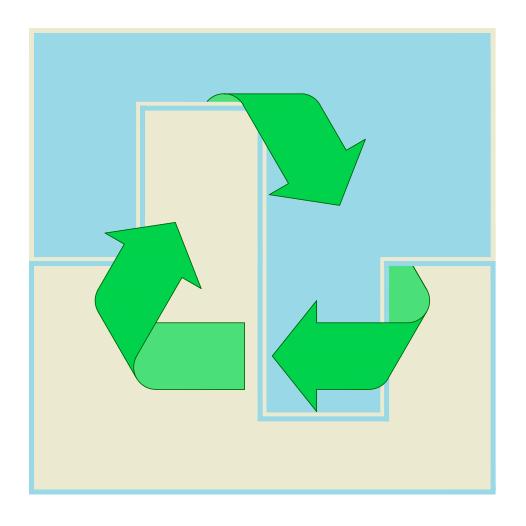
# Pulse Electric Field as a Pre-treatment Method of Wastewater Sludge prior to Anaerobic Digestion





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Water and Environmental Engineering Department of Chemical Engineering Master Thesis 2020

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by

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Master Thesis number: 2020-08

Water and Environmental Engineering
Department of Chemical Engineering
Lund University

June 2020

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Picture on front page: Drawing by Milton Matilde.

### **Preface**

This work marks the conclusion of my master's program in Water Resources Engineering at Lund University, where I was able to gain knowledge and experience in topics that are very important to me. Additionally, this experience has awarded me with new friendship, so thank you to all my classmates for these shared years. A hearty thanks to all the professors and lecturers that though us so much through this program.

I would like to express my gratitude to Arc Aroma Pure AB, specially to Fredrik Cedmert for allowing me to write this thesis on the work I have done with them and to Ivo achu Nges for all the support and guidance throughout this period.

To my supervisors Åsa Davidsson and Mirjam Victorin, thank you greatly for all the help, suggestions and for supporting the decision of finishing this work even during these adverse times. Thank you also to all the staff in the Department of Chemical Engineering at Lund University.

A loving thanks to my family, specially my mother Vânia, my father Jorge, my sisters Luciana and Isabella and my aunt Eliane, for believing in me and always being present, even from the other side of an ocean. And finally, to Julia Moreira, thank you for always being there and for every shared moment, you make wherever we are together home.

## Use of Pulse Electric Field as a method to increase biogas production from wastewater sludge

The process of treating wastewater generates sludge that needs to be disposed according to governmental legislations. Generally, the sludge needs to be further treated to ensure a number of parameters are below the allowed threshold values. One important parameter is the amount of organic material present in the sludge and degrading that material is also called sludge stabilization. The treatment and disposal of sludge can be the single most expensive step on wastewater treatment systems, for that reason, possibilities, methods and techniques that can help reducing that cost are constantly being pursued.

Anaerobic digestion (AD) degrades organic material generating biogas and it is one of the most common methods used within the European Union (EU) to stabilize wastewater sludge. This method transforms solid organic material (biomass) into biogas, decreasing the amount of organic material present in the sludge while producing clean energy. This process typically takes place in a digester, where sludge is submitted to a heated anaerobic environment (without oxygen). Pre-treatments are often used in order to optimize the anaerobic digestion maximizing biomass degradation and biogas production.

Pulsed Electric Fields (PEF) is a commonly used technology in the food industry to increase juice and olive oil extract, however, studies show that it also has the potential to be used as a sludge pre-treatment method as help increase biogas production. PEF consists of applying energy to the substrate being treated in order to generate pores into the cell's membrane (electroporation), making its content more easily available. Subjecting the wastewater sludge to this treatment prior to anaerobic digestion can help the microorganisms responsible for digesting the biomass have easier access to that organic material, increasing the overall biogas production.

PEF treatment can be characterized by a number of different parameters, one of them being treatment intensity, which is the energy input required from the treatment, generally expressed as energy/mass (J/kg).

This study compared the biogas production obtained from the digestion of sludge pre-treated by four different intensities with the gas produced from the digestion of untreated sludge and results showed that PEF-treated sludge generated up to 7.3% more biogas than untreated sludge and degraded around 7% more organic material.

Another impact of PEF treatment, besides electroporation, is the increase in the substrate's temperature. This can lower the energy required to heat the sub-sequent anaerobic digester, helping offset the energy required by the PEF treatment.

### Summary

This work analyzed the impact caused by different intensities of Pulsed Electric Fields (PEF) pre-treatment on the biogas production from small-scale anaerobic digesters of wastewater sludge under thermophilic conditions. PEF technology consists of passing the substrate through an electric field which generates a potential difference across the cells, resulting in electroporation.

Mixed sludge from Källby wastewater treatment plant was used and the biogas yield (Nm³/kg VS) obtained from the semi-continuous lab digesters with untreated sludge was similar to the yield registered by the plant's full-scale version. Substrate submitted to higher intensities exhibited higher biogas yield improvements. The digestion of PEF-treated sludge reached values of biogas production up to 7.4% higher than values obtained from the digestion of untreated sludge with the same solids content. No relevant difference was seen in the methane content of the biogas produced by treated and untreated substrate.

PEF treatment causes an increase in temperature in the substrate. That energy can be used to reduce the external heating requirements of the digester, contributing to offset the energy input required from the treatment. In this study, treatments with intensity around 70 kJ/kg were shown to increase the temperature of the sludge by around 18°C.

Higher cumulative biogas production and VS degradation were achieved by increasing treatment intensity. Increases of 7.3% in cumulative biogas production and 7% in VS reduction were obtained from the anaerobic digestion of mixed primary and secondary sludge pre-treated with intensity of 95 kJ/kg. The increase in substrate temperature caused by the pre-treatment can decrease the energy required for heating the subsequent anaerobic digester and contribute to offsetting the energy required from the pre-treatment.

The treatments conducted in this work only differed in treatment intensity. However, PEF treatment design consists of many other parameters. Thus, the relationship between these parameters and anaerobic digestion should be further investigated so that the true impact of Pulsed Electric Fields can be determined. Additionally, the performance of PEF varies from substrate to substrate, so methods to determine optimal parameters based on sludge type should be developed.

Key words: PEF, AD, biogas yield, sludge pre-treatment

#### **Abbreviations**

AD – Anaerobic Digestion

COD – Chemical Oxygen Demand

CSRT – Continuous Stirred-Tank Reactor

DOC – Dissolved Organic Carbon

EU – European Union

PEF – Pulsed Electric Field

SCOD – Soluble Chemical Oxygen Demand

SRT – Solids Retention Time

TS – Total Solids

VS – Volatile Solids

WAS – Waste Activated Sludge

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### 1 Introduction

The treatment and disposal of sludge can be the single most expensive step on wastewater treatment systems (Davis, 2010), for that reason, possibilities, methods and techniques to help reduce that cost are always being pursued.

Anaerobic digestion (AD) degrades organic material generating biogas and is commonly used in the EU as a sludge stabilization process at wastewater treatment plants (Hudcová et al., 2018) and one of its main advantages is having a positive energy balance (Nges, 2012). The production of clean energy has been in the interest of companies and governments due to the urgent need of replacing fossil fuels by more sustainable options, for example, in the EU there are various energy and climate policies that promote the use of biogas as energy source (Scarlat et al., 2018). In addition, the disposal of sludge will possibly become stricter in the future (Kelessidis and Stasinakis, 2012), forcing wastewater treatment plants to increase organic material degradation in order to be able to legally dispose their sludge. Thus, the need for new technologies that can contribute to the efficiency of anaerobic digestion.

