

Figure II.3. Hydrolysis 3: Filtered COD in mg/l as a function of time during the hydrolysis of drum filter sludge without respectively with chemical precipitation (6.5mg/l XL100, 2.5mg/l (6456)) (number of beakers within parenthesis).

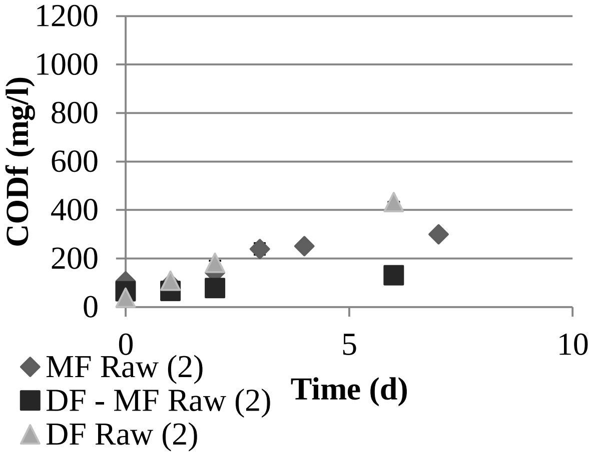

Figure II.4. Hydrolysis 4: Filtered COD in mg/l as a function of time during the hydrolysis of retentate from the membrane and drum filter sludge without chemical precipitation (number of beakers within parenthesis).

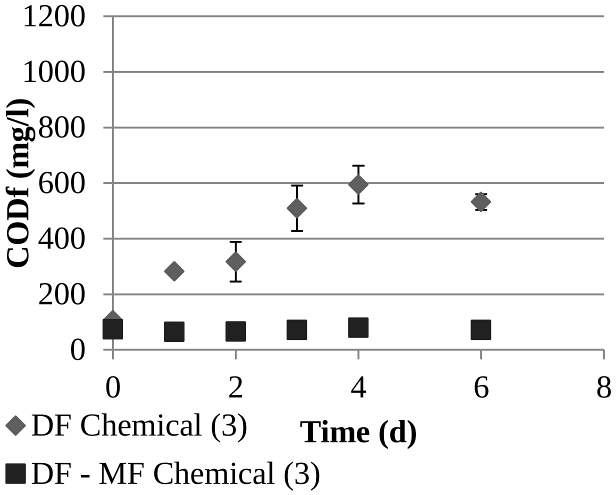

Figure II.5. Hydrolysis 5: Filtered COD in mg/l as a function of time during the hydrolysis of drum filter sludge and retentate from the membrane with chemical precipitation (number of beakers within parenthesis).

Appendix III

Figures illustrating the average concentration of the volatile fatty acids acetate and propionate in mg/l COD as a function of time in days, during the hydrolysis of the various sludge types with respectively without chemical precipitation and the number of beakers, from which samples were withdrawn and the average calculated, within parenthesis.

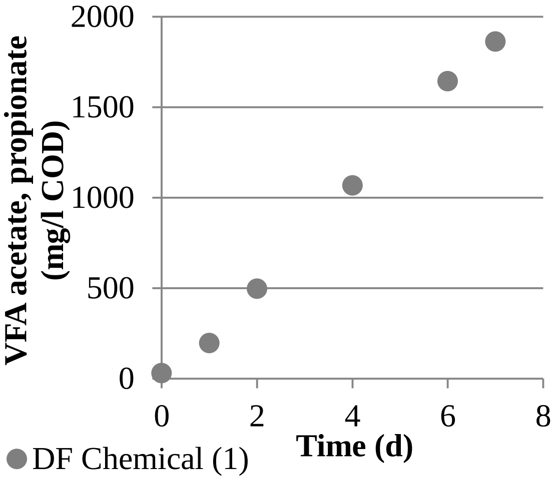

Figure III.1. Hydrolysis 1: The concentration of acetate and propionate (mg/l COD) in the chemically precipitated drum filter sludge as a function of the duration of the hydrolysis in days.

