

Utilisation of heat and organic wastes for energy recovery in the industry

Thesis by

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Abstract

Utilisation of heat and organic wastes for energy recovery in the industry

Food and drink processing industries are extremely large consumers of thermal energy as well as bio waste producer. There are huge opportunities to utilise the bio wastes for energy generation. In this project two cases were studied. Fermented grains and hops are used for the beer production and at the end the used materials are disposed with the yeast as organic wastes, in a similar way rice spirit is produced from a mixture of grains which are discarded at the end as spent grains. The feasibility of using the waste individually or in a mixture as feed for Anaerobic Digestion (AD) was evaluated. This project has considered the concepts of generating a more environmentally friendly thermal energy using solely the bio wastes from the industry and if applicable the low grade waste heat available to integrate a waste to energy system. In order to assess the viability of the use of the bio wastes, experimental work in the laboratory for the characterisation of the waste streams as well as an energy audit was performed. Continuous Stirred Tank Reactors (CSTR) were run for a period of time at mesophilic and thermophilic conditions, using the waste grains individually and in a mix. Once the bio methane potential was determined, simulations of the process were executed using engineering software and compared with the data gathered from the site visit. It is found that the figures of final composition of the product as well as the mass and energy balance were similar to the data obtained. The results showed the potential to use the spent grains as feed for the AD plant to produce biogas is positive, covering up to 70% of the total fuel required for production in the case of the brewery and 100 % in a spirit plant. It was also noticed that running the system at thermophilic temperature had a better performance in terms of methane content. The biogas could be used instead of the fossil fuel being used at the moment. Replacing the current use of fossil fuel will be a positive impact in CO₂ emissions reduction as well as economic performance for the brewery, since these bio wastes are often given away to farmers free of charge or more expenses would be incurred in discarding them from the site. This will lead to self-sustained production process and improve the waste management.

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- C.W. Chan n , E. Siqueiros, J. Ling-Chin, M. Royapoor, A.P. Roskilly (2015). **Heat utilisation technologies: A critical review of heat pipes.** Renewable and Sustainable Energy Reviews 50 (2015) 615–627
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Nomenclature

AD	Anaerobic Digestion
BMP	Bio methane potential, ml CH ₄ /g VS
<i>B_o</i>	Observed SMY, ml CH ₄ /g VS added
<i>B_u</i>	Ultimate SMY, ml CH ₄ /g VS added
COD	Degradable chemical oxygen demand, mg/l
COD _T	Total chemical oxygen demand, mg/l
<i>c_pw</i>	Specific heat capacity of water kJ/kg°C
CSTR	Continuously-stirred Tank Reactor
DGS	Distillers grains
<i>d</i>	(days)
<i>gCV</i>	caloorific value of the coal (kJ/kgK)
HRT	Hydraulic retention time, days
<i>H</i>	Enthalpy of steam (kJ/kg)
<i>h</i>	Entalphy of water (kJ/kg)
<i>I/A</i>	Intermediate Alkalinity, mg/l CaCO ₃
<i>l</i>	length, m
<i>m_w</i>	mass flow rate kg/h
<i>M</i>	Cumulative biogas production, l/(g VS) at any time <i>t</i>
<i>n</i>	Number of moles of gas
OLR	Organic Loading Rate, gVS/L-d
<i>P</i>	Biogas yield potential, l/(g VS)
<i>p</i>	Absolute pressure of gas, kPa

P/A	Partial Alkalinity, mg/l CaCO ₃
q	mass flow units (kg/h)
q _{tr}	Transient heat transmission, kW/R ²
Q _{con}	Heat for condensation kJ/h
r	radius, m
R	Maximum biogas production rate, l/(g VS d)
R	Universal gas constant, 8.3145 L kPa/K · mol
SMY	Specific bio methane yield, mL CH ₄ /g VS added
t	Time at which cumulative biogas production M is calculated, d
T	Absolute Temperature K
TA	Total Alkalinity, mg/l CaCO ₃
TAN	Total Ammoniacal Nitrogen, mg/l
TKN	Total Kjeldahl Nitrogen, mg/l
TS	Total Solids, %
TSS	Total Suspended Solids, %
V	Volume (ml)
VFA	Volatile Fatty Acids, mg/l
VS	Volatile Solids, %
VSS	Volatile Suspended Solids, %
WDG	Wet Distillers Grains
ΔT	change in temperature °C
α	Heat transfer coefficient (W/(m ² K))
λ	Duration of lag phase, days

Chapter 1. Introduction

1.1 Research Background

Food processing industries are extremely large consumers of thermal energy and bio waste generation and there are huge opportunities to improve its utilization. In the food and drinks industry sector, energy is often used inefficiently with great opportunity of improvement. However, the demand for energy from this sector will continue to increase due to economic growth. Currently the use of fossil fuels is the main tendency in most industrial processes even though the excessive use of these can lead to environmental issues. In the food industries a considerable amount of waste is generated as co-product. Normally the wastes don't receive further treatment being the most common practice to dispose them as animal feed or for landfill. As the constraints related to environmental issues are becoming more rigorous, it is necessary to develop optimized systems for food waste treatment. Among the several biological and chemical processes, combustion and co-firing seem to be a viable solution as an alternative to landfill. Wastes containing combustible material may be incinerated or combusted to produce heat and water vapour that can be utilized.

Fermented grains containing mainly sorghum, rice and corn are used for the spirit production. Even though they are re utilised during the process at the end are disposed as biological waste consisting mainly of wet distillers grains (WDG). In the Brewery, grains, hops and yeast are disposed as waste as well after the production process. The feasibility of using the bio waste as fuel for combustion/co-combustion in a boiler for the steam generation was evaluated. This applied to industries in China as part of the GLOBAL Sustainable Energy through China-UK Research Engagement (SECURE) project. Which counted with the research capabilities and expertise of staff at Newcastle University and a number of leading Chinese Universities. In order to address the globally important challenge of achieving sustainable and clean energy. China is one of the largest energy users in the world. In the United Kingdom (U.K.) the the food sector (excluding agriculture) increased by 67% between 2000 and 2014. In the study the main objective is to evaluate the use of bio waste produced during the production process

in combination with the waste heat generated to produce fuel using waste to energy technologies. This could be used as a complement or complete replacement of the fossil fuels for thermal energy generation in an industrial process of the drink industries. Another possible use for the WDG that was considered is as substrate to feed an Anaerobic Digestion (AD) process for biogas generation. This project has considered the concepts of generating a more environmentally friendly thermal energy. This could be achieved using solely the bio waste in combination with the waste heat, if applicable, from a spirit production plant and a microbrewery. A system to optimally integrate AD technology with combined heat and power to utilize the bio waste directly from the production to energy for the plant was considered.

In order to assess the viability of the use of the bio waste, samples were taken on-site of both production plants. Then they were characterised in the laboratory to determine the anaerobic biodegradability. A set of four reactors were installed and operated for 100 days. Finally simulations were performed using ECLIPSE package.

1.2 Aims and Objectives

The project aims to investigate and develop methodologies and systematic procedures for the optimum thermal energy and waste management. As well as maximising the energy utilization efficiency for heat recovery and storage from process waste streams of two study cases. In order to improve the thermal performance to obtain benefits in environmental, economic and social aspects. This will aim to reduce the dependence of fossil fuels which are currently the source for 100 % of the thermal energy utilization in the production process. That would lead to a more sustainable process and reducing the CO₂ emissions to the environment. This will be achieved using:

- Field study and energy audits of the case studies – analysis of energy consumptions and wastes in/from each process;
- Evaluation of the energy requirements for the processes by computational simulation applying the principle of energy and mass balance;

- Potential technologies to utilise the waste heat in these selected process industries to maximize the usage of energy;
- Laboratory experimental tests: Potential technologies to utilise the bio-wastes generated to produce renewable biogas as the fuel for generating heat and/or electricity for the production processes, to achieve sustainable energy supply from the renewable bio-wastes.

Some benefits expected when applying thermal energy and waste management in process industries and opportunities related to energy efficiency are:

- Increase of energy efficiency
- Cost reductions
- Reduction of harmful emissions, which have become a health risk in many metropolitan areas
- Conservation of domestic or imported energy resources for the national economy.
- Used to import energy can now be invested in more labour-intensive, energy-efficient technologies thereby creating additional jobs
- Efficient use of energy is the most economical and effective way of reducing the global warming effects and climate change caused by CO₂ emissions.

1.3 Methodology

The quantity of bio-waste produced by Food and drink industries is considered to be one of the most serious environmental issues [1]. These are large in bulk with their major outlet in agriculture specifically animal feed [2]. The major challenge is not only the bio-waste products produced but their bulk which if optimally utilized through anaerobic digestion or direct combustion will be capable of supplying all or part of the energy requirements of the industrial facilities.

1.3.1 Waste to energy

According to the Commission of the European Communities [3] the use of waste for energy production can contribute to achieving the 20% renewable energy goal and the 20% reduction of CO₂ emissions agreed upon at the European level . Waste-to-Energy (WtE) technologies have been studied and appear promising in terms of offering electricity, heat and transport fuels. There are different types of technologies such as thermo-chemical, bio-chemical and chemical conversion processes and they all depend on the type of substrate. In this study combustion (thermochemical) and Anaerobic Digestion (biochemical) are studied in order to be compared.

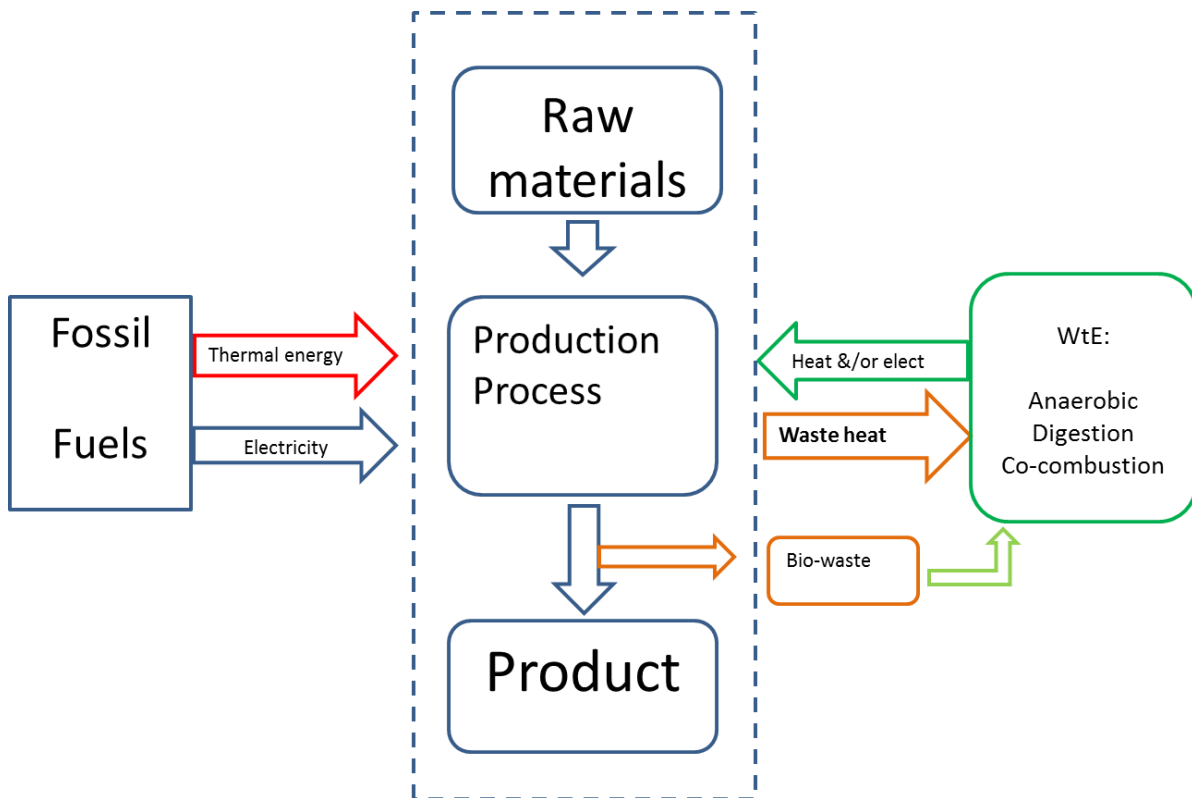


Figure 1-1 Schematic of general production process

A typical process can be described in Figure 1-1. It can be seen that raw materials are processed to obtain the required product but there is waste generation which is normally discarded. In a similar way energy is required within the process and some of it especially waste heat is also lost into the atmosphere. Waste to energy technologies can be used to recover this waste. The current energy losses could be recovered to run these technologies either as a pre-treatment for the waste such as drying or to maintain an anaerobic digestion system at the optimal conditions.

1.3.2 Investigation Of Energy Use In Food And Drink Industry

Two case studies were evaluated in this work. Both from the drink industry, in order to get accurate data each plant was visited for a period of days and all the process was mapped. During this stage the energy flows and consumption was determined.

1.3.3 Energy Audit For Industry

Using the energy audit methodology [4], data was collected within the visits. The steps followed for the audits included the ones described as level I and II, and when it was possible also in level III. Because the complexity of both processes was not high it was easy to focus just in the energy flow, as well as in the bio waste generation for each process.

1.3.4 Waste To Energy Technologies

During this work the concept of Waste to Energy technologies is going to be explored. These technologies are used to produce fuel in order to then be transformed in heat and/or electricity. This will represent the advantage that will also reduce significantly the amount of waste that is currently being discarded as landfill. In other words this kind of approach can give both a solution for waste minimization and energy production. That will lead to a sustainable production process with minimum or zero fossil fuels.

1.3.5 Experimental Investigation

Once the case studies were visited and the energy audit carried on, samples of the waste were taken for further study in the University. This study was not only limited to the characterisation of the waste, but also included an experimental work on the feasibility of

using the bio waste as feed for an Anaerobic Digestion process. A lab scale batch and continuous reactors were run at different conditions in order to determine which wastes could be suitable for biogas production. The bio gas could be used to replace the fossil fuels currently used. During these experiments the option of using residual heat from the process was explored. One of the conditions was run with a higher temperature to evaluate if this has a positive impact in the overall energy use.

1.3.6 Modelling and Simulation

The experimental work carried out in the university laboratories. Then simulations were done exploring different scenarios. Different arrangements were explored in order to use the bio waste or the biogas according to the experimental data obtained. The simulations were performed using a commercial software that has been used by several European projects. The validation of the simulations was done using the data obtained from the cases and also compared with literature. Finding out that the potential of the bio-wastes could be used to replace the fossil fuels currently used in the industries.

Chapter 2. Literature Review

2.1 Introduction

In this chapter a literature review on the current situation of the industrial energy consumption with a focus on the food and drink industry is going to be presented. Also a review on the current waste generation from this sector and the waste heat available in the processes are going to be considered. A review on waste to energy technologies as well as their integration with heat recovery systems was prepared. Finally a focus on anaerobic digestion being one of the most feasible WtE is presented. It was found that even though there are several studies with a focus on the reutilisation on the bio waste, they usually don't consider an integral approach in including a heat recovery from the same or even another process.

2.2 Energy consumption in food and drink industries

Food and drinks processing in the UK is a major industrial sector. It accounts for around 25% of industrial energy use (42 TWh/year) [5] [6]. Since last decades' research in heat recovery and utilization has been conducted because of the amount of low grade heat available from process industries. Different technologies have been studied [2, 7] in order to improve the thermal management and recovery from industrial process. Among the technologies studied steam Rankine, organic and supercritical Rankine cycles, trilateral cycle, and heat pipes have been extensively reviewed [7, 8]. It has been found that in most of the research the main focus is on electricity production from waste heat. As pointed by Chan et al.[7] there are still huge opportunities to improve their efficiency. The current technologies show a better performance with higher temperatures than the ones found in the food and drink industry (200 °C).