Pulsed Electric Field (PEF) technology has been practiced in the food and bioengineering industries since the 1960s (Toepfl et al., 2007), and more recently it has been thought as a pretreatment method for wastewater sludge to increase the degradation and biogas yield from anaerobic digestion (Kopplow et al., 2004). PEF consists of applying electric fields to a substrate and therefore generating a potential difference across its cells that can result in electroporation (Golberg et al., 2016).

Some studies have been published with analyses of the potential of PEF as a sludge pretreatment with promising results, such as Rittmann et al. (2008) and Salerno et al. (2009). However, more still needs to be learned about the impact this treatment can have on the anaerobic digestion of sludge, for example if there are specific conditions, such as temperature regimes, where this treatment can be expected to deliver higher or lower impacts or how to determine the best treatment parameters according to sludge characteristics.

Since PEF consists of applying energy to a substrate, aside from the main purpose of the treatment, it also causes an increase in temperature on the treated substrate. When applying this technology as a sludge pre-treatment method, the generated heat can be recovered in order to decrease the energy requirements to heat a subsequent digester, as argued by Salerno et al. (2009).

This study investigates the performance of a small-scale thermophilic digester semi-continuously fed with sludge treated using PEF technology and comparing them to the performance obtained from a second digester fed with untreated sludge. The sludge was obtained from Källby wastewater treatment plant in Lund, Sweden, and treated, as further detailed, in the lab at Arc Aroma Pure AB.

#### 1.1 **Aim**

The aim of this study is to analyze the impact of PEF treatment intensity on the biogas yield during the anaerobic digestion of wastewater sludge under thermophilic conditions. And, secondly, to investigate if the energy used for the treatment can be partially recovered to help off-set the energy requirements of heating the anaerobic digester.

### 2 Theoretical background

#### 2.1 Anaerobic Digestion

The biogas process can be divided into four steps and even though they are presented as being sequential in nature, all the steps happen simultaneously and synergistically in an anaerobic digester (Davis, 2010), for that reason all the different microorganisms have to be allowed enough time to perform their reactions. A pH between 6.5 and 7.5 is desirable to maintain equilibrium in the digesters, since acidogenesis is optimal at pH 6 while acetogenesis and methanogenesis are optimal around pH 7.5 (Davidsson, 2007).

#### 2.1.1 Hydrolysis

Polymeric compounds such as carbohydrates, proteins and lipids are the main components present in biomass (Li, 2017) and for that reason hydrolysis is often the limiting step when it comes to the anaerobic digestion of wastewater sludge (Speece, 1996). In this step these compounds are hydrolyzed into smaller particles by enzymes produced by microorganisms that can be obligated or facultative anaerobes (Davidsson, 2007), the facultative have an important role in this step since they consume the remaining oxygen in the system (Björnsson et al., 2000). The results are water soluble compounds like sugar, amino acids and long-chain fatty acids (Nges, 2012).

#### 2.1.2 Acidogenesis

A variety of facultative and obligates fermentative microorganisms transform the hydrolysis products into organic acids, alcohols, water and carbon dioxide. The organic acids generated by this step is also called electron sink (Nges, 2012). In a balanced anaerobic digestion of complex organic matter, acidogenesis is typically the fastest step (Vavilin et al., 1996).

#### 2.1.3 Acetogenesis

All microorganisms responsible for acetogenesis are strict anaerobes (Li, 2017). Those microorganisms react with the long-chain fatty acids and volatile fatty acids generated by the acidogenesis to form acetate, hydrogen gas and carbon dioxide (Davidsson, 2007). Hydrogen accumulation may inhibit acetogens (Nges, 2012).

#### 2.1.4 Methanogenesis

Methanogens, also obligated anaerobes, are divided into acetotrophic and hydrogenotrophic (Nges, 2012), the first uses acetate to produce methane and carbon dioxide while the second consumes carbon dioxide to produce methane and water (Davidsson, 2007). In anaerobic digesters acetotrophic methanogens are typically responsible for 70% of the total methane production (Li, 2017). Since methanogens are not able to utilize electron sink and the accumulation of hydrogen can inhibit acetogens it is important that these processes are well balanced in an anaerobic digester (Nges, 2012). It is during this step that true stabilization of the organic material occurs (Davis, 2010). Methanogenesis is considered the slowest step in the anaerobic digestion process (Anderson et al., 2003).

#### 2.2 Anaerobic Digestion of Wastewater Sludge

Anaerobic digestion is one of the most common methods used for stabilization of wastewater sludge among EU countries (Hudcová et al., 2018) and has the advantage of producing biogas while reducing organic content (Davidsson, 2007), other positive effects of this process are volume reduction of up to 50%, destruction of pathogens, removal of bad odor (Gebreeyessus and Jenicek, 2016) and improved efficiency of a possible subsequent dewatering step (Davis, 2010).

Two temperature regimes are used for anaerobic digestion, mesophilic (30°C – 38°C) and thermophilic (50°C – 57°C). Thermophilic conditions typically show higher reaction rate which allows for smaller digesters, higher solids and pathogens destruction. However, mesophilic temperature is used in most cases due to its lower energy requirements and higher process stability (Davis, 2010). On the other hand, thermophilic digesters are more aligned with modern environmental legislations (Gebreeyessus and Jenicek, 2016), for example, the United States Environmental Protection Agency says that substrate submitted to 50°C or higher for 20 minutes or longer can be classified as "Class A" bio-solid products (U.S. Environmental Protection Agency, 1994).

Wastewater sludge characteristics may vary between plants but always depend on the process that originates it. Conventionally, anaerobic digesters are fed with a mix of primary, biological and sometimes chemical sludges (Gebreeyessus and Jenicek, 2016). Although primary and waste activated sludge (WAS) have around the same VS/TS ratio of 60% to 80%, where VS is volatile solids and TS is total solids, the TS content in primary sludge ranges between 2% and 8% while WAS has around 1% (Davidsson, 2007), which indicates that primary sludge has an overall higher VS content. In addition, studies show that activated sludge with long sludge age show lower anaerobic degradability when compared to sludges with shorter solids retention time (SRT), that can be explained by the accumulation of inert materials from the influent and decay products of the treatment in the sludge (Ge et al., 2013). Anaerobic degradability of WAS is estimated to vary from 30% to 50% (Parkin and Owen, 1986).

The anaerobic process can be improved by pre-treating the substrate and increase accessibility of the organic matter to the microorganisms leading to improved hydrolysis (Davidsson, 2007) which can be of high relevance for wastewater treatment plants since, as mentioned before, that is typically the limiting step in anaerobic digestion of wastewater sludge (Speece, 1996). There are a large variety of pre-treatment methods that aim on decreasing final sludge amount and enhancing biogas production. Some other effects they may have are increased degradation rate, decrease required retention time and consequently digester volume (Carrere et al., 2010). Pre-treatments can be divided into three main groups: Chemical, Physical and Biological. Figure 2.1 shows some methods that can be applied to improve anaerobic digestion (Davidsson, 2007).