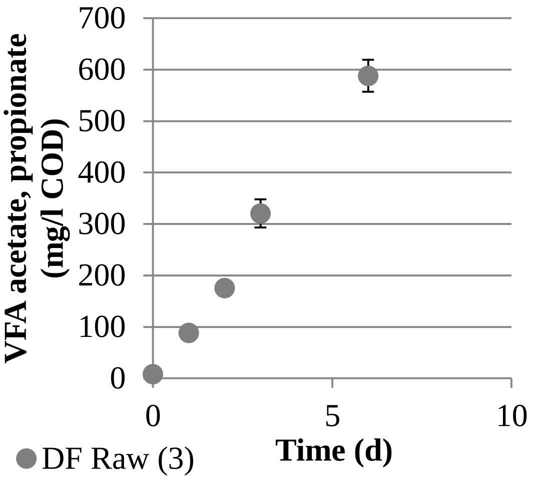

Figure III.2. Hydrolysis 2: The concentration of acetate and propionate (mg/l COD) in the raw drum filter sludge as a function of the duration of the hydrolysis in days.

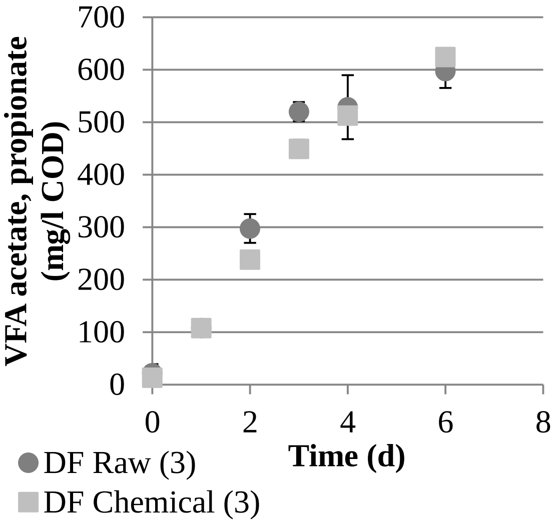

Figure III.3. Hydrolysis 3: The concentration of acetate and propionate (mg/l COD) in the raw and chemical drum filter sludge as a function of the duration of the hydrolysis in days.

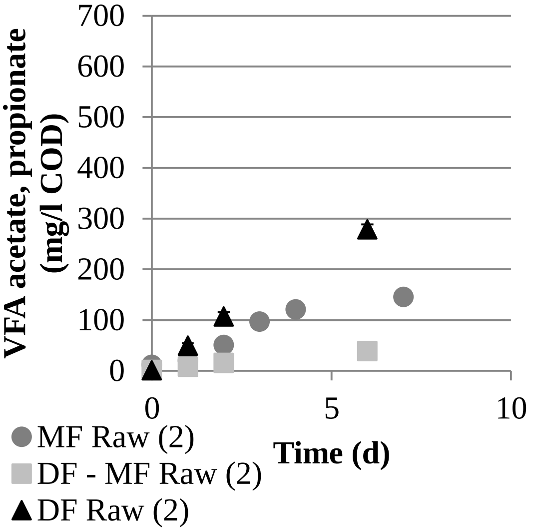

Figure III.4. Hydrolysis 4: The concentration of acetate and propionate (mg/l COD) in the raw and chemical drum filter sludge and MF retentate as a *function of the duration of the hydrolysis in days.*

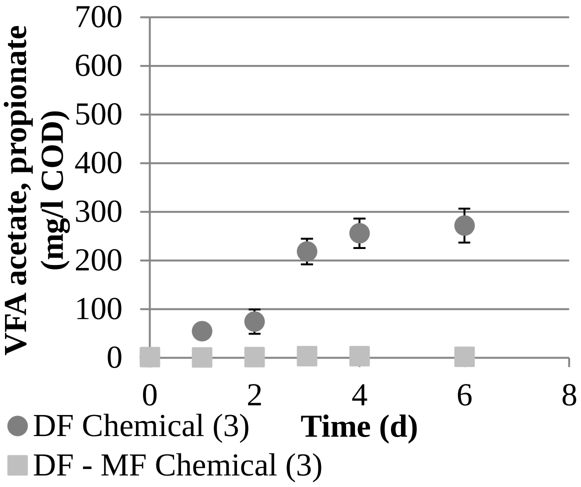

Figure III.5. Hydrolysis 5: The concentration of acetate and propionate (mg/l COD) in the drum filter sludge respectively the membrane retentate, with chemical precipitation, as a function of the duration of the hydrolysis in days.