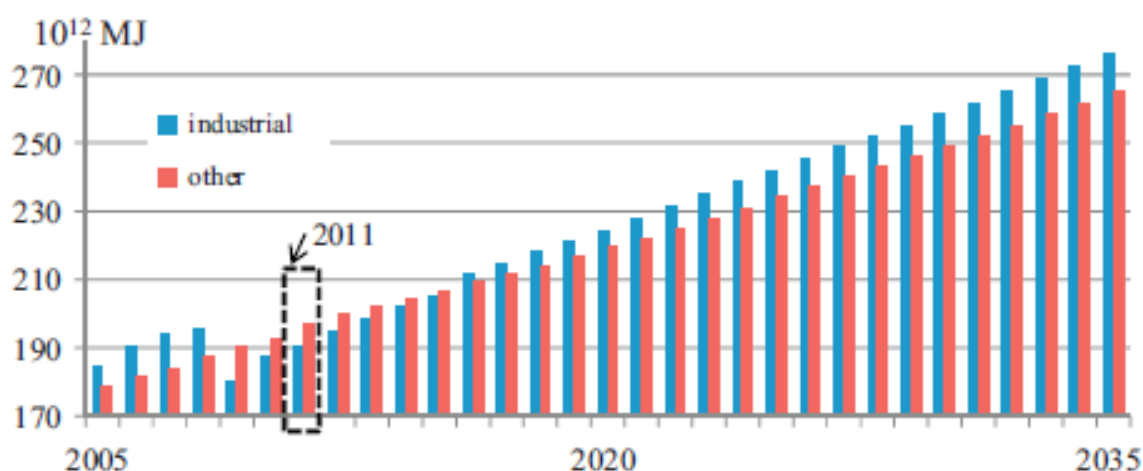


Figure 2-1 World energy consumption in the industrial and all other end-use sectors, 2005-2035 [9].

The chemical, food and drink, steel and iron and pulp and paper sectors of the process industries are substantial users and represented more than 50% of the industrial energy usage. Furthermore, energy cost represents a notable percentage of the total cost of production in these sectors .

Due to economic growth enterprises gain a competitive advantage and prepare for future environmental standards. Figures 2-1 and 2-2 show how the share of fuel will evolve from 2006 to 2030, according to [10]and the[11].

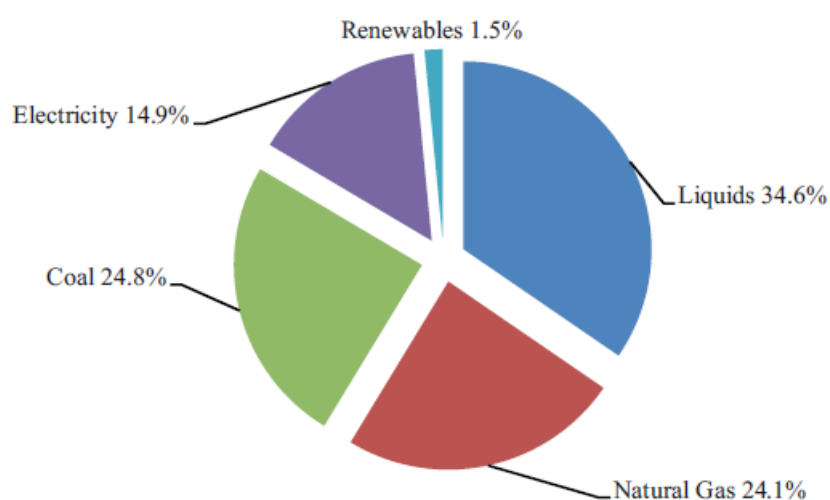


Figure 2-2 World industrial energy sector share by fuel in 2006 [9].

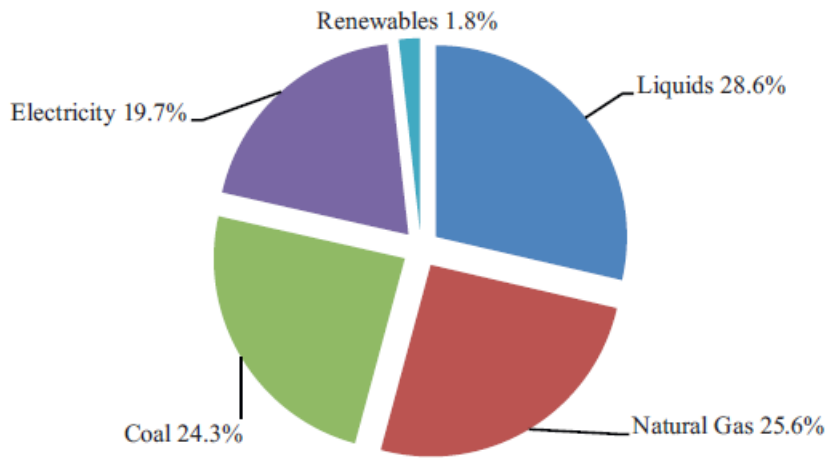


Figure 2-3 World industrial energy sector share by fuel in 2030 [9].

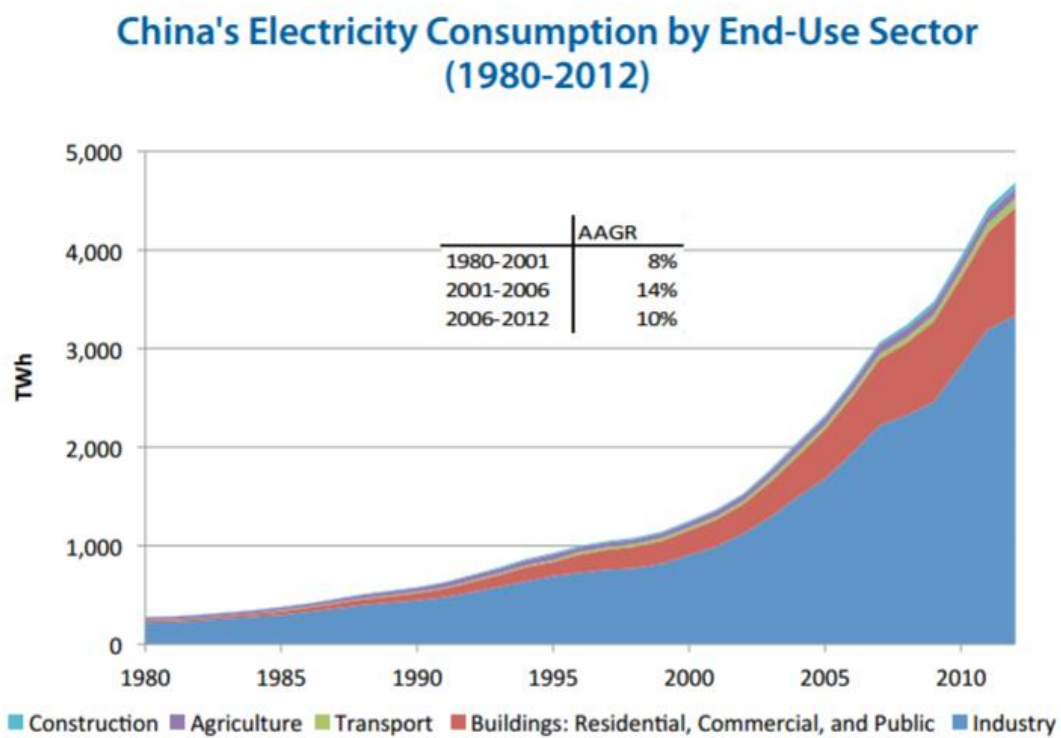


Figure 2-4 Share of Electricity consumption by end user in China in 1980-2012 [9].

China's Natural Gas Consumption by Sector (1980-2012)

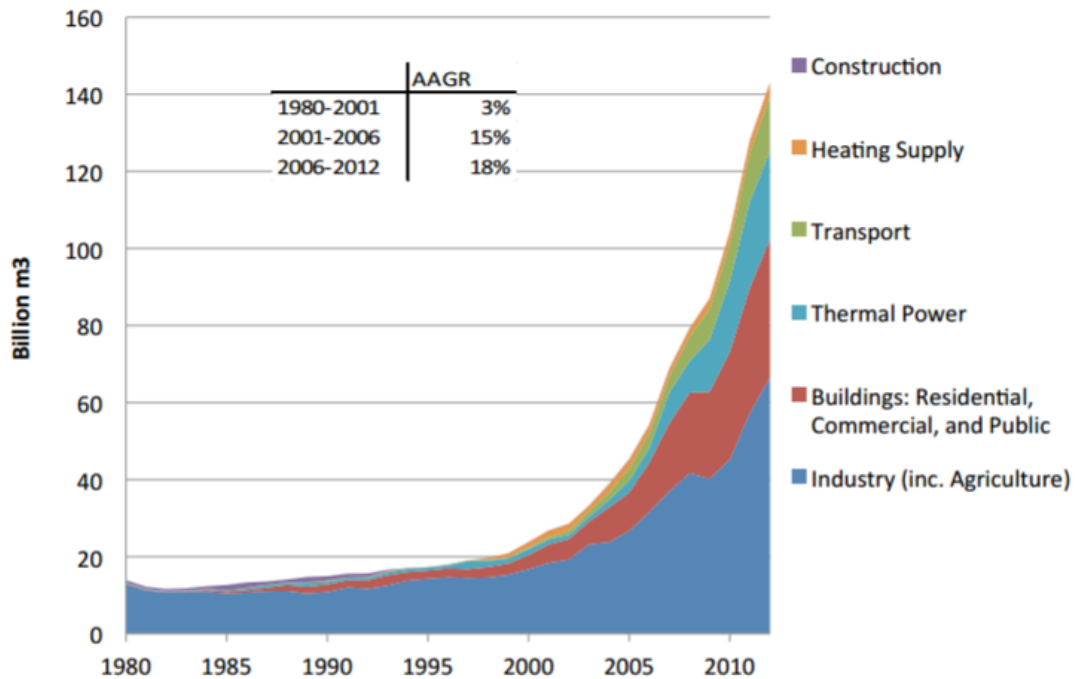


Figure 2-5 Share of gas consumption by end user in China in 1980-2012 [9].

As it can be seen in Figure 2-4 and 2-5 the share of energy consumption in the industrial sector in China is much has been increasing since 1980. Furthermore, energy cost represents a notable percentage of the total cost of production in these sectors.

Ammar et al [12] discussed the terms high grade heat and low grade heat which have been extensively used in the literature. High grade heat was referred to the heat which is viable for capture by the industrial processes, while low grade heat will be used to refer to that which it is not viable to recover within the processes and is rejected to the environment. Law, et al. [13] used the definition from [14] of low grade heat in terms of temperature as the one available from ambient temperature up to 260 °C. Therefore, technology investigation has to focus on that range of temperatures in order to find appropriate ways of reusing or recover the low grade waste.

2.3 Energy Audits

An energy audit consists in an inspection, analysis and survey of the energy flows to reduce the amount of energy input into the system. The fundamental goal of energy management is to help the industry to analyse its energy use and find energy saving opportunities and waste energy reduction. This is in order to produce goods and provide services with the least cost and least environmental effect [10] . Energy Audit is defined as “the verification, monitoring and analysis of use of energy including submission of technical report containing recommendations for improving energy efficiency with cost benefit analysis and an action plan to reduce energy consumption” [15].

The three top operating expenses that can be found in any industries are often found to be energy (including electrical and thermal), labour and materials. Energy management function constitutes a strategic area for cost reduction. They help to understand more about the ways energy and fuel are used in any process. They also help in identifying the areas where waste can occur and where scope for improvement exists[16], [17] .

The Energy Audit aim is to give a positive orientation of the energy utilization, preventive maintenance and quality control programmes. Which are vital for production and utility activities. An audit programme also helps to keep focus on variations which occur in the energy costs, availability and reliability of supply of energy, decide on appropriate energy mix. It also helps to identify energy conservation technologies and retrofit for energy conservation equipment. An Energy Audit can be considered as a tool for the translation of conservation ideas into realities. This is done by lending technically possible solutions with economic and other organizational considerations within a specified time frame. The primary objective of Energy Audit is to determine ways to reduce energy consumption per unit of product output or to lower operating costs. Energy Audit provides a reference point for managing energy in the organization and also provides the basis for planning a more effective use of energy throughout the organization [4].

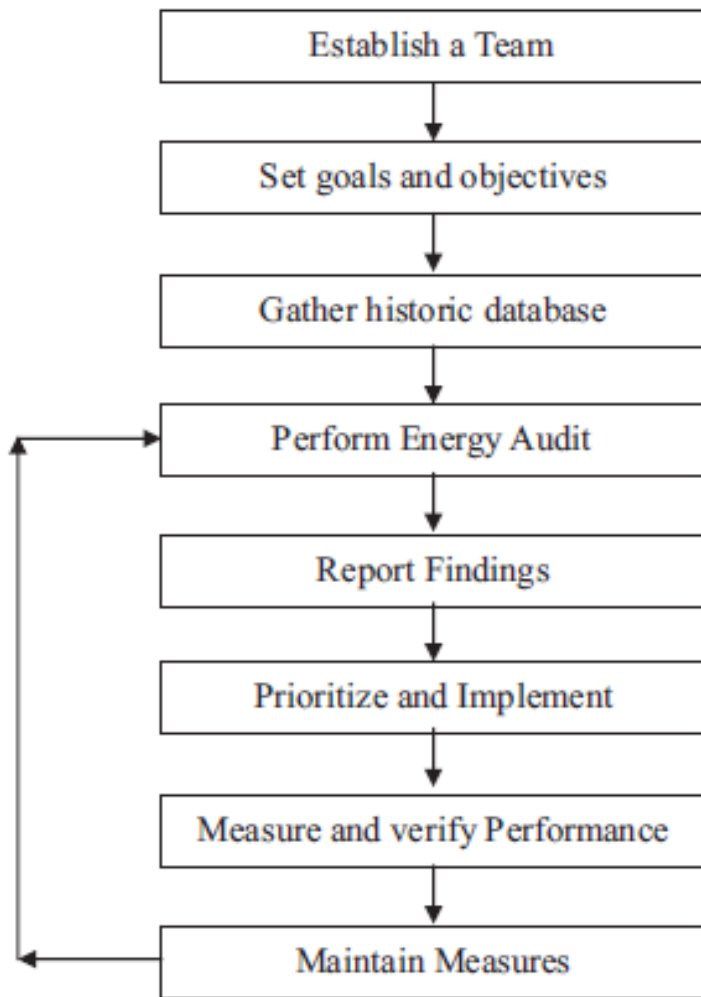


Figure 2-5 Typical energy audit [7]

Different types of Energy Audits can be found, in order to approach all the diverse industries and processes.

The type of Energy Audit to be performed mainly depends on:

- Function and type of industry
- Depth to which final audit is needed, and
- Potential and magnitude of cost reduction desired [16], [17].

Thus Energy Audit can be classified into the following three types.

- Preliminary Audit
- General Audit
- Detailed Audit

Preliminary Energy Audit

Is the simplest and cheapest that can be done, it doesn't require specific measurements or detailed data, it rather uses general energy bills, existing or easy data to obtain and may involve a quick visit to the facilities, and the aims of preliminary audits are:

- Establish energy consumption in the organization
- Estimate the scope for saving energy
- Identify the most likely (and the easiest) areas for attention
- Identify immediate (especially no-/low-cost) improvements/ savings
- Set a 'reference point' for comparison after the audit.
- Identify areas for more detailed study/measurement

The general audit based on the preliminary audit expands by collecting more detailed information about facility operation and performing more detailed interviews with the workers. An evaluation of energy conservation measures identified in the preliminary audit is normally conducted. In this stage utility bills are collected for a 12–36 months period. This is done in order to perform a breakdown of the energy use in the process, energy usage profiles and insight into variations in daily and annual energy consumption and demand. Measurements of specific energy-consuming systems can be performed as well [16], [17].