Chemical	Physical	Biological
NaOH	Thermal	Bacterial hydrolysis
КОН	Freezing/Thawing	Enzyme addition
Mg(OH) <sub>2</sub>	Ultrasonic treatment	Aerobic pre-treatment
Ca(OH)₂	Pulsed electric fields	
Acid	Milling	
Oxidation ( $O_3$ or $H_2O_2$ )	High pressure	
	Homogenisaton	
	Lysate centrifuge	

Figure 2.1. Sludge pre-treatment methods that can be used to improve anaerobic digestion. (Davidsson, 2007).

Most pre-treatment methods are aimed towards activated sludge because of its lower anaerobic degradability, however they are also used on primary sludge to increase dewaterability and pathogen removal (Carrere et al., 2010). According to Davidsson and la Cour Jansen (2006), thermal treating sludge at 70°C for 1 hour prior to anaerobic digestion can result in a 10%-20% increase in methane production. Enzyme addition can improve the biogas yield by up to 26% (Bonilla et al., 2018). And oxidation is reported by Carrere et al. (2010) to have increased methane production from anaerobic digestion of mixed sludge by 100%.

#### 2.3 Pulsed Electric Field

Pulsed Electric Field is a treatment method used in many different fields; it consists of applying high voltage pulses through the substrate which can be done by allowing it to pass between two electrodes. One outcome of this treatment is what is called electroporation (Golberg et al., 2016) represented in Figure 2.2. When subjected to high voltage, opposite electrical charges appear on each side of the cell membrane due to its electrically insulating properties. If a certain electric field intensity is reached, formation of pores can be observed due to the pressure on the membrane (Kopplow et al., 2004). When it comes to substrate characteristics, Rittmann et al. (2008) observed increases of 160% and 120% in soluble chemical oxygen demand (SCOD) and dissolved organic carbon (DOC), respectively when pre-treating a mix of primary and secondary sludge.

Electroporation can be reversible, when the cell is capable of restoring its original permeability or irreversible, when the pore formation is extreme enough that the cell is uncapable of restoring its membrane, resulting in the cells death (Golberg et al., 2016). Therefore, this method can also be used to kill desired microorganisms. The electric field intensities required to achieve reversible and irreversible electroporation depend on multiple parameters such as treatment time and cell size, shape and orientation (Valič et al., 2003).

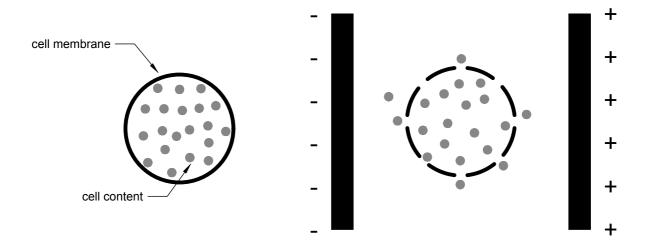


Figure 2.2. Formation of pores on cell membrane due to electric field – Electroporation.

Equations (1) and (2) show an analytical description derived by P. H. Schwan to determine the voltage through membrane assuming spherical shape, constant membrane thickness and conductivity. If a nonconductive membrane is assumed, f will assume the value of 1(one) (Schwan, 1957).

$$\Delta \Phi = \frac{3}{2} f E R \cos(\varphi) \tag{1}$$

Where  $\Delta\Phi$  is the induced transmembrane voltage, E is the external electric field, R is the radius of the cell and  $\Phi$  is the angle between the center of the cell and the direction of the field.

$$f = \frac{\sigma_e[3dR^2\sigma_i + (3d^2R - d^3)(\sigma_m - \sigma_i)]}{\left[R^3(\sigma_m - 2\sigma_e)\left(\sigma_m + \frac{1}{2}\sigma_i\right) - (R - d)^3(\sigma_e - \sigma_m)(\sigma_i - \sigma_m)\right]}$$
(2)

Where  $\sigma_i$ ,  $\sigma_m$  and  $\sigma_e$  are electric conductivities of the cytoplasm, cell membrane and external medium, respectively, and d is the membrane thickness.

According to Kotnik et al. (1997) the difference between the values obtained from the complete and simplified equations are considerably small making the use of the simplified version more reasonable. Further studies have been conducted by Kotnik and Miklavčič (2000) to derive a more complex description capable of determining transmembrane voltage on any spheroidal cell but are not going to be covered in this work.

#### 2.3.1 Electrical description

Electric pulses can be applied in several different shapes; according to Picart and Cheftel (2003) exponential decay and square pulses are the most common. In exponential decay pulses, the voltage is increased rapidly until it reaches the maximum value and slowly decreases to zero, therefore the substrate is not subjected to a specific electric field but a range of electric fields. On the other hand, when applying square pulses, the voltage is maintained at a maximum value for the duration of the pulse length allowing the substrate to be subjected to the maximum electric field for a longer period. Figure 2.3 illustrates the two types of pulses. Only square waves were used for the experiments described in this work.

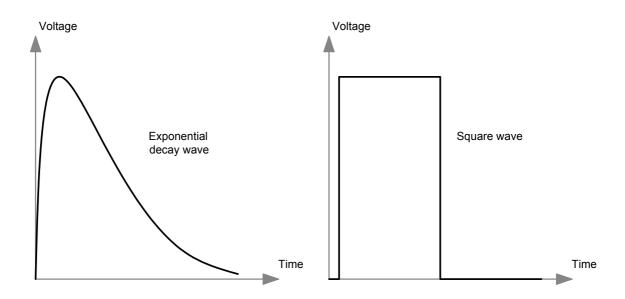


Figure 2.3. Illustration of exponential decay and square pulses.

The power consumption on a circuit with constant (steady) current can be expresses in Equation (3). This relationship can be used to determine the power consumption of a square pulsed electric field since square pulses are nothing more than a constant current being turned on and off repeatedly. For that description two other parameters are needed; frequency (F) which represents how many pulses are applied per unit of time and pulse length (w) which is the time between pulses start and stop, those parameters can be seen in Figure 2.4.

$$P_{steady} = V \cdot I \tag{3}$$

Where *P* is power in watts, *V* is voltage in volts, and *I* is current in amperes.

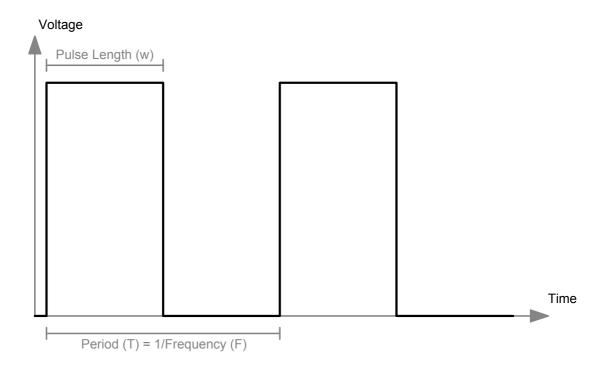


Figure 2.4. Illustration of Pulse Length and Frequency on square pulses.

Knowing the parameters listed before and that a pulsed circuit can be considered as a fraction of a constant circuit of same voltage and current, the power consumption of pulsed circuit can be described in Equation (4) with frequency expressed in hertz and pulse length in seconds.

$$P_{pulsed} = V \cdot I \cdot F \cdot w \tag{4}$$

Additionally, from Ohm's law, represented in Equation (5), the power consumption can also be expressed as a function of the substrate's resistance (*R*) expressed in ohms.

$$V = R \cdot I \tag{5}$$

Electric pulses can be monopolar or bipolar. On monopolar treatments one electrode is always charged as positive and the other as negative charge. Bipolar pulses are applied when alternating the electrodes charges between positive and negative. Figure 2.5 illustrates the relationship between electrodes charges and the formation of pulses.

