UNIVERSITÀ DEGLI STUDI DI PADOVA

DIPARTIMENTO DI INGEGNERIA INDUSTRIALE

CORSO DI LAUREA MAGISTRALE IN INGEGNERIA CHIMICA E DEI PROCESSI INDUSTRIALI

Master Thesis in Chemical and Industrial Processes Engineering

STEAM EXPLOSION: A WAY TO ENHANCE THE DIGESTION OF SEWAGE SLUDGE

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Academic year 2014– 2015

Sommario

Questo lavoro di tesi è stato svolto in Inghilterra, durante il periodo di Erasmus durato 5 mesi, in collaborazione con l'Università di Surrey situata a Guildford e con l'azienda Thames Water, nell'impianto situato a Reading. Questa azienda è una compagnia leader nel territorio inglese nel trattamento delle acque.

L'obiettivo della tesi è di capire se il processo di digestione anaerobica può essere migliorato utilizzando come pretrattamento l'esplosione di vapore. Si tratta di un tema sempre attuale nella prospettiva della produzione delle energie rinnovabili perché, in un mondo dove le fonti di energia fossili vanno man mano esaurendosi, è importante trovare delle fonti di energia che continuino a sostenere il nostro fabbisogno di carburante. I fanghi sono un fonte di energia che è sempre disponibile ovunque ci siano delle popolazioni e quindi è fondamentale continuare a sviluppare tecnologie che incrementino la resa dei processi che ne sfruttano il contenuto energetico. L'esplosione di vapore fu usata in principio come pretrattamento del legno, oggi è una tecnologia valida e conosciuta anche nel campo del pretrattamento dei fanghi, per incrementare la resa di biogas prodotto dalla digestione anaerobica. Ci sono molti impianti in funzione in tutto il mondo che usano questa tecnologia ed è importante che si continui a lavorare per ottenere delle rese migliori in modo da fare diventare i fanghi una fonte di energia rinnovabile sempre più di diffusa.

L'elaborato si compone di quattro capitoli nei quali è stata trattata la produzione di biogas a partire da fanghi, usando come pretrattamento degli stessi la tecnologia dell'esplosione di vapore. Sono stati fatti quattro esperimenti eseguiti su un impianto pilota, ognuno dei quali è stato descritto e analizzato nel dettaglio.

Nel primo esperimento è stata analizzata la produzione di biogas a partire da campioni esplosi a 3 bar e 6 bar per capire se effettivamente essa aumenta in modo significativo al variare della pressione.

Nel secondo esperimento sono stati analizzati dei campioni di fanghi esplosi a 3 bar e 4.5 bar. Da questi due esperimenti si può ricavare un'idea di quanto la variabilità nelle proprietà del fango influisca nella produzione di biogas. Entrambi gli esperimenti sono stati fatti su due campioni per ogni tipo di fango e due campioni per l'inoculo, che sono stati poi utilizzati come bianco nell'analisi dei dati successiva.

Nel terzo esperimento si sono studiate 3 diverse pressioni di esplosione: 3, 4.5 e 6 bar. In questa analisi sono stati usati tre campioni per ogni tipo di fango esploso e due campioni per il fango inesploso per confrontare le quantità di biogas prodotto, più un campione di inoculo da usare sempre come bianco nelle successive analisi.

I dati di questo terzo esperimento sono stati elaborati in vario modo, e precisamente con:

1. un'analisi statistica dove si sono analizzati gli incrementi di produzione del biogas rispetto ai campioni di fango esplosi a 3 bar e si sono individuati gli intervalli di confidenza per la

produzione di biogas per i campioni esplosi a 4.5 e 6 bar usando anche i dati ottenuti dai primi due esperimenti.

2. l'analisi della domanda chimica di ossigeno (COD) e degli acidi grassi volatili (VFA) dove si è studiata la correlazione che c'è tra i suddetti parametri e le diverse pressioni di esplosione.
3. un'analisi al microscopio, dove si è misurata la dimensione delle particelle di fango in funzione delle diverse pressioni di esplosione.
4. un'analisi idrodinamica: dove è stato messo a punto un modello semplificato per la predizione della pressione finale all'interno del serbatoio nel quale avviene l'esplosione di vapore, e del tempo di scarico del liquido all'interno nel serbatoio.

Il quarto esperimento è basato su un'idea, mai provata prima, secondo cui il fango è sottoposto oltre che all'esplosione di vapore anche alla cavitazione, provocata da una strozzatura nel condotto di uscita, in modo da diminuire ulteriormente la dimensione delle particelle di fango. Per questo esperimento sono state fatte le analisi del punto 2 e 4, sopra descritte.

Il primo capitolo della tesi comprende l'analisi della letteratura riguardante la tecnologia dell'esplosione di vapore, seguita dalla descrizione dei tipi di fanghi usati negli esperimenti. Si descrivono inoltre le varie configurazioni possibili per questo tipo di processo, e si riassumono gli aspetti fondamentali del processo di digestione anaerobica.

Nel secondo capitolo vengono descritti, dapprima l'impianto dove sono stati eseguiti gli esperimenti, e il processo di esplosione di vapore. Vengono poi riportate le procedure per la preparazione dei campioni, e tutte le analisi che sono state svolte sui campioni analizzati.

Il terzo capitolo mira ad esporre e discutere i risultati ottenuti partendo con la produzione di biogas per chiudere con l'analisi idrodinamica.

Per concludere è stata sviluppata una parte economica del processo al fine di individuare quelli che sono i costi di investimento e i costi operativi per diverse configurazioni attualmente disponibili sul mercato. E' stato calcolato il tasso interno di rendimento (IRR) per ognuna delle configurazioni, in presenza e in assenza degli incentivi ed il tempo di recupero dell'investimento su una tecnologia di questo tipo.

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Introduction

This work aims to examine if the digestion of activated sludge from wastewater treatment can be improved by steam explosion. This topic is important in the perspective of renewable energy supply because, in a world where fossil fuel energy resources are slowly decreasing, it's important to find other energy sources to cover this deficit - which increases year by year.

Activated sludges are a source of energy that is always available wherever there is population, so it's important to continue developing technologies to increases the yield of energy recovery from them, and to make these technologies the foundations for a new and better future. Steam explosion is a process originally developed for wood, and has subsequently been used even to improve the biogas production from the sludge, and has been known and used as a pretreatment for sludge digestion for many years.

There are many plants running with this technology all over the world, and this reinforces the need to keep improving this technology to achieve a better yield and to make sludge an increasingly important source of renewable energy.

This work has been developed in Reading, United Kingdom, during an Erasmus student exchange program. The project is the result of cooperation between a world leading water company, Thames Water, and the University of Surrey in Guildford.

This work is comprised of four chapters. The first is used to frame steam explosion, and to describe the basis of this process. Furthermore, the digestion process is described in detail.

In the second chapter, the experiments that have been undertaken are reported, together with all the analysis that has been made on the sludge samples.

In the third chapter, the results from all the experiments and all the analysis made are discussed. The fourth chapter is used to make an economic analysis of this project to estimate the Internal Rate Return and the payback time for this process.

I wish to acknowledge those who helped me in this work. Above all my supervisor, prof. Bertucco who helped me in this route and gave me the opportunity to do this work abroad with the Erasmus program at the University of Surrey. I would like to thank prof. Thorpe for his help during the five months I spent in England, Aurelien Perrault, project manager in Thames Water, for his cooperation during the experiments and Dejene Tilahun, laboratory technician in Thames Water, for the invaluable help who gave me during all the project's duration.

Chapter 1

Sewage sludge, pretreatment processes and anaerobic digestion

1.1 The company

Thames Water, is the private utility company responsible for the public water supply and waste water treatment in large parts of Greater London, the Thames Valley, Surrey, Gloucestershire, Wiltshire, Kent, and some other areas of the United Kingdom. Thames Water is the UK's largest water and wastewater services company, and supplies 2.6 gigalitres of drinking water per day, and treats 4.4 gigalitres of wastewater per day. Thames Water's 15 million customers comprise 27% of the UK population.

Thames Water Innovation is very interested in this project because although THP is a well-known process, there are still some uncertainties around steam explosion and its benefits. The idea really is to try to optimise AD process by increase gas generation and volatile matter destruction and we think we can achieve this by improving the understanding of the process and operational envelop.

1.2 Steam explosion literature's review

Steam explosion was patented as a pre-treatment method for biomass by Mason et al. in 1926. It's a physicochemical pre-treatment method that can be undertaken with or without the addition of acid as a catalyst. In this process the biomass is heated to about 160 °C and 6 bar with steam for 30 minutes. After, it is subjected to an explosive decompression which, in a few seconds, brings the flash vessel back to 1 bar. When the biomass is exploded, the water that is deeply penetrated into its structure is subjected to flash, and this allows for the destruction of the main internal structure of the biomass, making it possible to achieve more surface area for the next stage, Anaerobic Digestion.

The steam explosion is used mostly as a pre-treatment method for the wood, and in fact most of the literature refers to that kind of biomass. In some cases, steam explosion with acid as catalyst was used in the wood industries, e.g. SO_2, H_2SO_4, because it was useful for improving the hydrolysis rate of hemicelluloses during the steam explosion (Boussaid et al., 2000; Shevchenko et al, 2001; Bura et al., 2002). Steam explosion for biomass can be used as an environmentally friendly pulping process (Kokta and Ahmed, 1998) as pre-treatment for microbial bioethanol or biogas production (Cara at al., 2006; Dererie at al., 2011). It is also used for the pre-treatment of wood prior pelletization to increase heating value, bonding properties and hydrophobicity of the wood (Adapa et al. 2010; Adapa et al.2010a, Lam et al. 2011). Steam explosion treatment has also been suggested as a pre-treatment process in the wood pellet industry, as a method to get dimensionally stable and durable pellets (Obernberger and Thek, 2010). The bulk density of steam-exploded pellets is relatively high in comparison to conventional wood pellets, and different values were found in literature, ranging between 630 to 750 kg/m^3. This is similar to what happens in the steam explosion for the sludge, where more surface area is achieved.

The advantages of using steam explosion as a pre-treatment method are:

- no chemicals are used except water;
- larger surface area for the anaerobic digestion process;
- there isn't the equipment corrosion that occurs when acid pre-hydrolysis is used in the wood industries;
- the process comes for free once that the desired pressure is reached, so, no more additional cost, aside from pressurizing the tank;
- increased Volatile Solids Destruction (VSD);
- the process allows increased loading (i.e. throughput) in existing assets reducing capital costs.

The steam explosion processes can be run both in continuous or batch, depending on the purpose. In the lab scale, the batch mode is used, while for larger scale projects, the continuous mode is more often applied. The batch systems are usually used as pre-treatment in laboratory experiments and the configuration is largely similar to the one represented in Figure 1.1 (Lam, 2011). The continuous systems are instead used in industrial production processes. Figure 1.2 (Lam, 2011) shows an example of continuous steam explosion system from a pilot plan located in Sherbooke, Quebec, Candada.

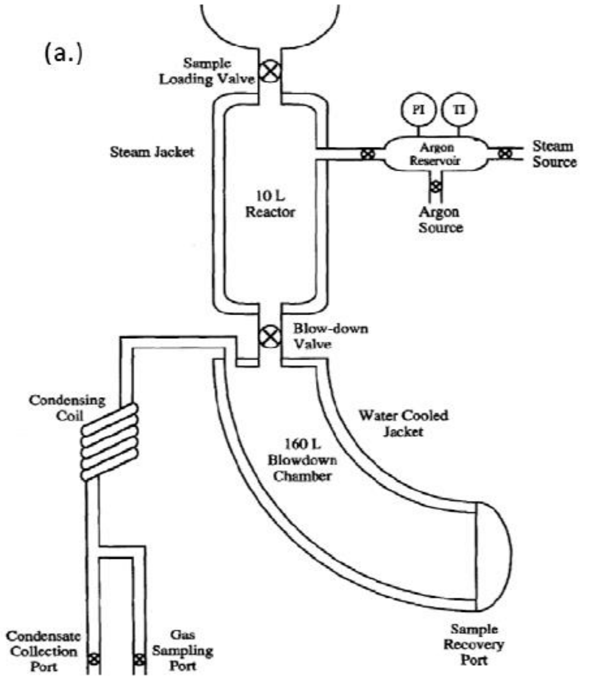

Figure 1.1 *Standard configuration for the batch system.*

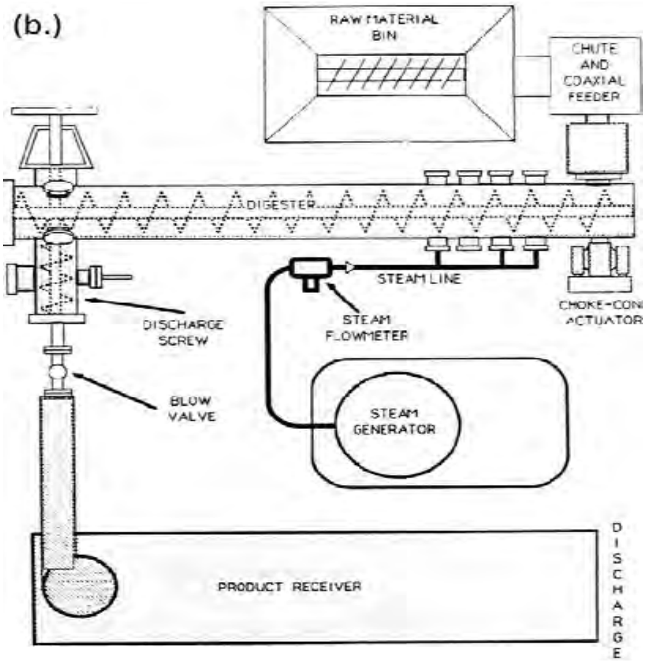

Figure 1.2 *Configuration for the continuous system.*

Steam explosion is influenced by several parameters such as the residence time in the reaction, moisture content of the feed, the reaction pressure and the reaction temperature. If just one of these conditions during the process changes, the whole process is influenced. In order to optimize the process Overend and Chornet (1987), developed a severity index R_0 (Equation 1.1) (Overend at al., 1987), commonly used to optimize the steam explosion.

$$R_0 = \int_0^t exp\left(\frac{(T-100)}{14.75}\right) dt \tag{1.1}$$

Where t is the residence time and T is the reaction temperature in Celsius degree.

This model doesn't take into account the moisture content of the feedstock: a limitation, as this factor influences the whole process. The process parameters and also the severity index are adjusted based on the aim of the process. For example, in the wood, biomass destructions start for R_0 around 2 and for $R_0 > 4$ the reaction of condensation and dehydration begin (Lam, 2011).

Thames Water started the conversion of their normal Anaerobic Digestion plant to a steam explosion plant in 1993, with the plant located in Chertsey. So far, Thames Water have converted six plants, scattered throughout England, to this technology and are planning to convert other plants in the coming years.

The improvement that has been so far achieved with the steam explosion plant is that the biogas production grew from 300 m^3/TDS to 450 m^3/TDS (TDS stand by tons of dry solids).

1.3 Sewage sludge

Sewage sludge is a semi-solid material that is produced as by-product during wastewater treatment of industrial or municipal wastewater. It is the part removed in sewage treatment plants, during the various solids separation steps: mechanical, biological and chemical. These treatments are necessary for waste disposal without causing changes to the ecosystem. There are mainly two types of sludge: Primary sludge and Secondary Activated Sludge (SAS).

1.3.1 PRIMARY SLUDGE (PS)

Primary sludge (Figure 1.3) is the term for the settleable suspended solids present in the raw wastewater. It is the part of the sludge that can be separated without any biological treatments.

Primary sludge is mainly composed of:

- easily degradable organic compounds (cellulose, proteins, sugar);
- inert inorganic compounds (sand, carbonates);
- not readily biodegradable compounds (fibers, rubber).

Figure 1.3 *Primary sludge.*

1.3.2 SECONDARY ACTIVATED SLUDGE (SAS)

Secondary activated sludge (Figure 1.4) contains the substances that are sedimented in secondary decanters. These substances are formed during the biological treatment which the sewage undergoes before the secondary decanters. The secondary activated sludge is formed by microorganisms that feed on the substances present in the sewage and then, growing, form the SAS.

The SAS are mainly composed of:

- the settleable suspended solid fraction that escaped the primary sedimentation;
- solids produced in the biological reactor, comprised of: suspended solids (not settleable and not biodegradable), suspended solids (not settleable but biodegradable) and biodegradable dissolved solids.

Figure 1.4 *Secondary activated sludge.*

1.4 Steam explosion pre-treatment configuration schemes

The configuration of the plants for the pre-treatment of sludge has changed over the years in order to produce more biogas. There are many configurations that treat the primary and the secondary sludge in different way.

1.4.1 THERMAL HYDROLYSIS PROCESS (THP)

The thermal hydrolysis process is a pre-treatment method used to dewater, solubilize and homogenize the sludge so that the organic matter becomes more available, so as to make the sludge more digestible. This technique is becoming increasingly widespread because it allows, as reported by Thorpe at al. (2013), increase of the biogas yield as well as volatile solids destruction (VSD). In THP the dewatered sludge is heated at about 160°C and 6 bar for 30 minutes and then moved to a flash vessel at atmospheric pressure to then be sent to the digestion step. This treatment is basically the same with respect to steam explosion, but was designed before, and in the beginning the release of pressure was gradual. Nowadays most of the THP plants use steam explosion.

Biogas production for this process is typically 450 m^3/TDS. Figure 1.5 summarizes a block flow diagram of this process.

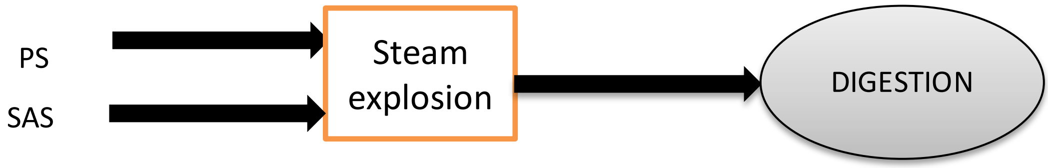

Figure 1.5 *Flowsheet configuration for the THP process.*

1.4.2 INTERMEDIATE THERMAL HYDROLYSIS PROCESS (ITHP)

In this case, the THP process is the middle of two digesters. The first stage is a normal digestion which exploits the organic matter of the sludge without any treatment, whereas in the second stage the sludge is hydrolysed with THP, and is then digested again. With this scheme, the THP plant is smaller because of the previous digestion and the consequent dewatering. The two stages combined together bring to 500 m^3/TDS, showing a 20% improvement with respect to the conventional THP. The combined action of these two factors lead to a self-sufficient plant in terms of heat. Figure 1.6 shows a raw flowsheet of this process.

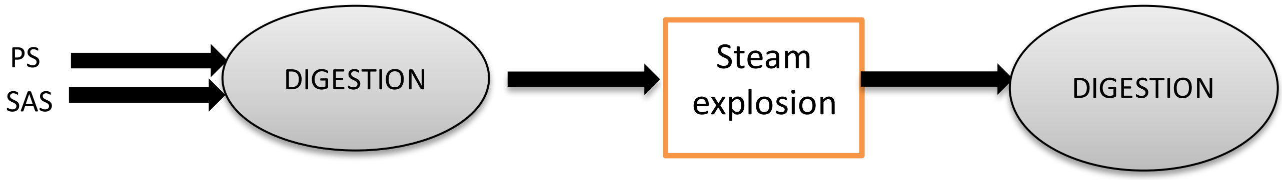

Figure 1.6 *Flowsheet configuration for the ITHP process.*

1.4.3 SAS only Thermal Hydrolysis Process

In this kind of pre-treatment, only SAS is hydrolysed. The Primary Sludge is added to the SAS once it has been hydrolysed. The advantage is that the THP plant size is smaller and the steam needed is reduced so that no additional fuel is required. The performance of the process is reduced because the Primary sludge is not hydrolysed and this involves a loss in biogas production. This method achieves a volatile solids destruction of 54% and around 421 m^3/TDS of biogas as demonstrated by Shana at al. (2013) in laboratory trials. Figure 1.7 shows a block diagram of this process.

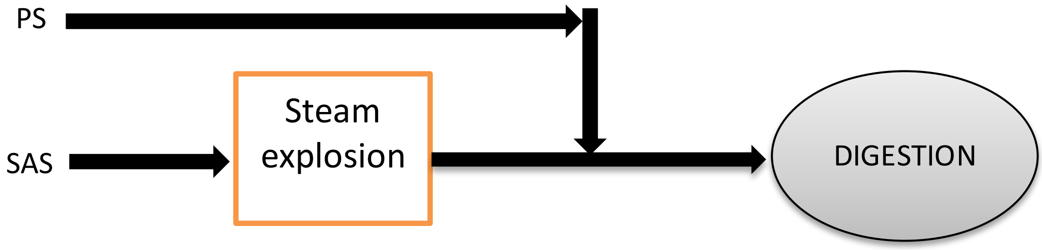

Figure 1.7 *Flowsheet configuration for the SAS only THP process.*

A configuration used in a real Thames water's plant is represented in Figure 1.8. Here the whole flowsheet for the THP process is represented, with the indication of temperature and the DS contents in the crucial step of the process to allow a better understanding.

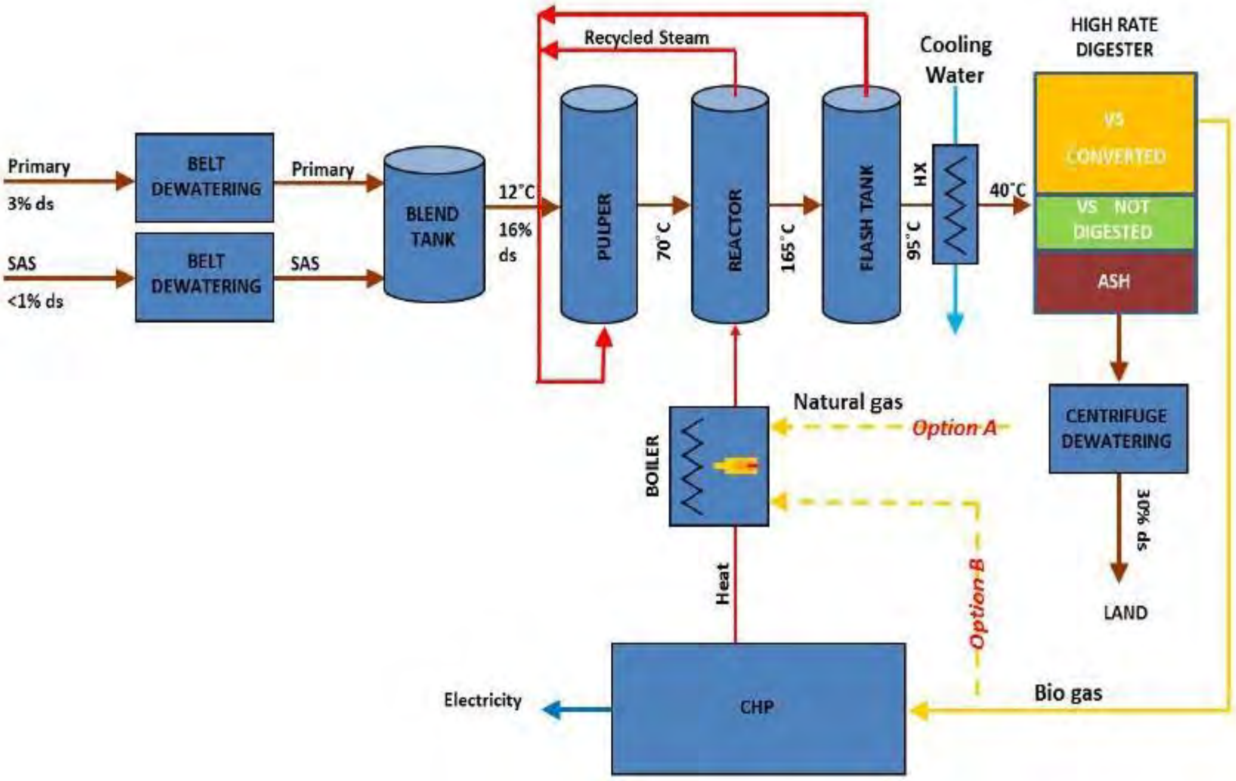

Figure 1.8 *Flowsheet in a real plant.*

Clearly, the sludge is first mixed and then blended. Then, it is conveyed to the pulper tank where the sludge is heated to 70 °C, and goes to the reactor tank where it is heated with steam to 165 °C and sent to the flash tank where the pressure is released. After the flash, the sludge is cooled to 40 °C with water and sent to the high rate digester. The biogas produced is sent to the CHP unit (Combined Heat and Power) in order to produce electricity, power, and heat to power the boiler. The sludge that is not converted into biogas can be disposed of as fertilizer. In order to make steam, the power given from the CHP unit is insufficient, so an energy supply is required. There are two options for this matter: option A, which implies an external gas supply that must be bought, or option B, where part of the biogas produced is sent to the boiler to supply the lack of energy.