A detailed audit evaluates all major energy using systems. This is performed in order to provide a comprehensive energy project implementation plan for a facility. This is the type of audit which requires more information and time. In some cases in order to get the data

special equipment is needed, in the other hand it also offers the most accurate estimate of energy savings and cost opportunities. In a detailed audit, based on the data acquisition and information gathered as well as the inventory of energy using systems the energy balance is done. This is then compared to utility bill charges. Detailed energy auditing is carried out in three phases: Phase I, II and III. [16], [17].

Phase

I

Plan and organize in site audit informal interviews with process

Step 1 engineer(s), Production manager

Step 2 Meeting with divisional heads

Phase

II Primary data gathering, process flow diagram and energy utility

Step 3 diagram

Step 4 conduct survey and monitoring

conduct of detailed trials for the highest energy consumption

Step 5 equipment

Step 6 analysis of energy use identification and development of energy

Step 7 conservation opportunities

Step 8 Cost benefit analysis and reporting and presentation to

Step 9 management

Phase

III Post Audit phase

Step 10 Implementation and follow up

Figure 2-6 Phases of an Energy Audit [7]

Food processing involves several physical unit operations, microbiological, biochemical and chemical processes. All of them aim at preservation and improvement of food quality, or conversion to safe nutritional food products in large, economic scale. Food engineering has evolved into an interdisciplinary area of applied science and engineering. The food processing unit operations have been primarily adapted from chemical engineering. Due to diversity of food processes and food products, several specialized unit operations have been developed. Based on their purpose, these unit operations were classified into three broad groups, separation, assembly and preservation [18].

2.4 Types of bio waste

Bio waste can be considered within the biomass resources which include various natural and derived materials. Such materials can include woody and wood wastes, bagasse, agricultural and industrial residues, waste paper, municipal solid waste, sawdust, bio solids, grass, waste from food processing, animal wastes, aquatic plants and algae etc.[19]. There have been some classifications of the bio wastes depending on their characteristics. Primary residues like by- products of food crops, secondary residues which includes the waste from the food and drink industry and biomass derived commodities

Bio-wastes in the context of this review will be interpreted to mean the biodegradable fraction of products and residues from organic non-fossil materials of biological origin. Considering that they are readily available in a renewable and sustainable basis which can be used as a liquid, solid or gas bioenergy source through the state-of-art technology of thermal, chemical and biological conversion techniques. The quantity of bio-waste produced by Food and drink industries is considered to be one of the most serious environmental issues. They represent a large in bulk with their major outlet in agriculture, specifically animal feed. The major challenge is not only the bio-waste products produced but their bulk which if optimally utilized through the appropriate waste to energy technology will be capable of supplying all or part of the energy requirements of the industrial facilities.

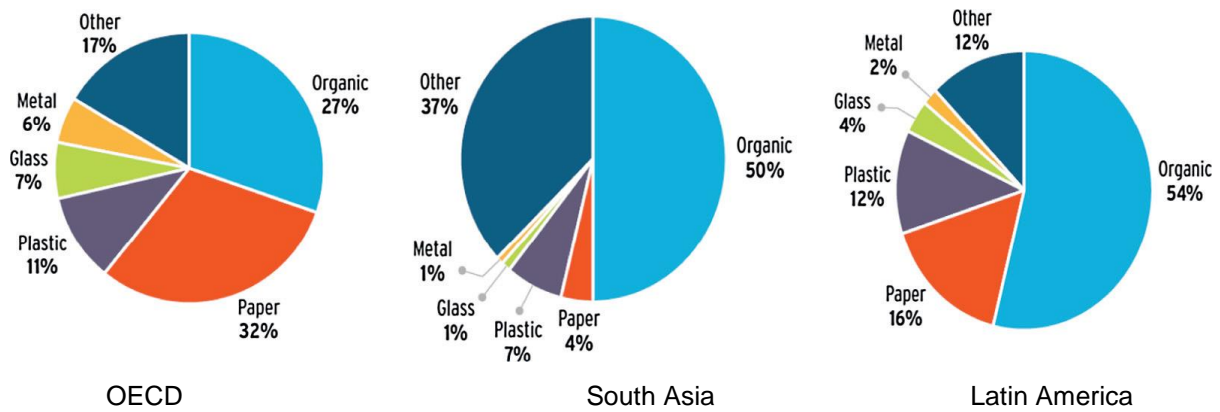


Figure 2-7 Composition of municipal solid waste in different regions of the world. [20]

2.4.1 Distillers Grains (DGS)

Most alcoholic spirit drink plants in the world are dry grind facilities which use starch from Corn, Sorghum, Rice, etc. to produce ethanol. The remainder of the grains is used to produce a variety of wet and dried distillers grains co-products including DGS. However the yield of biogas from anaerobic digestion for various categories of bio wastes from food industry (including wine industry) could be up to 0.4-0.8 m³biogas/kg and crops in the range of 0.35-0.4m³/kg. Sorghum is known to be an annual plant of tropical origin. Recently a lot of research has been undertaken in the EU and other countries to explore its biomass productivity and energy potential under various conditions[21] [22]. The fuel characteristics of Sorghum is 44.0%C, 6.25%H, 0.20%N, and 0.9%S, hence 48.65%O with average calorific value of 4100kcal/kg [23]. On the other hand brewers' spent grain (BSG) is the main by-product of the brewing industry, representing around 85% of the total by-products generated [24-26].

2.5 Waste to energy technologies

There have been studies exploring the different waste to energy technologies which include a wide range of systems and processes.

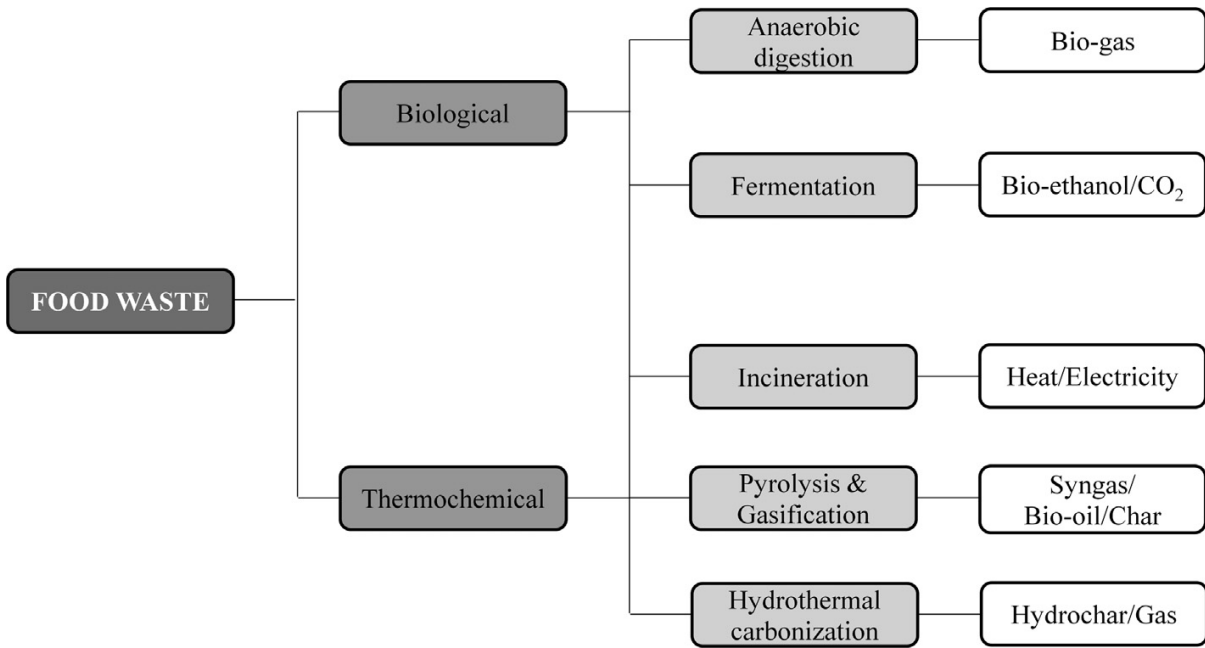


Figure 2-8 Waste to Energy Technologies [27]

2.5.1 Thermochemical Conversion Processes

The thermochemical processes take place at high temperatures these include Combustion, Gasification, and Pyrolysis among others.

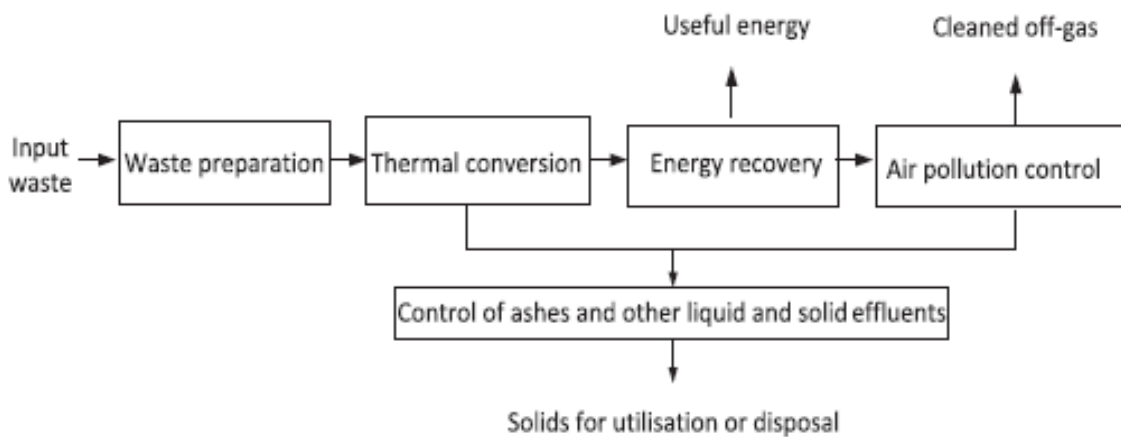


Figure 2-9 Generic waste to energy system based on thermal conversion[28]

2.5.1.1 Combustion

This method also known as incineration is the earliest and still one of the most used in order to converting bio waste to energy. As study conducted by Wang et al. [29] found that since 2009 the number of papers published in incineration technologies has steadily increased. China is the country with more research on this area among other developed countries, which means that in the future it could be considered as an alternative to fossil fuels and waste reduction in that country.

Its principle is a direct and complete oxidation in which carbon in bio waste is oxidized to carbon dioxide, hydrogen to water, sulphur to sulphur dioxide and nitrogen to nitrogen oxide. One of the disadvantages of combustion is related to toxic air emissions generated from the earlier equipment and technologies as pointed by[30].

Biomass co-firing in large coal based thermal power plants has been considered as an opportunity to increase the share of renewable energy sources in the primary energy balance and the share of electricity from Renewable Energy in gross electricity consumption for a country. Among the advantages of co-firing biomass is the reduction of CO₂ and SO₂ and NO_x emissions. It also represents a near-term, low-risk, low-cost and sustainable energy development. Biomass co-firing has been successfully demonstrated in over 150 installations worldwide for most combinations of fuels and boiler types in the range 50–700 MWe[31].

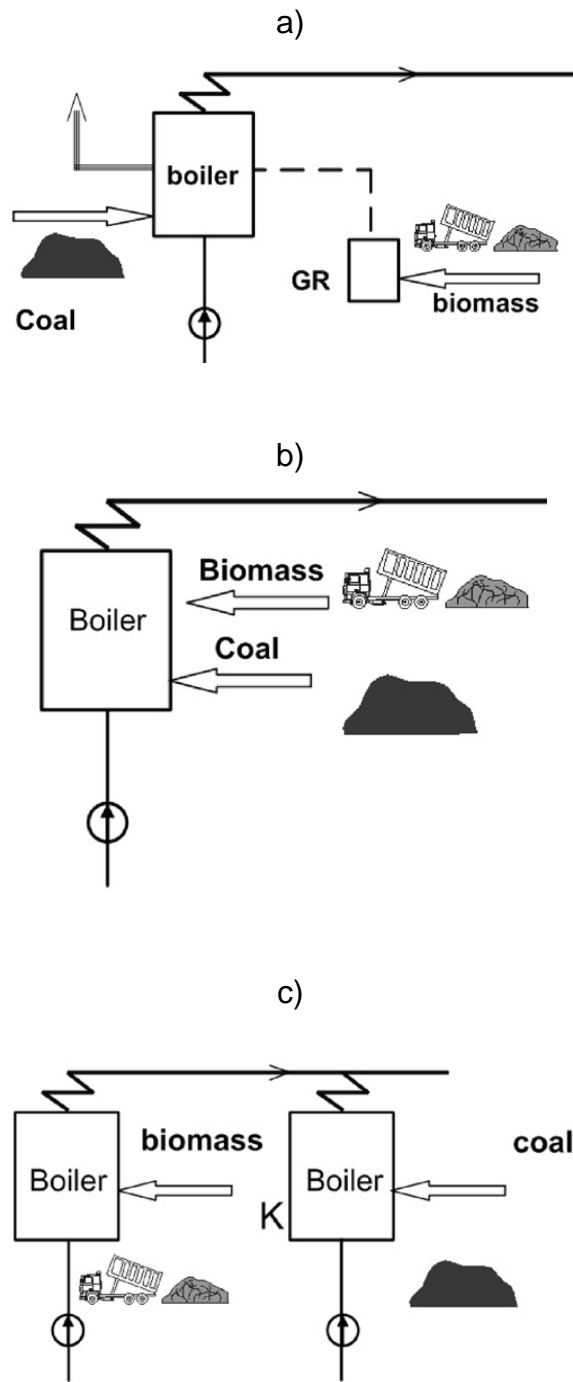


Figure 2-10 Common biomass co-firing technologies [31].

Even though the research on combustion has been increasing recently there are still opportunities to improve the current technologies as suggested by [32]. These technologies could be adapted for organic waste incineration improving the efficiency of the process. Recent studies [33], found that waste incineration technologies are more mature and could potentially be economically feasible. While another study suggest that

there is still an opportunity to investigate the interplay between municipal and industrial waste incineration in order to have a better understanding on the economic advantages of combustion [34].

2.5.1.2 Pyrolysis

Pyrolysis represents a process of thermal degradation where the material is heated at high temperatures (500-550 °C) in the total absence of air in order to produce recyclable products. These products includes char, oil/wax and combustible gases. It has been used to produce charcoal from biomass for thousands of years. When applied to waste management, municipal solid waste (MSW) can be turned into fuel and safely disposable substances (char, metals, etc.) a pyrolysis reactor acts as an effective waste-to-energy convertor. There are different configurations for the pyrolysis system and reactors, fixed-bed reactors, rotary kiln, fluidised bed reactor and tubular reactors have been used for MSW treatment [35].

Furthermore a review on different types of pyrolysis was performed by Tripathi et al. [36] where slow, fast, flash, intermediate, vacuum and hydro pyrolysis were compared and reviewed as well as the main parameters affecting the process. It was reported that temperature is the major parameter that affects the biochar yield and quality. Uslu *et al* noted, the yields of fast pyrolysis are 40-65% for organic condensates, 10-20% char, 10-30% gases and 5-15% water based on dried bio waste[37] [38].

Chen et al. [39] presented a review where the reported pyrolysis temperature varied from 300 to 900 °C, being the typical running temperature around 500–550 °C with liquid products as major portion of products. At temperatures higher than 700 °C, syngas is the main product, due to the fact with the oil they are more valuable research interest has focused in these two. They also reported that residence time of the materials in the reaction zone is an important parameter; which was reported to be in the range of a few seconds up to 2 h.