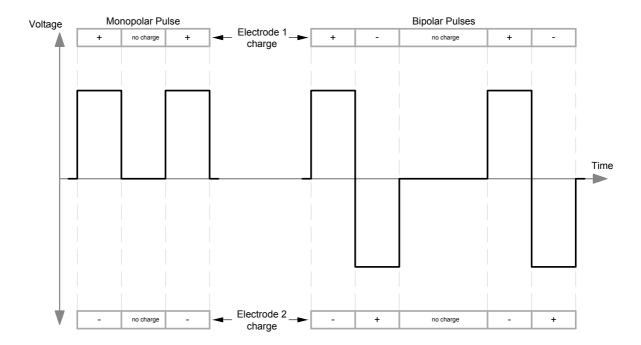


Figure 2.5. Relationship between Monopolar and Bipolar pulses and electrode charges.

Additional parameters are the gap between the electrodes (L) and, when applying PEF treatment through a continuously flowing substrate, the active volume of the treatment chamber  $(V_A)$  and the substrate flow rate (Q) through the chamber. With those parameters it is possible to determine the electric field strength (EF) and the treatment time (t), i.e. the average time each cell remains inside the active volume, according to Equations (6) and (7), respectively.

$$EF = \frac{V}{L} \tag{6}$$

$$t = \frac{V_A}{Q} \tag{7}$$

The PEF treatment intensity (TI), i.e. energy consumption is expressed in J/kg and is defined in Equation (8).

$$TI = \frac{P}{O} \tag{8}$$

### 3 Methodology

#### 3.1 PEF Treatment

The Pulsed Electric Field treatment was conducted in Arc Aroma's laboratory using a dynaCEPT® designed and produced by Arc Aroma Pure AB, Figure 3.1 is an illustration of the machine. As mentioned before, only square waves were used in this work.

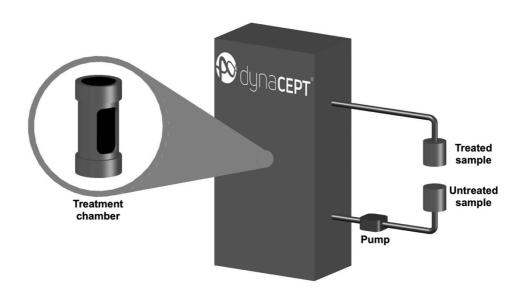


Figure 3.1. Illustration of dynaCEPT®.

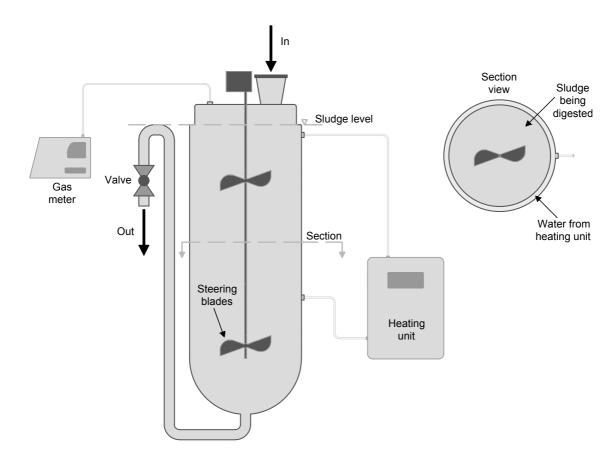
During the period where the lab digesters were running, sludge was collected from Källby every three days. A fraction of it was pre-treated and the remaining fraction was passed through the dynaCEPT® with the PEF technology turned off. This procedure was chosen in order to minimize differences in experimental error between the untreated and pre-treated substrate. Then, both treated and untreated samples were stored in the fridge and used as feed substrate to the digesters for the next couple of days. The intensities analyzed are described in Table 3.1.

Additionally, the temperature of a series of samples were measured before and after PEF treatments with intensity of around 70 kJ/kg in order to evaluate the energy recovery potential of the treatment.

#### 3.2 Anaerobic Digesters

In order to perform this experiment, two continuous stirred-tank reactors (CSRT), each with 10 liters capacity, were set up at Arc Aroma's laboratory. They were fed every day in a semi-continuously manner with sludge collected from Källby. The retention time chosen for this test was 10.5 days, to mimic the average retention time at the thermophilic process at Källby wastewater treatment plant. Thus, 952 mL of sludge were fed to each digester every day while digested sludge was removed in order to maintain the same volume (10 L). The digesters were sealed to avoid aerobic environment. The inlet at the top was covered by a metal lid with rubber seal and the outlet was controlled by a valve. They remained completely closed and were only opened during the feeding.

The digesters were kept at a temperature of  $55^{\circ}$ C by a water bath connected to a heating and pumping machine and a gas meter (model  $\mu$ Flow from Bioprocess Control) was attached to each digester in order to read the produced biogas. Figure 3.2 is a representation of the digesters' setup.



*Figure 3.2. Representation of the digester's setup and its components.* 

This work was divided into five parts; start-up and phases A through D according to Table 3.1. During the Start-up both digesters were filled with inoculum (digested sludge from Källby thermophilic digester) and fed daily with untreated sludge for a month to ensure similar initial conditions, values for the biogas production obtained from both reactors during the Start-up period can be found in Figure A.1 of the Appendix. Then, pre-treatment intensities of 4, 20, 70 and 95 kJ/kg were analyzed, each for 21 days (two times the retention time). One of the digesters

kept being fed with untreated sludge (control) while the other was fed with the PEF-treated samples.

Table 3.1. Chronological description of phases.

Phase	Treatment intensity (kJ/kg)	Length (days)	Start	End
Start-up	0	31	Day (-30)	Day 0
Phase A	4	21	Day 1	Day 21
Phase B	20	21	Day 22	Day 42
Phase C	70	21	Day 43	Day 63
Phase D	95	21	Day 64	Day 84

The TS and VS content of both digested sludge and the feed substrate were measured following Table 3.2 for all four phases and according to APHA's standard methods (American Public Health Agency, 2017). The methane content of the produced gas was also constantly measured by injecting a known volume of the gas in a 3 M NaOH solution where the CO<sub>2</sub> would be dissolved and the CH<sub>4</sub> would be read.

Table 3.2. Days from each phase where TS and VS measurements were taken.

Task	Day of phase
TS and VS from substrate	$1^{st} - 6^{th} - 11^{th} - 16^{th}$
TS and VS from digested sludge	$6^{th}-11^{th}-16^{th}-21^{st}$

#### 4 Results and discussion

#### 4.1 Substrate characteristics

Figure 4.1 summarizes the characteristics of the sludge substrate obtained from Källby and fed to the digesters throughout the experiment. The sludge was a mix between primary and secondary sludges and presented a VS/TS ratio considerably constant with an average of 75.9%.