1.5 Other pre-treatments

Together with steam explosion, there are several others pre-treatment methods for the sludge. The most widespread are:
- biological;
- mechanical;
- chemical.

These pre-treatments will be shortly discussed (Carrère et al., 2010).

1.5.1 BIOLOGICAL PRETREATMENT

Biological pretreatment aims to enhance anaerobic digestion by improving the hydrolysis process in an additional stage prior to the main digestion process. The most common method is temperature phased anaerobic digestion (TPAD), which uses a higher stage at either thermophilic (around 55 °C) or hyper-thermophilic (between 60 and 70 °C) conditions, anaerobic and aerobic. Thermophilic processes, and particularly thermophilic hydrolytic activity of bacterial populations, are the most widespread, mainly at a temperature of 55 °C. Thermophilic conditions generally result in an increase of the organic solids destruction rate, attributed to increased hydrolytic activity. Usually with this kind of pretreatment an increase of 25% of biogas production is achieved.

1.5.2 MECHANICAL PRETREATMENT

The most popular mechanical pretreatment is ultrasonic pretreatment. This process allows disruption within the cell structure and floc matrix. The ultrasonic method is based on two main mechanisms: cavitation, and chemical reaction due to the formation of OH•, HO_2• and H• radicals at high frequencies. This process allows the attainment of higher volatile solids destruction and the achievement of a sensible improvement in biogas production.

1.5.3 CHEMICAL PRETREATMENT

In this kind of pretreatment, the most widely used is ozonation. Ozonation allows the sludge to be well solubilized, whilst the yield increases together with the ozone dose. This process has been commercialized by the Japanese Kurita company. This pretreatment increases significantly in rate both biogas production and COD removal.

1.6 Anaerobic digestion (AD)

The Anaerobic digestion process is the manner by which microorganisms destroy biodegradable materials in the absence of oxygen. Anaerobic digestion occurs when the bacterial hydrolysis of the input materials begins. Acidogenic bacteria then, convert the sugars and aminoacids into carbon dioxide, hydrogen, ammonia, and organic acids. These bacteria convert these resulting organic acids into acetic acid, along with additional ammonia, hydrogen, and carbon dioxide. Methanogens convert these products to methane and carbon dioxide. The methanogenic archaea populations play an indispensable role in anaerobic wastewater treatments.

Anaerobic digestion is widely used as a source of renewable energy. It produces a biogas, consisting of methane, carbon dioxide and traces of other contaminant gases. This biogas can be used directly as fuel, in combined heat and power gas engines.

The steps that occur during anaerobic digestion are:

- hydrolysis;
- acidogenisis;
- acetogenesis.
- methanogenesis

There are several families of bacteria that are responsible for the whole mechanism:

- hydrolitic bacteria;
- acid forming bacteria;
- hydrogenotrophic bacteria.

1.6.1 HYDROLYSIS

During hydrolysis, hydrolytic bacteria transform the particulate organic substrate into soluble monomers and polymers. Proteins, carbohydrates and fats are transformed to aminoacids, monosaccharides and fatty acids respectively. Reaction 1.1 and reaction 1.2 show a typical hydrolysis reaction for a protein (cysteine) and a fatty acid.

$$4C_3H_7O_2NS + H_2O \rightarrow 4CH_4 + 6CO_2 + NH_3 + H_2S + CH_3COOH \tag{1.1}$$

$$C_xH_yO_z + (x - y/4 - z/2)H_2O \rightarrow (x/2 - y/8 + z/4)CO_2 + (x/2 + y/8 - z/4)CH_4 \tag{1.2}$$

1.6.2 ACIDOGENESIS

In the second stage, acid-forming bacteria transform the products of the first step into short chain volatile acids, alcohols, hydrogen and carbon dioxide. The principal acidogenesis stage products are shown in reactions 1.3, 1.4, 1.5. From these products, hydrogen, carbon dioxide and acetic acid bypass the acetogenesis, and can be utilized directly by the methanogenic bacteria in the final stage. Three typical acidogenesis reactions are showed below:

$$C_6H_{12}O_6 \leftrightarrow 2CH_3CH_2OH + 2CO_2 \tag{1.3}$$

$$C_6H_{12}O_6 + 2H_2 \leftrightarrow 2CH_3CH_2COOH + 2H_2O \tag{1.4}$$

$$C_6H_{12}O_6 \rightarrow 3CH_3COOH \tag{1.5}$$

1.6.3 ACETOGENESIS

In acetogenesis, the rest of the acidogenesis products are transformed by acetogenic bacteria into hydrogen, carbon dioxide and acetic acid. Hydrogen plays an important intermediary role in this process, as the reaction will only occur if the hydrogen partial pressure is low enough to thermodynamically allow the conversion of all the acids. Such lowering of the partial pressure is carried out by hydrogen scavenging bacteria, thus the hydrogen concentration of a digester is an indicator of its health.

Characteristic reactions of this stage are:

$$CH_3CH_2COOH + 3H_2O \leftrightarrow CH_3COOH + H_2CO_3 + 3H_2 \tag{1.6}$$

$$C_6H_{12}O_6 + 2H_2O \leftrightarrow 2CH_3COOH + 2CO_2 + 4H_2 \tag{1.7}$$

$$CH_3CH_2OH + 2H_2O \leftrightarrow CH_3COOH + 2H_2 \tag{1.8}$$

1.6.4 METHANOGENESIS

During this stage, microorganisms convert the hydrogen and acetic acid formed by the acid formers to methane gas and carbon dioxide. The bacteria responsible for this conversion are the hydrogenotrophic bacteria and they are strict anaerobic. Waste stabilization is accomplished when methane gas and carbon dioxide are produced.

The methane is produced in two ways. The first is described by reaction 1.9 and is the result of the bacteria's work that cause the anaerobic oxidation of the H_2 to occur, whilst the second is the "acetoclastic way", represented by reactions 1.10 and 1.11, where the anaerobic dismutation of acetic acid occurs.

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O \tag{1.9}$$

$$2C_2H_5OH + CO_2 \rightarrow CH_4 + 2CH_3COOH \tag{1.10}$$

$$CH_3COOH \rightarrow CH_4 + CO_2 \tag{1.11}$$

Figure 1.9 shows a summary of the whole anaerobic digestion process:

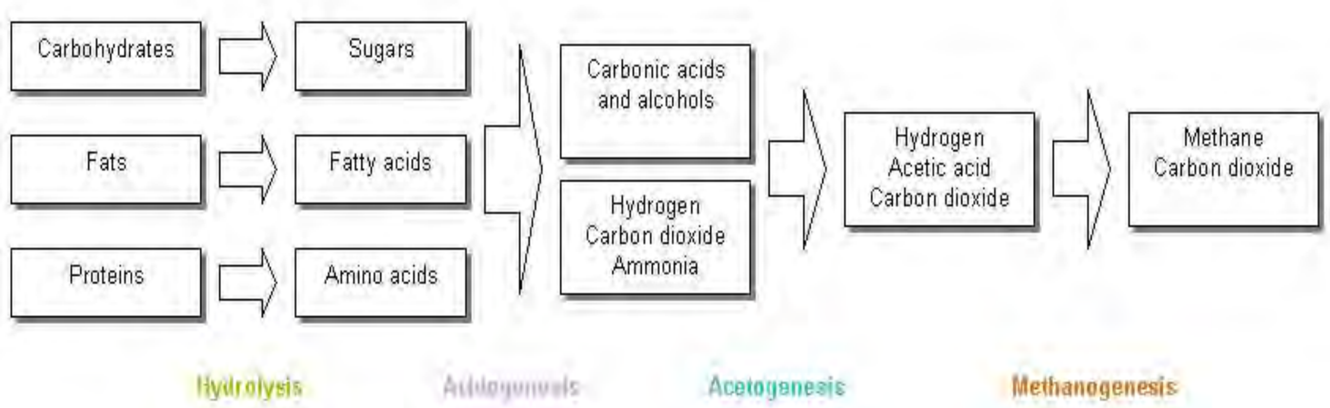

Figure 1.9 *Summary of the anaerobic digestion process.*

1.7 Aim of the work

This work aims, primarily, to explain the steam explosion process of sludge, and use the principles behind it to enhance the production of biogas through anaerobic digestion. A secondary aim is to see whether or not steam explosion is affected by cavitation, and to see how this physical phenomenon process could affect the production of biogas which, after all, is the final goal.

Chapter 2

The experiments

Three sets of experiments for the steam explosion pre-treatment method were prepared. The first involved two steam explosions of sludge at 3 and 6 bar. In the second, sludge was exploded from 3 and 4.5 bar. The third was conceived to draw comparisons between these three explosion pressures, all with the same sludge sample and even a sample of sludge that was not exploded.

The first two experiments the different response of the sludge to the three different pressures was investigated, and the changes in physic-chemical properties of the sludge over time, to see how those properties can affect biogas production.

In the third one, a series of data was obtained that are mutually comparable to each other and where is possible to see the real difference between the exploded sludge and the not exploded one in terms of biogas production.

A fourth experiment was set up, but the aim of this one was different, drawn from an original idea based on a smaller phenomenon that occurs during the steam explosion process, namely cavitation. In order to verify this idea, the fourth experiment was set up using the valve between the reaction tank and the flash vessel. The experiment includes an explosion at 6 bar, but with the valve opened in three different positions: 25%, 50% and 100%. In this way, it was possible to see whether cavitation has an influence over the steam explosion or not.

After the third and fourth experiments the samples were subjected to three analyses:

- Chemical oxygen demand (COD) measurement;
- Volatile fatty acids (VFA's) measurement;
- Microscopic analysis, for the third one alone.

At the end, all the experimental results were subjected to a statistical analysis to see whether they are statistically acceptable.

In order to understand the differences between the three different explosion pressures in a mechanical way, a hydrodynamic analysis was undertaken and a model has been developed. The same model has been used to investigate the same differences in the fourth experiment where the opening percentage of the valve has been changed.

2.1 The steam explosion plant

The experiments were performed in the Thames Water's plant, located in Reading, where there has been made available a pilot plant (Figure 2.2). This plant consists of a boiler (Figure 2.1), which produces the required steam, a reaction vessel (Figure 2.3), where hydrolyzation occurs, the flash tank (Figure 2.4), where the exploded sludge is collected, and the feed pump (Figure 2.5). There is also a control panel (Figure 2.6) from which it's possible to control the flash valve and the bottom valve of the flash tank for sludge collection. Additionally, it’s possible to monitor the pressure through digital pressure indicators. Figure 2.7 shows the P&ID of the pilot plant.

Figure 2.1 *Boiler.*

Figure 2.2*Steam explosion pilot plant.*

Figure 2.3 *Reaction vessel.*

Figure 2.4 *Flash tank.*

Figure 2.5 *Feed pump.*

Figure 2.6 *Control panel.*

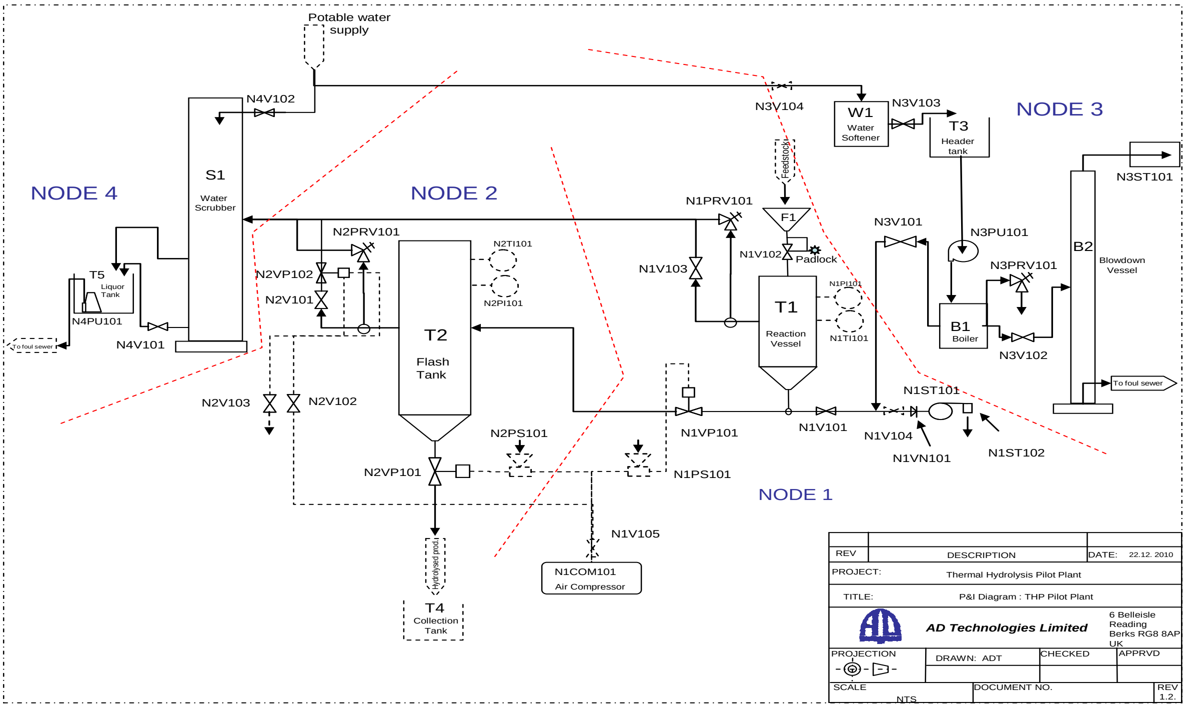

Figure 2.7 *P&ID of the pilot plant.*

The P&ID is divided in four nodes, one node for each part of the process: production of steam (node 3), reaction (node 1), the flash (node 2) and the purge (node 4). In each node there are the equipment used in that particular part of the plant, labelled with a serial code that depends on the node.

2.2 Samples preparation

In both experiments a 60:40 mixture, based on dry solids (DS),of primary and secondary sludge, respectively, was prepared. In order to produce the samples it was necessary to undertake DS analysis. This type of analysis measures the quantity of dry solids that the sludge contains. This requires weighing one sample each of primary and secondary sludge into two different crucibles, and placing them in the oven at 105 °C for 24 hours. In order to make calculations for the AMPTS (the acronym AMPTS stand by Automatic Methane Potential Test System) we also needed the volatile solids (VS). The procedure necessary to obtain this data involved placing the crucible containing the dried sludge in the muffle furnace at 550 °C for another 24 hours, immediately after removing it from the oven.

For the first experiment a sample with 9.93% of DS for the primary sludge and 7.03% of DS for the secondary activated sludge was obtained. In order to make the 60:40 mixture the total quantity of sludge to put inside the reactor was decided. Relying on previous experiments, approximately 20 kg was used. Thus, 12 kg of primary sludge were weighed and mixed with 11.4 kg of secondary sludge. In the second experiment a sample with a DS of 7.04% for the primary sludge and 6.7% for the secondary sludge was obtained. In the third experiment the DS for the primary sludge was 12.13% and 11.92% for the secondary.

The next step was to dewater the sludge in order to produce a mixture with reasonable DS value. To do this, it was necessary to leave the sludge to lose water for a couple of days, and undertake the DS analysis again to be sure that the total DS was correct, before doing the steam explosion.

2.3 Steam explosion

The procedure for the steam explosion experiments was the same in each experiment. The feed was loaded by the pump, located at the top of the rig, and once the boiler was ready to use, the reactor was filled with steam until the pressure reached 6 bar. At this point it was necessary to keep the pressure constant during all the 30 minutes required for the sludge to be hydrolysed. To do this, the manual flow control valve of the steam must be adjusted. This is the crucial phase of the experiment because, if the pressure inside the reactor vessel is not controlled, the sludge will not be adequately hydrolysed and this will cause problems in the subsequent phases of the experiment. After 30 minutes, if the explosion must be made at 6 bar, the on/off valve between the reaction vessel and the flash vessel is opened, causing the steam explosion of the sludge in the flash vessel. At this point the sludge sample is collected from the bottom of the flash vessel, opening its on/off valve. If the explosion has to be made at 3 or 4.5 bar, after the 30 minutes, the pressure can be adjusted by opening the purge valve on the reaction vessel until the desired pressure is reached. After that, the valve between the reaction vessel and the flash vessel can be opened for steam explosion to occur. The ideal pressure vs time profiles for these experiments are represented in Figure 2.8, Figure 2.9 and Figure 2.10.

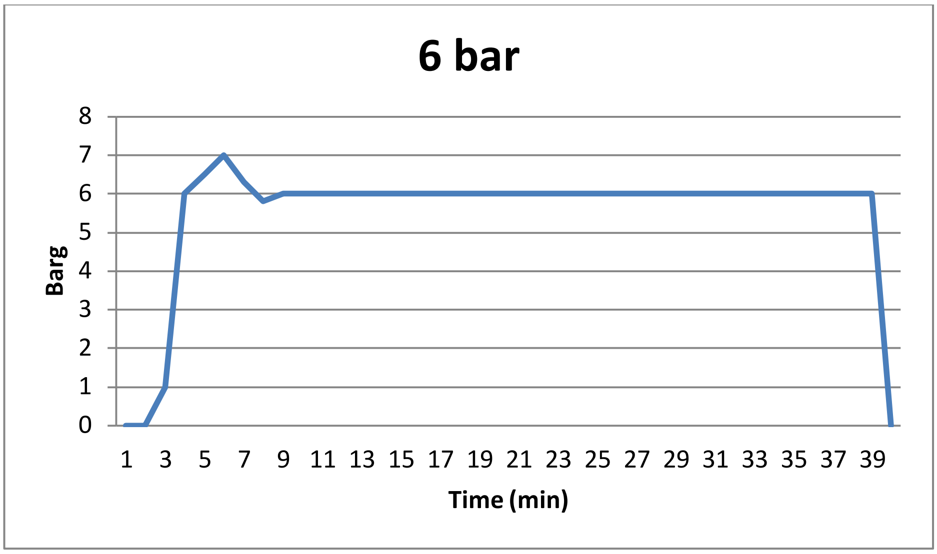

Figure 2.8 *Ideal pressure graph for the 6 bar experiment.*

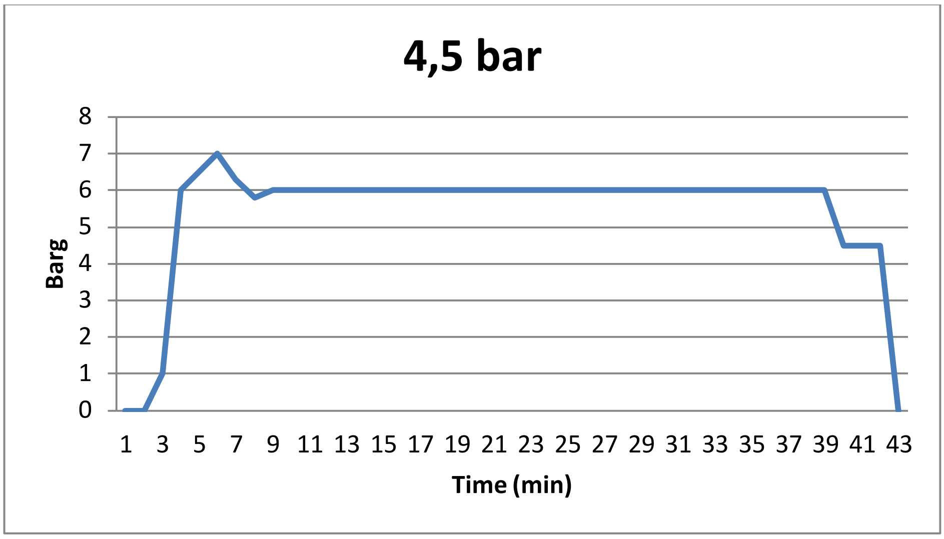

Figure 2.9 *Ideal pressure graph for the 4.5 bar experiment.*

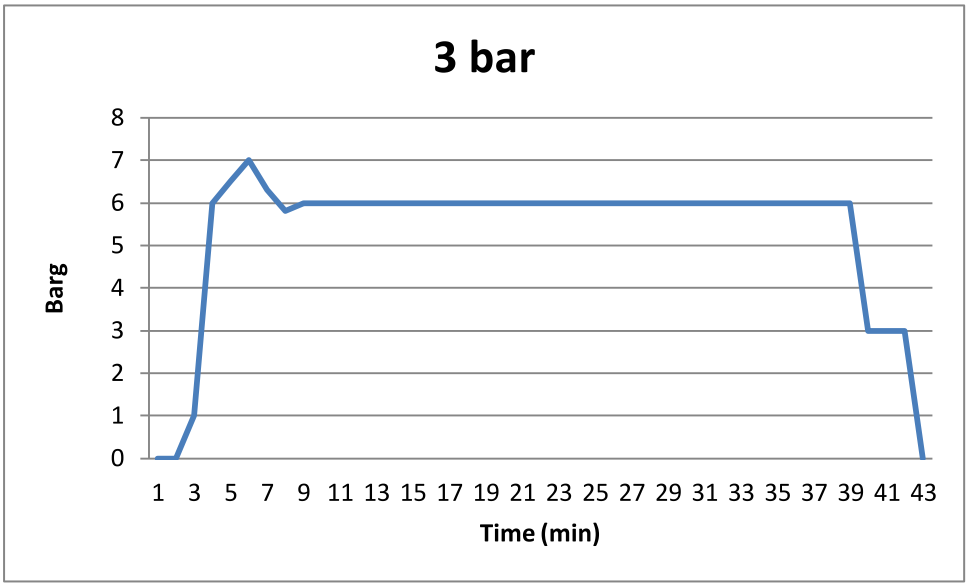

Figure 2.10 *Ideal pressure graph for the 3 bar experiment*

2.4 AMPTS test

After steam explosion, the sludge samples were combined with inoculum and distilled water in a convenient amount, the value of which was calculated based on the DS and VS percentage of the exploded sludge and the inoculum, so that there is the same amount of organic load for each sample in order to have comparable results.

In the first and second experiment 6 bottles of 2 liters each were used. For the first experiment two bottles for the 6 bar exploded sludge, two bottles for the 3 bar one and two bottles for the inoculum were used.