Appendix IV

Table IV.1. The estimated respectively measured TS and VS content in BMP test 1 expressed as a percentage.

Notation	**Content**	**Estimated values**		**Measured values**	
		TS (%)	**VS (%) of the wet weight**	**TS (%)**	**VS (%) of the wet weight**
Y1	Inoculum	5.23	3.42	5.18	3.20
Y2	Inoculum	5.23	3.42	5.18	3.20
Y3	Inoculum	5.23	3.42	5.18	3.20
C1	Cellulose	99.00	94.00	99.00	94.00
C2	Cellulose	99.00	94.00	99.00	94.00
C3	Cellulose	99.00	94.00	99.00	94.00
R1	Raw DF sludge	0.50	0.40	4.30	0.33
R2	Raw DF sludge	0.50	0.40	4.30	0.33
R3	Raw DF sludge	0.50	0.40	4.30	0.33
K1	Chemical DF sludge	0.80	0.50	4.20	0.31
K2	Chemical DF sludge	0.80	0.50	4.20	0.31
K3	Chemical DF sludge	0.80	0.50	4.20	0.31
B1	Mixed sludge from Källby WWTP	6.00	4.44	7.30	5.92
B2	Mixed sludge from Källby WWTP	6.00	4.44	7.30	5.92
B3	Mixed sludge from Källby WWTP	6.00	4.44	7.30	5.92

Table IV.2. The estimated respectively measured TS and VS content in BMP test 2 expressed as a percentage.

Notation	**Content**	**Estimated values**		**Estimated values**	
		TS (%)	**VS (%) of the wet weight**	**TS (%)**	**VS (%) of the wet weight**
I1	Inoculum	5.03	3.20	4.89	3.17
I2	Inoculum	5.03	3.20	4.89	3.17
I3	Inoculum	5.03	3.20	4.89	3.17
H1	Hydrolysed raw DF sludge	0.44	0.33	0.27	0.21
H2	Hydrolysed raw DF sludge	0.44	0.33	0.27	0.21
H3	Hydrolysed raw DF sludge	0.44	0.33	0.27	0.21
H4	Hydrolysed chemical DF sludge	0.42	0.31	0.34	0.24
H5	Hydrolysed chemical DF sludge	0.42	0.31	0.34	0.24
H6	Hydrolysed chemical DF sludge	0.42	0.31	0.34	0.24

Appendix V

Case study for Källby WWTP

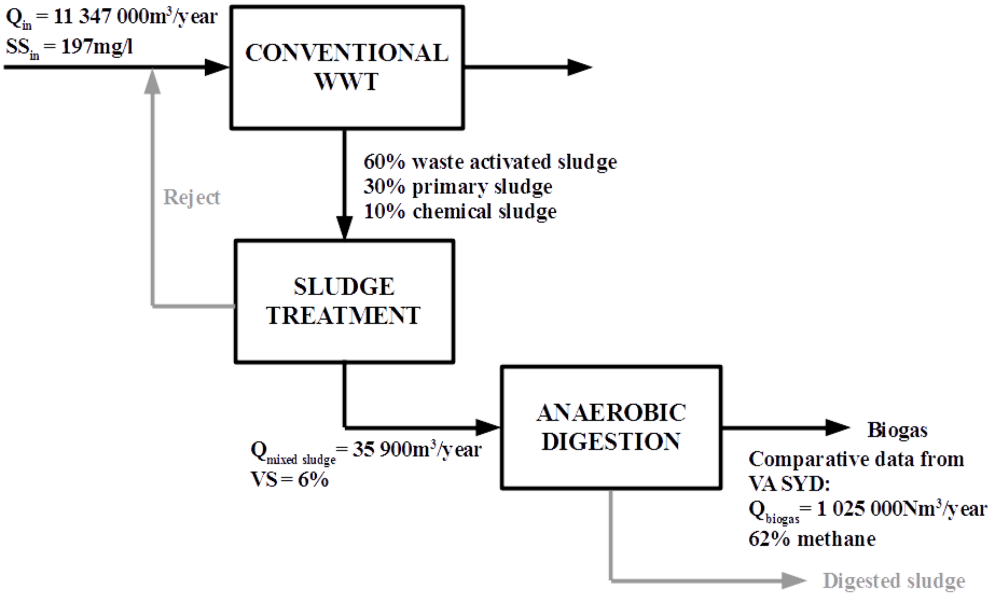

Figure V.1. A simplified process scheme of the conventional waste water treatment process at Källby WWTP and the figures used for the calculations (Hey, 2015; Murto, 2015).