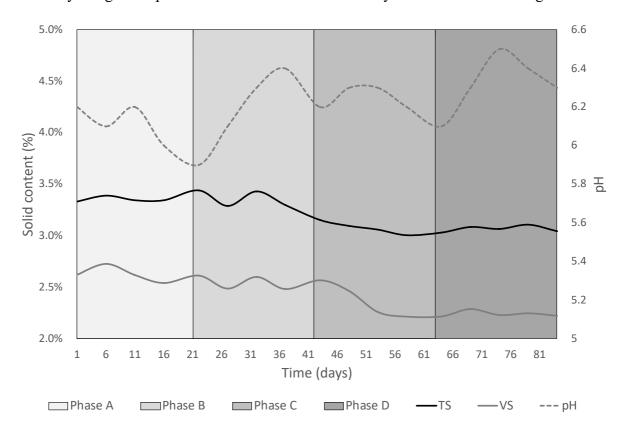


Figure 4.1. pH, TS and VS content of the sludge substrate obtained from Källby throughout the experiment and the indication of which treatment intensity was under analysis.

#### 4.2 Biogas production and VS reduction

Figure 4.2 shows the biogas production obtained along the period equivalent to one retention time for all the intensities analyzed compared with the production obtained from the digestion of untreated sludge (control). The pH values in the reactors ranged from 7.4 and 7.6 which indicates a reasonable environment for anaerobic digestion, as mentioned before pH values from 6.5 to 7.5 are considered ideal for AD (Davidsson, 2007). There were no significant variations in temperature, the water bath temperature was kept constant at 55°C±1. The variation on daily biogas production from the two digesters were similar, which can be an indication that these variations were due to changes in the substrate collected from Källby. In this study, the substrate was only analyzed for pH, TS and VS content. Substrate characterization regarding organic matter, such as chemical oxygen demand (COD) and SCOD,

combined with carbohydrates, lipids and protein composition, could have helped to correlate these daily variations with the changes in substrate.

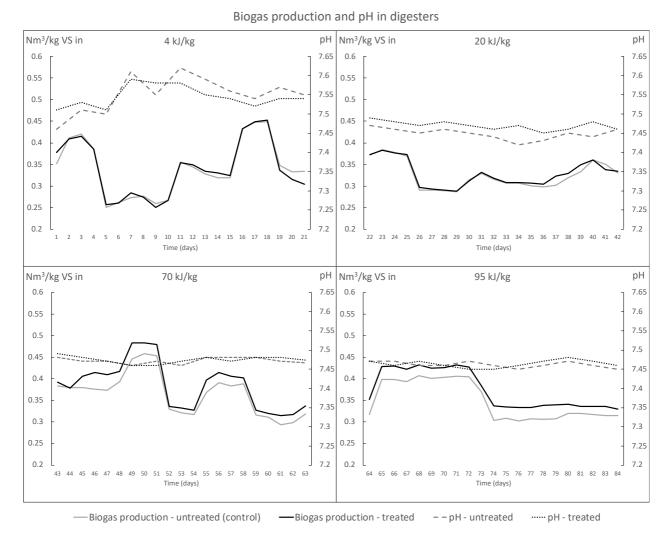


Figure 4.2. Comparison between biogas production in the digesters fed with untreated and treated sludge and pH of digested sludges for each of the intensities analyzed (4, 20, 70 and 95 kJ/kg). The gas production is expressed in normalized cubic meters (Nm³) per kilogram of VS added to the reactor.

An increase of 7.3% was obtained when pre-treating the sludge with an intensity of 95 kJ/kg (Figure 4.3). The biogas production improvement was obtained by subtracting the accumulated gas production from both digesters, during the 21 days, and dividing by the accumulated gas production from the digester with untreated sludge (control). The methane content of the biogas produced by the digestion of treated and untreated sludge were very similar, therefore the relative increase in methane production can be related to the increase in total biogas. The methane content ranged between 60-65%. VA SYD reported an average methane content of 64% in the biogas produced by Källby during 2018 (VA SYD, 2019).

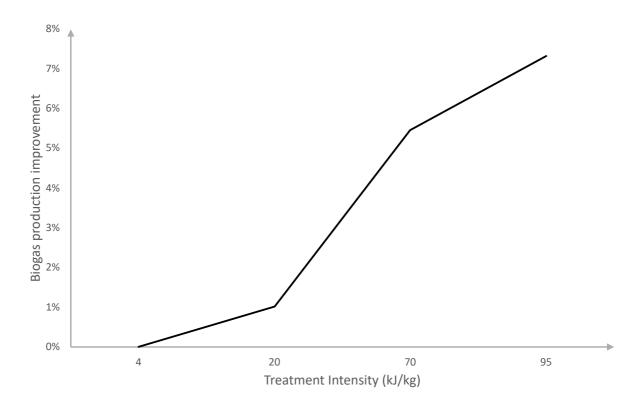


Figure 4.3. Improvement in cumulative biogas production from anaerobic digestion of PEF-treated sludge as a function of treatment intensity.

The relative increase in organic matter degradation, shown in Table 4.1, are aligned with the biogas production increase, reinforcing that higher treatment intensity resulted in increased VS reduction. The values for VS removal are the difference between VS in and out of the digesters divided VS in.

Table 4.1. TS and VS degradation from anaerobic digestion of untreated and treated sludge with intensities of 4, 20, 70 and 95 kJ/kg.

		4 kJ/kg	20  kJ/kg	70 kJ/kg	90 kJ/kg
	TS removal	17%	28%	14%	16%
Untreated	VS removal	29%	36%	29%	28%
7D ( )	TS removal	17%	28%	14%	17%
Treated	VS remove	29%	37%	31%	30%
Removal	TS	0%	0%	0%	6%
increase	VS	0%	3%	7%	7%

More information about the solids content of the digested sludge can be found in section A.2 of the Appendix.

The biogas yield in relation to the amount of VS added to the small-scale digesters is analyzed in Table 4.2 while Table 4.3 shows the gas production obtained from Källby during the year of 2018 found in the plant's environmental report (VA SYD, 2019).

Table 4.2. Comparison between the average biogas yield obtained from the small-scale digesters when fed with untreated and treated sludge.

		4 kJ/kg	20 kJ/kg	70 kJ/kg	90 kJ/kg
Accumulated biogas	Untreated	0.179	0.165	0.174	0.156
prod. in 21 days (Nm <sup>3</sup> )	Treated	0.179	0.167	0.183	0.168
VS in substrate (% wb)		2.63	2.55	2.38	2.24
VS load (kg/day)		0.0250	0.0243	0.0227	0.0213
Average biogas yield	Untreated	0.341	0.324	0.365	0.349
(Nm³/kg VS)	Treated	0.341	0.328	0.385	0.375

The values for VS content in the above table are averages obtained from the measurements described in Table 3.2. The VS load was calculated by multiplying the VS content in the substrate by the mass of substrate added every day (952 g) while the biogas yield was determined by multiplying the average biogas production by the reactor's volume (10 L) and dividing by the VS load.

Table 4.3. Average biogas yield obtained from the full-scale digester at Källby during the year of 2018 obtained from VA SYD (2019).

Källby's biogas production during 2018

Yearly biogas production	651 300	Nm <sup>3</sup>
Average daily flow into the digester	1.3	L/s
Average TS content in sludge	6.9	%
Average VS/TS ration	74	%
Estimated biogas yield	0.311	Nm³/kg VS

Values for the biogas yield obtained from the digestion of untreated sludge are aligned with the yield obtained from the full-scale plant. It can also be seen that the PEF treatment seems to have a positive, however small, impact on the biogas yield, which can be related to the physical impact of generating pores in the cell's membrane and, as discussed by Kopplow et al. (2004), making the organic matter present in the sludge more easily available.