The second experiment follows the same scheme, two bottles for the exploded sludge at 4.5 bar, two bottles for the 3 bar and two bottles for the inoculum.

The third experiment was set up with three bottles for each samples of exploded sludge. To compare the results with the not exploded sludge, also two bottles of raw sludge and one of inoculum has been analysed. In the third experiment the seed was a mixture of exploded seed derived from the exploded digested sludge and the normal digested sludge. It was a 50:50 mixture based on the DS and was used for all the bottles. Thus twelve bottles of 2 liters were used. In all these experiments the inoculum was used as a blank, a reference sample of which was then subtracted from the results.

Once the samples were ready they were put in the AMPTS. A set of equipment is presented in Figure 2.11: it comprises of a water bath where the bottles are immersed at 38 °C (Figure 2.12), a CO_2 stripping device (Figure 2.13), a biogas measuring device (Figure 2.14) and a data logger, which is a laptop with the programme needed for data collection. On the top of the 2 litre bottles there is a cap which contains a motor for stirring the sludge while it undergoes the Anaerobic Digestion process.

The CO_2 stripping device consist of a set of bottles of 100 ml each with a 3M NaOH solution inside. The solution is dosed with 0.1% alizarin yellow R solution for indication of the pH value. When the solution turns from red to yellow it means that it is not absorbing CO_2 anymore and must be replaced with the fresh one. The water bath is composed of a basin with a capacity of 5 liters surrounded by internal electrical resistances, the temperature of which can be adjusted from an analogic interface placed on the side of the basin.

Figure 2.11 *Set of equipments that composed the AMPTS.*

Figure 2.12 *Water bath with the 2 liters bottles inside.*

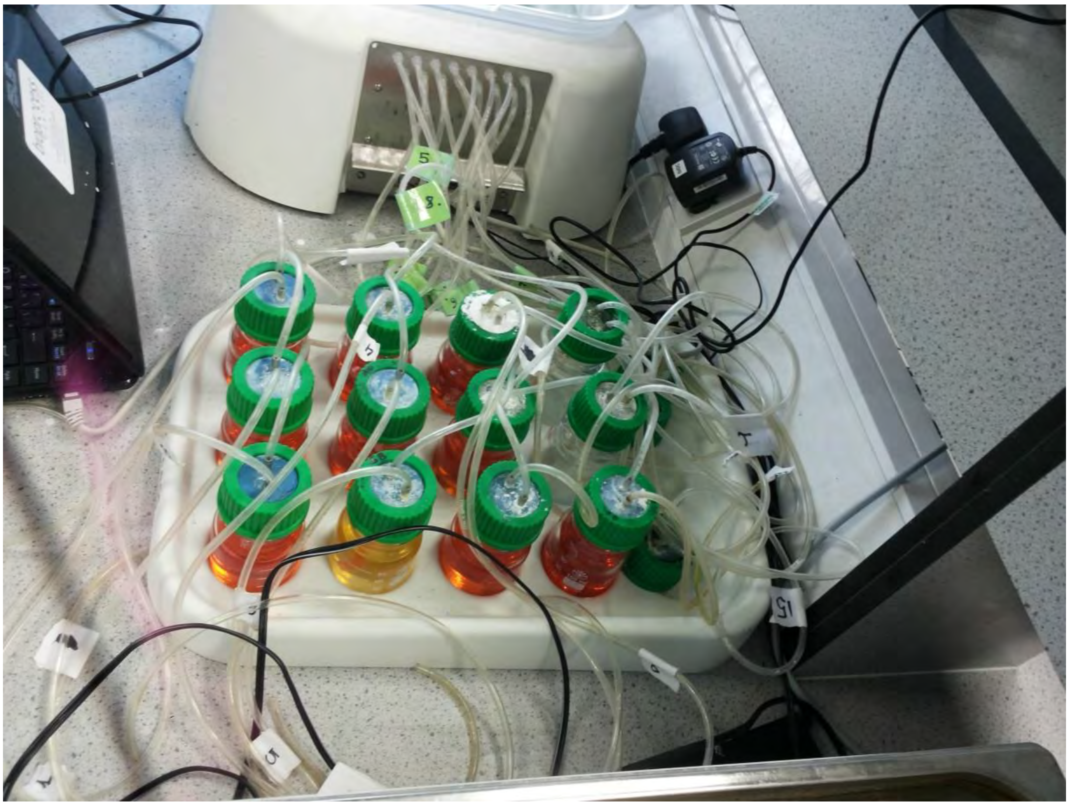

Figure 2.14 *CO_2 stripping device.*

Figure 2.14 *Biogas measuring device.*

2.5 COD analysis

All the sludge samples must be diluted with distilled water in a 1:10 ratio before starting the analysis: 10 ml of sludge in 90 ml of distilled water. Then, in order to make the COD analysis, the test kit LCK 514, produced by the Hach Lange GMBH, has been used. To run the analysis there is a procedure to follow, using the instructions given by the company and the chemicals that are included in the test kit, and then heating the samples for 15 minutes at 170 °C. Then, the test tubes with bar codes are inserted in the machine to give the results of the COD in mg/l.

2.6 VFA analysis

Before running this analysis, the sludge must be centrifuged for one hour to 4000 rpm. This was done with a Centrifuge 5810, produced by Eppendorf. Then, the test kit LCK 365, produced by the Hach Lange GMBH, was used. There is always a procedure to follow, given by the company, with different chemicals. Before reading the VFA's, the samples must be heated up to 100 °C for 10 minutes. After that it's possible to insert the test tubes with the bar code in the machine in order to have the results of the VFA's analysis in mg/l.

Figure 2.15 shows the machine used to read the results, a Hach Lange DR3900. This machine reads the bar code on the test tubes to see what kind of analysis must be run, and then shows the results on the display.

Figure 2.15 *The machine used to read the results of the COD and VFA analysis.*

2.7 Microscope analysis

The microscope analysis has been undertaken in order to understand whether the higher explosion pressure causes a crushing in the particle size of the sludge. However, these are purely qualitative results. To run this analysis the microscope Polivar Met, produced by Reichert-Jung (Figure 2.16), equipped with a camera (Canon EOS 50D) was used.

The sludge samples have been put over a laboratory slides and spread to achieve a better particle distribution under the microscope. The microscope has been set with 5x0.10 Epi resolution.

Figure 2.16 *Microscope Polivar Met.*

2.8 Statistical analysis

In order to see if the results achieved in this work are reliable, a statistical analysis was applied. An analysis of the standard error has been done comparing the increasing rate of biogas production. The increasing rate is calculated as follows:

$$x_i = \frac{y_j}{y_k} \tag{2.1}$$

Where:

- x_i is the increasing rate.
- y_j is the amount of biogas produced at the pressure *i*.
- y_k is the amount of biogas produced at the pressure k. Pressure k is the reference pressure.

All the parameters are taken each time from the same experiment and then all the increasing rate for all the experiment are used to calculate the standard error σ_e.

$$\sigma_e = \sqrt{\left(\frac{\sum(x_i - \bar{x})^2}{(n(n-1))}\right)} \tag{2.2}$$

Two analysis have been done, one for the 6 bar pressure and one for the 4,5 bar pressure.

At the end we can estimate the confidential interval for the increasing rate for each pressure analysed can be estimated.

2.9 Hydrodynamic analysis

The hydrodynamic analysis was made in order to analyse the pressure drop inside the reactor tank and see how the discharge time of the liquid changes when the driving force changed in the third experiment. The same model has been used to analyse the discharge time and the pressure in the reactor tank when the pressure drop along the pipes changed in the fourth experiment (due to the different opening of the valve near to the flash tank).

This model can be used to calculate the discharge time of the liquid. The liquid inside the reactor is composed of water, the condensed steam used to reach the final temperature, and the sludge.

In order to calculate the quantity of water inside the reactor, the mass balances have to be written. Overall mass balance (Equation 2.3) and the mass balance based on the DS (Equation 2.4) are as follow:

$$m_{sludge} + m_{water} = m_{tot} \tag{2.3}$$

$$m_{sludge} DS_{sludge}^{in} = m_{tot} DS_{sludge}^{out} \tag{2.4}$$

Where m_{sludge} is the total mass of the sludge charged in the reactor, m_{water} is the amount of water used in the experiment, DS_{sludge}^{in} is the DS value of the sludge before the steam explosion and DS_{sludge}^{out} is the DS value of the sludge after the steam explosion.

Combining these two equations (2.3 and 2.4) the mass of water can be calculated:

$$m_{water} = m_{sludge} \frac{(DS_{sludge}^{in} - DS_{sludge}^{out})}{DS_{sludge}^{out}} \tag{2.5}$$

So it results that there are 6,5 kg of water in the reactor.

To develop the model, to calculate the pressure drop in the circuit, the velocity heads method has been used:

$$\frac{\Delta P}{\rho g} = k \frac{u^2}{2g} \tag{2.6}$$

$$k = k_{on/off\ valve} + 3k_{standard\ elbows\ 90°} + k_{pressure\ drop\ distributed} + k_{gate\ valve} \tag{2.7}$$

Where ΔP is the pressure drop between the reactor tank and the flash tank, ρ is the density of the water, g is the gravitational acceleration and u is the velocity. The parameter k (2.7) is the sum of all the resistances that the flow has to face during the route between the reactor tank and the flash tank.

Table 2.1 shows the values for each *k*:

Table 2.1 *k values.*

$k_{\text{on/off valve}}$	0,15
$k_{90° \text{ standard elbows}}$	0,8
$k_{\text{pressure drop distributed}}$	0,34
$k_{\text{gate valve } 25\%}$	112
$k_{\text{gate valve } 50\%}$	36
$k_{\text{gate valve } 100\%}$	9

Then, knowing the pressure drop we can calculate the initial velocity u_0 as:

$$u_0 = \sqrt{\frac{2\Delta P}{\rho k}} \tag{2.8}$$

It's clear that this velocity will change in every integration step because of the pressure that decrease step by step. Once that the starting velocity is known, is possible to calculate the volumetric flow rate:

$$\dot{V}(t) = u_0 A \tag{2.9}$$

Then, the volume variation can be calculated as :

$$\frac{dV}{dt} = \dot{V}(t) \tag{2.10}$$

So, once that the liquid volume variation is known, the corresponding increase of the steam volume in the reactor can be calculated, and the new pressure using the ideal gas law, since the number of mole in the vapour phase *n,* and the temperature *T,* are assumed constant:

$$P_1 V_1 = nRT \tag{2.11}$$

And now is possible to calculate the new velocity from the 2.1 and start the procedure all over again until the liquid in the tank is completely discharged.

The equation model was integrated numerically, by means of the Euler's method. It was checked that the results obtained are the same when the integration step was 0.01s and 0.001s, so that an integration step of 0.01s was chose (Δt=0.01s) in order to achieve good accuracy in the results with less iteration.

Below, as an example, will be shown the first step of the model calculation.

1. Find u_0 : $u_0 = \sqrt{\frac{2\Delta P_0}{\rho k}}$ where ΔP_0 is 600000 Pa, the initial pressure drop.
2. Find $\dot{V}_0$: $\dot{V}_0 = u_0 A$ where A is the area of the pipe.
3. Find the residual volume of the liquid and the steam: - $L_1 = L_0 - \dot{V}_0 \Delta t$;
   - $V_1 = V_0 - \dot{V}_0 \Delta t$.
4. With V_1 we can calculate the new pressure: $P_1 = \frac{nRT}{V_1}$.

With P_1 we can find the new velocity and start the procedure again, until L_n=0.

Chapter 3

Results and discussion

In this chapter the results obtained will be described and discussed through the interpretation of the graphs measured in each experiment, then the results will be compared in order to make the final discussion and comments on the whole process.

All these graphs show the biogas production for each experiment. In the first ones the biogas production per day or per hour is represented, showing how fast the biogas is produced and in how many days or hours it is produced. This is useful in order to compare the different pressures to see which one is more effective. The second graphs show the cumulative biogas production, i.e., the total amount of biogas produced in the whole time of the experiment.

In all the graphs the average of the samples for each pressure is plotted.

In this chapter the results of the COD, VFA's and microscope analysis will be shown.

3.1 First and second experiment

3.1.1 FIRST EXPERIMENT

Figure 3.1, Figure 3.2 report the results of the first experiment.

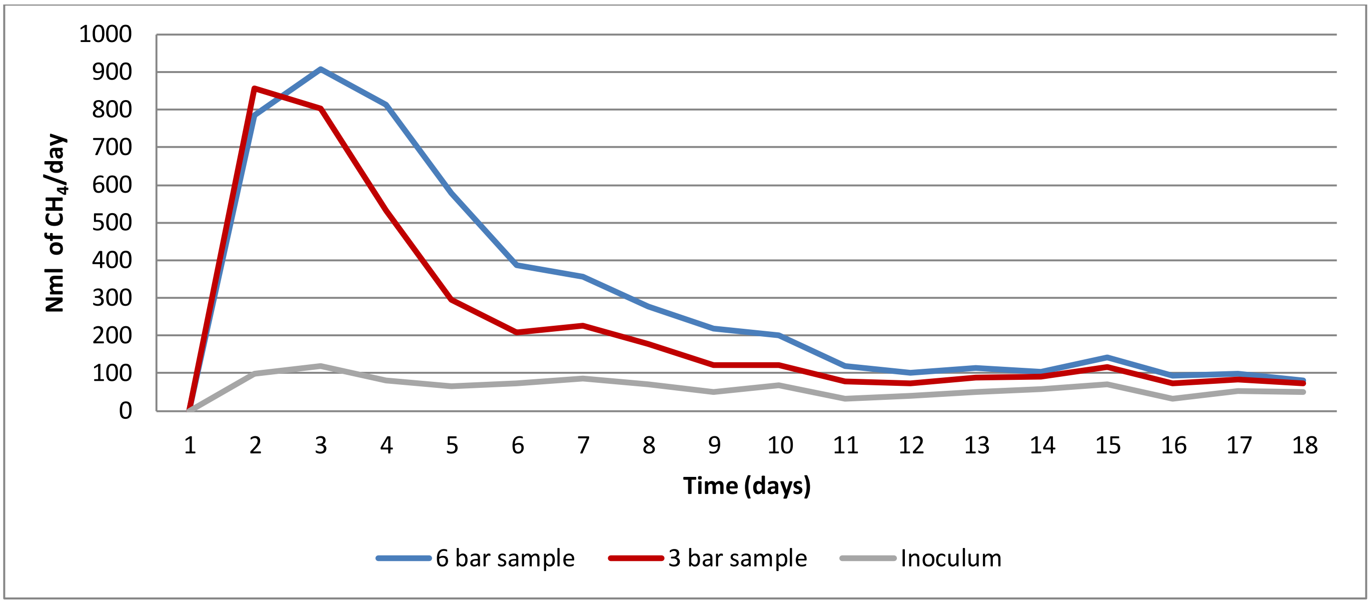

Figure 3.1 *Instantaneous biogas production per day for the first experiment.*

The production of biogas of the 3 bar and the 6 bar samples. It's clear that after the first couple of days the 6 bar sample starts to produce more biogas than the 3 bar one, as expected. After four days the biogas production starts to slow down more and more until the samples reach more or less the same amount of gas produced per day.

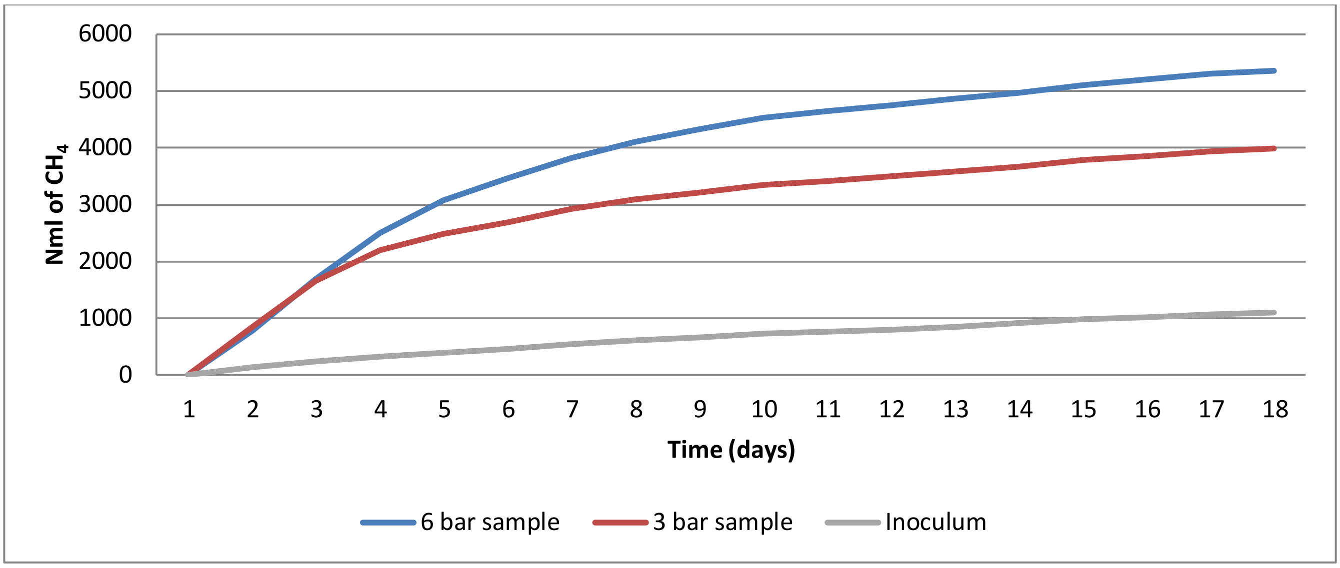

Figure 3.2 *Cumulative biogas production for the first experiment.*

However, the total amount of gas produced in the whole time of the experiment and the difference between the two samples is substantial as the sludge exploded at 6 bar produce one liter of CH_4 more than the sludge exploded at 3 bar (Figure 3.2).

3.1.2 SECOND EXPERIMENT

Figure 3.3 shows the production of the biogas per day of the sludge exploded at 3 bar and 4.5 bar. This time the difference between them is not so big even if the 4.5 exploded sludge produce more biogas anyway.

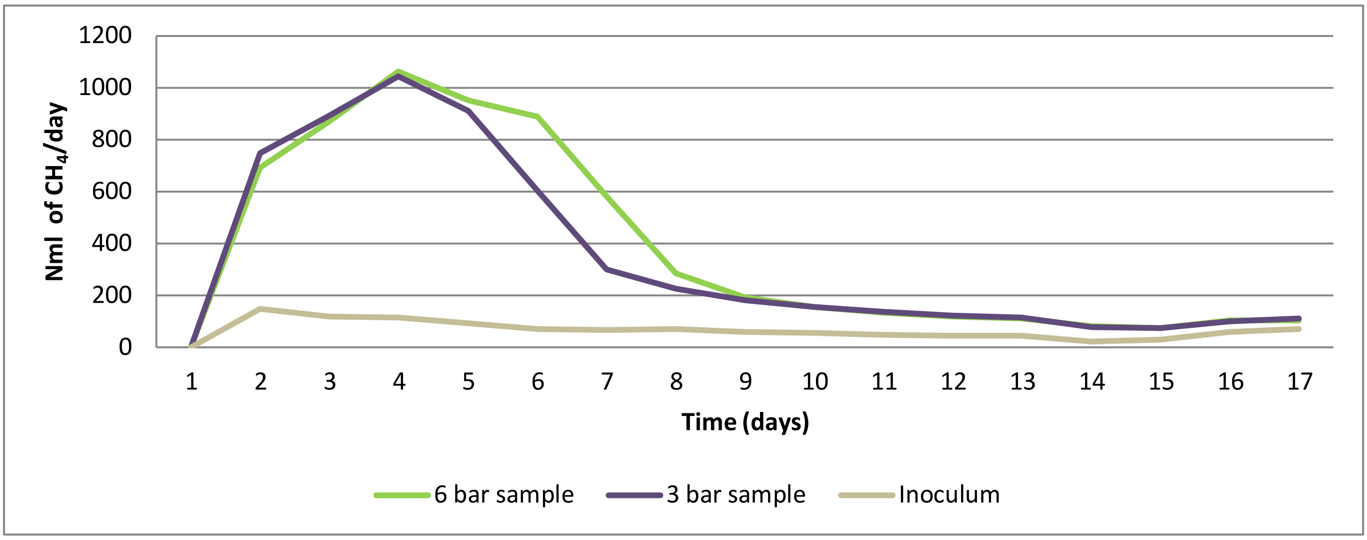

Figure 3.3 *Instantaneous biogas production per day for the second experiment.*

Figure 3.4 shows the different amount of biogas produced by the two samples, the difference is not as remarkable as it was in the previous experiment with the 6 bar exploded sludge.

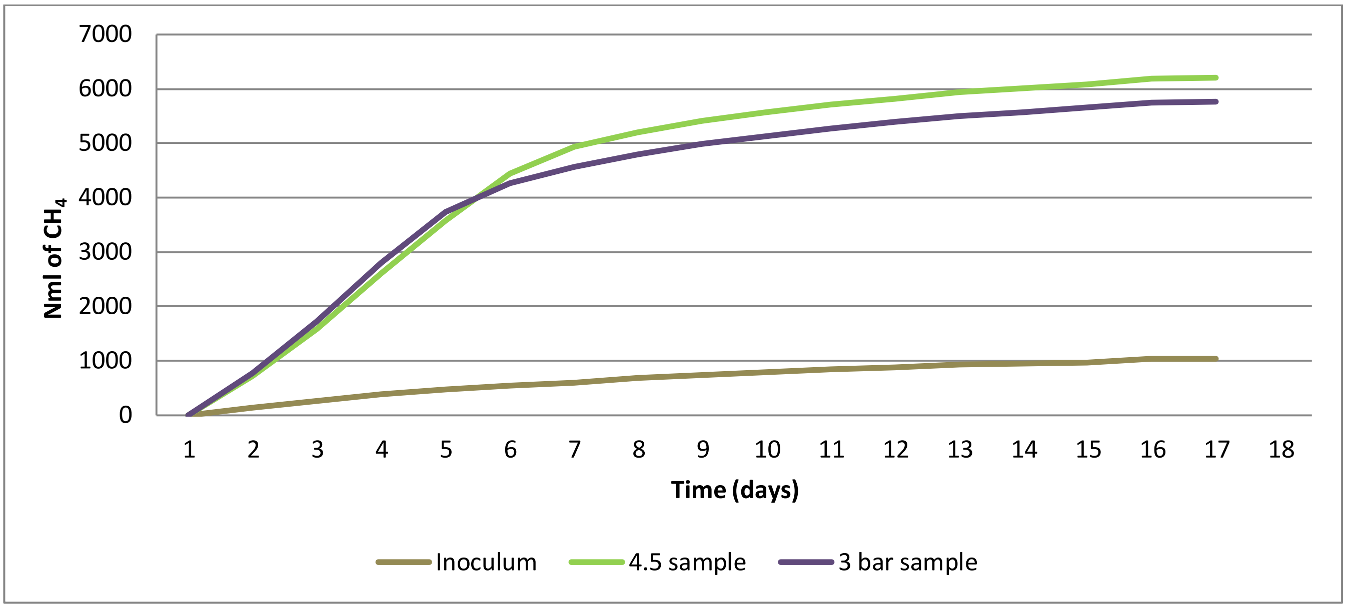

Figure 3.4 *Cumulative biogas production for the second experiement.*

3.1.3 COMBINED RESULTS OF THE FIRST AND SECOND EXPERIMENT

The graphs below (Figure 3.5 and Figure 3.6) show the combined results of the first and second experiment. There are two main point worth to analyse:

1. The 3 bar exploded samples from the two experiment produce a quite different amount of biogas even if the sludge collecting point was the same.
2. The sludge exploded to an higher pressure produce more biogas both in terms of cumulative amount and punctual amount in both experiments.