A study for woody type bio waste found [40] that the chemical composition under slow pyrolysis is crucial and could affect the behaviour of the process. As an alternative for

self-sustainable process, pyrolysis has been demonstrated for vineyard residues, using a mobile pilot paralyzer [41] reported that it was feasible to produce biochar with little pre-treatment in a vineyard in Spain.

2.5.1.3 Gasification

In gasification, the bio waste is converted by partial oxidation at high temperature into a gas known as product gas or syngas comprising carbon monoxide, hydrogen, carbon dioxide, water, methane, higher hydrocarbons and tar. The syngas can be used as fuel in Internal Combustion Engine or gas turbine to power tri generation in food industry. A study from You et al.[42] showed that food wastes are more favourable than sewage sludge for co gasification based on residue generation and energy output. It was also found that the gasification-based schemes are financially superior to the incineration-based

2.5.1.4 Biological processes

In biological processes micro-organisms convert bio waste to biofuels through microbiological method of either fermentation or anaerobic digestion technique.

2.5.1.5 Fermentation

The fermentation of bio wastes involves the production of bio ethanol. First generation bio ethanol was initially obtained from edible sources such as corn, sugar cane and other edible crops. In order to avoid competition with these edible sources the second generation bioethanol was developed and obtained from non-edible materials such as agricultural waste, lignocellulosic from weeds, micro algal biomass etc. Numerous food wastes have been utilized for the production of bio-ethanol, including grape and beetroot pomace[43], potato peel waste, citrus waste [44, 45] and household food waste [46] All of them finding positives results in the bioethanol production.

Since anaerobic digestion plants started to gain a lot of interest in the last decades, it was noticed by some researchers that the AD fibres found in the sludge still contains a notable quantity of cell wall polymers making them interesting for bio refinery integration[47]. The proper combination of bioethanol and biogas production processes has been explored by some researchers and can be regarded as a suitable option to attain a greater bioenergy potential from biomass, improving both the mass and energy balances of single processes [48, 49].

2.5.1.6 Anaerobic Digestion of wastes

Anaerobic digestion is the decomposition of biomass through bacterial action in the absence of oxygen. It is essentially a fermentation process and produces as product biogas, which is a mixture of methane and carbon dioxide with traces of hydrogen sulphide (0-1.5%) and ammonia (0-0.5%). This can be operated in batch reactors, one-stage continuously fed system and two-stage (or multi-stage) continuously fed system with batch operated system being the most simple and efficient [50, 51].

The formation of Methane and Carbon dioxide by the anaerobic digestion of industrial bio waste was first recognised and reported during the latter part of the nineteenth century. This mixture of gases essentially about two-third Methane (66%) and one-third (34%) Carbon dioxide with small amounts of Hydrogen, Nitrogen, Organic Sulphides and Hydrocarbons is known as Biogas[52]. Anaerobic digestion also involves a 4-stage process: hydrolysis, acidification, acetogenesis and methanogenesis as indicated in the figure below.

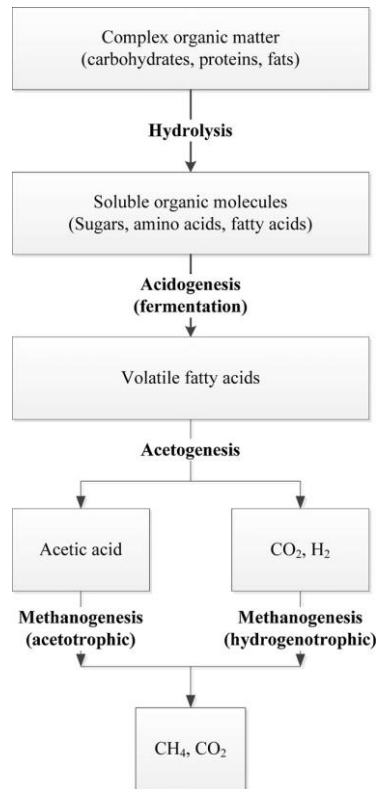


Figure 2-11 Steps of anaerobic digestion [50]

In the first phase anaerobic bacteria use enzymes to decompose high molecular organic substances such as proteins, carbohydrates, cellulose and fats into low molecular compounds. During the second phase acid forming bacteria continue the decomposition process into volatile fatty acids (VFA's), organic acids, carbon dioxide, hydrogen sulphide and ammonia. The formation of the various acids results in a lowering of the pH. During the acetogenesis phase acid bacteria form acetate, carbon dioxide and hydrogen from the VFA's. The methanogenesis phase involves methane forming bacteria producing methane, carbon dioxide and alkaline water and has an optimum pH range of 6.5-7.5 [53].

2.6 Anaerobic digestion technologies

There can be found several configurations and equipment for anaerobic digestion plants. These include a range of reactors, in a wet or dry basis, single stage or multi stage, batch

or continuous, all of these depending on the type of waste to be treated [54]. As mentioned before, moisture is an important characteristic of the waste in the digester [55]. It is essential to maintain the correct water content in order for the activity of the anaerobes to be at an optimal level and maintain biogas production. Digesters can be classified by their state as wet or dry [53]. Wet reactors are those with a total solids value of 16% or less. Whilst dry reactors have between 22% and 40% total solids and those that fall between wet and dry are considered semi-dry. Although other researchers as [56] state that biomass with moisture content in excess of 75% can be effectively processed.

Hydraulic retention time (HRT) can be defined as the volume of the tank divided by the daily flow. Digesters can also be defined by their rate as low rate or high rate. A low rate system is one with long hydraulic retention times while high rate systems will present shorter hydraulic retention times. Where lower performance on long-sludge age activated sludge and higher performance on primary sludge can be found. Low rate systems are generally used for feedstock with a relatively high solid content which require longer to be broken down sufficiently. High rate systems are used for quite dilute feedstock such as waste water where the digestion process occurs a lot faster. In a straight mesophilic anaerobic digestion process with a HRT of 20 days are common, with a conversion of organics to gas is typically between 25–60% [57, 58].

2.7 Factors affecting anaerobic digestion

2.7.1 Temperature

Temperature is one of the most important parameters to look after in an anaerobic digestion process. The microorganisms that will produce the biogas are highly sensitive to it [59]. There are three ranges of temperatures where AD can be implemented. These include a low temperature range where psychrophilic bacteria are present, a medium range temperature for mesophilic operation and a high temperature known as thermophilic. From these three the mesophilic and thermophilic are the most utilized [60]. Mesophilic process takes place at lower temperatures with a high hydraulic retention time (15-30 days). The temperature range is from 30–38°C, and the thermophilic 44–57°C [61].

Thermophilic process takes place at higher temperatures with a shorter hydraulic retention time (12-14 days).

Studies have shown that thermophilic operation usually performs better in terms of biogas production and composition [62] [63]. The biogas presents a higher methane content as well as an increased destruction rate of organic solids, improved solids–liquid separation, and increased destruction of pathogenic organisms. But the drawback is that a thermophilic operation can be more instable and is sensible to temperature changes that could affect the overall performance [60, 63-65]. An important factor for the operation conditions rely on the bacteria and how they can adapt some of them have an optimal temperature with a range of 10 °C [66].

2.7.2 Organic Loading rate

The organic loading rate can be defined as the weight of organic matter (influent waste) per day applied over a surface area. It indicates the amount of volatile solids to be fed into the digester each day [67]. It is most commonly expressed in terms of kg influent waste/m³/day or sometimes as kg VS/m³/day. It can also be expressed as the inverse of the hydraulic retention time multiplied by the influent waste concentration (in grams).

The organic loading rate (OLR) is one of the key factors in determining the system conversion efficiency and hence the quantity of biogas which is yielded. Generally a lower organic loading rate will result in a higher conversion [62].

The organic loading rate highly relies on the feedstock, a study found that for food wastes ORL of 5 and 6 kgVS/m³ presented better performances than higher ones at 9 kgVS/m³ [68]. Another study found that methane yield decreases with the increase of the OLR [69]. [70] reported that at low organic rates the methane production was higher. Similar results were reached in a study comparing the methane yields at different OLR's finding that at 2 kgVS/m³ and 3 kgVS/m³ for municipal solid waste the methane content was the highest compared with higher loading rates [71].

2.7.3 C/N Ratio

Every feedstock used in the AD process is different. Each will have varying quantities of carbon and nitrogen content. It is essential that this ratio is evaluated so that the correct balance can be achieved in order to accomplish optimum performance in the AD process[72]. Otherwise one of these substances will represent the limiting factor.

Whereas carbon constitutes the energy source for the micro-organisms, nitrogen serves to enhance microbial growth. If the amount of nitrogen is limiting, microbial populations will remain small and it will take longer to decompose the available carbon. Excess nitrogen, beyond the microbial requirement, is often lost from the process as ammonia gas and it reflects on the system performance [73].

2.7.4 pH, Alkalinity

Alkalinity is an important parameter to monitor during the anaerobic digestion process. It measures the ability of a solution to neutralize acids and can be expressed using several different units (typically calcium carbonate). In Anaerobic Digestion processes it is strongly influenced by the presence of several components such as carbonate and bicarbonate ammonia, phosphate and volatile fatty acids. The determination is generally done by titration using 0.1 N HCl (or another strong acid) at two equivalent points: pH 5.75 partial alkalinity (PA) pH 4.3 or total alkalinity (TA).

2.8 Numerical modelling of waste to energy technologies

Because of the complexity of Anaerobic digestion the development of mathematical models, predicting the dynamic process behavior has attracted considerable attention in the last two decades[74]. Back in the 1970's the first modeling approaches focused on describing the limiting step of the process like one developed by Hill and Barth [75]. These kind of models were simple and easy to use but were incapable to effectively describe the process performance.

The incorporation of the acidogenesis and acetogenesis steps led to a new generation of models. These were more focused on the concentration of volatile fatty acids [76] and had a better representation of the process.

Finally with the increase knowledge on the microbiological interactions another generation of models was developed. These models incorporated additional processes and species, more detailed kinetics with inhibition. In 2002, the International Water Association (IWA) Task Group for Mathematical Modelling of Anaerobic Digestion Processes developed the Structured Anaerobic Digestion Model no.1 (ADM1) [77]. Since then it has become widespread and generally accepted. However, one of the limitations is that it does not distinguish between microorganisms performing the same reaction.

Process simulation model using commercial software represent difficulties because of all the kinetics and interactions of Anaerobic digestion. Nevertheless few attempts have been made. A simulation model using ASPEN was developed by Rajendran et al. [78], using 46 reactions including kinetics for the acidogenic, acetogenic and methanogenic steps. Finding an average difference in biogas production from real scenarios of up to $\pm 20\%$ change in substrate composition and the extent of the reaction is 5.279%. Super pro design software has also been utilised to simulate anaerobic digestion with reliable results [79].

2.9 Summary

In this chapter a literature review of the different aspects that have been studied for waste to energy technologies. It can be seen that these technologies are promising with several options have shown positive results for waste reduction and also fuel production. From the operating conditions from the literature review the thermophilic systems have shown a better performance in the biogas yield, having a higher methane composition and also other advantages such as shorter HRT.

Chapter 3. Study Cases

3.1 Introduction

This chapter is dedicated to the description of the two process plants from the drink industry located in China and UK that were visited and studied. Energy audits were performed in both sites and samples were collected for further analysis at the University. The wastes that were studied are the dried waste from the spirit plants which includes a combination of rice, sorghum and corn. For the brewery, three wastes were evaluated separately, yeast, hops and spent barely grains.

3.2 Spirit production plant

The Spirit plant is located in the province of Hunan, at Jishou city. It has 2000 employees and an annual turnover of 164, 455 \$/year. The total annual production is 8,900 tons where the rice spirit is the main product. The company production process is a non-continuous batch, having 2 shifts of seven hours a day with total working days through the year of 218.

The production process used is a traditional technique. This is important as there is great concern that any changes during the process may affect the quality of the product which is among the top priorities from the business point of view. The challenge that this represents is big because even though there is interest in improving the energy management in the site, there are limited opportunities within the process as the priority is to achieve the desired final quality of the spirit. There is no cold generation in the plant and for the moment a heat recovery system for the effluent gases from the boiler has not been considered. Currently the inclusion of renewable energies in the process has not been explored, being the coal the only source of fuel for the thermal energy.

The production facilities consist of:

- 8 workshops with 4 production lines each.

- Each line has 2 Distillers, an air cooling unit, and 120 fermentation pools.
- There are 38 working groups of 12 people each.

The operation consists in four main processes; all of the work is done manually as there is not any kind of automation in the process. The first step is the steaming or cooking of the grains. In this step 2000 kg of raw materials containing the grains; sorghum, rice and corn in a 75:25:5 % ratio are poured into an open tank. Then, 2000 kg of water is added at ambient temperature (22 °C) and 360 kg of steam at 2.5 bars and 127 °C pass through the bottom of the container for four hours (90 kg/h) . The container is a cylindrical concrete open tank with no insulation, causing that all of the steam not absorbed by the material to be lost to the ambient at approximately 100 °C. Figure 3-1 shows the steaming/cooking step. There is no waste generation in this first stage.



Figure 3-1 Steaming step in spirit production.

The next step is to manually put the steamed grains into another pot where air is used for cooling it down to 22 °C, as seen in Figure 3-2. Then distillers yeast is added so the saccharification can begin, and this last for 48 hours.



Figure 3-2 Preparation for cooling.

After that, the fermentation step takes place and it last for 60 days. It is done at ambient temperature, and the grains are covered with a layer of wet clay. In this stage there is a generation of waste of 3, 600 kg comprising wet grains.

Material input for the fermentation:

- 3260 kg of grains cooled down
- 480 kg rice husks
- 10 kg distiller yeast
- 17, 988 kg of fermented grains discarded from previous batches.

The final step is the distillation. The fermented grains are mixed with husks and manually transferred to the distiller, which is an uninsulated stainless steel tank. The distillation occurs by adding steam to the material, as in the first step, the steam flow is controlled manually to get 80 °C inside the distiller and the ethanol is evaporated. Cooling water is used in the condenser at 18 °C. Figure 3-3 shows the distillation unit.

Material input for the distillation:

- 22,778 kg fermented grains

The energy input is of 1,620 kg of steam at 2 bars which represents 983 MJ/h and the waste generated 17,988 kg wet distillers grains to re use in next batches.



Figure 3-3 Distillation Unit

Once the distillation is finished the final step is to bottle. In the figure below a process flow diagram of the operation is shown.