The results show that higher improvement in biogas production is obtained with the increase in treatment intensity, however the increase seen in these experiments are very low, especially

when comparing with values from other similar studies, such as Rittmann et al. (2008) and Salerno et al. (2009). The former reached up to 45% increase in cumulative gas production and 8% increase in VS removal while the latter achieved 80% and 42% increase in gas production and VS removal, respectively. A possible suspicion is that the substrate analyzed from Salerno et al. (2009) above had a higher recalcitrant fraction, thus, the PEF treatment was able to increase VS availability to a higher degree when compared to Rittmann et al. (2008) and the results showed in this report.

Different reasons can arguably be given as possible explanations for this high difference between the current study and the ones mentioned above. The first one is that both of the aforementioned studies were conducted under mesophilic conditions, while, in this study, thermophilic digesters were used. The increased reaction rate might contribute to a lower potential of improvement from PEF pre-treatment. One could think that due to the low retention time used in this experiment (10.5 days) the methanogens are not able to digest all the available substrate and increasing the hydrolysis rate would have little effect on the final gas production, however, if that was the case, it would cause an increase in the VFA amount which would be indicated by a drop in pH, which according to the pH measurements did not occur.

Arguments can be given about the pulsed electric fields treatment itself. Although in this work the only parameter that is explicitly given about the applied PEF treatment is the intensity, other parameters are also very important and changing each of them might have different impacts on AD. Table 4.4 shows two hypothetical set of values for PEF treatments that require identical energy inputs; however, these treatments have completely different values for voltages (V), frequency (F) and pulse length (w). Additionally, changes in chamber volume and gap between electrodes can impact the electric field.

Table 4.4. Hypothetical sets of PEF parameters. Treatment intensities were calculated according to Equations (4) and (8). Mass flow and substrate resistance were assumed constant to exclude impacts from the substrate characteristics.

Parameters	Set 1	Set 2
Voltage (volts)	10	6
Frequency (Hertz)	20	30.9
Pulse length (seconds)	0.005	0.009
Current (amperes)	10	6
Mass flow (kg/s)	1	1
Substrate resistance (ohms)	1	1
Treatment intensity (J/kg)	10	10

#### 4.3 Energy recovery

Table 4.5 shows the temperature increase in samples before and after being treated with intensities around 70 kJ/kg.

Table 4.5. Temperature of samples before and after PEF treatment with intensity around 70 kJ/kg.

Sample no.	Before (°C)	After (°C)	Increase (°C)
1	15.8	32.0	16.2
2	16.0	35.0	19.0
3	18.3	38.6	20.3
4	15.2	34.8	19.6
5	16.5	33.3	16.8

Average increase (°C): 18.4

Equation (9) obtained from Davis (2010) estimates the heat addition required to increase the sludge temperature, while Equation (10) defines treatment intensity.

$$q_r = M_{sl} C_p \Delta T \tag{9}$$

$$TI = \frac{q_r}{M_{sl}} \tag{10}$$

Where  $q_r$  is the heat required in kJ/day,  $M_{sl}$  is the mass of sludge in kg/day,  $C_p$  is the specific heat of sludge in kJ/kg·°C,  $\Delta T$  is the temperature change in °C, and TI is treatment intensity in kJ/kg as already defined previously.

$$\Delta T = \frac{TI}{C_p} \tag{11}$$

Assuming the specific heat of sludge to be the same as water (4.186 kJ/kg·K), by combining Equations (9) and (10) and isolating the temperature rise to obtain Equation (11), it can be found that the treatment intensity of 70 kJ/kg would represent an increase in sludge temperature of about 16.7 °C.

Changes in temperature between the measurement before and after the treatment, together with imprecision and a slight change in the sludge's actual specific heat might have contributed to the difference between theoretical and obtained values.

However slightly different from the value calculated using Equation (11), the obtained values are reasonable and, according to the theoretical method shown, increased treatment intensity generates higher increase in substrate temperature.

By taking advantage of the temperature increase caused by the PEF treatment, energy requirements for the subsequent anaerobic digester can be decreased, contributing positively to the system's energy balance. Table 4.6 displays a comparison between the energy requirements of heating a full-scale digester with volume of 3000 m³ to 55°C when fed with treated and untreated substrate. The substrate temperature assumed in this energy balance are the averages between the values shown in Table 4.5. Considering the density and specific heat of sludge to be the same as water, 1000 kg/m³ and 4.186 kJ/kg·K, respectively. The flow into the digesters is assumed 100 m³/day. The energy required to treat 100 m³/day of substrate with a PEF intensity of 70 kJ/kg is 7x10<sup>6</sup> kJ/day, which is slightly lower than the recovered value obtained in the following table, that is another indication that there was some imprecision when measuring the temperature increase caused by the pre-treatment.

Table 4.6. Comparison between energy requirements to heat digesters receiving treated and untreated substrate.

Substrate	Before (°C)	After (°C)
Substrate temperature (°C)	16.4	34.7
Energy required (kJ/day)	$16.2 \times 10^6$	$8.5 \times 10^6$
Decrease in energy required	7.7 >	x 10 <sup>6</sup>
to heat digester (kJ/day)		

The calculations show that up to  $7.7x10^6$  kJ/day could theoretically be recovered from the treatment. It is relevant to note, however, that losses in temperature during the transport of sludge from the PEF treatment to the digester will decrease the energy recover ratio.

Table 4.7 displays the yearly energy obtained from biogas production at Källby wastewater treatment plant during 2018 and how much an increase of 7.3% would represent.

Table 4.7. Simulation of biogas production increase from Källby's full-scale digester. The yearly biogas production is obtained from VA SYD (2019).

	kWh	kJ
Total produced biogas in 2018	4 037 200	$1.45 \times 10^{10}$
Average daily biogas production	11 061	$3.98 \times 10^7$
7.3% of average daily biogas production	807	2.91 x 10 <sup>6</sup>

A 7.3% increase in the biogas produced by Källby's full-scale digester during 2018 would represent an increase in energy production of 2.91x10<sup>6</sup> kJ/day. That is roughly one third of the energy required to treat 100m<sup>3</sup>/day with PEF intensity of 70 kJ/kg. Therefore, when assuming no energy is recovered from the pre-treatment, the low increase in biogas production obtained in this study indicate the use of PEF as a pre-treatment to be not feasible.

#### 5 Conclusion

Although Pulsed Electric Field is fairly known and used in other fields, this technology is still new as a pre-treatment of wastewater sludge. However, different studies indicate an existing potential for this application to significantly improve the anaerobic digestion process of wastewater sludge increasing biogas production and organic matter degradation.

The results obtained from this work show that higher PEF treatment intensities result in higher values for cumulative biogas production from anaerobic digestion. The experiments reached up to 7.3% higher cumulative biogas production values from the digestion of sludge treated by 95 kJ/kg when compared to the digestion of untreated sludge with similar conditions. The obtained improvements can be considered very low when compared to results obtained from other similar studies and other traditionally used pre-treatment methods.