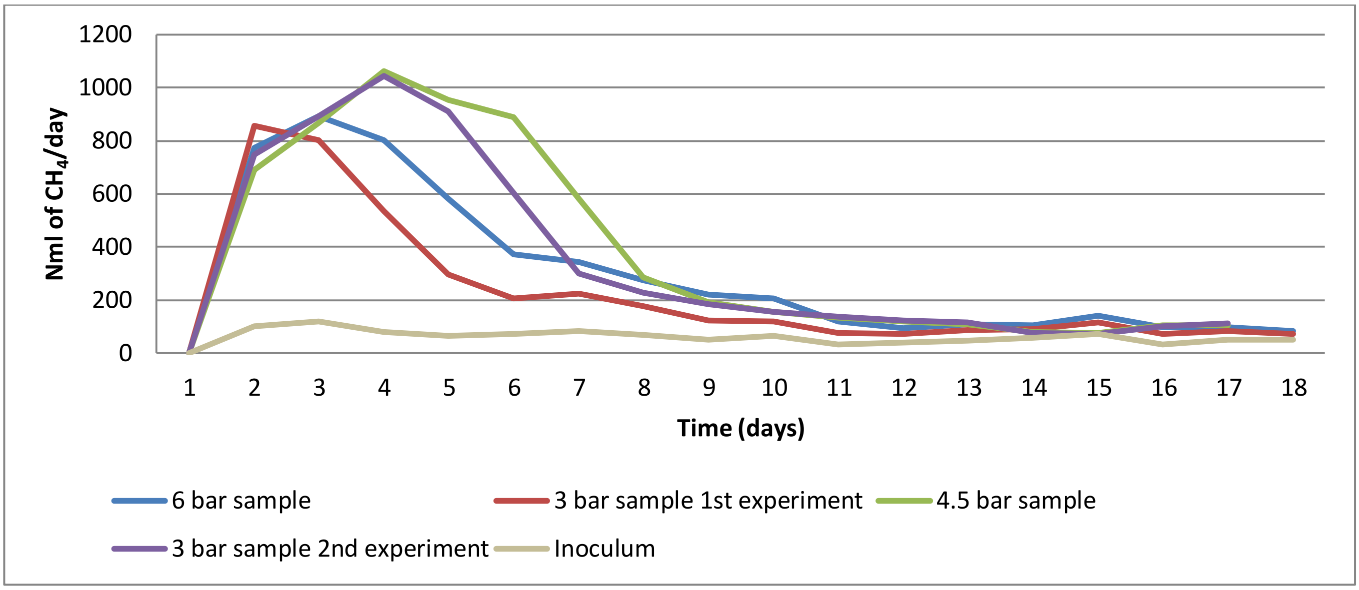

Figure 3.5 *Combined instantaneous biogas production per day.*

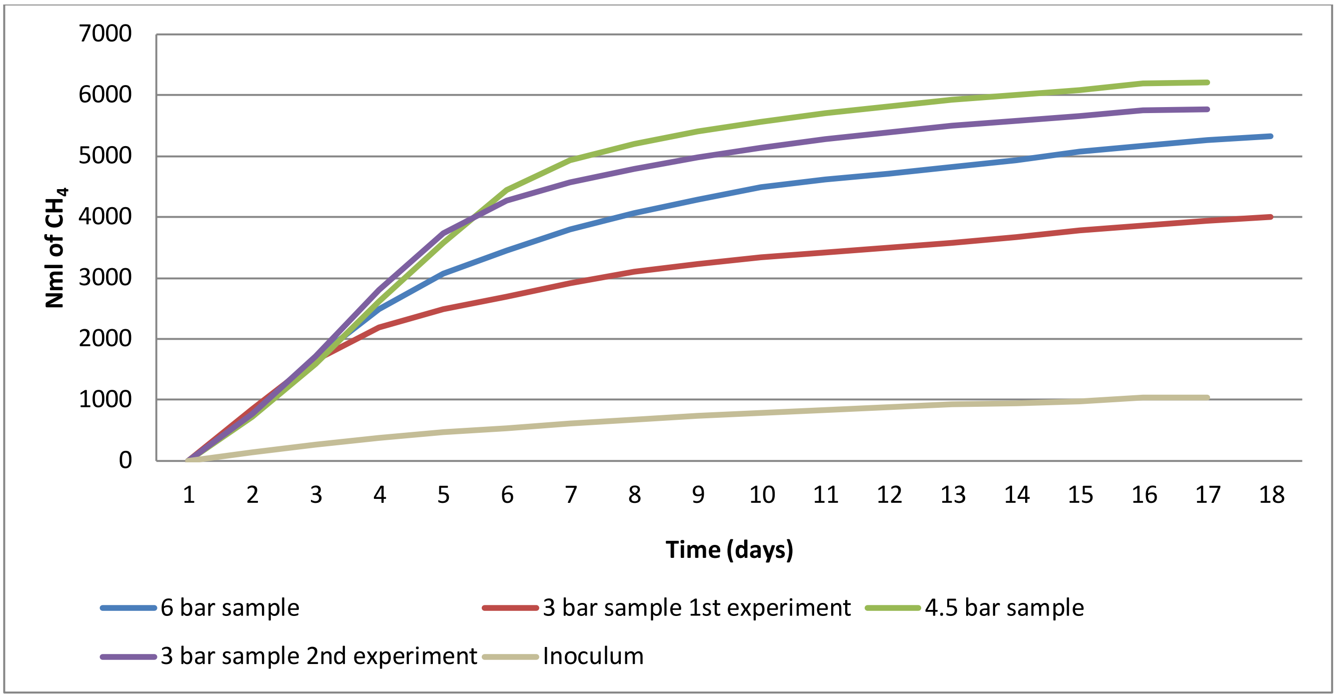

Figure 3.6 *Combined cumulative biogas production.*

3.2 Third experiment

3.2.1 INOCULUM AND UNEXPLODED FEED SAMPLES

Figure 3.7 and Figure 3.8 show both the instantaneous and cumulative amount of biogas produced by the normal samples (the unexploded sludge) and the inoculum.

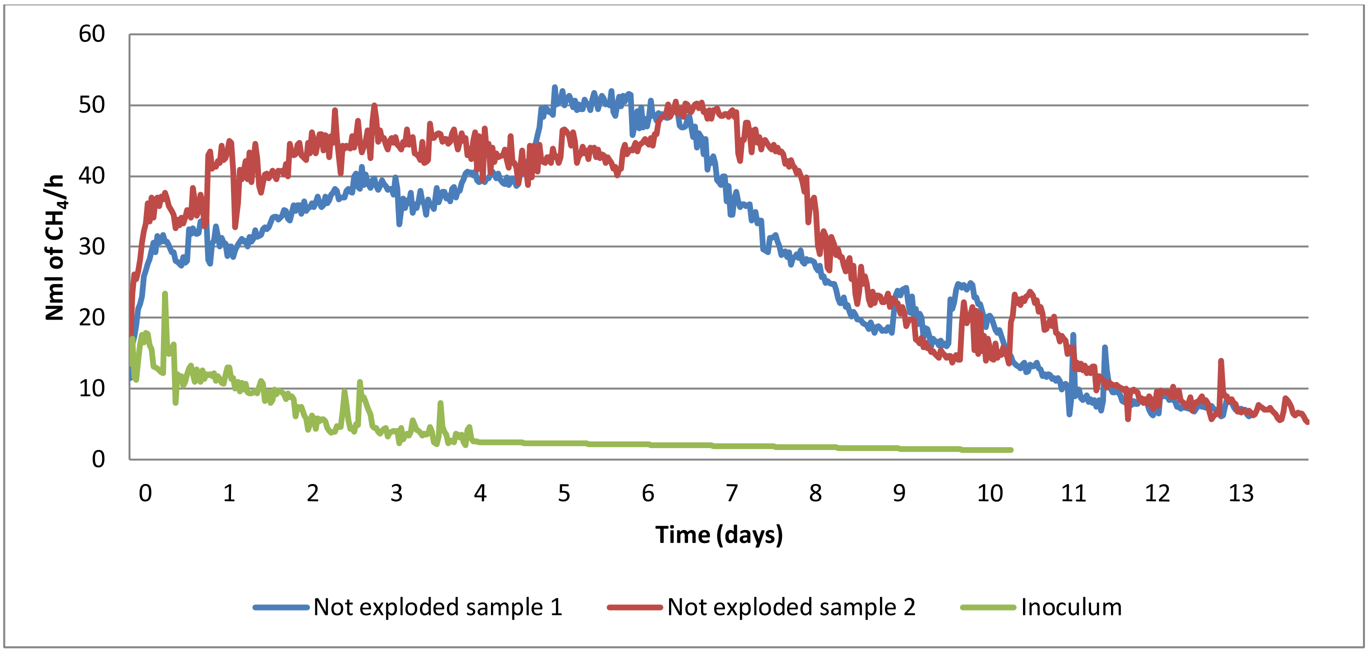

Figure 3.7 *Instantaneous biogas production for the unexploded feed and inoculum.*

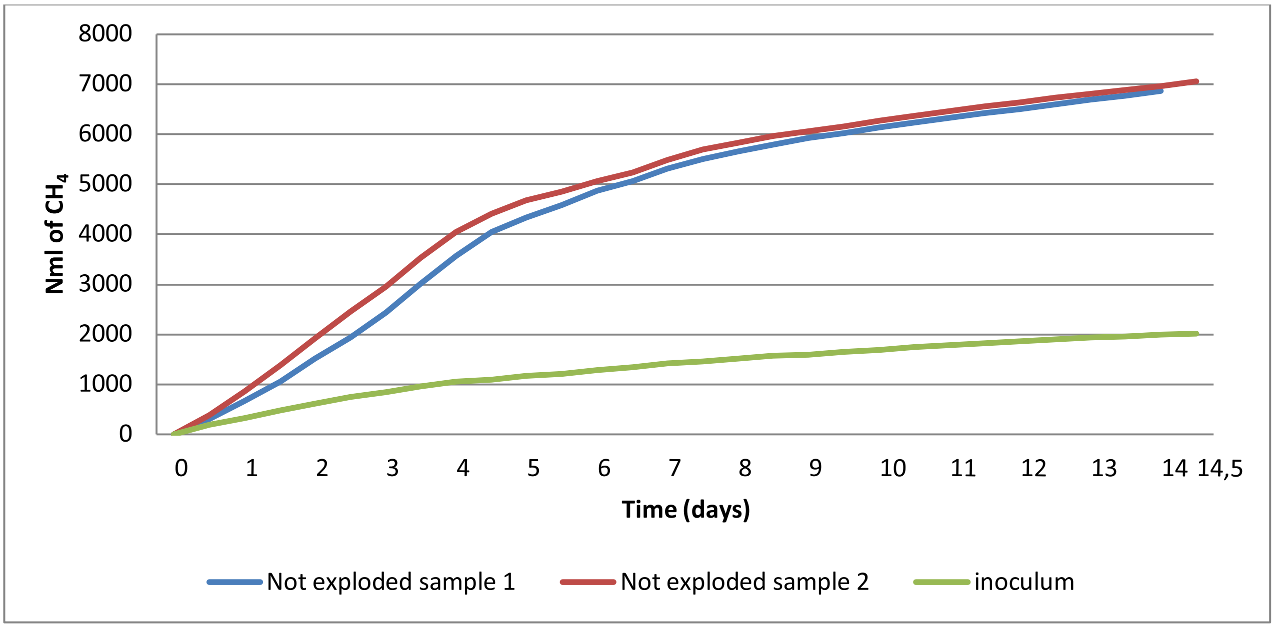

Figure 3.8 *Cunulative biogas production for the unexploded feed and inoculum.*

3.2.2 3 BAR SAMPLES

Figure 3.9 and Figure 3.10 show both the instantaneous and cumulative amount of biogas produced from the 3 bar samples.

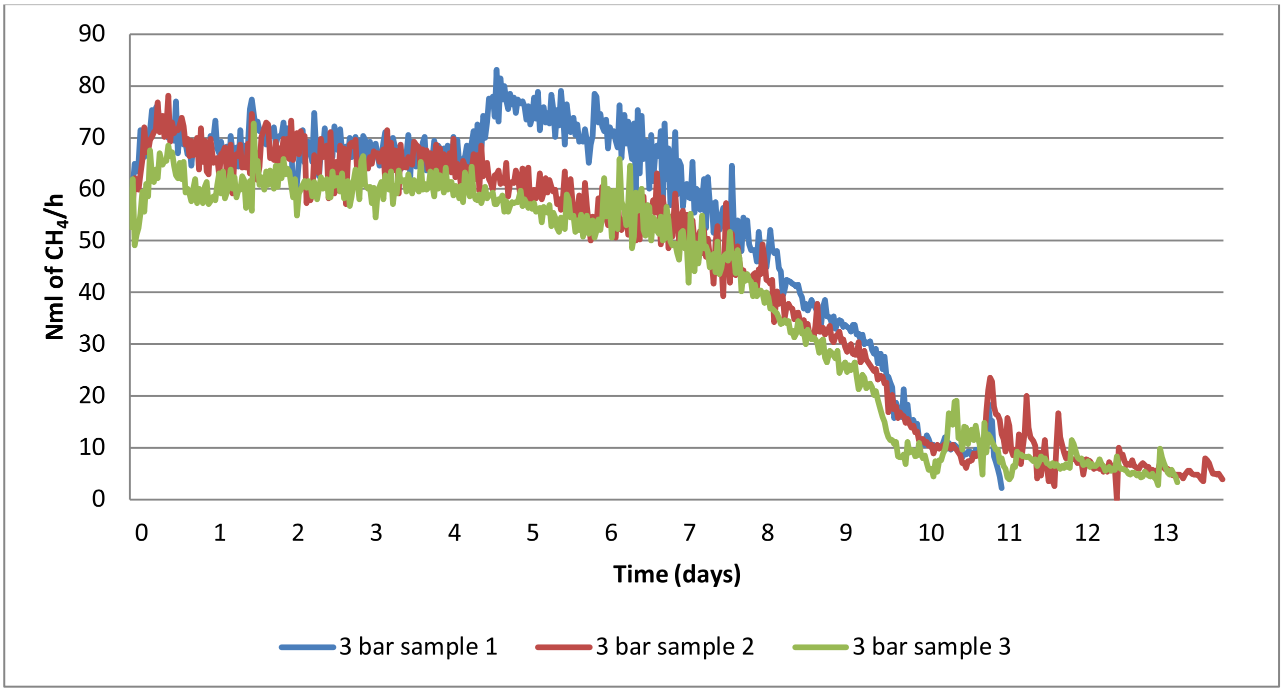

Figure 3.9 *Instantaneous biogas production for the 3 bar samples.*

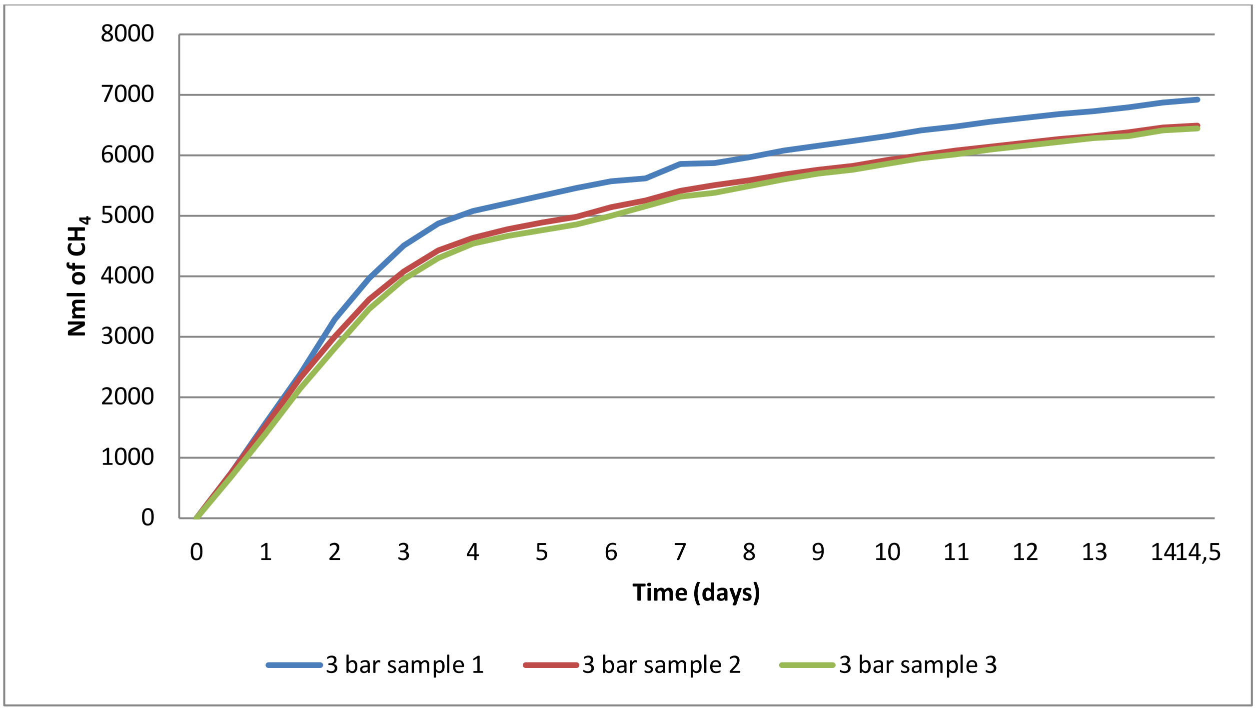

Figure 3.10 *Cumulative biogas production for the 3 bar samples.*

3.2.3 4.5 BAR SAMPLES

Figure 3.11 and Figure 3.12 show both the instantaneous and cumulative amount of biogas produced from the 4.5 bar samples.

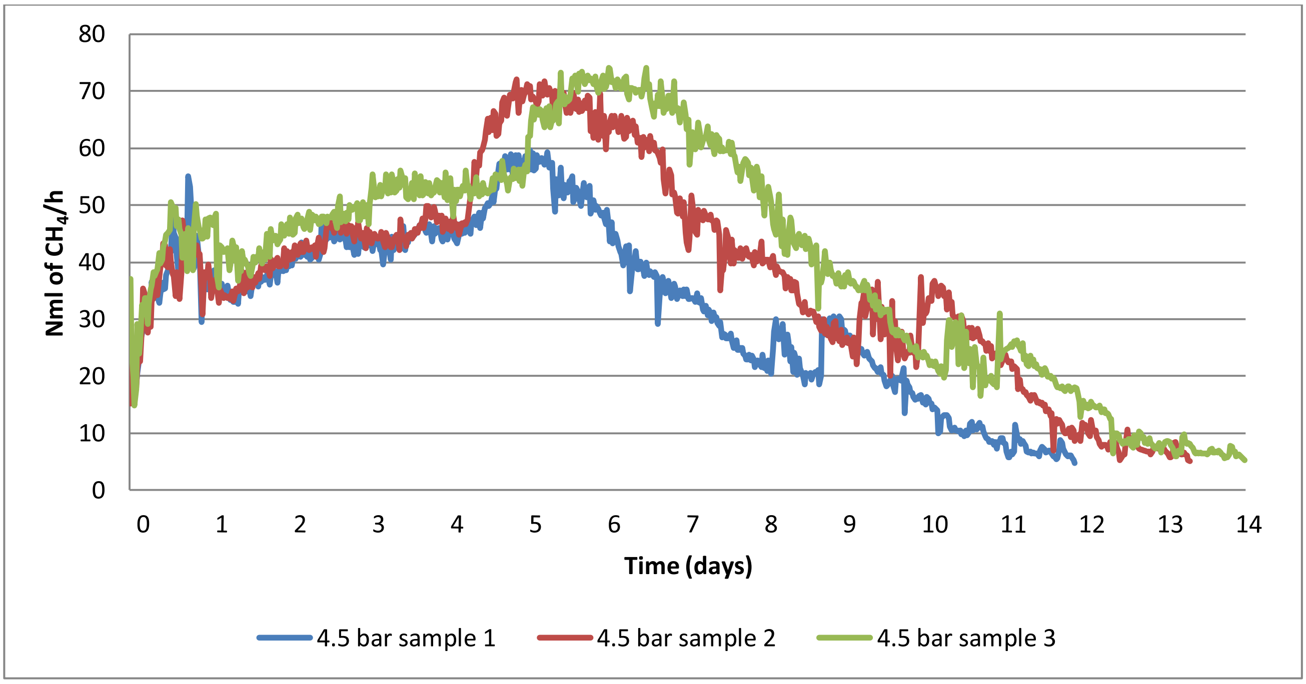

Figure 3.11 *Instantaneous biogas production for the 4.5 bar samples.*

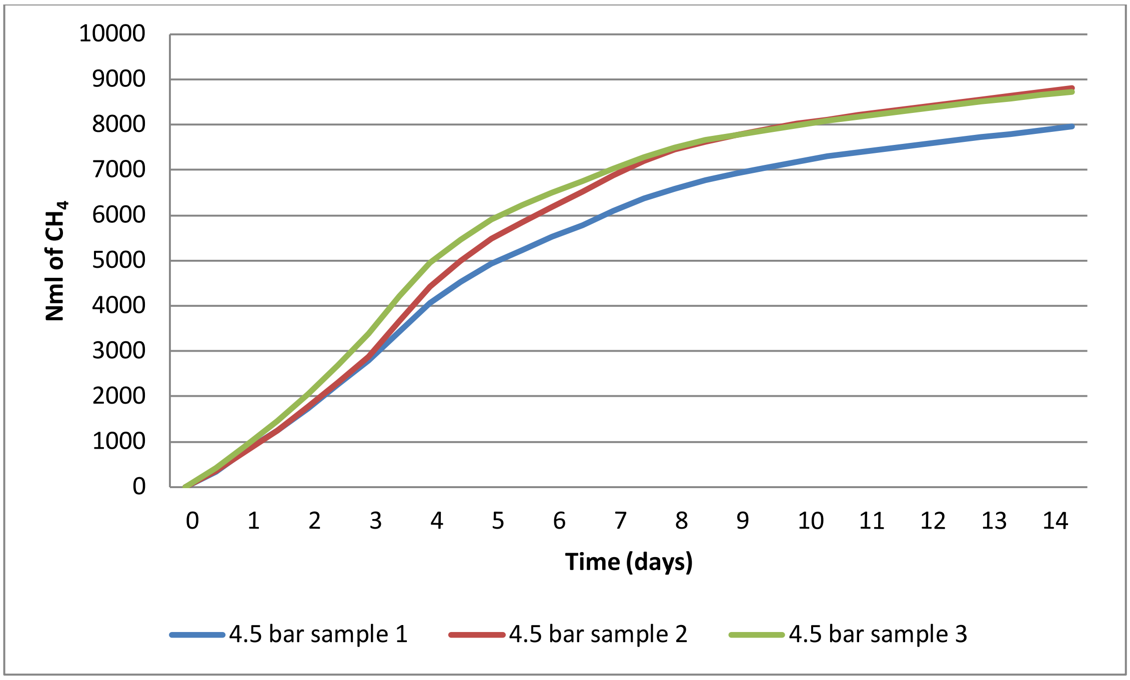

Figure 3.12 *Cumulative biogas production for the 4.5 bar samples.*

3.2.4 6 BAR SAMPLES

Figure 3.13 and Figure 3.14 show both the cumulative and instantaneous amount of biogas produced from the 6 bar samples.