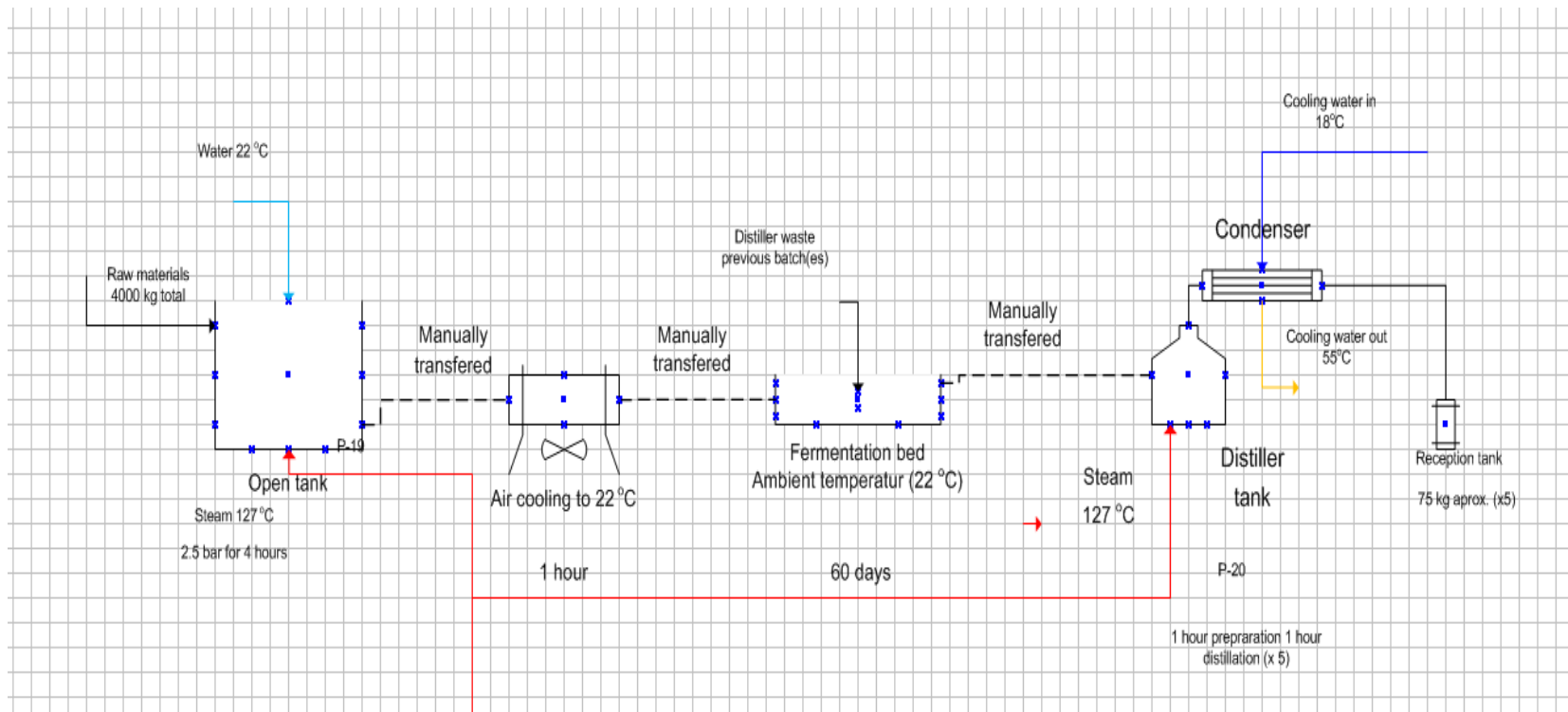


Figure 3-4 Process flow diagram for the spirit production

3.3 Energy demand and heat loss calculations

The energy demand calculations were performed assuming steady state of the process. Steam supply calculations were based on the theoretical energy demand of each production step. It was assumed that there were no losses in the heat transfer and that the condensate left the steam heat exchanger at a temperature of 98 °C and was discarded. The steam supply temperature used was 127 °C, which is the temperature at which it arrives to the process at 2.5 bars. Losses on site due to leakages or insulation were not considered for the calculation purposes. The mass of steam used was directly based on the sum of all theoretical energy demands and heat losses in each stage evaluated (**correction #21 assumption stated**). The calculation for the theoretical steam demand was done using the following equation:

$$m_s = \frac{Q_{dem}}{(\Delta h_e + cp_s * \Delta T)} \quad (3.1)$$

m_s Mass flow rate kg/h

Q_{dem} Heat demand kJ/h

Δh_e Change in enthalphy kJ/kg

ΔT Change in temperature °C

cp_s Specific heat capacity kJ/kg°C

The total heat demand was calculated adding all the energy consumptions including the heat losses during the process given by:

$$Q_{dem} = Q_{st} + Q_{dist} + Q_{loss} \quad (3.2)$$

Q_{dem} Heat demand kJ/h

Q_{st} Heat for steaming kJ/h

Q_{dist} Heat for distillation kJ/h

Q_{loss} Heat loss kJ/h

For the heat loss calculations of the discarded condensate the following equation was used:

$$Q_{con} = m_w * cp_w * \Delta T \quad (3.3)$$

Q_{con} Heat for condensation kJ/h

m_w mass flow rate kg/h

ΔT change in temperature °C

cp_w Specific heat capacity of water kJ/kg°C

The heat loss calculation through the sides of the tanks was calculated given the characteristics of the concrete tank, with a thickness of 20 cm and the distillation unit.

$$q_{tr} = \frac{2\pi l(T_{in} - T_{out})}{\frac{1}{r_{in}\alpha_{out}} + \frac{\ln\left(\frac{r_{out}}{r_{in}}\right)}{\lambda_s} + \frac{1}{r_{out}\alpha_{out}}} \quad (3.4)$$

q_{tr} Transient heat transmission, kW/R²

l length, m

r radius, m

α Heat transfer coefficient (W/(m² K))

Since the steaming tanks are not covered the heat loss to the ambient room was calculated using natural convection. The ambient room temperature was assumed to be 25 °C in average, according to the local weather at the time of the visit and the plant conditions.

Finally the losses from the surface temperature from the mix were given by:

$$q_{tr} = A * k_{mix} \Delta T \quad (3.5)$$

The mapping of the energy flows were determined through the mass balance and the temperatures at which the operation takes place. In most of the cases the temperature was measured in site using the thermal camera to validate the ones given by the industry, finding that they were in within range of +- 3 °C

The losses through radiation were not calculated in this study as in comparison with the other losses they weren't significant.

Steaming step energy consumption calculation

The heat demand for the steaming step was calculated using:

$$Q_{st} = \Delta T * (m_w cp_w + m_{sor} cp_{sor} + m_r cp_r + m_c cp_c) \quad (3.6)$$

For the cooling step it was assumed that only the grains were cooled and there was not loss of water. The temperature of the steam supplied at 2.5 bars is 127 °C.

The fermentation step is done at ambient temperature and no heat input is required.

Theoretical distillation energy demand

The final step of distillation heat demand is given by the latent heat of vaporization of the ethanol at ambient pressure using:

$$Q_{dist} = m_{et} * cp_{et} * \Delta T + m_{et} * \Delta h_{e,et} \quad (3.7)$$

Heat loss from the distiller and condenser surface to the ambient was calculated using equation (3.4) and condensate water discarded with equation (3.5).

Steam generation equipment

The steam for the process is provided by coal boilers with a capacity to generate 10 tons per hour at 6.5 bars. Figure 3-5 shows the schematic diagram of the boiler. The boilers are operated manually as well.

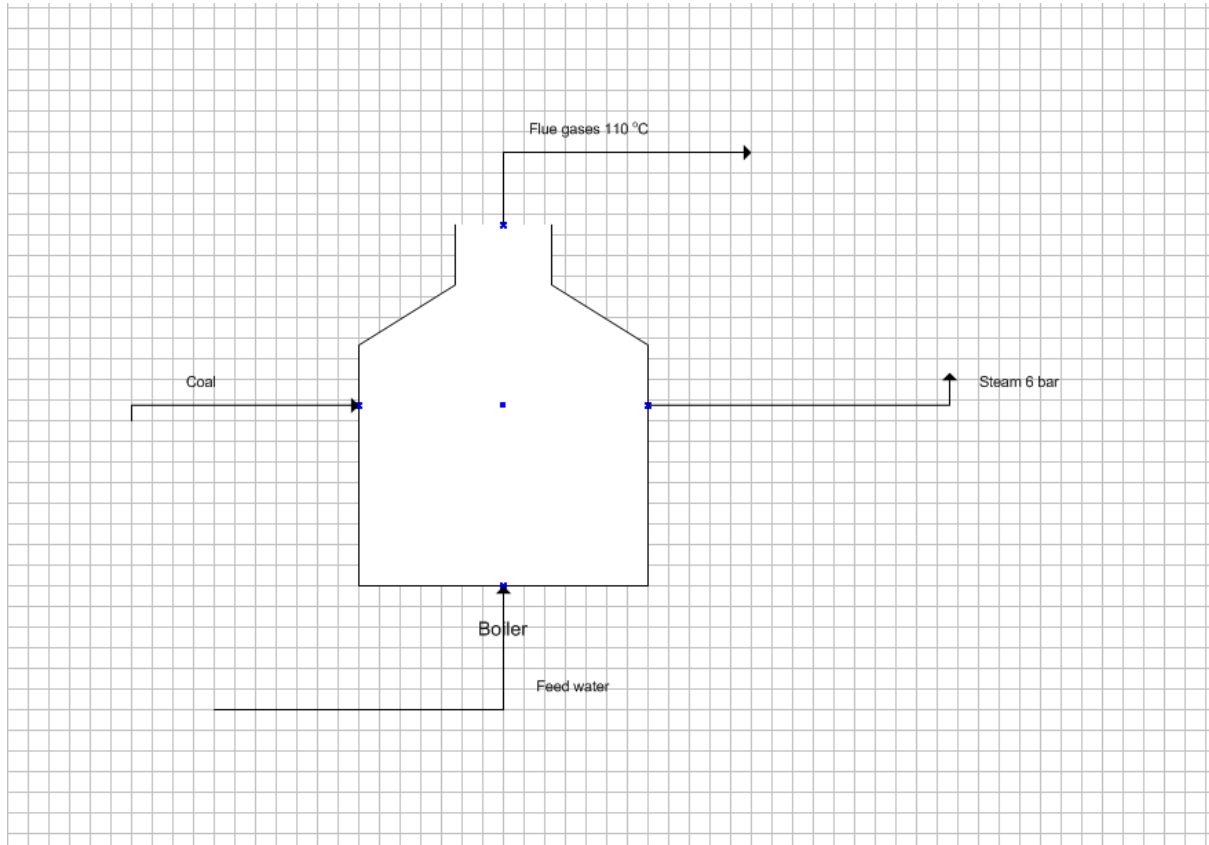


Figure 3-5 Diagram of the boiler

The boiler efficiency can be calculated using the direct method with the following equation:

$$Boiler_{eff} = \frac{Q * (H-h) * 100}{q * GCV} \quad (3.8)$$

H-Enthalpy of steam (kJ/kg)

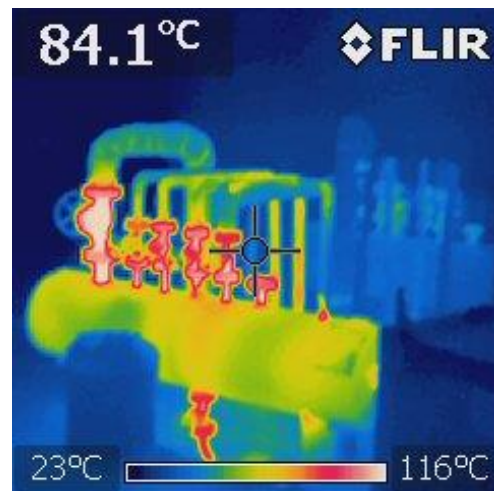
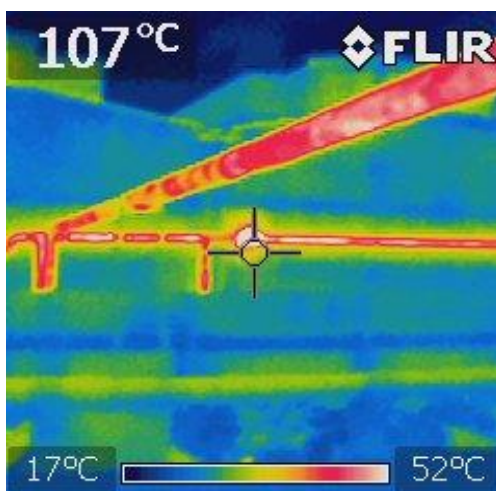
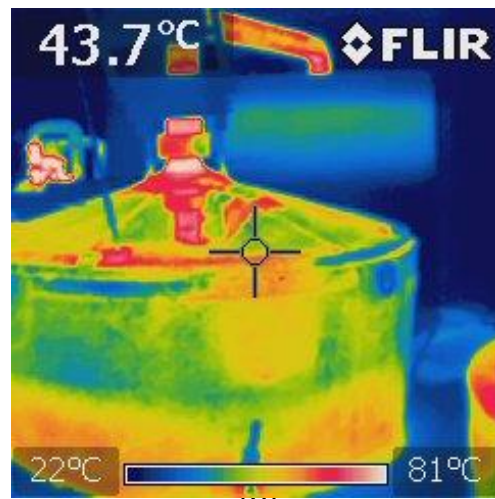
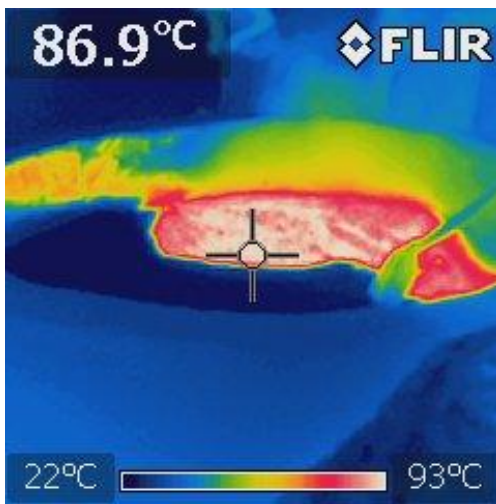
h- Entalphy of water (kJ/kg)

gCV-caloorific value of the coal (kJ/kgK)

q- mass flow units (kg/h)

Since all of the information can be taken from steam tables for enthalpies and the mass flow of the steam produced is 10000 kg/h for each boiler, it was possible to determine the boiler efficiency, which turned to be 56 %.

During the energy audit in site, a thermal camera was used to take pictures of relevant areas, (FLIR model TR650, UK) finding opportunities to reduce the heat losses to the ambient due to lack of insulation or deteriorated insulation.



(c)

(d)

Figure 3-6 Thermal images of the spirit process.

Among the opportunities found to improve the energy use, the steam distribution system represents an important amount of losses to the ambient. This is mainly due to poor insulation as it can be seen in the photos taken with the thermal camera. In Figure 3.6 the red spots represent the hot areas of the distribution system which accounts for important losses of heat to the ambient. Part a) represents the steaming stage, as it can be seen it is done open to the ambient having spots at 89 °C during the process that last an hour. Part b) of figure 3-6 shows the distillation unit, it can be noticed that there are spots that require a better insulation where temperatures up to 80 °C can be appreciated. Finally parts c) and d) it can be seen that the steam distribution system insulation needs to be improved in order to avoid thermal losses of up to 52 °C to the ambient.

During the energy audit it was possible to map the process by comparing the information previously gathered with measurements of the main steps of the operation. This allowed to identify how much energy is used for the actual production and the amount that is lost due to inefficiencies in the process as mentioned above. In Table 3.1 the annual consumption for the company is shown. It can be seen that coal is the only fuel that is used for the production while the electricity is supplied by a local company.

Table 3-1 Fuel used in the production process

Fuels used		Coal	Electricity
	unit	Tonnes	MWh
Annual consumption	units/year	9, 291	4,480
	MWh / year (LCV)	1,170,666	4,480

Table 3.2 shows the cost of the energy for the process being the coal the one that represents higher costs. The utilization of coal is through the boilers with a consumption of 10 tons/hour to generate steam at 6.5 bars which be further used in the process.

Table 3-2 Energy costs for the Spirit Plant production process

		Coal	Electricity
Fuel price	€/kWh LCV	101.2	0.1049
Annual energy cost	€/year	1,012,000	469,952

After the calculations were done, it was possible to construct a Sankey diagram for the energy flow. From figure 3.7 it can be noticed the there is a huge opportunity for heat recovery in the process as almost 50% is lost. The steam that is used for the process only represents 63% of the steam available, and during the steaming step another 60% is lost because the vats remain open to the atmosphere while the grains are cooking.