PEF treatment requires energy and the use of such treatment is only feasible if its energy input can be balanced by its advantages. Aside from the increase in biogas production discussed before, the increase in the sludge's temperature caused by the treatment can reduce the need of external heating and contribute to offsetting the treatment's energy requirements. A 16.4 °C increase in substrate temperature can represent a reduction of up to  $7.7 \times 10^6 \, \text{kJ/day}$  in the energy required to heat a 3000 m³ digester.

#### 6 Future studies

This work only differentiated the PEF treatments conducted by treatment intensity i.e. energy input. It is important to note, however, that there are a variety of other parameters which can be changed when applying pulsed fields technology, such as voltage, frequency, pulse length and chamber volume. Therefore, in order to have a deeper understanding of the impact Pulsed Electric Fields can have on the anaerobic digestion of wastewater sludge, more studies should be conducted to further investigate the influence of each of these parameters.

Additionally, the sludge being treated can influence the optimal parameters mentioned before, thus investigating the relationship between those parameters and membrane destruction can, subsequentially, help develop ways to determine a set of parameters that work best according to sludge characteristics, for example, TS and VS content, average cell size and electrical resistance or resistivity.

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# **Appendix**

### A.1 Digesters during start-up

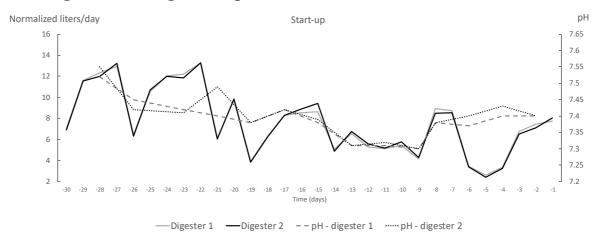


Figure A.1. Gas production and pH in small-scale digester start-up period. During start-up both digesters were fed with the same untreated sludge. After (from day "0" onwards) Digester 1 was used as control and Digester 2 started to be fed with treated substrate.

### A.2 Solids content in substrate, treated and untreated digested sludges

Table A.1. Solids content and standard deviations from substrate, untreated and treated digested sludges during 4 kJ/kg treatment.

### 4 kJ/kg

Day	sub	strate	untı	reated	tre	eated	sub	strate	unt	reated	tro	eated
-	TS	StDev	TS	StDev	TS	StDev	VS	StDev	VS	StDev	VS	StDev
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
1	3.33	0.01										
6	3.39	0.11	2.73	0.02	2.66	0.05	2.62	0.03	1.85	0.06	1.85	0.10
11	3.34	0.06	2.65	0.04	2.73	0.10	2.73	0.02	1.80	0.10	1.84	0.02
16	3.34	0.00	2.84	0.01	2.90	0.02	2.62	0.11	1.95	0.02	1.92	0.10
21			2.89	0.10	2.82	0.08	2.54	0.03	1.86	0.01	1.84	0.04
	Averag	ge:	Average:		Average:		Averag	ge:	Averag	ge:	Averaş	ge:
	3.35%		2.78%		2.78%	2.78%		2.63%			1.86%	
					•							
	TS remaining		82	2.9%	82	2.9%	VS ren	naining	70	).9%	70	0.7%
	TS dige	ested	17	7.1%	17	7.1%	VS dig	gested	29	9.1%	29	9.3%
	increas	e in TS d	legrada	tion:	0.0%		increas	se in VS	degrada	ition:	0.69%	

Table A.2. Solids content and standard deviations from substrate, untreated and treated digested sludges during 20 kJ/kg treatment.

#### 20 kJ/kg

Day	sub	strate	unt	reated	tre	eated	sub	strate	unt	reated	tro	eated
	TS	StDev	TS	StDev	TS	StDev	VS	StDev	VS	StDev	VS	StDev
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
22	3.44	0.12										
27	3.29	0.08	2.39	0.09	2.30	0.09	2.62	0.11	1.61	0.11	1.54	0.10
32	3.43	0.09	2.39	0.13	2.31	0.13	2.49	0.12	1.56	0.05	1.54	0.08
37	3.30	0.01	2.31	0.08	2.33	0.12	2.60	0.03	1.67	0.07	1.66	0.10
42			2.60	0.07	2.71	0.02	2.48	0.08	1.65	0.07	1.70	0.03
	Averag	ge:	Averag	ge:	Averag	ge:	Averag	ge:	Averag	ge:	Averag	ge:
	3.36%		2.42%		2.41%	2.41%		2.55%			1.61%	
	•		•									
	TS rem	naining	72	2.0%	71	.8%	VS ren	naining	63	3.7%	63	3.2%
	TS dige	ested	28	3.0%	28	3.2%	VS dig	gested	36	5.3%	36	5.8%
	increas	e in TS o	degrada	tion:	0.71%		increas	se in VS	degrada	ition:	1.38%	

Table A. 3. Solids content and standard deviations from substrate, untreated and treated digested sludges during 70 kJ/kg treatment.

## 70 kJ/kg

Day	sub	strate	unti	reated	tre	eated	sub	strate	unt	reated	tre	eated
,	TS	StDev	TS	StDev	TS	StDev	VS	StDev	VS	StDev	VS	StDev
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
43	3.15	0.05										
48	3.09	0.07	2.68	0.13	2.66	0.08	2.57	0.04	1.73	0.01	1.70	0.06
53	3.06	0.11	2.71	0.03	2.71	0.08	2.46	0.03	1.74	0.00	1.70	0.03
58	3.00	0.09	2.61	0.10	2.60	0.13	2.26	0.09	1.60	0.06	1.57	0.07
63			2.63	0.01	2.60	0.02	2.21	0.03	1.65	0.04	1.62	0.10
	Averag	ge:	Average:		Average:		Averag	ge:	Averag	ge:	Averag	ge:
	3.08%		2.66%		2.64%		2.38%		1.68%		1.65%	
<u>,                                      </u>									-1			
	TS rem	naining	86	5.4%	85	5.9%	VS ren	naining	70	).7%	69	9.3%
	TS dige	ested	13	3.6%	14	1.1%	VS dig	gested	29	0.3%	30	).7%
	increas	e in TS d	legrada	tion:	3.68%		increas	se in VS	degrada	tion:	4.78%	

Table A. 4. Solids content and standard deviations from substrate, untreated and treated digested sludges during 95 kJ/kg treatment.

#### 95 kJ/kg

Day	sub	strate	unti	reated	tre	eated	sub	strate	unt	reated	tro	eated
	TS	StDev	TS	StDev	TS	StDev	VS	StDev	VS	StDev	VS	StDev
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
64	3.03	0.11										
69	3.08	0.13	2.66	0.11	2.57	0.12	2.22	0.10	1.69	0.03	1.63	0.02
74	3.06	0.04	2.55	0.09	2.53	0.09	2.29	0.10	1.58	0.03	1.55	0.08
79	3.11	0.07	2.56	0.03	2.56	0.07	2.23	0.04	1.55	0.09	1.52	0.12
84			2.56	0.07	2.57	0.07	2.25	0.05	1.60	0.05	1.55	0.07
	Averag	ge:	Averag	Average:		Average:		Average:		ge:	Averag	ge:
	3.07%		2.58%		2.56%	2.56%		2.24%		1.60%		
	TS remaining 84.1%		1.1%	83	3.3%	VS ren	naining	7	1.5%	69	9.6%	
	TS dig	ested	15	5.9%	16	5.7%	VS dig	gested	28	8.5%	30	0.4%
	increas	se in TS d	legrada	tion:	5.03%		increas	se in VS	degrada	ition:	6.67%	

### A.3 Biogas production

Table A.5. Gas production and solids content from substrate, untreated and treated digested sludges during 4 kJ/kg treatment.