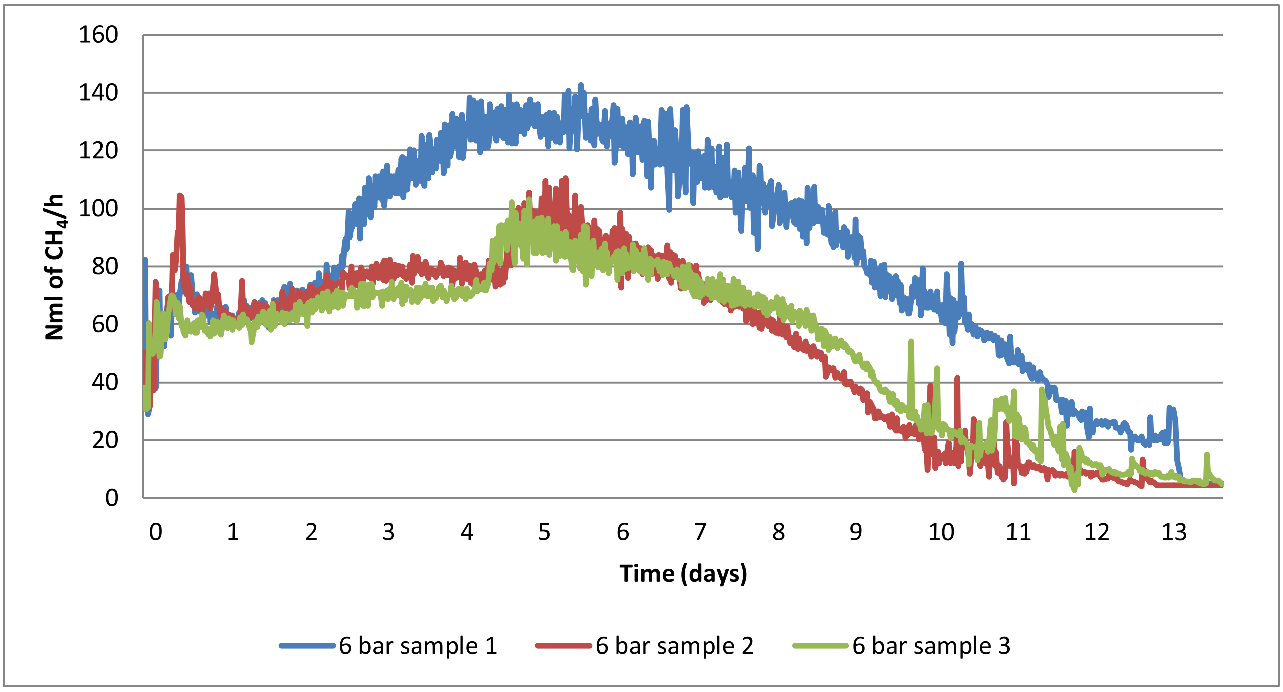

Figure 3.13 *Instantaneous biogas production for the 6 bar samples.*

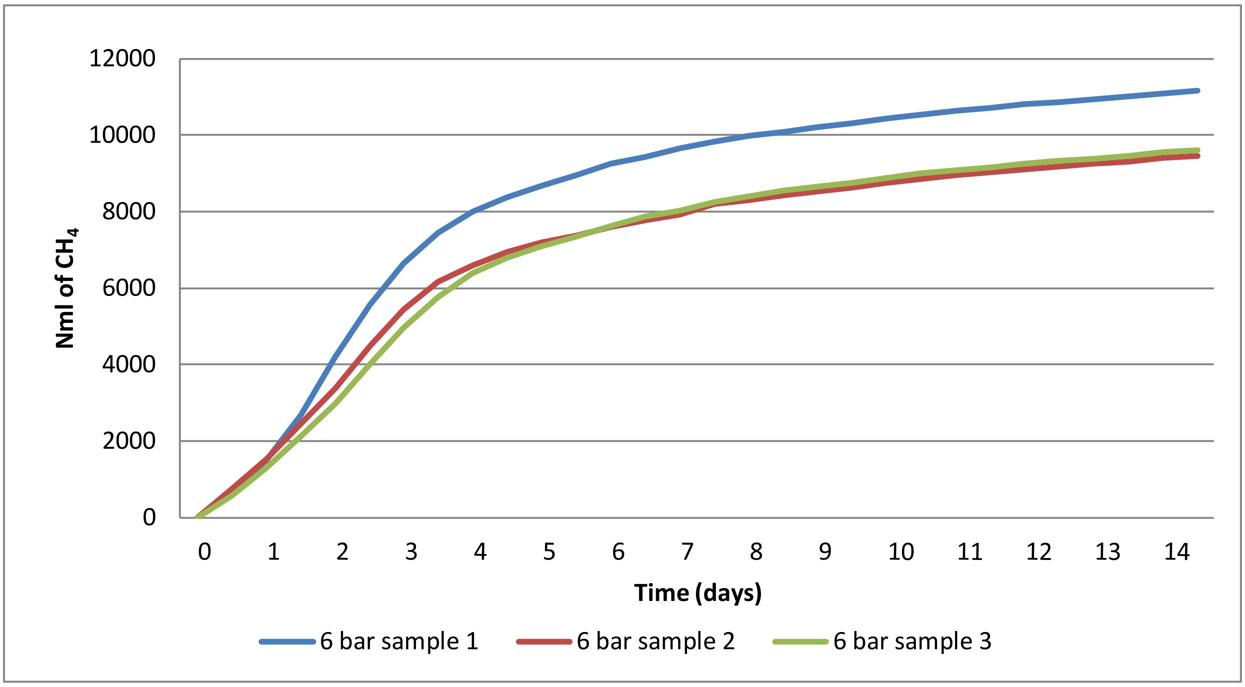

Figure 3.14 *Cumulative biogas production for the 6 bar samples.*

3.2.5 COMBINED RESULTS

The results of the third experiment are here compared and discussed. Figure 3.15 and Figure 3.16 show both the instantaneous and cumulative profiles of biogas amounts from all the samples together. The average values of the previous samples from each pressure are reported, reduced by amounts of biogas produced from the inoculum.

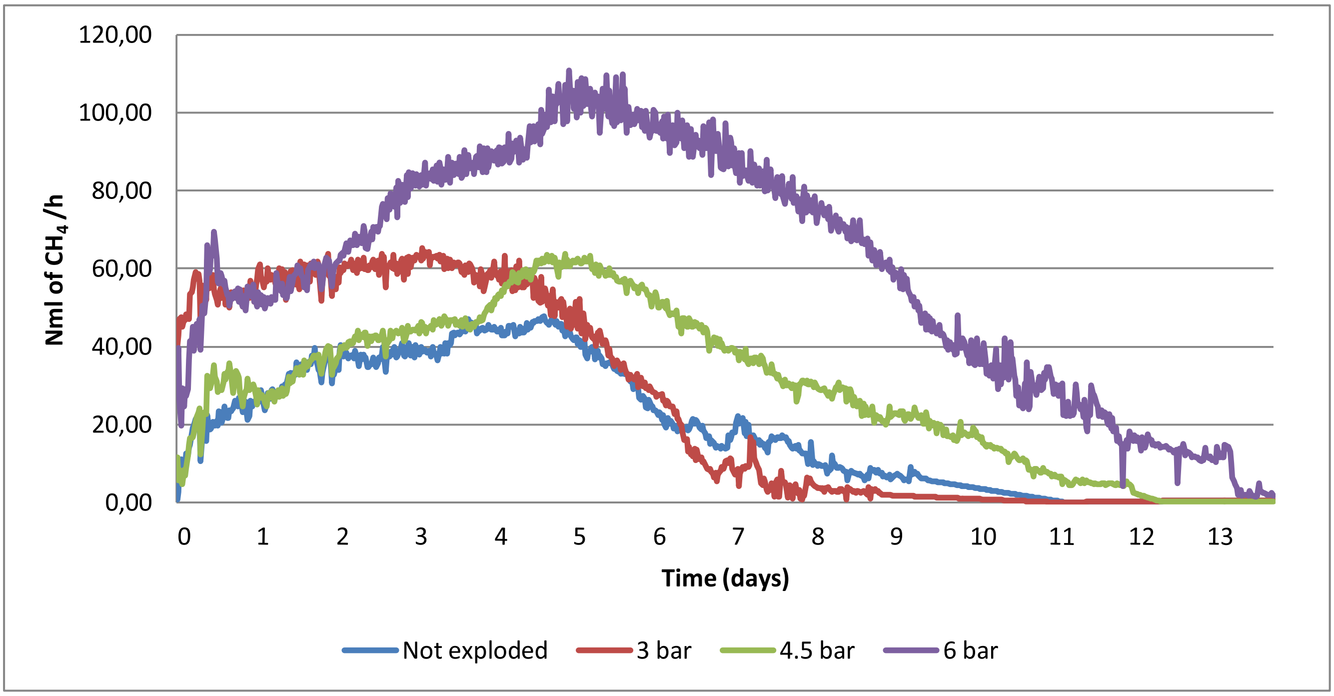

Figure 3.15 *Combined instantaneous biogas production.*

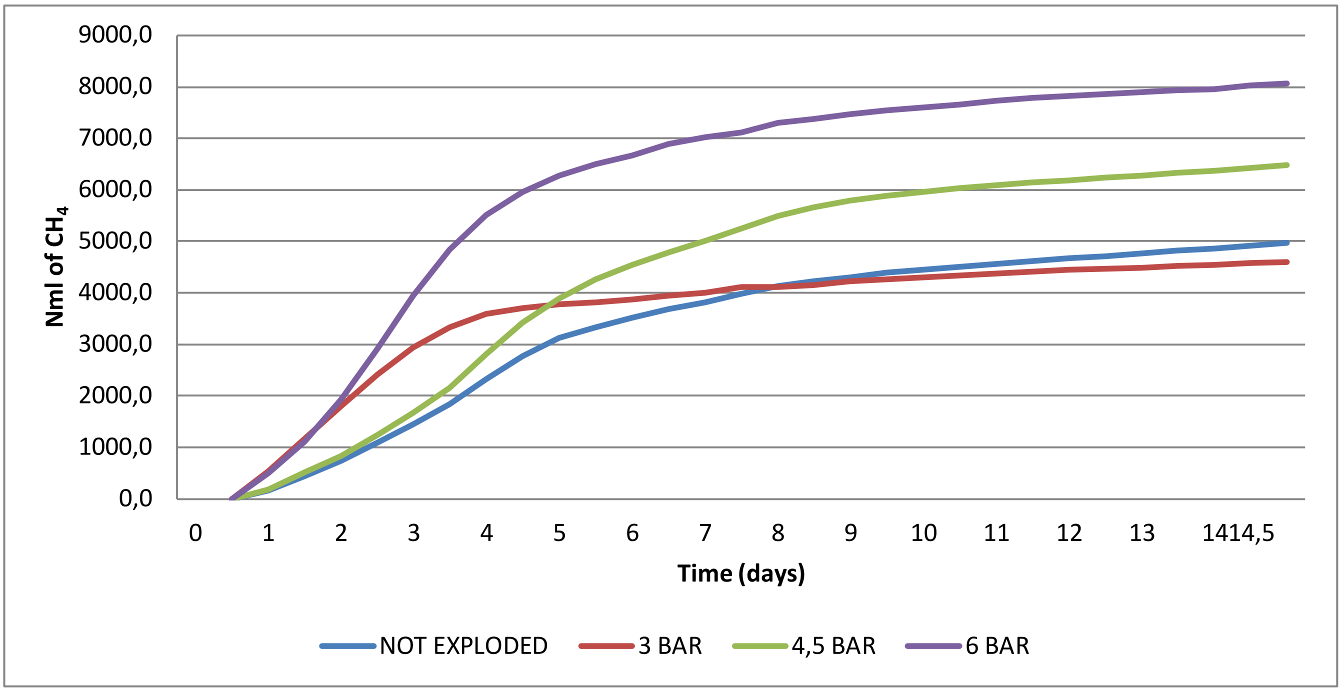

Figure 3.16 *Combined cumulative biogas production*

As can be seen, the amounts of biogas produced from samples obtained at the 3 different pressures is very different. It increase with the explosion pressure, both in terms of instantaneous and cumulative production, and the explosion pressure. These results confirm and strengthen the results achieved so far.

The plots of Figure 3.17 and Figure 3.18 show the biogas production for all the average samples normalized to the organic load of the third experiment.

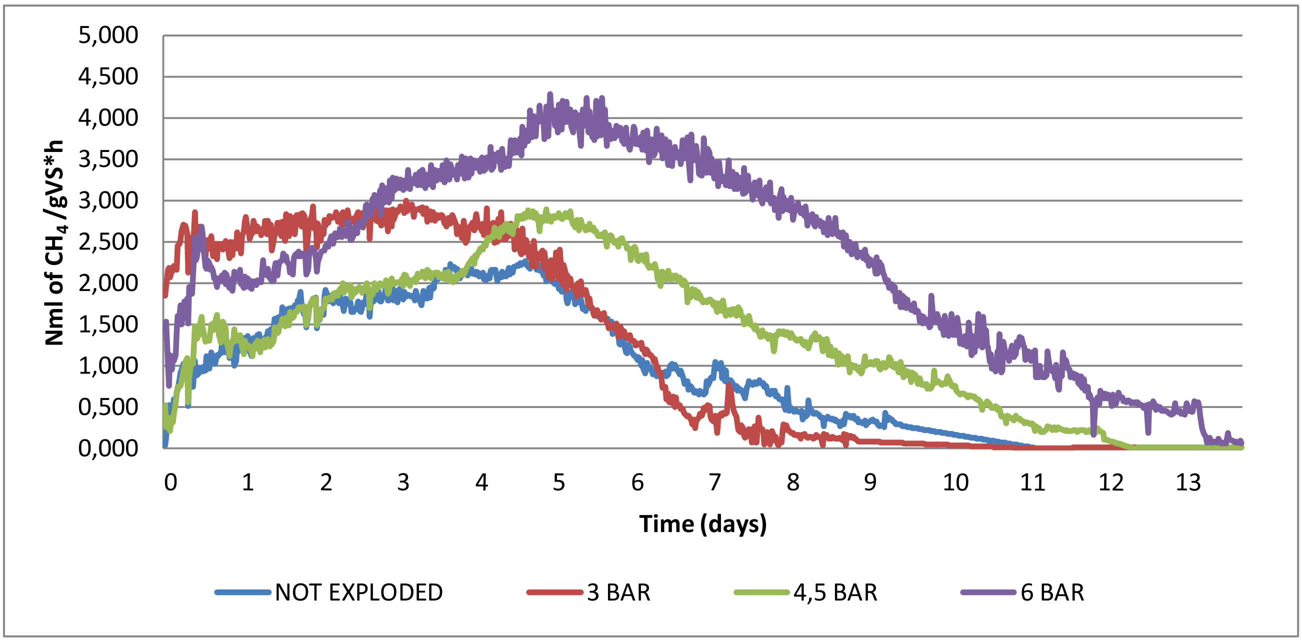

Figure 3.17 *Normalized instantaneous biogas production.*

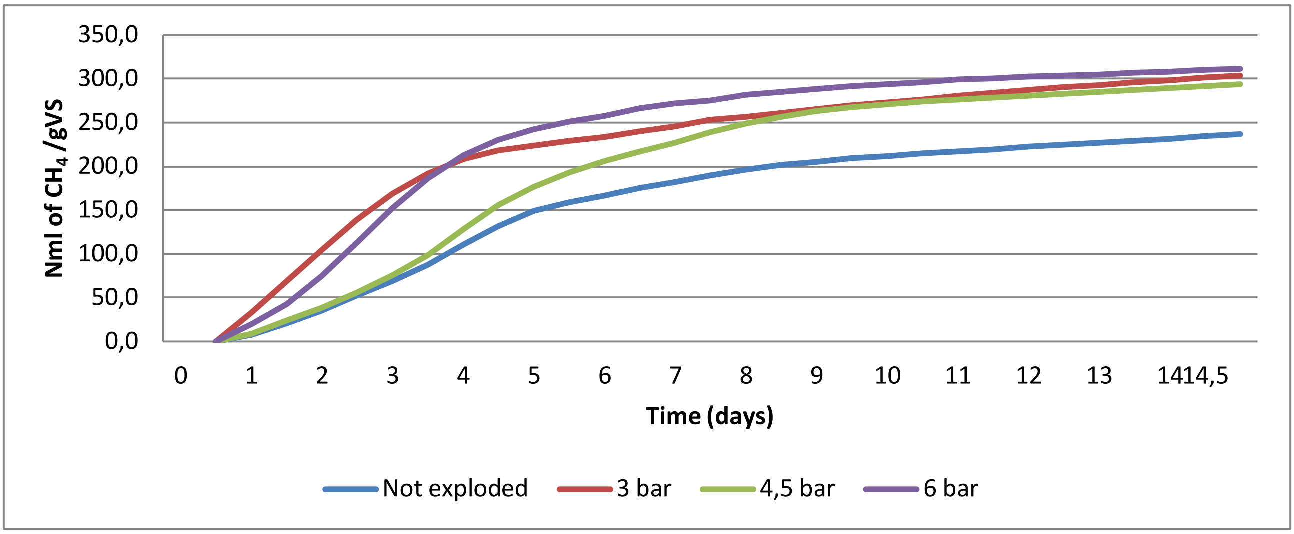

Figure 3.18 *Normalized cumulative biogas production.*

3.3 Fourth experiment

In this experiment the effect of cavitation on the production of biogas has been investigated. Three steam explosion test have been made changing the opening percentage of the valve between the reactor tank and the flash tank.

The valve positions that has been investigated are:

- 25%;
- 50%;
- fully opened

3.3.1 UNEXPLODED FEED AND INOCULUM

Figure 3.19 and Figure 3.20 show both the instantaneous and cumulative amount of biogas produced by the normal samples (the unexploded sludge) and the inoculum.

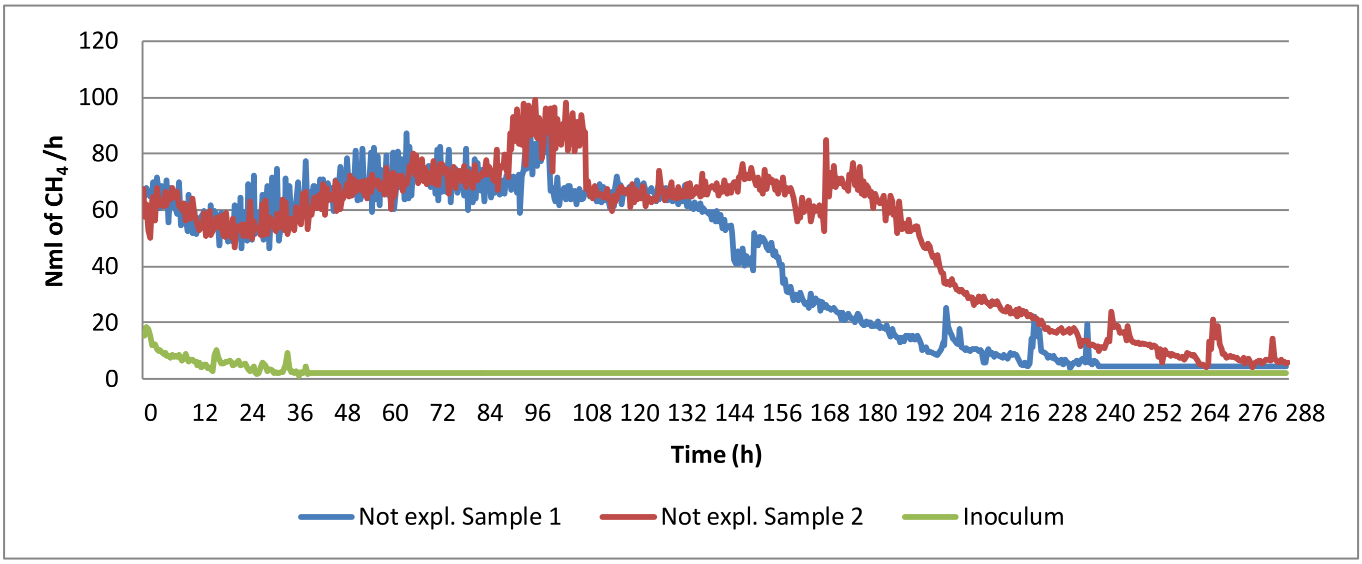

Figure 3.19 *Instantaneous biogas production for the unexploded feed and inoculum.*

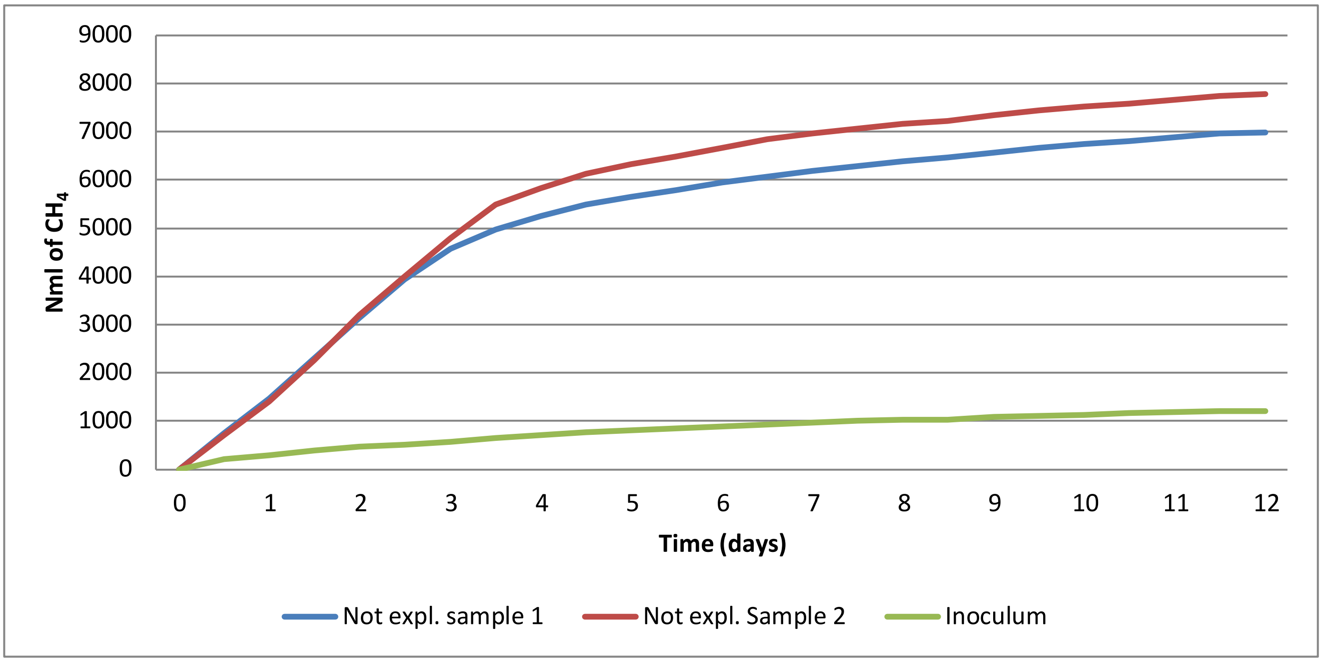

Figure 3.20 *Cunulative biogas production for the unexploded feed and inoculum.*

3.3.2 VALVE OPENED AT 25%

In Figure 3.21 and 3.22 both the instantaneous and cumulative amount of biogas produced by the sludge exploded with the valve opened at 25% are reported.