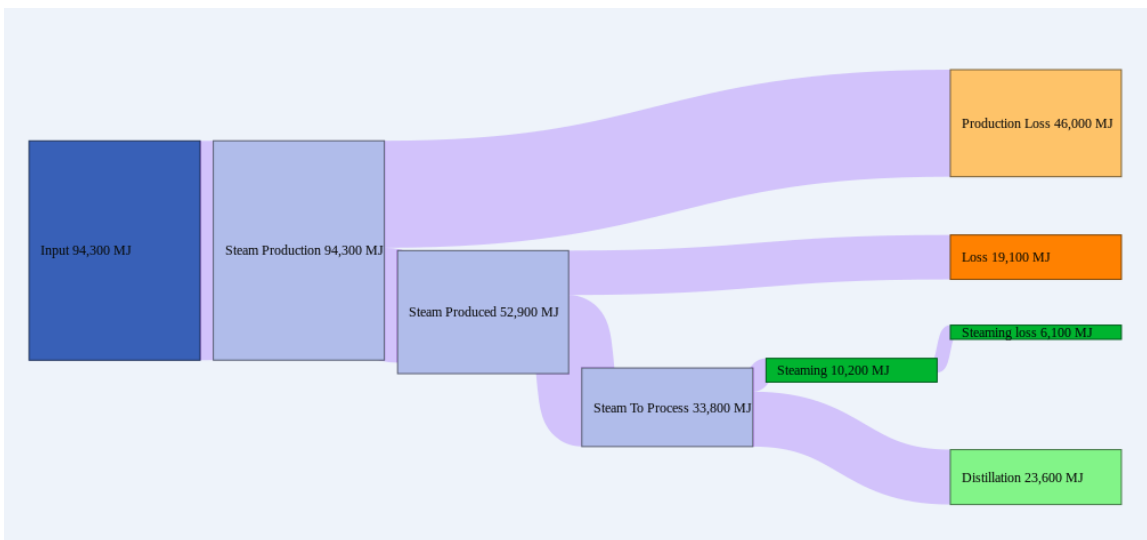


Figure 3-7 Sankey diagram for the steam production and utilization in the spirit plant.

Currently the wastes generated are given away to local farmers with no cost as there is no plan to using them. In this work the potential use of these wastes was evaluated. The feasibility of biogas production from the bio waste or its utilisation as biomass for direct combustion in order to replace partially or even totally the coal which is used was studied. This will lead to a more environmentally friendly operation.

Table 3-3 Energy costs for the Spirit Plant production process

Operation	heat demand kWh	heat loss kWh
Steaming	251.20	
cooling		69.46
distillation	229.40	
condensation		84.50

3.4 Brewery

The second study case is a small scale Brewery within the industry and it's located in the North East of UK. The first visit to the brewery was in the 19th of March 2014, and then in a two months basis in order to collect samples. Its operation is on a batch production process. The beer production process can be represented in four main steps[24], as shown in Figure 3-8.

The first step is mashing, which takes place in a stainless steel tank where approximately 1600 L of water previously heated to 70 °C is mixed with 500 kg of malted barley. This process breaks down the starch into simple fermentable sugars to form the mash. It is critical to keep the temperature at 65.5 ± 1 °C during this period in order to achieve the breakdown of the starch and this is achieved by adding more hot water heated with steam.

Next step is boiling, wort is transferred from the mashing tank to the copper. At this stage the spent grain are then scraped out of the tank and discarded. At the moment is given to local farmer where it is later used as animal feed. Once in the copper, the wort is heated up to a 100 oC and brought to boil after the addition of 50 kg of aromatic hops. Approximately 100 L of the wort evaporates to the environment during this time via an externally vented flue.

Wort is then transferred to the fermentation vessels and it's cooled down by cooling water in a heat exchanger. Yeast is then added to the fermentation vessel(s) and the liquid is

maintained at 21 °C for the fermentation process to occur. After the copper has cooled, the hops are scraped out and discarded, receiving the same treatment as the grains.

Once the beer has fermented a chiller is used to bring the temperature down to 11 °C to stop the fermentation process. The liquid is then transferred to conditioning tanks in a cold room held at 10 °C. There it is stored for a number of days, depending of the beer produced, to let the yeast settle and the beer to mature and stabilise. From there the beer is barrellled and stored in a refrigeration room (storage room) for a short period before it is transported.

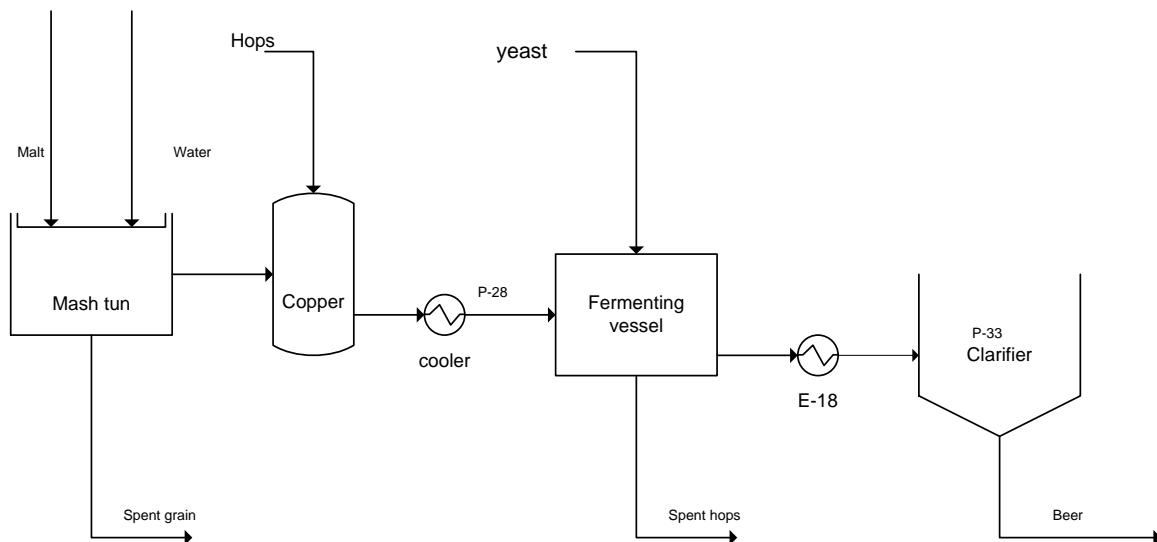


Figure 3-8 Process flow diagram of the brewery

3.4.1 Heating

For this case study a similar approach to the spirit plant analysis was made. In this occasion it was possible to compare the data based on the previous work of [80] and [81]. The energy requirements for the mashing step were calculated using equation 3.6 based on the data given by the brewery during the visit and the properties of the grains. Equation 3.7 was used to determine the heat demand for the boiling at the copper including the

heating of the wort from 60 °C to 101 °C. The heat losses through tanks were calculated with equation 3.4 and the losses for condensation with equation 3.3.

In order to provide the steam required for the heating processes the brewery is currently using a Fulton 20E oil fed boiler. The boiler runs for 5 days a week and approximately 5 hrs per day. The steam is fed through steel pipe work into both the mash tank and Copper as required via a system of ball valves. The data that was proportioned during the audit indicated that over the last 5 years (2009-2014) an approximately amount of 50, 000 litres of oil (l) have been used. Resulting in an average of 10, 000 l a year and this figure gives a monthly consumption of around 830 litres. Based upon the recorded oil consumption, an average use of 1,568 litres per month is considered. This is considering that the boiler is running for 5 hours per day and 5 days per week. The boiler rating required for the process has been calculated at 131.3 kW. This is 34.3 % lower than the currently installed capacity. According to the audit calculations a rating of just 91.2 kW is actually required to fulfil the demand. This indicates that the boiler system is actually running at an efficiency of 69.6 %, due to the losses previously mentioned.

3.4.2 Electricity consumption

A relatively large amount of electricity is required during the brewing process; including the running of pumps and motors, chillers, air conditioners, lighting and office equipment. It was possible to collect meter readings from the previous five years, 2009 to 2014, which have enabled a better level analysis of their usage on a month-to-month basis. It also gives a good estimate of daily usage levels.

According to the meter readings the brewery uses an average of 242.1 kWh/day. The break down is based upon all equipment running over an 8 hr working day for 5 days a week. While cooling equipment (chillers and air conditioners) run for 24 hrs, 7 days a week, this yields a required electrical rating (power) of 19.8 kW. The data collected and calculated from the audit agrees with this rating to within 0.2 %. It also shows that the total electrical requirement is slightly higher than that of the average from the meter

readings. This is because the calculation takes into account both the electricity used for the maximum cooling and heating demands at the same time.

3.4.3 Cooling

Contrary to the spirit production plant there is also a substantial cooling requirement for the brewery; cooling fermentation tank temperatures in the brew hall, cold liquor and conditioning vessels in the cold room and finished product in the refrigeration room. There are 3 glycol chillers and 5 air conditioners throughout the brewery, all of which are electrically powered. In each case half of the rated capacity was used and based upon 24 hr running as this best represented an average demand. This yields a total installed cooling capacity of 567.7 kWh, which equates to a 23.7 kW rating. However, the calculated cooling requirement for the brewery is much lower at just 182.6 kWh, a rating of 7.6 kW. This is just 32.2 % of the total installed capacity. This could be misleading due to the thermal losses from the vessels, boiler, steam transfer pipes during the process is so high that a much larger proportion of the installed capacity is actually utilised. The audit calculations make clear that 62.4 % of the electricity used (168.8 kWh) is in fact for the cooling requirement of the brewery.

The resulting energy demand can be summed up to an average of 131 kW thermal energy considering that the boiler is used 16 h a day for heating, 24 kW thermal energy for cooling for 24 hours 7 days a week and 20 kW electrical power over the same period of time of heating which is 16 h per work day. Figure 3-9 shows the distribution of energy usage in the brewing process.

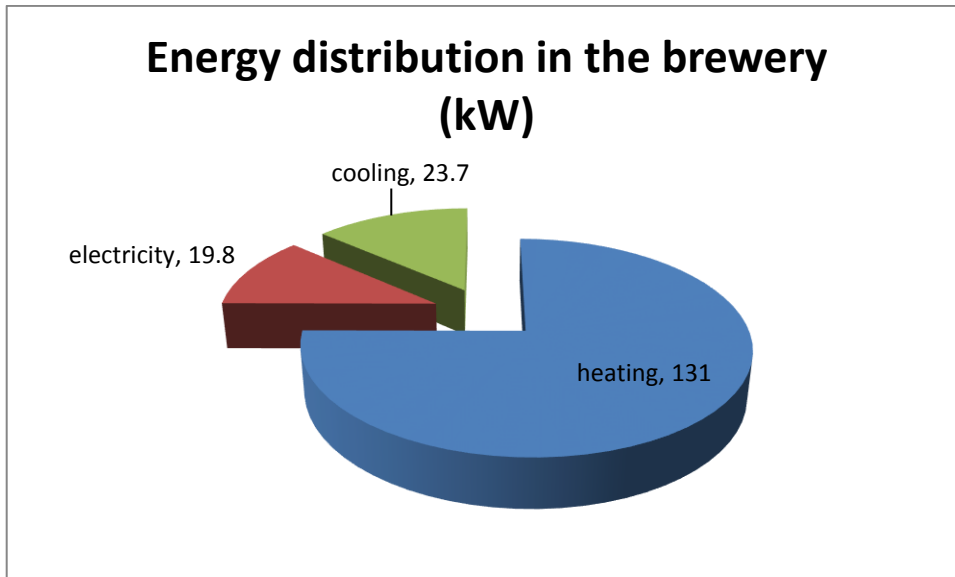


Figure 3-9 Energy distributions in the brewing process.

3.4.4 Bio waste generation

Two stages of bio waste generation were identified. The first one can be found after the mash tun, where approximately 1,500 kg of grains and 150 kg of spent hops are discarded on a daily basis. This amounts to approximately 1,650 kg of spent by-products per day. However, the results from the experimental characterisation which will be presented in the chapter 4, show that there are approximately 350 kg of volatile solids (VS) for the grains and 25 kg of VS which are actually biodegradable. The second stage where biowaste is disposed is after clarification when the yeast is discarded. Literature suggests that a maximum of 4-8 m³ of biogas can be produced for every m³ of digester that is available [82]. As the calorific value of biogas (20 MJ/m³) is lower than the heavy oil (38 MJ/m³), in order to cover the 131 kW which corresponds to 480 MJ/h and considering that the boiler is running for 8 hours per day and 5 days per week at full capacity, 192 m³ of gas would be required per day, which represents 50,000 m³ per year. If the daily biogas requirement can be assumed as stated above then the necessary digester size can be determined. This figure considers replacing the total consumption of fuel oil. The experimental work will determine the maximum

biodegradability of the substrates in order to find out how much of the current utilization can be replaced.

3.4 Summary

This chapter was used to describe the study cases. Both of them belong to the drinks industry. An energy audit was carried out in both plants and relevant measurements and information of usage was collected for future study. It was found that the main energy usage is on the thermal side, but with low temperatures compared with other industrial sectors. During the process bio waste is generated, consisting mainly in wet spent grains, throughout the visits it was possible to collect samples of these wastes at each stage for further analysis at the University. At the moment neither case is re using them. In terms of waste heat it was noticed that there are opportunities in both plants, being the boiler and the cooking step the ones with more waste heat generation. In the future chapters the use of these wastes and the integration of the waste heat will be explored.

Chapter 4. Experimental set up

4.1 Introduction

This chapter describes the experimental set up and methods that were used to characterise the samples collected in the energy audits. The biogas production potential of each of the substrates was determined under two different conditions. Analytical methods were used and two experiments were run in order to have a feasibility evaluation for the use of the wastes to produce bio fuel in order to replace the fossil fuels currently used. One of the experiments was the bio methane potential determination which was done in a batch set up. The other experiment that was run was a semi continuous one, using Continuous Stirred Tank Reactors.

4.2 Samples collection and characterization

The sample collection was done in site during the energy audit and in follow up visits for the brewery. The samples were taken from the place they are stored just before they got discarded. Then taken to the University and stored in the cold room at 5 °C. Once the samples were stored the characterisation of the substrate was carried out at the University facilities. Physical and chemical properties were determined in the Environmental Laboratory of Civil Engineering. The characterisation included Calorific Value (CV), elemental analysis, Total Solids (TS) and Volatile Solids (VS), Total Kjeldahl Nitrogen(TKN) and Total organic carbon (TOC).

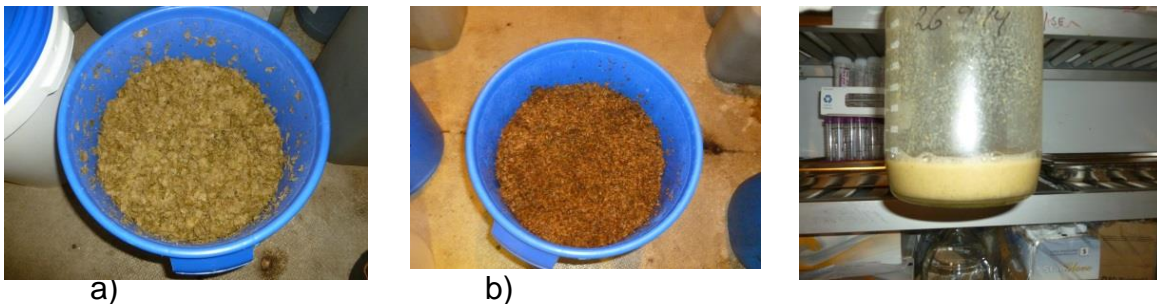


Figure 4-1 Wastes from the brewery . a) Hops, b) spent grains, c) yeast

The figure 4.1 shows the different samples collected from the brewery, as mentioned before each substrate is discarded at a different stage of the process, these samples were taken directly from the plant.