The incoming amount of suspended solids in metric tons per year is 2 235 tons/year and by assuming a density of 1 000 kg/m^3 of the sludge the mass of mixed sludge going into the anaerobic digesters is 35 900 tons/year with a VS content of 6%. Taking into consideration the methane potential for the mixed sludge (307 Nm^3/ton VS) from the first BMP test in this study, the yearly production of methane is

$$35\,900 \cdot 0.06 \cdot 307 = 661\,278 Nm^3/year$$

By comparing the estimated methane production to the current methane production at Källby WWTP, which is 62% of 1 025 000 Nm^3/year i.e. 635 500 Nm^3/year, it can be seen that the actual methane production is 96% of the estimated. A reasonable result as the methane production achieved in a BMP test is usual higher than the one achievable in reality, due to a prolonged retention time and optimal conditions.

Concerning the amount of sludge produced by the drum filter, a removal efficiency of approximately 98% has been shown. By utilising the same data as for the incoming waste water characteristics to the conventional waste water treatment process, an incoming amount of suspended solids of 2 191 tons/year is found. After having converted the amount of suspended solids into the amount of volatile suspended solids (based on the measurements made) and performed the same calculations as for the conventional process, the resulting yearly production of methane in Nm^3 can be found in Table V.1.

Table V.1. The conversion and calculation of the yearly production of methane for each sludge type.

Sludge type	kg TS/kg SS	kg VS/kg SS	ton VS/year	Methane potential (Nm^3/ton VS)	Yearly methane potential (Nm^3/year)
DF raw	1.14	0.86	1 884	366	689 530
DF chem	1.09	0.80	1 753	457	800 902
DF raw hydrolysed	1.24	0.80	1 753	512	897 291
DF chem hydrolysed	1.20	0.84	1 840	462	850 148

Due to the methane potential of the microfiltration membrane retentate not having been measured, assumptions have to be made. When utilising direct membrane filtration a removal efficiency of nearly a 100% of the suspended solids was achieved. Consequently, a larger amount of organic matter is extracted from the waste water into the sludge, most likely leading to a higher methane potential as well. The methane potential would evidently depend on the chemical precipitation applied, whether biological sludge hydrolysis is utilised as well as on the microorganisms present. However, a methane potential of 480 Nm^3/ton VS is assumed as it is within the interval for the chemically precipitated drum filter sludge and the hydrolysed raw drum filter sludge. Under the assumption of the raw MF retentate having a methane potential of 480 Nm^3/ton VS and a removal efficiency of 100% the following results in Table V.2 are found.

Table V.2. The conversion and calculation of the yearly production of methane for the retentate from the membrane.

Sludge type	kg TS/kg SS	kg VS/kg SS	ton VS/year	Methane potential (Nm^3/ton VS)	Yearly methane potential (Nm^3/year)
MF raw	1.38	0.81	1 811	480	869 108

As a consequence of there being no values for the content of SS, TS and VS in the sludge for chemically precipitated MF retentate, due to lack of time, calculations are only performed for the raw MF retentate.

A summary of the estimated yearly methane potential in Nm^3/year for each sludge type is shown in Table V.3, starting with the lowest methane production. The values in the third column were obtained by multiplying the yearly methane potential with an estimated factor of 0.9, which has to be taken into consideration as the methane potential achieved in BMP tests is higher than in reality due to the retention time being longer and more optimal process conditions. As the actual methane production from the conventional mixed sludge at Källby WWTP represented 96% of the methane potential of the same sludge in the BMP test, a factor slightly higher than normal was assumed. Further, due to the sludge having a retention time of 30 days in the anaerobic digesters at Källby WWTP a higher factor can be considered as justified.

Table V.3. The yearly estimated methane production in Nm^3/year for each sludge type as well as the results multiplied with a factor of 90%.