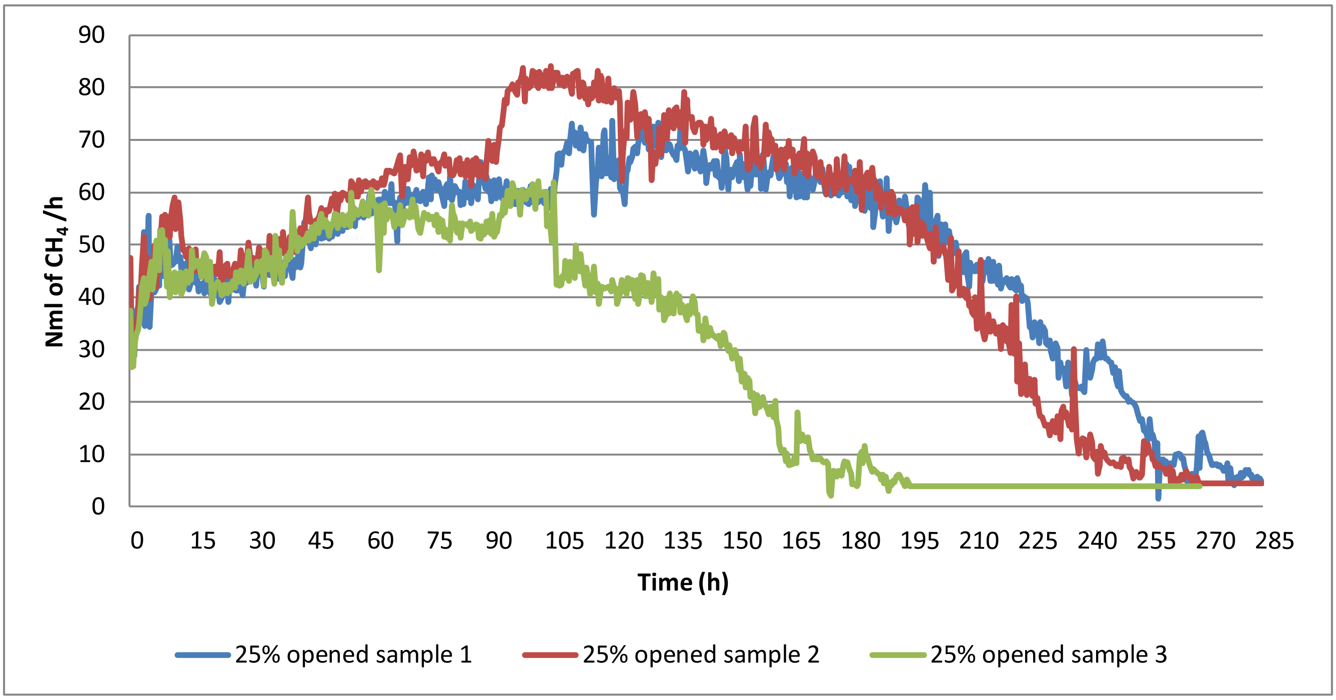

Figure 3.21 *Instantaneous biogas production for the sludge exploded with the valve opened at 25%.*

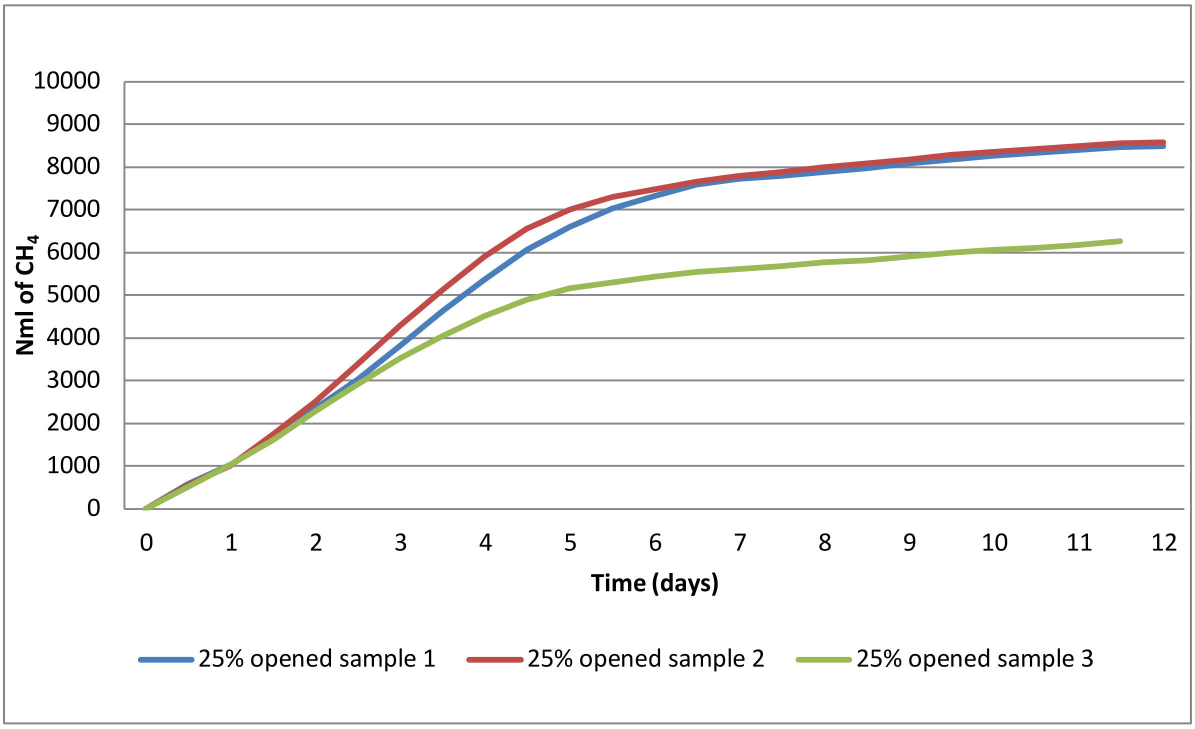

Figure 3.22 *Cunulative biogas production for the sludge exploded with the valve opened at 25%.*

This biogas production experiment was repeated three times. However, the third sample lead to a biogas amount totally different to the two others samples. So it was not considered.

3.3.3 VALVE OPENED AT 50%

The graphs in Figure 3.23 and 3.24 show both the instantaneous and cumulative amount of biogas produced by the sludge exploded with the valve opened at 50%. In this case all the three samples gave reproducible results.

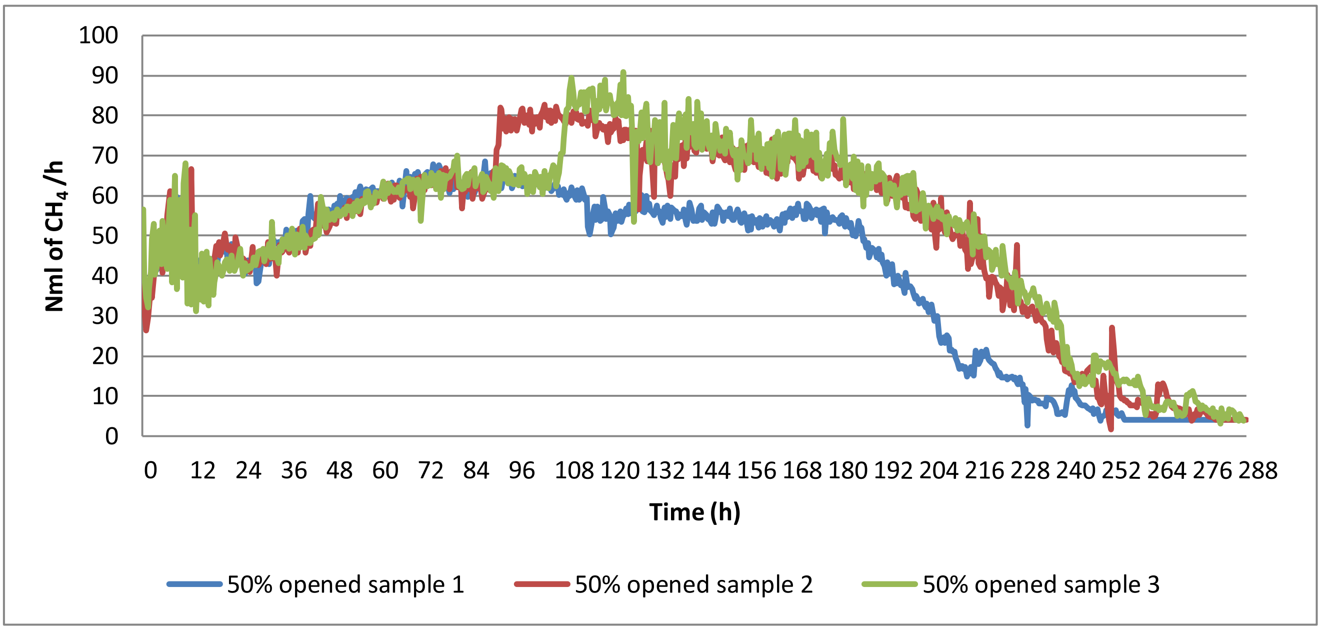

Figure 3.23 *Instantaneous biogas production for the sludge exploded with the valve opened at 50%.*

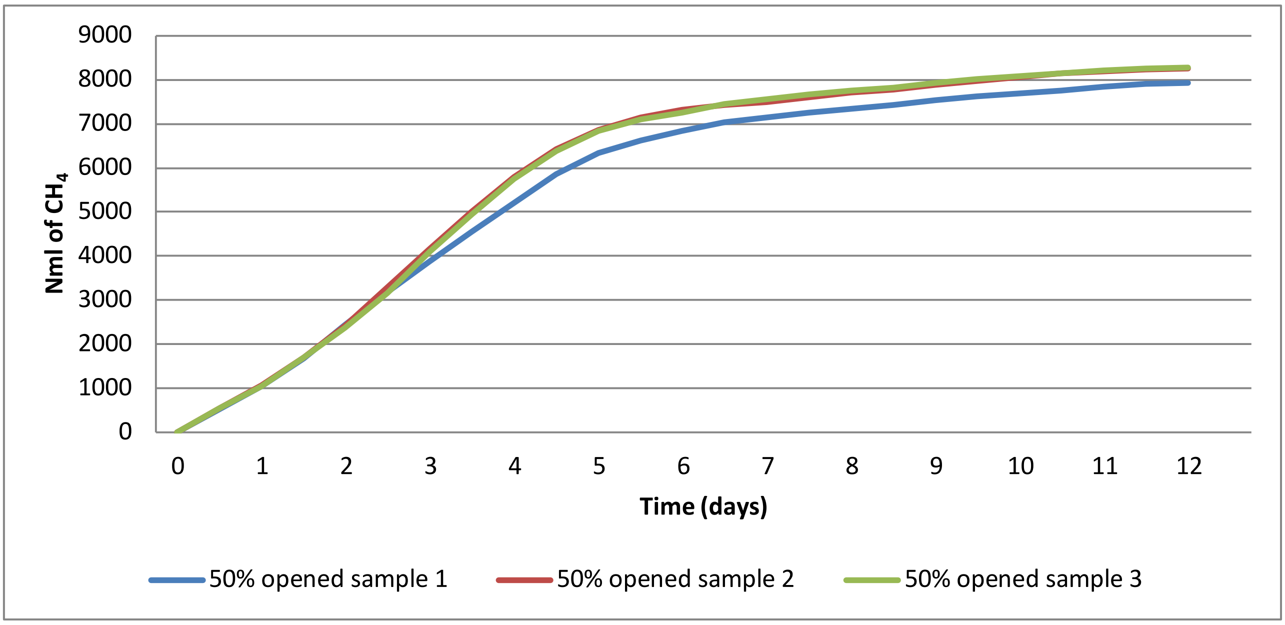

Figure 3.24 *Cumulative biogas production for the sludge exploded with the valve opened at 50%.*

3.3.4 VALVE FULLY OPENED

In Figure 3.25 and Figure 3.26 both the cumulative and instantaneous amount of biogas produced by the sludge exploded with the valve fully opened are displayed. Also in this case the total amount of biogas are comparable.

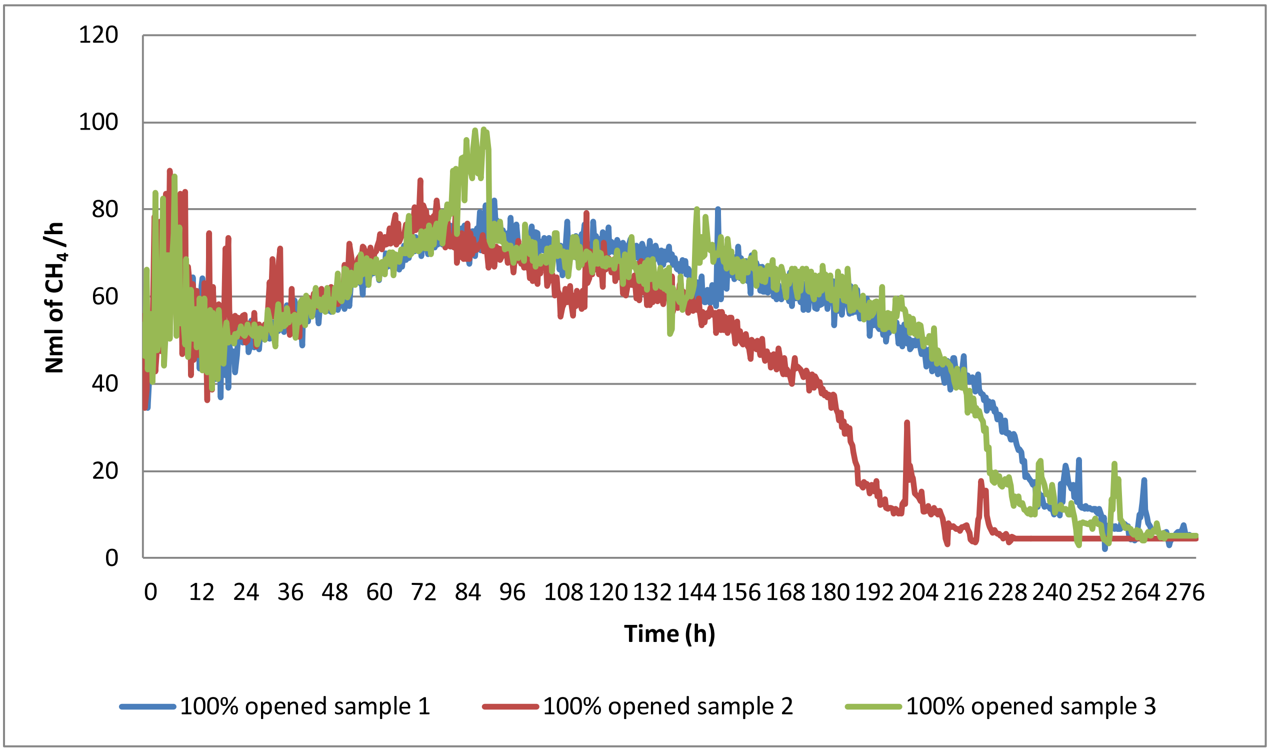

Figure 3.25 *Instantaneous biogas production for the sludge exploded with the valve fully opened.*

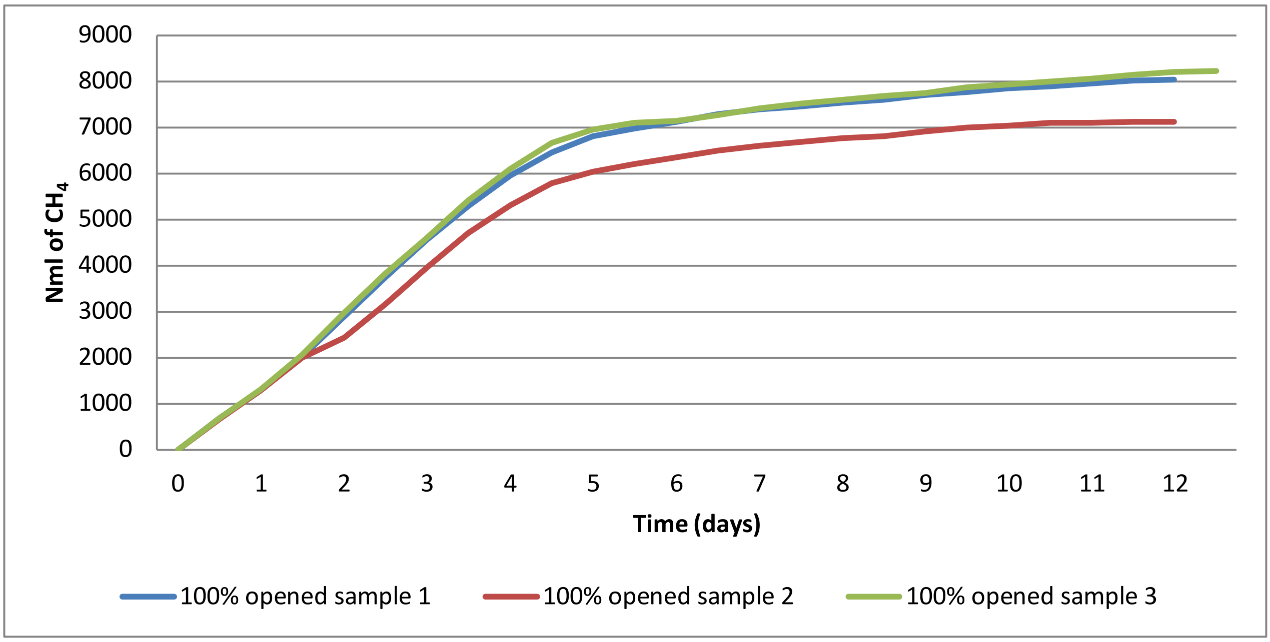

Figure 3.26 *Cunulative biogas production for the sludge exploded with the valve fully opened.*

3.3.5 COMBINED RESULTS OF THE FOURTH EXPERIMENT

All the results above are now combined for comparison in Figure 3.27 and 3.28 with respect to the instantaneous and cumulative amount of biogas produced respectively. In the graphs it is reported the average of the results of each valve opening, substracted by the amount of biogas produced by the inoculum.

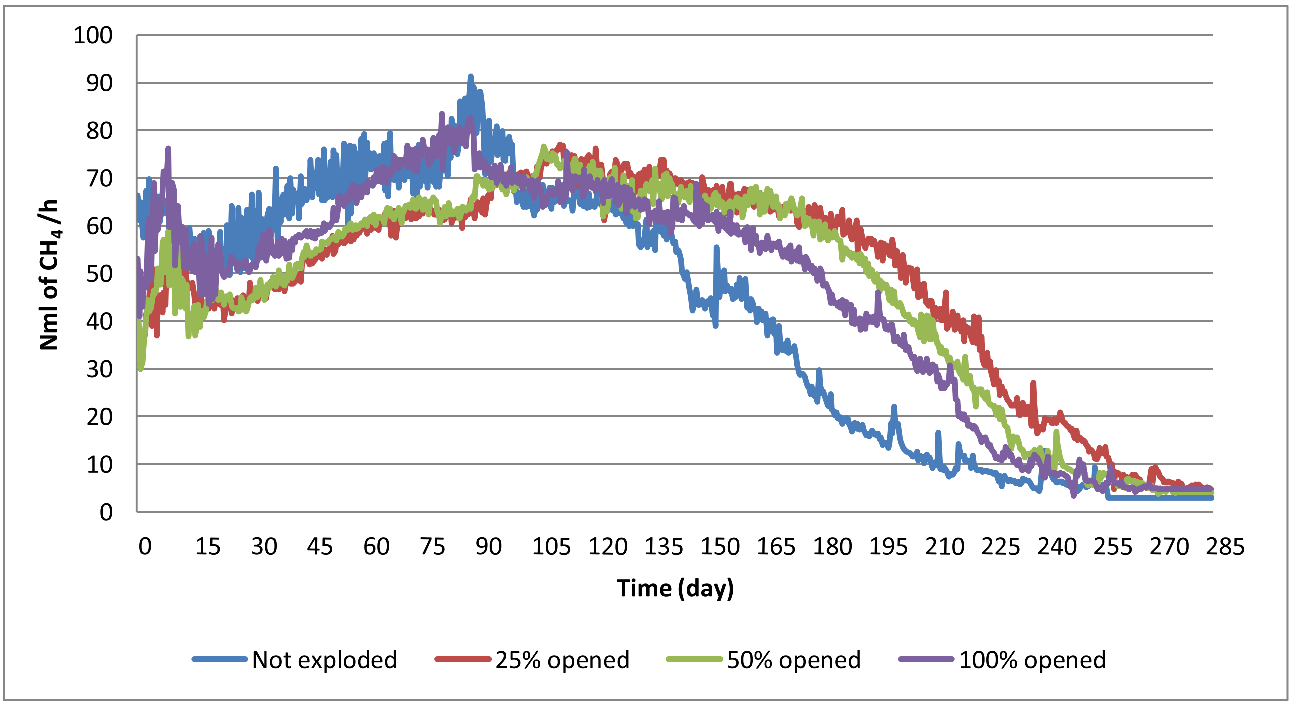

Figure 3.27 *Combined instantaneous biogas production.*

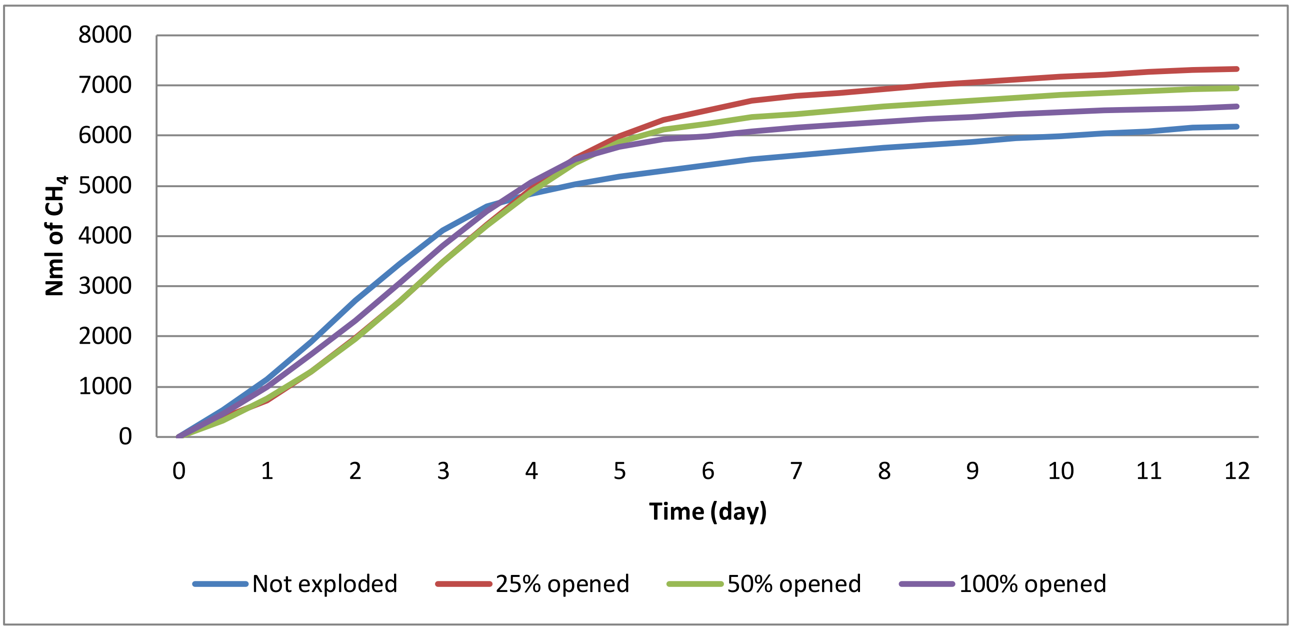

Figure 3.28 *Combined cumulative biogas production.*

These results are of note, because the quantity of biogas produced by the 25% opened valve is higher, followed by the 50% opened and the 100% opened. So, it seems that there is a correlation between the opening percentage of the valve and the biogas production. The reasons why the 25% produced more biogas may be due merely to variance, as these results are based on just one experiment, or may be due to the higher cavitation that occurs when the passing section narrows. So these results could be an incentive for future investigations as to whether or not, when the cavitation increases, the sludge is subjected to a particle size reduction that leads to more biogas production.