4.2.1 Elemental analysis (CHN)

Prior to this analysis the samples were dried and ground. The wastes were dried at 60 °C during 48 hours in an incubator and then grinded using mortar and pestle. Then the elemental analysis was performed by Chemical Analysis Service of School of Chemistry in Newcastle University using Carlo Erba 1108 Elemental Analyser (Carlo-Erba, Italy). The principle of Dumas method is applied which involved the complete and instantaneous oxidation of the sample by flash combustion[83]. The products of combustion are separated by chromatographic column and detected by thermal conductivity detector.

4.2.2 Calorific value determination

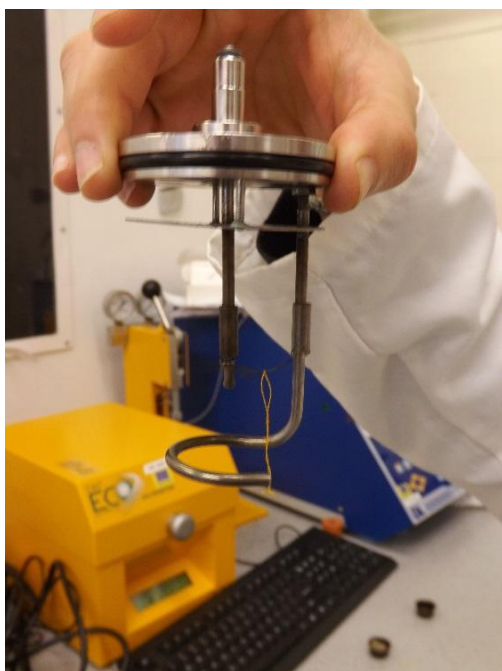
In the same way as the elemental analysis the samples were first dried at 60 °C in the oven. Then they were ground using mortar and pestle and then sieved through a mesh 60. The calorific value was determined in a laboratory in School of Chemical Engineering and Advanced Materials. A CAL2K ECO Bomb Calorimeter, shown in Figure 4.2 was used to obtain the calorific value of the waste from spirit plant. The bomb calorimeter was used following the instruction provided in the operating manual by the manufacturer and described below.



a)



b)



c)

Figure 4-2 a) CAL2K ECO Bomb Calorimeter, b)Filling station, c)Vessel cap with firing cotton

First the pre-cut length of Firing Cotton (CAL2K-4-FC) is looped over the firing wire and the ends twisted together. Then the sample is weighed using a laboratory microbalance (Metler Toledo, United Kingdom). In these experiments, quantities around 0.5 grams of sample were used for each analysis.

Secondly, the weighted crucible and sample are inserted into the outside electrode's crucible holder, ensuring that the firing cotton touches the sample. Then the electrode assembly is introduced into the vessel body and the cap is screwed down. Once this is done, the vessel is placed under the Filling Station and 3000 kPa oxygen is used to fill the vessel then it can be removed from the Filling Station and introduced into the Calorimeter. At this moment the mass of the sample previously weighted is introduced in the screen. The equipment then proceeds with the following steps: INITIAL where a temperature stabilisation phase will be carried out, the duration of this phase is set for the equipment to be 10 minutes. FIRING, when the pre-set initial conditions have been met, the pre-set firing voltage is applied to the firing wire. FINAL, this is initiated immediately after a successful firing phase. During this phase, a CV figure is calculated every 6 seconds taking into account the calibration curve, CV corrections and sample mass. DONE, on successful completion of the FINAL phase, a final CV result is displayed and stored.

4.2.3 Total Solids and Volatile Solids

The samples of the substrates were first characterised by evaluating the total solid (TS) and volatile solids % (VS). This was undertaken in triplicate according to the APHA standard method [84]. Figure 4-3 shows the test equipment which was an oven (Morgan and Grundy Ltd, United Kingdom) set to 104 °C. An empty crucible is weighted in a microbalance (Metler Toledo, United Kingdom) and the reading is recorded. Approximately 5 g of each solid sample is weighted and for the liquid samples 20 ml are poured into each crucible. The mass of sample and the crucible is recorded. Crucible is placed into heater oven at 104°C for 24 hours.



Figure 4-3 Oven used for Total Solids determination.

This step removes any water in the sample so there is only solid remain in the crucible. The crucible with dried sample is weighted using the same weighting scale and its mass is recorded. The total solids are then calculated by relation of the dried crucible between the one with the sample and expressed as a percentage. The equation 4.1 was used for the determination.

$$\% \text{Total solids} = \frac{W_{\text{total}} - W_{\text{crucible}}}{W_{\text{sample}} - W_{\text{crucible}}} \quad (4.1)$$

Where: W_{total} = weight of dried residue and crucible (mg), W_{sample} = weight of wet sample and crucible (mg) and W_{crucible} = weight of empty crucible (mg)

Then the crucible with dried solid is ignited at 550 °C for 15 minutes and then allowed to cool. Once it is cooled it is weighted again with the balance and the results are recorded. Finally the crucible is placed into oven at 550 °C for further 5 minutes to remove any

volatile residues and weighed again, this could be done two or three times or until both weights do not differ in more than 0.05%.



Figure 4-4 Furnace for volatile solids determination.

Once the weights are recorded the calculation can be completed for the VS using the equation 4.2:

$$\% \text{Volatile solids} = \frac{W_{\text{total}} - W_{\text{volatile}}}{W_{\text{total}} - W_{\text{crucible}}} \quad (4.2)$$

Where: W_{total} = weight of dried residue and crucible (mg), W_{volatile} = weight of the residue and crucible after ignition (mg) and W_{crucible} = weight of empty crucible (mg)

4.2.4 Total Kjeldahl Nitrogen

Total Kjeldahl Nitrogen (TKN) and Total Ammoniacal Nitrogen (TAN) $\text{NH}_3\text{-N}$ was determined using a Vadopest 30S steam distillation unit (Gerhardt, UK). According to APHA standard [84], the method is to place 10 ml of sample in a digestion tube and add 14 ml of concentrated sulphuric acid, add two Kjeltabs and digest. Once the digestion is completed the tubes need to cool down to room temperature. Then the distillation can be

done and finally using N/50 sulphuric acid a titration step has to be carried out. The digestion step is not required for the Ammoniacal Nitrogen determination. After preparation, the sample then only needs to be distilled and titrated with N/50 sulphuric acid. Finally the organic nitrogen can be determined subtracting the ammoniacal nitrogen from the total Kjeldahl nitrogen.

Total organic carbon (TOC) was analysed using a LECO CS244 carbon analyser (LECO Ltd., UK). All samples were undertaken in triplicate and calibrated against a known commercial standard (Calcium carbonate, 99% purity). A blank consisting of distilled water was used as well.

A semi-continuous solvent extraction known as Soxhlet method was used for the lipids content determination. The samples were first dried and then ground into small particles and placed in a porous thimble. The thimble was placed in an extraction chamber, which is suspended above a flask containing the solvent and below a condenser. The flask is heated and the solvent evaporates and moves up into the condenser where it is converted into a liquid that trickles into the extraction chamber containing the sample. Eventually, the solvent builds up in the extraction chamber and completely surrounds the sample. The extraction chamber is designed so that when the solvent surrounding the sample exceeds a certain level it overflows and trickles back down into the boiling flask. As the solvent passes through the sample it extracts the lipids and carries them into the flask. The lipids then remain in the flask because of their low volatility. At the end of the extraction process, which normally lasts a few hours, the flask containing the solvent and lipid is removed, the solvent is evaporated and the mass of lipid remaining is measured. The percentage of lipid in the initial sample can then be calculated.

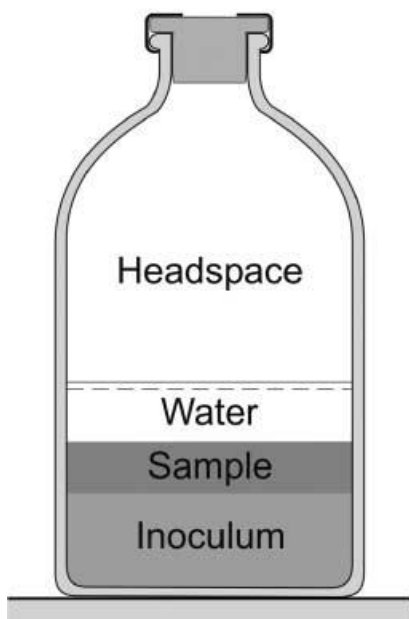
4.3 Test plan and procedure

The test plan procedure was defined to run a batch experiment to determine the bio methane potential (BMP) of the different substrates. A semi continuous experiment was also run using CSTR's to evaluate the feasibility of the substrates in a continuous process.

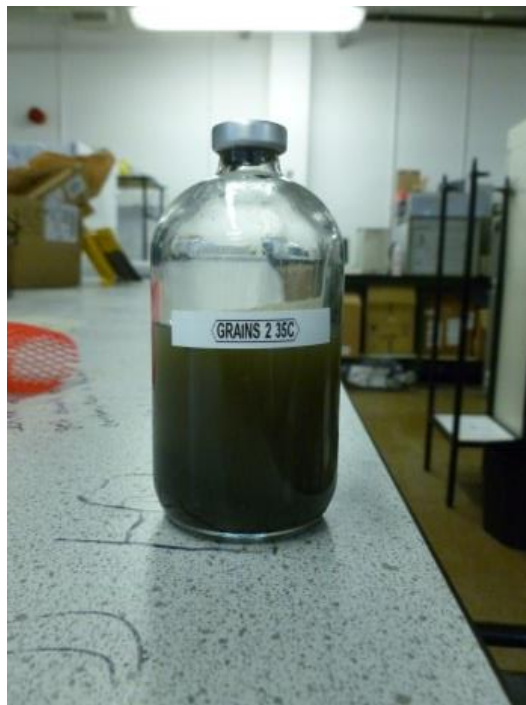
4.4.1 Batch Test

These assays were used to establish the anaerobic biodegradability for determination of the ultimate methane potential (BMP) of organic wastes [85]. But they are also used for determination of the rate of the biodegradation in general. In this study the guidelines from the [86] and [85] were followed to perform the evaluation of potential anaerobic biodegradability of organic waste under specific conditions. The conditions evaluated were, mesophilic and thermophilic at 35 °C and 55 °C respectively.

The BMP assay should be designed to provide ideal anaerobic conditions and prevent any form of inhibition. In order to accomplish this, appropriate microbial community, enzyme pool, and nutrients should be present and the substrate and intermediate product concentrations should be kept below inhibitory/toxic levels. The duration of the test was of 16 days. Digested inoculum, containing low (<10 mg/L) concentrations of inorganic carbon (IC), was incubated at the temperatures mentioned above, in sealed 100 ml vessels with the test substance at a ratio of 1 to 3 as recommended by Angelidaki et al. [85] 0.2g/V_S of substrate and 0.6 g/V_S of inoculum. In order to measure the activity of the sludge one parallel blank control with sludge inoculum in the medium and just water was also prepared. Figure 4-5 shows batch sample preparation of spent brewery grains.



a)



b)

Figure 4-5 a) Batch example by Angelidaki b) batch preparation of spent brewery grains.

4.4.1.1 Inoculum

The sludge used for the mesophilic tests was a digested sludge consisting of 50% crushed granular sludge from a UASB at mesophilic temperatures (38°C). It was fed on

papermill wastewater and combined with 50% of mesophilic sludge from Laboratory CSTR systems fed on cellulose rich autoclaved municipal solid waste. Moreover, for the thermophilic bottles 100% thermophilic sludge from laboratory CSTR systems was used. The sludge was fed on cellulose rich autoclaved municipal solid waste operated at 50 °C.

4.4.1.2 Anaerobic media

Anaerobic media consist of several mineral and synthetic medium should be added to assure that the necessary nutrients and micronutrients which are required by the microorganisms for optimal function are present. Anaerobic medium described by Angelidaki [85] was prepared in the laboratory with added nutrients, micronutrients and buffer vitamins, as shown in Table 4-1. The chemicals required were available in the laboratory. Analytic scales were used to weigh the materials and all the stock solutions required. Two litres of the media were prepared to have enough for the experiments.

Table 4-1 Anaerobic media used for the bio methane potential

Stock Solution A	
chemical	g/l
NH ₄ Cl	100
NaCl	10
MgCl ₂ 6H ₂ O	10
CaCl ₂ 2H ₂ O	5
Stock solution B	
	g/l
K ₂ HPO ₄ 3H ₂ O	200
Stock solution D	
	g/l
FeCl ₂ 4H ₂ O	2
H ₃ BO ₃ Boric ac.	0.05
ZnCl ₂	0.05
CUCl ₂ 2H ₂ O	0.038
MnCl ₂ 4H ₂ O	0.05
(NH ₄) ₆ Mo ₇ O ₂₄ 4H ₂ O	0.05
AlCl ₃	0.05
CoCl ₂ 6H ₂ O	0.05
NiCl ₂ 6H ₂ O	0.092
ethylenediaminetetraacetate	0.5
conc. HCl	1
Na ₂ SeO ₃ 5H ₂ O	0.1
Stock solution E Vitamin mixture (mg/l)	
	mg
Biotin	2
folic acid	2
pyridoxine acid	10
ridoflavin	5
thiamine hydrochloride	5

cyanocobalamine (B12)	0.1
nicotinic acid	5
P-aminobenzoic acid	5
lipoic acid	5
DL-pantothenic acid	5

g/l

Cysteine Hydrochloride	0.5
NaHCO ₃	2.6
dissolve in 10 ml of dist water	

4.4.1.3 Assay experimental setup

As suggested by [85] the assay was performed in triplicate at each condition used, as shown in Table 4-2, in total 36 vessels of 100 ml were used for the test.

Table 4-2 Conditions and replicates for the tests

Condition/No.		
of replicates	35 °C	55 °C
Spirit waste	3	3
Grains	3	3
Yeast	3	3
Hops	3	3
Cellulose	3	3
Blank 1	3	3

The assay vessels were flushed with nitrogen before transferring the substrate and inoculum by weight. Also nitrogen was flushed in the headspace of the inoculum storage vessel. The use of the medium prepared and described previously and of this gas mixture allows for keeping the pH at neutrality at the beginning of the assays. The inoculum,

substrate and medium were poured into the assay vessels. Then each vessel was closed with a thick butyl rubber stopper and held in place by sealing with an aluminium crimp. An incubator set to 35°C was used to store the vessels prepared for mesophilic conditions. A second incubator for the thermophilic vessels was set at 55°C. In both incubators the bottles were mixed in a swirling motion mixing plate.

The biogas composition was measured every day to monitor the production curve. The sample was taken directly from the bottle using a 100 µl pressure lock gas tight syringe (SGE, Australia) and injecting directly into a Carlo Erba HRGC S160 GC (see Figure 4-6). The GC was equipped with an Agilent HP-PLOTQ column (0.32 mm diameter, 30 m length and 20 µm film, Agilent, UK) connected to a Flame ionisation detector (FID). The carrier gas was hydrogen (250 ml/min) with an oven temperature held isothermally at 35°C. Gas samples were compared to a known standard concentration of gas (Scientific technical gases, UK). The bio methane potential was determined by multiplying the value of the methane content (%) by the headspace volume. This figure indicates the amount of methane produced by mg of VS solid used .



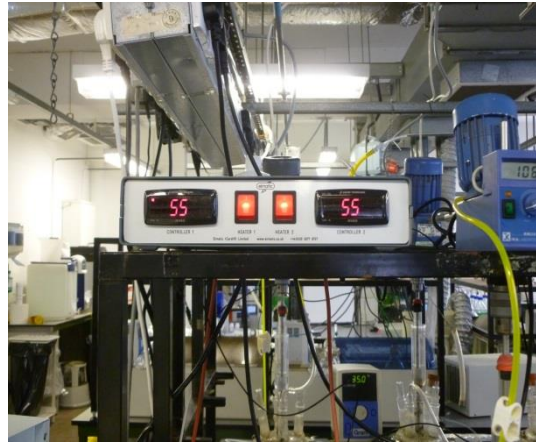
Figure 4-6 Gas chromatograph for methane content determination.