Day	•	Substr			Untreat	ed (cor	ntrol)		Tı	eated		
		influe	nt		Gas production	Dige	ested s	sludge	Gas production	Dig	ested s	sludge
	TS	VS	pН			TS	VS	рН		TS	VS	pН
	(%)	(%)			(NmL)	(%)	(%)		(NmL)	(%)	(%)	
1	3.33	2.62	6.2		8785			7.46	9438			7.51
2					10300				10230			
3					10500			7.51	10390			7.53
4					9650				9638			
5					6276			7.5	6435			7.51
6	3.3%	2.73	6.1		6547	2.73	1.85		6506	2.66	1.85	
7					6834			7.61	7106			7.59
8					6932				6866			
9					6473			7.55	6272			7.58
10				ρp	6680				6680			
11	3.34	2.62	6.2	4 kJ/kg	8890	2.65	1.80	7.62	8860	2.73	1.84	7.58
12				4	8619				8719			
13					8208			7.59	8347			7.55
14					7995				8271			
15					7996			7.56	8110			7.54
16	3.34	2.54	6		10836	2.84	1.95		10827	2.90	1.92	
17					11189			7.54	11239			7.52
18					11214				11310			
19					8711			7.57	8416			7.54
20					8328				7887			
21					8361	2.89	1.86	7.55	7615	2.82	1.84	7.54
			·		1							

Averages	Accumulated biogas	Avera	iges	Accumulated biogas	Averages	
TS in: 3.35	179324 NmL	TS out:	2.78	179162 NmL	TS out:	2.78
VS in: 2.63		VS out:	1.86		VS out:	1.86

Biogas production improvement: -0.09

Table A.6. Gas production and solids content from substrate, untreated and treated digested sludges during 20 kJ/kg treatment.

Day	,	Substr			Untreat	ed (co	ntrol)		Tr	eated		
		influe	nt		Gas production	Dig	ested :	sludge	Gas production	Dig	ested s	sludge
	TS	VS	рН			TS	VS	рН		TS	VS	рН
	(%)	(%)			(NmL)	(%)	(%)		(NmL)	(%)	(%)	
22	3.44	2.62	5.9		9016			7.47	9022			7.49
23					9304				9269			
24					9164			7.46	9128			7.48
25					8943				9037			
26					7043			7.45	7205			7.47
27	3.29	2.49	6.1		7035	2.39	1.61		7110	2.30	1.54	
28					7015			7.46	7036			7.48
29					6961				6985			
30					7601			7.45	7555			7.47
31				õ	7969				8035			
32	3.43	2.60	6.3	20 kJ/kg	7642	2.39	1.56	7.44	7695	2.31	1.54	7.46
33				20	7448				7457			
34					7433			7.42	7456			7.47
35					7293				7422			
36					7219			7.43	7384			7.45
37	3.30	2.48	6.4		7307	2.31	1.67		7821	2.33	1.66	
38					7727			7.45	7987			7.46
39					8066				8452			
40					8733			7.44	8742			7.48
41					8481				8185			
42					8002	2.60	1.65	7.46	8102	2.71	1.70	7.46
	<b>A</b>	varagas			Accumulated	,	Voro	g o g	Accumulated	,	Voro	g o c

Averages	Accumulated biogas	Avera	ges	Accumulated biogas	Averages		
TS in: 3.36	165402 NmL	TS out:	2.42	167085 NmL	TS out:	2.41	
VS in: 2.55		VS out:	1.62		VS out:	1.61	

Biogas production improvement: 1.02

Table A.7. Gas production and solids content from substrate, untreated and treated digested sludges during 70 kJ/kg treatment.

Day Substrate influent		Untreate	ed (cor	ntrol)		Treated						
		influe	nt		Gas production	Dige	ested	sludge	Gas production	Dig	ested	sludge
	TS	VS	pН			TS	VS	pН		TS	VS	pН
	(%)	(%)			(NmL)	(%)	(%)		(NmL)	(%)	(%)	
43	3.15	2.57	6.2		8655			7.48	8864			7.49
44					8595				8549			
45					8583			7.47	9185			7.48
46					8511				9386			
47					8429			7.47	9263			7.47
48	3.09	2.46	6.3		8888	2.68	1.73		9437	2.66	1.70	
49					10084			7.46	10920			7.46
50					10355				10914			
51					10235			7.47	10838			7.46
52				8	7444	2.71	1.74		7607	2.71	1.70	
53	3.06	2.26	6.3	70 kJ/kg	7246			7.46	7506			7.47
54				70	7163				7399			
55					8322			7.48	8988			7.48
56					8831				9379			
57					8665			7.48	9182			7.47
58	3.00	2.21	6.2		8770	2.61	1.60		9084	2.60	1.57	
59					7159			7.48	7408			7.48
60					7030				7236			
61					6651			7.47	7122			7.48
62					6752				7167			
63					7208	2.63	1.65	7.466	7621	2.60	1.62	7.474
	A	verages	1		Accumulated biogas	A	Avera	ges	Accumulated biogas	A	Avera	ges

Averages	Accumulated biogas	Avera	ages	Accumulated biogas	Averages	
TS in: 3.08	173576 NmL	TS out:	2.64	183055 NmL	TS out:	2.62
VS in: 2.38		VS out:	1.66		VS out:	1.63

Biogas production improvement: 5.46

Table A.8. Gas production and solids content from substrate, untreated and treated digested sludges during 95 kJ/kg treatment.

Day	,	Substr			Untreat	ed (cor	ntrol)		Tr	eated		
	TS VS pH			Gas production	Dig	ested s	sludge	Gas production	Dig	ested	sludge	
	TS	VS	рН			TS	VS	рН		TS	VS	рН
	(%)	(%)			(NmL)	(%)	(%)		(NmL)	(%)	(%)	
64	3.03	2.22	6.1		6780			7.47	7525			7.47
65					8507				9147			
66					8495			7.47	9179			7.46
67					8404				9005			
68					8690			7.46	9238			7.47
69	3.08	2.29	6.3		8554	2.66	1.69		9071	2.57	1.63	
70					8601			7.46	9089			7.46
71					8676				9227			
72					8652			7.47	9107			7.45
73				50	7888				8177			
74	3.06	2.23	6.5	kJ/kg	6480	2.55	1.58	7.46	7194	2.53	1.55	7.45
75				95	6596				7155			
76					6446			7.45	7125			7.46
77					6551				7135			
78					6526			7.46	7215			7.47
79	3.11	2.25	6.4		6570	2.56	1.55		7264	2.56	1.52	
80					6827			7.47	7280			7.48
81					6821				7188			
82					6779			7.46	7187			7.47
83					6730				7187			
84					6715	2.56	1.60	7.45	7038	2.57	1.55	7.46
		Varagas			Accumulated		Voro	g06	Accumulated		Vora	goe

Averages	Accumulated biogas	Avera	iges	Accumulated biogas	Aver	ages
TS in: 3.07	156288 NmL	TS out:	2.58	167733 NmL	TS out:	2.56
VS in: 2.24		VS out:	1.60		VS out:	1.56

Biogas production improvement: 7.32