As summary the following tables (Table 3.1, Table 3.2, Table 3.3 and Table 3.4) show the yield, expressed as ml CH_4/gDS, for each experiment.

Table 3.1 *First experiment.*

	DS loaded	Biogas producted(ml)	yield (ml/gDS)
3 bar	20,5	2912	142
6 bar	20,54	4248	207

Table 3.2 *Second experiment.*

	DS loaded	Biogas producted(ml)	yield (ml/gDS)
3 bar	19,98	4726	237
4,5 bar	19,04	5171	272

Table 3.3 *Third experiment.*

	DS loaded	Biogas producted(ml)	yield (ml/gDS)
3 bar	25,61	4601	180
4,5 bar	25,93	6476	250
6 bar	30,43	8062	265

Table 3.4 *Fourth experiment.*

	DS loaded	Biogas producted(ml)	yield (ml/gDS)
25% opened	24,68	7321	297
50% opened	24,36	6944	285
100% opened	25,68	6575	256

3.4 COD and VFA's analysis results

In this section the results of the COD and VFA's analysis for the third and fourth experiment are summarized.

3.4.1 THIRD EXPERIMENT

Table 3.5 shows the COD values. Two readings have been taken for each sample and then averaged and plotted.

Table 3.5 *COD analysis results for the third experiment.*

	COD (mg/l)	COD (mg/l)	Average (mg/l)
6 bar	61410	44210	52810
4,5 bar	7646	72230	74345
3 bar	93500	86860	90180
Not exploded	94000	128750	111375

Figure 3.29 shows the COD trend compared to the explosion pressure.

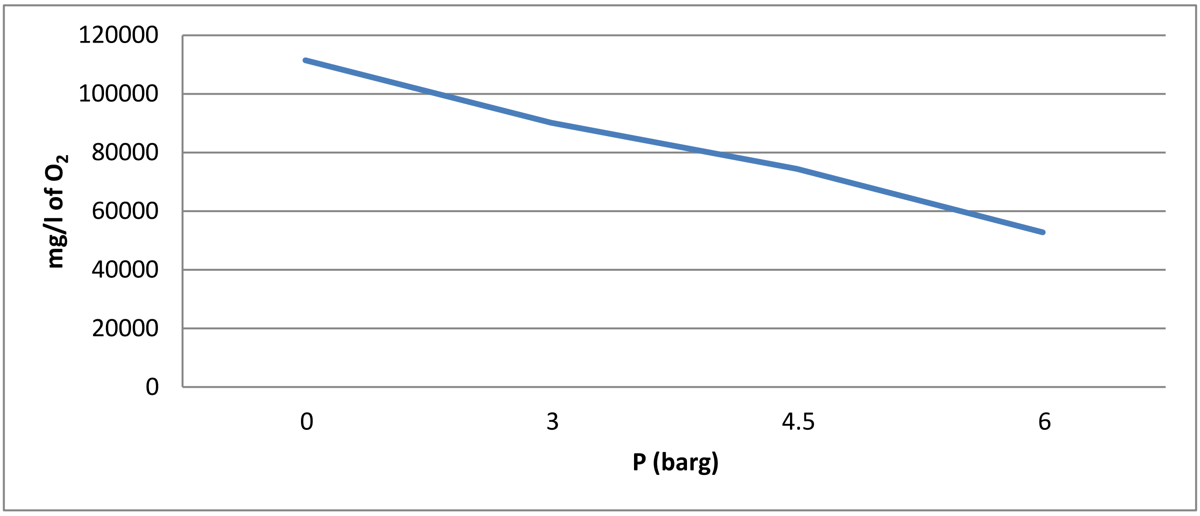

Figure 3.29 *COD trend as a function of steam explosion pressure.*

Table 3.6 shows the results of the VFA's analysis which are also displayed in Figure 3.30.

Table 3.6 *VFA's analysis results for the third experiment.*

	VFA's (mg/l)
6bar	2968
4,5 bar	2653
3 bar	2395
Not exploded	1947

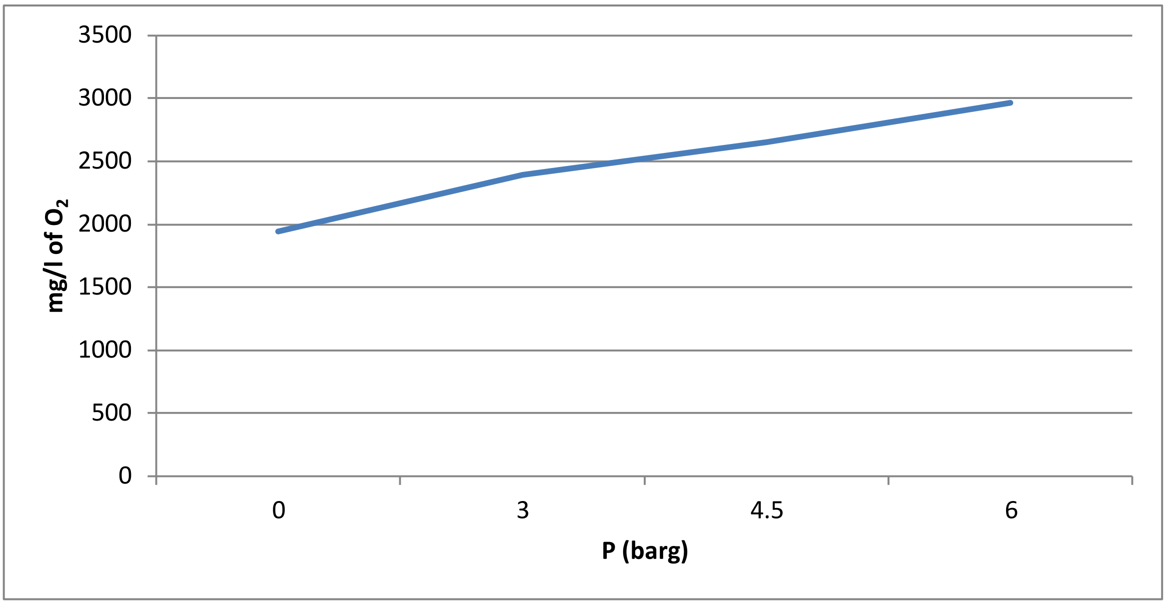

Figure 3.30 *VFA's trend as a function of the explosion pressure.*

These results shows a clear dependence between the COD value and the explosion pressure. It seems that more the pressure increases, the more the COD decreases. The VFA's instead, seems to have a completely different trend. In fact, the more the pressure increases, the more the VFA's increases.

3.4.2 FOURTH EXPERIMENT

Table 3.7 shows the results of the COD for the fourth experiment.

Table 3.7 *COD analysis results for the fourth experiment.*

	COD (mg/l)	COD (mg/l)	Average (mg/l)
6 bar 100% opened	108080	146250	127165
6 bar 50% opened	105300	95130	100215
6 bar 25% opened	104520	108150	106335
Not exploded	94000	128750	111375

Figure 3.31 shows the trend of the COD compared to the opening percentage of the valve.

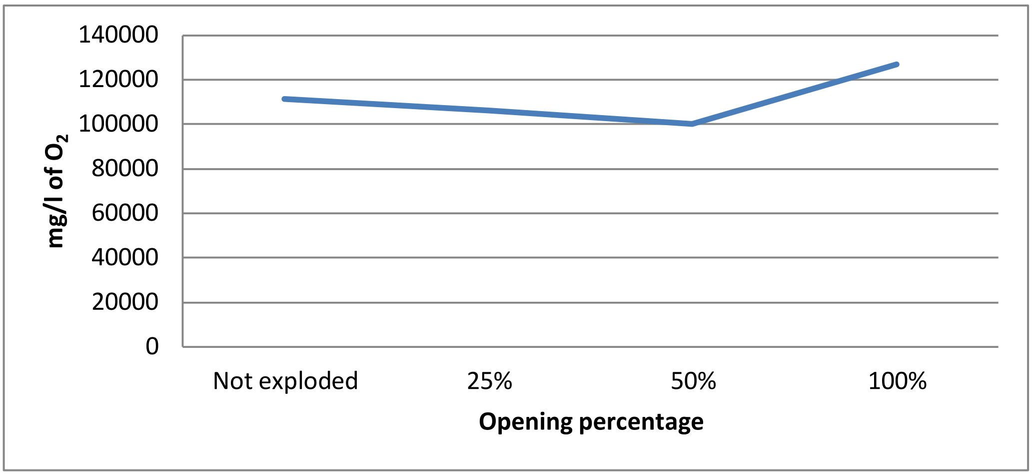

Figure 3.31 *COD trend as a function of a pressure.*

Table 3.8 shows the results of the VFA's analysis of the fourth experiment.

Table 3.8 *VFA's analysis resultsof the fourth experiment.*

	VFA's (mg/l)
6 bar 100% opened	2124
6 bar 50% opened	2559
6 bar 25% opened	2593,5
Not exploded	1424,5

Figure 3.32 shows the VFA's trend compared to the opening percentage of the valve.

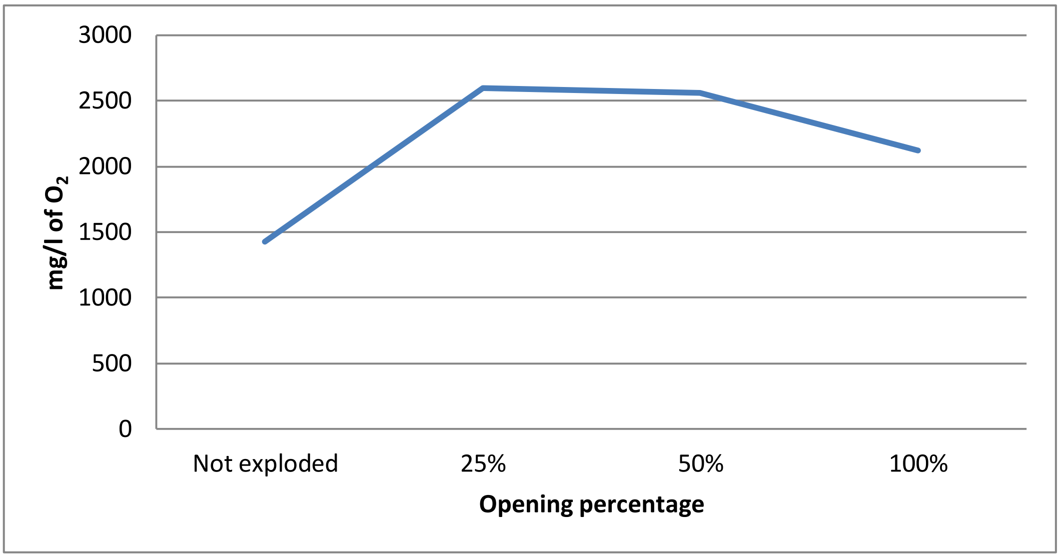

Figure 3.32 *VFA's trend as a function of a opening percentage of the valve.*

These results doesn't actually give a trend to analyse. So it seems that there isn't a correlation between the values of COD and VFA's and the opening percentage of the valve.

3.5 Microscope analysis results

In the third experiment the sludge samples has also been analysed by an optical microscope.

Figure 3.33 shows the image obtained from the raw sludge (the sludge not exploded).

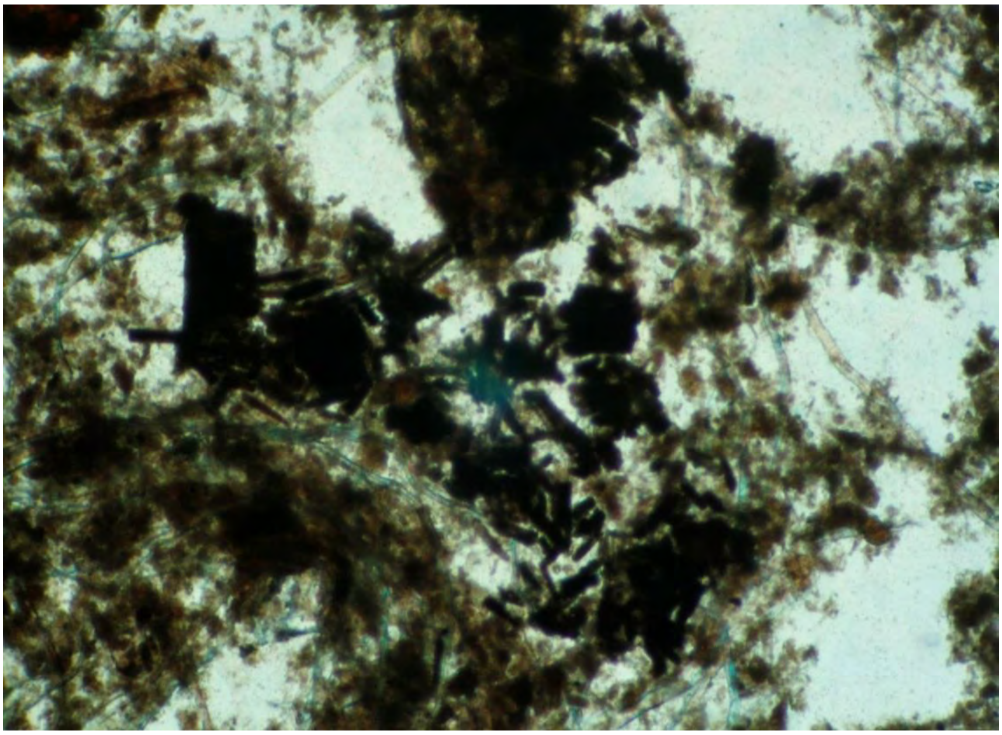

Figure 3.33 *Raw sludge.*

Figure 3.34 shows the image obtained from the sludge exploded at 3 bar.

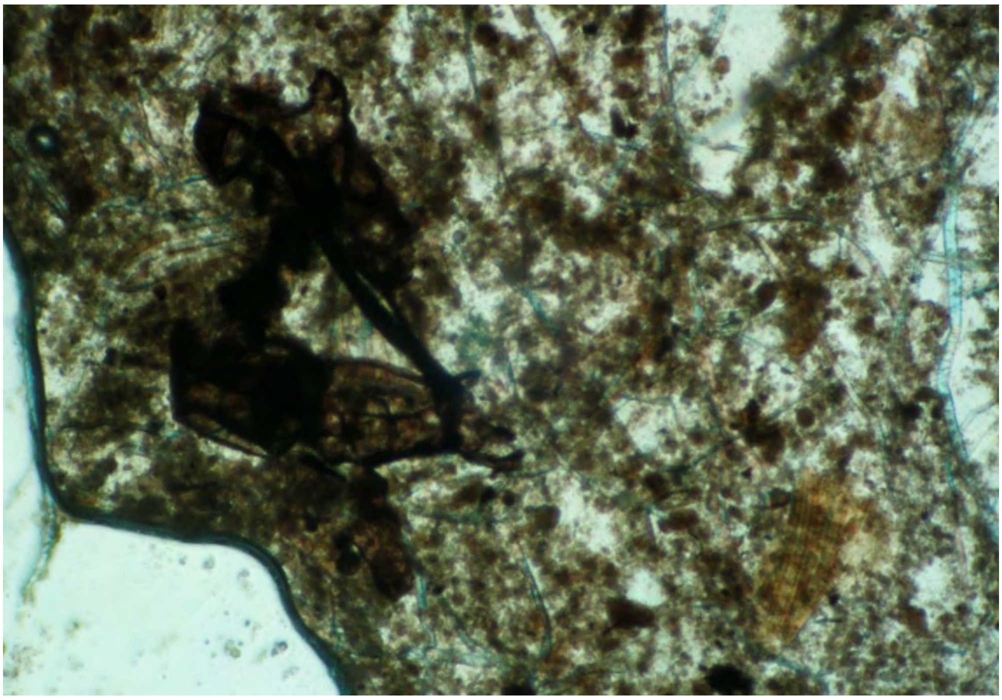

Figure 3.34 *Sludge exploded at 3 bar.*

Figure 3.35 shows the image obtained from the sludge exploded at 4.5 bar.

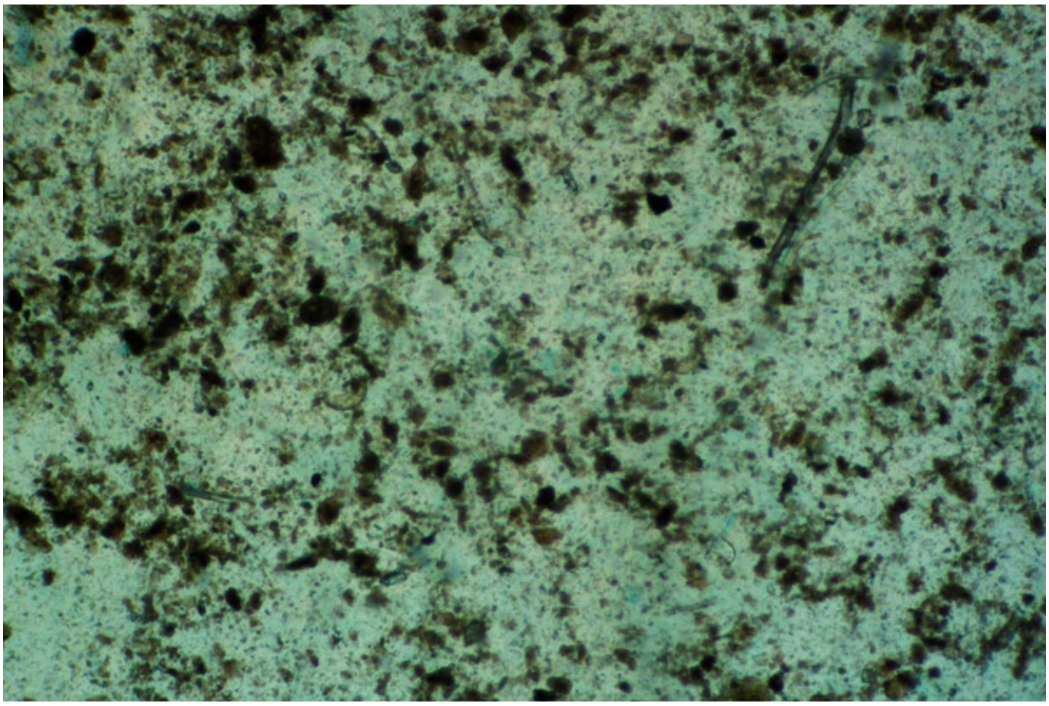

Figure 3.35 *Sludge exploded at 4.5 bar.*

Figure 3.36 shows the image obtained from the sludge exploded at 6 bar.

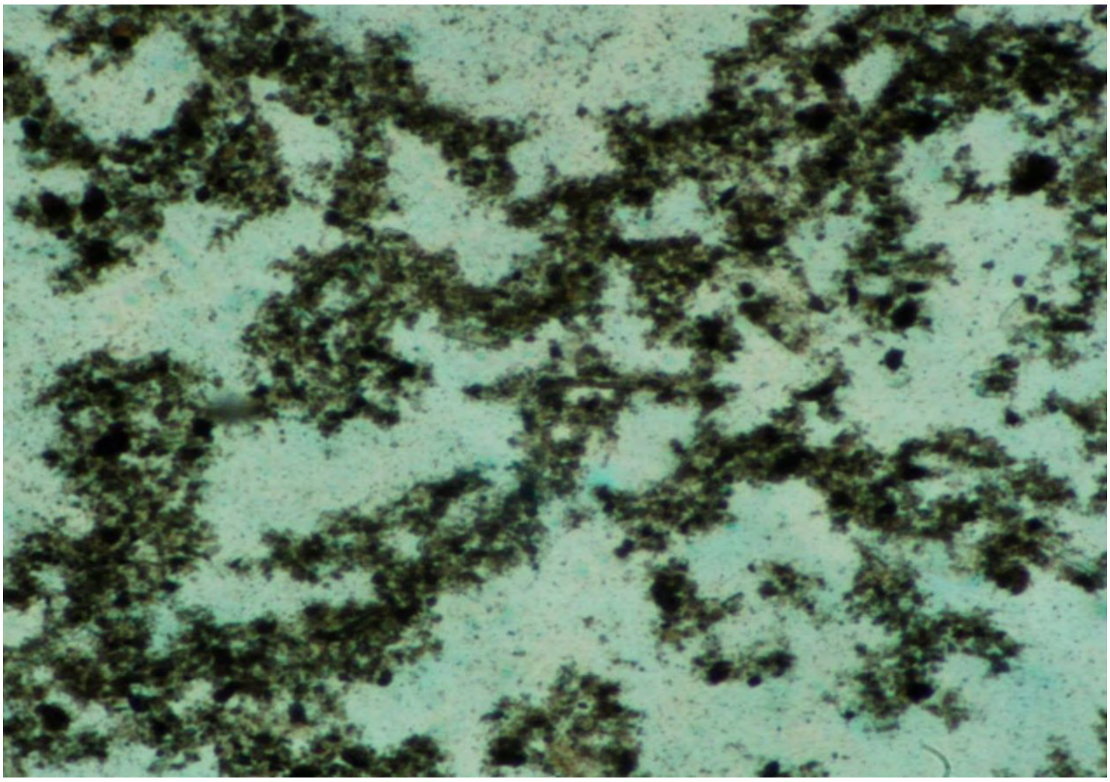

Figure 3.36 *Sludge exploded at 6 bar.*

These figures show that more the pressure increases, more the size of the agglomerates decreases. This is could be the reason why, during the experiment, the sludge exploded at the highest pressure seems to be more fluid and less viscous. Practically, it seems to have a different rheology.

This particle size reduction could explain as well why the sludge exploded at highest pressure produces more biogas then the others.

3.6 Statistical analysis

The statistical analysis has been performed on the first three experiments and the standard error has been calculated in order to determine the 95% confidential interval (equation 4.1) for the increasing percentage of the biogas production for the 6 bar and 4.5 bar pressure.

$$95\%\ C.I. = Mean\ \pm\ \sigma_e * t \qquad (4.1)$$

Where t is a coefficient taken from the table of values t for the Student distribution and it depends from the number of the samples used in the analysis. In this case the t=2.776.

Table 3.9 shows the increasing percentage of biogas production for the 6 bar and 4.5 bar samples respect to the 3 bar samples. The parameter x_i is already been defined in chapter 2, equation 2.1 .

Table 3.9 *Increasing percentage for the 6 bar and 4.5 bar samples.*

	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4(%)	sample 5 (%)	Mean (%)
$x_{6\ bar}$	46.94	44.78	98.86	61.67	65.15	63.48
$x_{4.5\ bar}$	11.96	6.88	29.02	47.44	45.81	28.22

Table 3.10 shows the results of the standard error calculation for the 6 bar and 4.5 bar.