Sludge type	Yearly methane potential (Nm^3/year)	Yearly methane potential (Nm^3/year)
Data from Källby WWTP	635 500	Not multiplied with a factor
Mixed sludge from Källby WWTP	661 278	595 150
DF raw	689 530	620 577
DF chemical	800 902	720 812
DF chemical hydrolysed	850 148	765 133
DF raw hydrolysed	897 291	807 562

Appendix VI

Basic statistics

Table VI.1. Parameters and formulas used for statistics analysis (Gordus, 1985).

Parameter	Formula
Average (arithmetic mean):	$\bar{x} = \frac{\sum x_i}{N}$
Standard deviation:	$s_x = \sqrt{\frac{\sum(x_i - \bar{x})^2}{N-1}}$

Appendix VII – Popular science article in Swedish

Ökad biogasproduktion med innovativ kompakt avloppsvattenrening

Ökande utsläppskrav i takt med ökande belastning på reningsverken, leder till högre kostnader och att mer energi och kemikalier kommer att krävas med dagens reningsmetoder. Ett nytänkande, kompakt reningskoncept som inte bara resulterar i renare vatten utan även i att mer biogas kan utvinnas borde väl därmed vara intressant?

Algblomning orsakad av övergödning är ett årligen återkommande problem i Östersjön med konsekvenser som dålig badvattenkvalité och bottendöd. Varje år släpps en stor mängd näringsämnen och organiskt material ut till våra vattendrag med avloppsvattnet, vilket bidrar till övergödningen. Näringsämnen som istället hade kunnat tillvaratas för att återcirkuleras och spridas på åkrar i syfte att ersätta konstgödsel och andra på kemisk väg framställda ämnen.

Vidare innehåller avloppsvattnet inte bara näringsämnen utan även läkemedelsrester och tungmetaller som inte går att ta hand om med nuvarande reningsmetoder. Detta i samband med att städerna breder ut sig, markpriserna stiger samt kostnaderna, belastningen och utsläppskraven på reningsverken ökar, gör att nya innovativa och resurseffektiva lösningar krävs.

I projektet *Den varma och rena staden* testas därför för närvarande en innovativ pilotanläggning för kompakt avloppsvattenrening på Källby avloppsreningsverk i Lund. Tanken är att organiska föroreningar ska kunna avskiljas mekaniskt med hjälp av tre olika reningssteg.

Pilotanläggningen består av ett trumfilter, vilket är en roterande trumma klädd med en filterduk som filtrerar bort grövre partiklar ur avloppsvattnet, följt av ett mikrofiltermembran, som avskiljer resterande finare partiklar i avloppsvattnet samt ett biomimetiskt membran för osmos. Det biomimetiska membranet är ett cellmembran med transportproteiner, så kallade aquaporiner, och liknar de cellmembran som finns i njurarna på oss människor för att koncentrera urinen. Tanken är att endast vattenmolekyler ska kunna passera, vilket gör att bara rent vatten (och avloppsslam) kommer ut från pilotanläggningen.

En av fördelarna med kompakt avloppsvattenrening är att mer partiklar och organiskt material kan tillvaratas än med konventionella reningsmetoder. Vanligtvis omvandlas nämligen, genom energikrävande luftning av bassängerna, en stor del av det organiska materialet och kvävet till koldioxid och kvävgas som avges direkt till atmosfären och bidrar till växthuseffekten.

Partiklarna som avskiljs i reningsprocessen bildar istället ett slam ur vilket värdefulla näringsämnen kan utvinnas. Slammet kan sedan rötas för att producera biogas. Biogasen kan i sin tur sedan uppgraderas för att användas som förnyelsebar energikälla och drivmedel för bilar.

För att ytterligare öka mängden producerad biogas alternativt för att snabba på rötprocessen kan så kallad biologisk hydrolys användas som förbehandling av slammet. Hydrolysen är det första steget i rötprocessen, där stora partiklar såsom kolhydrater, proteiner och fetter bryts ner till mindre partiklar av bakterier och andra mikroorganismer.

Rötförsök och hydrolysförsök har genomförts i labbskala för att utvärdera biogaspotentialen för slammet från den kompakta pilotanläggningen, vilket har visat på positiva resultat. Både ohydrolyserat och hydrolyserat slam från den kompakta pilotanläggningen hade högre biogaspotential än slammet från konventionellt renat avloppsvatten.

Kompakt avloppsvattenrening skulle därför kunna utgöra en tänkbar lösning för att minska övergödningen i våra vattendrag och öka mängden producerad biogas.

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