Table 3.10 *Standard error values.*

$\sigma_e^{6\ bar}$	0,09698
$\sigma_e^{4.5\ bar}$	0,08363

So, the confidential intervals are:

- $95\%\ C.I. = 63.48\ \pm 26.92$ %, for the 6 bar;
- $95\%\ C.I. = 28.22\ \pm 23.22$ %, for the 4.5 bar.

This leads to do not take into account anymore the sample 3 that it’s the sample 1 of the 6 bar in the third experiment, the rest of the data are in the confidential interval for both the experiments. So, they have statistical significance.

3.7 Hydrodynamic analysis

In this section will be exposed the results obtained from the model developed will be discussed

.

3.7.1 RESULTS FROM THE SIMULATION OF THE THIRD EXPERIMENT

Figure 3.37 shows the results from the simulation of the third experiment in order to compare the pressure drop and the discharge time. The results achieved are close enough to the reality that when the driving force (the pressure drop) decreases, the time needed in order to discharge the tank increases appreciably. It worthwhile to note that the experimental values are close to those obtained from this model.

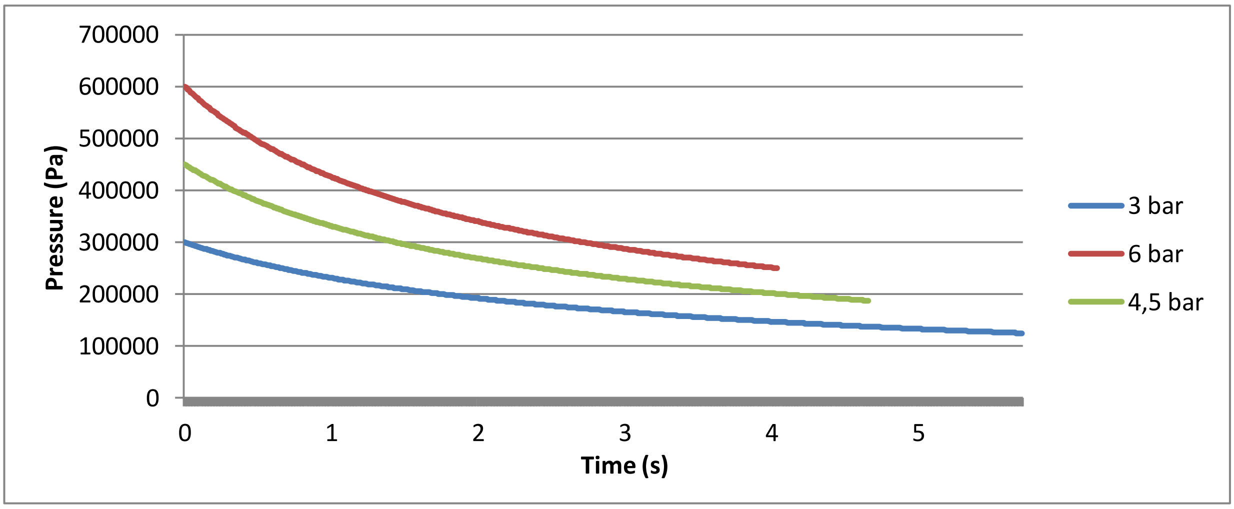

Figure 3.37 *Simulation results from the third experiment.*

3.7.2 RESULTS FROM THE SIMULATION OF THE FOURTH EXPERIMENT

Figure 3.38 shows the results from the simulation of the fourth experiment.

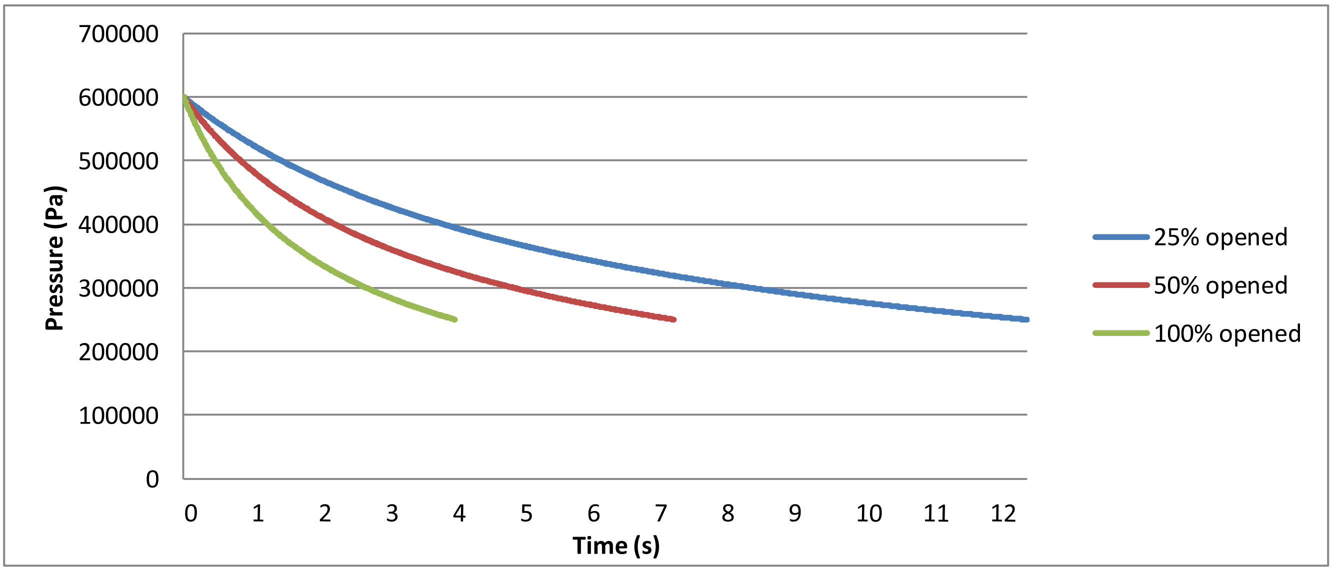

Figure 3.38 *Simulation results from the fourth experiment.*

This graph shows that the final pressure when all the liquid has been pushed out of the tank is the same for each trial, but the time needed to discharge the liquid from the tank is different because of the increases in the pressure drop due to the different opening percentage of the valve.

3.8 Concluding remarks

This chapter aims to outline as many points of view as possible as to why the steam explosion achieves that kind of performance when used as a pretreatment for sludge digestion. In order to answer this question, many analyses were performed:

- VFA's analysis;
- COD analysis;
- Microscope analysis;
- Statistical analysis.

It is concluded that, the higher the explosion pressure, the higher the biogas production achieved, represented in Figure 3.5 and Figure 3.15 in which the samples are compared together. Figure 3.5 shows as well that the biogas production is highly affected by the variability of chemical and physical properties of the sludge, but also that the biogas production is always higher with a higher explosion pressure.

Figure 3.29 and Figure 3.30 show that there is an inverse linear dependence between the explosion pressure and the COD values, supporting the idea that if the sludge is exploded at higher pressure it is more digestible.

The microscope analysis may explain why this is the case, because it is possible to see that when the pressure increases, the particle size decreases and this means that there is more surface area available for the bacteria to eat the sludge and produce more biogas.

All these analyses confirm that steam explosion is able to achieve a very good result if used as a pretreatment for sludge digestion, since it allows the increase of biogas production, which is also related to the explosion pressure used in the experiments.

Chapter 4

Economic analysis

This chapter aims to give an economic overview of the possible configurations available nowadays on the market for the steam explosion process of sludge. These technical alternatives depend on the different use of the by-products coming from the anaerobic digestion. All the data used in this chapter has been taken from Mills et al.: *"Environmental & economic life cycle assessment of current & future sewage sludge to energy technologies"* (Mills et al., 2014).

The technologies considered are:

- Conventional Anaerobic Digestion (AD) with Combined Heat and Power (CHP) and land recycling of the digestate to agriculture.
- Thermal Hydrolysis Process (THP) AD with CHP and land recycling of the digestate to agriculture (THP AD CHP).
- THP AD with bio-methane injection or Gas to Grid (GtG) and land recycling of the digestate to agriculture (THP AD GtG).
- THP AD with CHP, drying for fuel (THP AD CHP + Drying for fuel).
- THP AD with CHP, drying and pyrolysis with CHP (THP AD CHP + Drying, Pyrolysis and CHP).

As already told, the differences in these technologies are just on tale of the product chain, where the biogas can be used to produce electricity or directly injected in the gas network, and the by-products must be disposed.

The most commonly used method is to use the biogas to produce renewable electricity. This method in the UK is incentivised by the Renewable Obligation Scheme which gives a supplemental income to the generators of renewable energy. Lately, a new practice is taking place in England, the Gas to Grid method, that aims to inject the biogas produced in the AD directly to the national gas network. In England this method is incentivised by the Renewable Heat Incentive (RHI). The biogas must be cleaned and diluted with propane and odorant before be injected in the network. The big disadvantage of this method is that the heat required for the whole process can't be taken from a waste source and must be supplied by burning some biogas or purchasing supplementary natural gas.

In order to recover the part of energy that the sludge maintains after the AD, the sludge can be dried to produce a fuel that can be either sold as a combustible or be employed in a pyrolysis plant and used again in the CHP.

4.1 CapEx analysis

To calculate the CapEx for each scenario a lot of assumptions needs to be done which are reported in Mills et al. (2014).

The CapEx for a plant with a capacity of 100 TDS/d are reported below in Table 4.1.

Table 4.1 *CapEx data.*

Component	CapEx (£)	Size	Units
Pre-treatment and thickening	2,743,151	100	TDS/d
AD	5,972,063	22000	m3
THP	6,086,669	100	TDS/d
Dewatering and cake storage	3,939,311	60	TDS/d
Odour treatment	687,338	100	TDS/d
CHP and electrical	5,719,973	5000	kWe
Control and instrumentation	815,715	100	TDS/d
General	2,099,387	100	TDS/d
Sub total	28,063,607		
Contractor management (20%)	5,612,721		
Client overheads (10%)	3,367,633		
Total	37,043,961		
Additional options (before contractor and client overheads)			
GtG	4,774,740	5000	kWe
Drying plant	3,225,370	3200	kgH2O/h
Pyrolysis	5,413,554	60	TDS/d
Pyrolysis CHP	5,003,201	4000	kWe

*TDS stand by Tons of Dry Solids

4.2 OpEx analysis

The Opex for this process has been taken from Mills, 2012b that deeply analyse this topic taking into account several parameters and comparing the CHP units available on the market in order to compare them and see which one give the most valuable scenario for this process.

In order to calculate the OpEx a lot of assumptions needs to be done. These values are summarized below: Table 4.2 shows the THP AD process specific, Table 4.3 shows the assumptions made for the THP AD process, Table 4.4 shows the financial assumptions, Table 4.5, instead, shows the parameters for the CHP unit and Table 4.6 shows the assumptions made for the bio-methane injection on the network.

Table 4.2 *THP AD process specific.*

Bio gas yield	420	m^3/TDS
Solids destruction in the AD	40	%
Cake dry solids	42	% DS

Table 4.3 *THP AD assumptions.*

Thickening – electrical demand	60	kW h/TDS
Thickening – polyelectrolyte consumption	2,2	kg/TDS
Input:output – dry solids	1%:5%DS	
THP – electrical demand	50	kW h/TDS
THP pre dewatering – polymer demand	5	kg/TDS
THP pre dewatering input:output	5%:16.5%DS	
THP steam demand @12barg	1	kg/kgDS
AD electrical demand	40	kW h/TDS
Dewatering – electrical demand	50	kW h/TDS
Dewatering – polymer demand	10	kg/TDS
Polymer GHG emissions	2	kgCO2e/T
Liquor treatment energy demand	1,05	kW h/m^3

Table 4.4 *Financial assumptions.*

Electricity cost	75	£/MW h
ROC value	45	£/MW h
Polymer value for the floculation	2	£/kg
Natural gas	29	£/MW h
RHI incentive value	65	£/MW h
Sludge recycling	18	£/wet
Maintenance	3%	Of CapEx
Discount rate	8%	
Value of dried sludge fuel product	60	£/TDS

Table 4.5 *CHP assumptions.*

CV of biogas TW typical	23	MJ/Nm^3
CHP electrical efficiency	38%	
CHP high grade heat efficiency	18%	
CHP low grade heat efficiency	20%	
Bio-gas leaks (CH4)	2%	
CO_2 exhaust emissions	175	g/kW h
CO exhaust emissions	986	mg/kW h
Dust exhaust emissions	164	mg/kW h
NOx exhaust emissions	821	mg/kW h
NMVOCs emissions	136	mg/kW h
SO2 emissions	439	mg/kW h

Table 4.6 *Bio-methane injection assumptions.*

Electrical demand	0,05	kW h/m^3
Propane required	0,101	kg/m^3
CO2 emissions	105	g/kW h
CV of grid ready bio-methane	37	MJ/m^3

With all these data it's possible to calculate the OpEx for all the configurations:

Table 4.7 *Opex for each scenario.*

Scenario	OpEx (£ pa) (No Incentives)	OpEx (£ pa) (Incentives)
Conv. AD CHP	6,902,150	4,582,575
THP AD CHP	5,657,500	3,809,383
THP AD GtG	7,317,033	1,885,833
THP AD CHP and drying	6,713,567	4,865,450
THP AD CHP and drying + pyrolysis and CHP	9,429,167	5,619,783

Table 4.7 shows the OpEx with and without incentives, and it's clear that the best scenario, with and without the incentives, is the one where, after the AD, the remaining sludge is dried and then subject to pyrolysis in order to recover as much energy as possible from the sludge.

4.3 Cost benefit analysis

Figure 4.1 shows the IRR for each scenario, with and without the incentives:

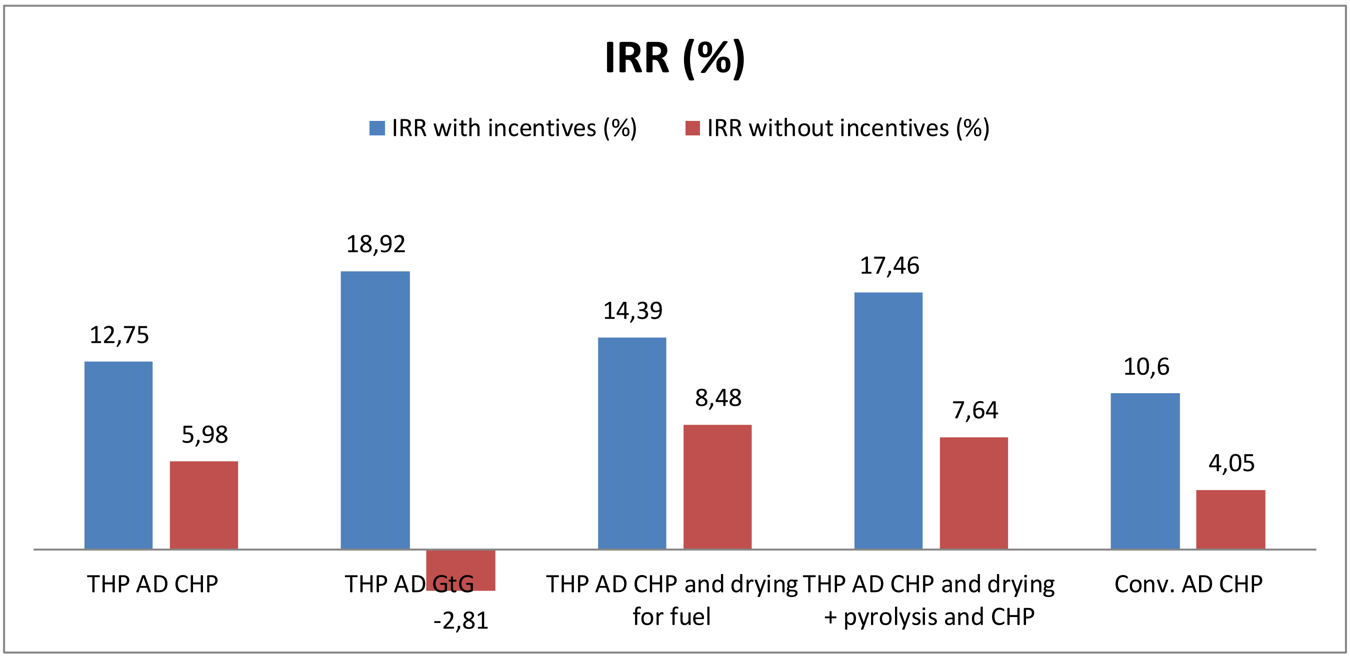

Figure 4.1 *IRR with and without the incentives for each scenario.*

These results show that the best investment would be the gas to grid with the incentives, but if for some reasons the government decides to cancel the incentives, then the investors will not be able to see a profit on their investment. It means that this is a risky investment. The best option in terms of IRR after the GtG it seems to be the THP AD CHP and drying with the pyrolysis plant, this solution is the most expensive one, but it will give most profit in the long run. The next profitable scenario is the THP AD CHP and drying for fuel followed by the simple THP AD CHP.

In order to make a comparison between the real data obtained in this work and the assumptions made in the paper, an economic analysis has been done to see how many years an investors have to wait before the investment is paid off. The two scenarios analysed are:

- 300 m^3/TDS;
- 420 m^3/TDS.

The first data comes from the best scenario analysed in this thesis (Table 3.4) and second one is the assumptions made in the paper used as a reference to write this chapter. It worth stating that this analysis is just qualitative, inasmuch as it has been made with raw economic data.

It has been hypothesized an AD THP CHP and drying plant of 100 TDS/day, a production of 1,02 MWh/TDS and a discount rate of 8%.

Figure 4.1 shows the results of this analysis.

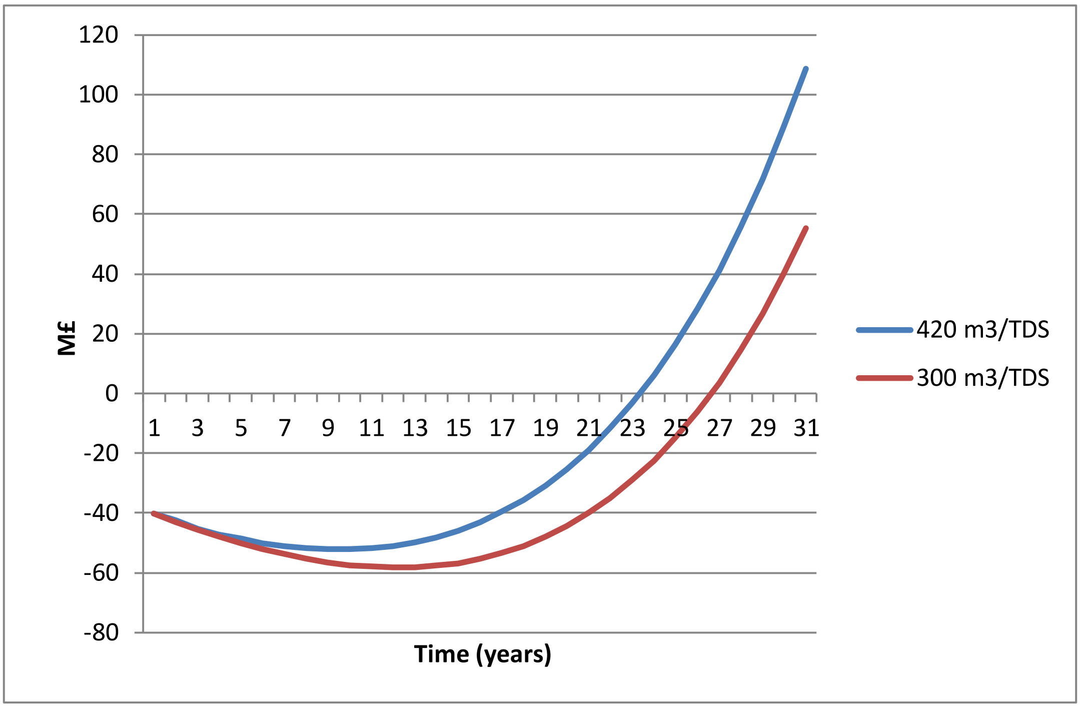

Figure 4.1 *Investment recovery time.*

These results give an estimation of the recovery time for this plant. In the first scenario the recovery time is 26 years, in the second scenario instead is 23 years. These numbers, as already mentioned, are purely qualitative.

The results achieved are not so bad, considering that, starting from an initial investment of £40 million, the investors are able to recover their capital in a little more than 20 years.

Conclusions

This work has analysed in many ways the biogas production from sewage sludge with the steam explosion as a pretreatment. Biogas production in four experiments has been analysed, and all of them lead to the conclusion that the quantity of biogas produced is somehow function of the explosion pressure thanks to which the sludge is capable to achieve a better yield. To strengthen these results a statistical analysis has been done to calculate the confidential intervals for the increase of biogas production.

A new methodology has been proposed wherein the sludge is subjected, as well as the explosion, to the cavitation caused by varying the opening percentage of the valve before the flash tank. This experiment has produced good results. Figure 3.28 shows that more cavitation produces more biogas. Although it is only one experiment, this could be a new technology that is worth further analysis and improvement in future experiments to see whether the results achieved in this thesis are reliable or not.

The COD and VFA analysis has been done and these lead to the conclusion that, as it possible to see in Figure 3.29 and 3.30, an increasing explosion pressure decreases the COD values. For the VFA instead, it's the reverse, if the explosion pressure increases, the VFA value increases as well.

A microscopic analysis on the sludge samples exploded at different pressures revealed that there is a strong correlation between the particle size of the sludge and the explosion pressure. The more the pressure increases, the more the particle size decreases, and this could be the reason why the sludge exploded at higher pressure produces more biogas, in fact, small particles mean more available area for the bacteria that are able to ensure an higher efficiency during the anaerobic digestion.

A hydrodynamic analysis to understand what occurs inside the reactor tank and to see how fast the reactor is discharged has been made by developing a simple hydrodynamic model to calculate the discharge time and the final pressure inside the reactor once all the liquid is removed.

Finally, an economic analysis has been made to understand the real cost in a big plant for the steam explosion of sludge, to see the internal rate of return of this process, and to understand the amount of time it would take for hypothetical investors to reach the break-even point on their investment.

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Ringraziamenti

Desidero ringraziare i miei genitori, Orazio e Maria Anna, che non senza sacrifici mi hanno permesso di arrivare fin qui, e insieme a mio fratello Glauco, mi hanno insegnato ad essere la persona che sono oggi.

Voglio ringraziare mia zia, Cinzia, che insieme ad Alberto, mio cugino Mariano e mia nonna Corinda mi hanno ospitato per questi 5 anni, accogliendomi e supportandomi sempre in tutte le situazioni.

Un ringraziamento speciale va alla mia ragazza, Annarita, che in tutti questi anni mi è rimasta affianco e mi ha aiutato ad affrontare le difficoltà che mi si sono poste davanti.

Un pensiero va anche a quelli con cui ho condiviso questo percorso, Francesco e Luca, i quali ringrazio per le nostre infinite sessioni di studio, e non, che ci hanno accompagnato in questo tragitto.

Un ringraziamento va a tutti i miei amici che nonostante fossi lontano, hanno continuato a starmi vicino, in particolar modo a Matteo che dall'Italia mi ha aiutato a risolvere diversi problemi, legati anche a questo elaborato.

"Il tempo è relativo, il suo unico valore è dato da ciò che noi facciamo mentre sta passando"
A. Einstein