4.4.2 Continuous Stirred Tank Reactors

A total of six 5L CSTR laboratory scale reactors were operated for the semi continuous experiment. These were constructed out of borosilicate quickfit culture vessels (Scilabware, UK). Four of the six reactors at mesophilic conditions were operated at 35 °C, held in a Grant water bath. Daily monitoring was done for the temperature and for the level of the water bath to keep a stable operation. The other two thermophilic reactors were held at 55 (+2 °C) controlled using heating jackets (Elmatic, UK).



a)



b)



c)

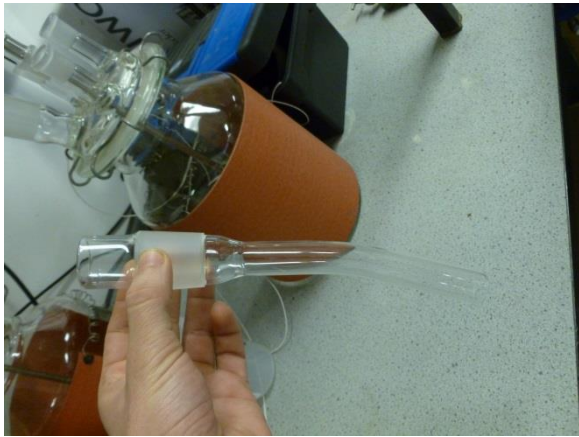
Figure 4-7 Thermophilic Reactors set up a) Insulated heating jackets b) thermostats c) 5 L gas bag

The inoculum used for the mesophilic reactors was digested sludge consisting of 50% crushed granular sludge from a UASB at Mesophilic temperature (38°C). Then fed on papermill wastewater and 50% Mesophilic sludge from Laboratory CSTR systems which were fed on cellulose rich autoclaved municipal solid waste, operated at 35 °C. Furthermore, 100% thermophilic sludge from laboratory CSTR systems fed on cellulose rich autoclaved municipal solid waste operated at 50 °C was used for the thermophilic

reactors. The Organic Loading Rate (OLR) for the experiment was determined to be 5gVS/d based on a previous experiment conducted in similar conditions. A Hydraulic Retention Time (HRT) of 25 days was set for the experiment.

The reactors were operated in daily batch fed mode. A volume of 200 ml of mixed liquor volatile suspended solids (MLVSS) was abstracted and replaced with 200 ml of the daily feed. The feed was prepared on a regular basis every 72 hours. The determination of the amount of the feed was done using the TS and VS values that were taken weekly. In a first attempt the amount required of feed was weighted and poured directly into the reactors and then completed with 200 ml of water. But it was noticed within days that some of the grains were not mixed properly and the decision to blend them prior was taken. The amount feed previously calculated was added into a blender and blended using water in order to reduce the particle size and to make up the 200 ml.

In the first attempt the feeding of the reactors was done by opening them and extracting the sample followed by adding the feed. This operation was tried to be done as quick as possible to avoid a long exposure to oxygen. A modification to the reactors headspace was performed to minimize this exposure. A new sampling device was introduced. This was determined by measuring the level and the length required for the level to be kept at 5 l a glass tube was ordered and made by the University Glass blower.



a)



b)

Figure 4-8 Modification to the sampling port of the reactor.

The operation conditions are shown in Table 4-3.

Table 4-3 Operation conditions for the Continuous Stirred Tank Reactors

Description	Name	Feed	Volume	Temperature	HRT	OLR	Stirrer rpm
Mesophilic Reactor 1	R1	Grains	5 L	35 °C	25 days	5g VS	100
Mesophilic Reactor 2	R2	Hops	5 L	35 °C	25 days	5g VS	100
Thermophilic Reactor 1	TR5	Grains	5 L	55 °C	25 days	5g VS	100
Thermophilic Reactor 2	TR6	Hops	5 L	55 °C	25 days	5g VS	100

4.4.3 Operation and monitoring of the Reactors

The sludge was acclimatised for 15 days at the operation temperature of 35 °C for the mesophilic reactors and 55 °C for the thermophilic reactors. Then the operation began by adding just 1 gVS for the first two weeks. The quantity of feed was then increased from

1 gVS to 3 gVS after the first two weeks. Finally reaching the 5gVS at which the experiment was carried out.

The operation and monitoring of the reactors consisted in daily sampling and feeding at the ORL and HRT mentioned. Daily gas production in all reactors was collected using 5 L gas bags (Supelco, Sigma Aldrich, UK) and measured volumetrically using a 1 liter acrylic syringe (SGE, Australia). This measure was carried out at the same time each day, in order to allow 24 hours between each measurement. The room temperature was taken and recorded every time and the data of the pressure was also recorded. This information was used in further calculations of dry volume, dry methane and standard conditions of temperature and pressure.

Methane content was analysed by taking 100 µl of sample direct from the each gas bag using a pressure lock gas tight syringe (SGE, Australia) and injecting directly into a Carlo Erba HRGC S160 GC. The GC was equipped with an Agilent HP-PLOTQ column (0.32 mm diameter, 30 m length and 20 µm film, Agilent, UK) connected to a Flame ionisation detector (FID). The carrier gas was hydrogen (250 ml/min) with an oven temperature held isothermally at 35 °C. A calibration curve was first obtained for each measurement by using a known standard concentration of gas (Scientific technical gases, UK). This curve was constructed by injecting in triplicates five known volumes, 20, 40, 60, 80 and 100 µl. The area of the curve was recorded and then plotted to obtain the equation that would be later used to compare to the gas samples collected from the reactors.

Reactors pH was measured daily prior to feeding according to APHA standard method 4500-H+B [84]. A Jenway 3010 pH-meter (Jenway, UK) equipped with double junction electrode (VWR, UK) was used. The equipment was calibrated prior to use with commercial certified standards, pH 4 and pH 7 (VWR, UK).

Volatile fatty acids (VFAs) were measured in a weekly basis. First they were prepared by diluting the samples 1:50 V:V with deionized water. Then the samples were spinned in a centrifuge. Formerly the aqueous samples were filtered using 0.22 µm Polyethylene sulphone syringe filter (VWR international, UK). This was then diluted 1:1 V:V with 0.1N Octane sulfonic acid (Thermoscientific, UK). Finally a sonication for 45 minutes was carried out to drive of carbonate (50/60 Hz, Decon Ultrasonics Ltd, UK). The VFAs were then measured in duplicate using liquid Ion Chromatography (Dionex ICS-1000).

Equipped with an Ionpack ICE ASI column, with heptafluorobutyric acid as the eluent and tetrabutylammonium hydroxide as the regenerant). Calibration was undertaken using a range of VFAs prepared to a range of concentrations. Detectable range was >2ppm and to a maximum of 500 ppm without dilution. Total Solids and Volatile Solids were also monitored in a weekly basis following the protocol mentioned in Chapter 3.

Other analyses that were carried out during the operation were the weekly determination of TS and VS of each reactor in order to monitor the biodegradation of the VS added. In a similar way the determination of TSS and VSS was carried out. Also a measurement of Total Alkalinity (TA) was done in a weekly basis for the 2nd and 3rd HRT. This was mainly to cross check with volatile fatty acids concentration and the pH. These analyses were made according to the following table.

Table 4-4 Frequency of analysis for the CSTR's operation

Solid phase	
Analysis Substrates	Frequency
TS	weekly
VS	weekly
TSS	once
VSS	once
elemental analysis (C,N,H)	once
Lipids	once
TAN (mg/l)	once
TKN (mg/l)	once
Calorific Value (kJ/kg K)	once
Carbohydrates	
Lignin	
COD	
Liquid phase	
Analysis Reactors	Frequency
pH	daily
VFA's	weekly
TS	twice a month
VS	twice a month
TSS	twice a month
VSS	twice a month
TAN (mg/l)	once
TKN (mg/l)	once
Gas phase	
Analysis Biogas	Frequency
gas volume production	daily
methane content	three times a week

4.4 Summary

In this chapter the experimental set up was described. A batch run was set up with the different samples in order to determine the bio degradability of the substrates. A semi continuous experiment was also set up, in both of them the focus was to determine the biogas production at mesophilic and thermophilic conditions.

Chapter 5. Experimental results and discussions

5.1 Introduction

In this chapter the results from the experimental set up will be presented. A batch experiment was set under mesophilic conditions of 35 °C and was run for 20 days. In the same manner another experiment at thermophilic temperature of 55°C was operated. A semi-continuous experiment was also prepared and ran for a period of over 100 days. This experiment was performed using the 5L CSTR's described in chapter 4. In both cases the biogas production and composition was thoroughly studied as well as other parameters that affect the operation.

5.2 Results for the samples characterization

Firstly, the results for the characterization of the samples are going to be presented. These results were determined in the University facilities. The values were then used to determine the theoretical biomethane potential as well as the quantities for the experimental work.

5.2.1 Waste from study cases

In order to get reliable results, characterisation of the substrates was carried out. Physical and chemical properties were determined in the laboratory. The characterisation included: Calorific Value (CV) determination, elemental analysis (CHN), Total Solids (TS) and Volatile Solids (VS). The elemental analysis was performed by Chemical Analysis Service of School of Chemistry in Newcastle University using Carlo Erba 1108 Elemental Analyser as described in Chapter 4. The determination was made in duplicate for each substrate. With this value it was possible to get the Carbon/Nitrogen relation (C/N) and compare it with other substrates. In the literature it was seen that the brewery and spirit waste presented a low C/N ratio compared with wastes such as cow manure (13:1), pig

manure (12:1) or food waste (35:1)[87, 88]. The elemental analysis results for the substrates are listed in Table 5-1.

Table 5-1 Elemental analysis results for the substrates

	Nitrogen	Carbon	Hydrogen	C/N
Yeast	6.07	42.05	6.57	6.96
Grains	4.76	45.16	6.67	9.80
Hops	4.66	49.25	6.70	10.58
Spirit	3.65	46.36	8.91	5.77

The determination of the elemental analysis was also useful to perform the theoretical calculations which are based solely in the chemical composition of the substrates.

The Calorific Value obtained for the spirit waste in the laboratory as described in Chapter 4 is shown in the table below. The values obtained are in accordance to the ones presented by [25] and [89] where they reported values between 18 and 20 MJ/kg.

Table 5-2 Calorific Value results for the spirit plant waste

Fuel	Spent Grains	
	mass	CV
test	(g)	(MJ/kg)
1	0.5008	18.33
2	0.5037	18.52
3	0.5021	18.56
average	0.5022	18.47

The samples of the substrates were further characterised by evaluating the total solid (TS) and volatile solids (VS) content. The standards procedures supplied in the laboratory manual and APHA, 2005 [57] were followed. The results are shown in Table 5-3. The substrates studied showed results for TS and VS according to the ones reported in literature such as [90] where the values for VS were in the range of 90 to 95 %.

Table 5-3 Results for Total and Volatile solids of the substrates.

	TS %	VS%
Grains	25.25%	95.40%
Hops	12.24%	96.34%
Yeast	13.02%	90.40%
Spirit	90.98%	8.30%

Further to the initial characterisation, a determination of TS and VS for the substrates was done on a weekly basis following the same technique. This was done in order to monitor any change and make necessary adjustments in the feed preparation. Lipids content for each substrate was also determined in the University as well as the Total Kjeldahl Nitrogen and Total Amomiacal Nitrogen, following the procedure described in Chapter 4.

Table 5-4 Summary of the characterization of the samples

	Grains	Hops	Yeast
TS	25%	16%	9.94%
VS	96%	96%	82.07%
C	45.16	49.25	42.05
N	4.76	4.66	6.07
H	6.67	6.70	6.57
C/N	9.49	10.58	6.93
Lipids	6.60%	6.01%	1.02%
TAN	218.4	257.6	140
TKN	17,920	20,020	8,260

In the Table 5-4 a summary of the characterization for each substrate is presented. From this table it can be noticed that the grains and the hops have the higher volatile solids percentage, which means that more organic matter is available to degrade. It also shows a low Carbon to Nitrogen ratio for the three substrates, compared with other substrates that may affect the digestability, being the yeast the lowest. This also applies for the lipids content where the yeast presents the lowest value compared with the grains and the hops and also for the TAN and TKN. Having a complete characterisation is important to begin the experiments, as with it then is possible to determine the operation parameters such as Organic Loading Rate and Hydraulic Retention Time.

5.2.2 Inoculum characterization

To perform anaerobic digestion, inoculum is required for introduction of microbial activity to the sample. The inoculum for this experiment was supplied from the research team of Newcastle University who works on anaerobic digestion in the environmental school of Civil Engineering. The sludge for the mesophilic experiment at 35 °C was available in the laboratory to use. The sludge at 55 °C for the thermophilic conditions had to be first acclimatized for two weeks using one of the incubators available in the Pilot Lab. The

Total solid content and Volatile solid for inoculum were obtained as method explained in Chapter 4. The results are shown in Table 5-5.

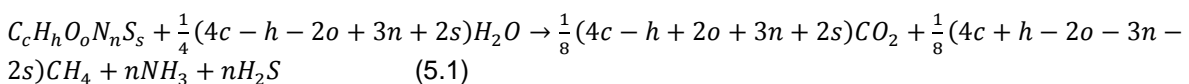
Table 5-5 Total Solids and Volatile solids for inoculum

	TS %	VS%
Inoculum		
35 °C	2.89%	67.68%
Inoculum		
55 °C	1.95%	45.82%

With the values for the TS and VS of the inoculum was possible to work out the best ratio of substrate-inoculum for the experiments. VDI recommends a relation of 3to 1 and in order to get this it was necessary to get the values.

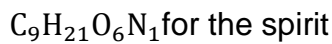
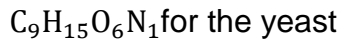
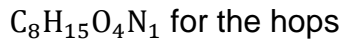
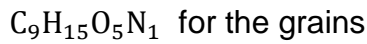
5.3 Theoretical Determination of Biogas and Energy Yield from the Waste

The Buswell equation [91] is commonly used to calculate biogas and energy yield from waste. The calculation is given by the chemical elements percentage composition, empirical formula and atomic weight of the substrate. The equation is presented as:



This value is the ultimate quantity of methane that a certain waste product can produce. The main assumption is that all the matter contained is biodegradable and converted into methane. The compound element composition by weight can be calculated given the elemental analysis. That analysis was performed in the samples where it was possible to determine the Carbon (C), Hydrogen (H), Oxygen (O), Nitrogen (N) content.

Assuming there are 100g of the compound element, and with their atomic weight (C=12, H=1, O=16, N=14, S=32), the empirical formula of the compound was obtained as:



By applying the Buswell equation, the biogas composition is obtained as shown in the following table:

Table 5-6 Theoretical biogas composition calculated from Buswell equation

	CH₄ %	CO₂ %
Grains	49%	43%
Hops	46%	47%
Yeast	46%	43%
spirit	53%	40%

The results from the theoretical biogas composition based on the compositional analysis show the spirit waste has a better potential for bio methane production. The spent grains from the brewery are the second highest according to this theoretical determination. This biogas determination is just an approximation as many other aspects have to be taken in account in order to define if a substrate can be suitable for anaerobic digestion. Due to the fact that it is a simple and fast way of having an idea it could be used as a guide. Studies such as [92] and [93] have used this kind of theoretical calculation finding good accordance when comparing with experimental values.

5.4 Batch results.

The biogas composition was measured on a daily basis for the batch experiment. Before each measurement a calibration curve was obtained using a calibration gas of known

composition. The curve was constructed using three injection points for each known volume. The biogas measurements were then done also in triplicates for each bottle, having 9 points for each condition. Because the experiment was run in closed bottles the volume of the headspace, which was previously determined to be 60 ml was used for the calculations of the volume of methane produced. The figures below show the different curves for each substrate.

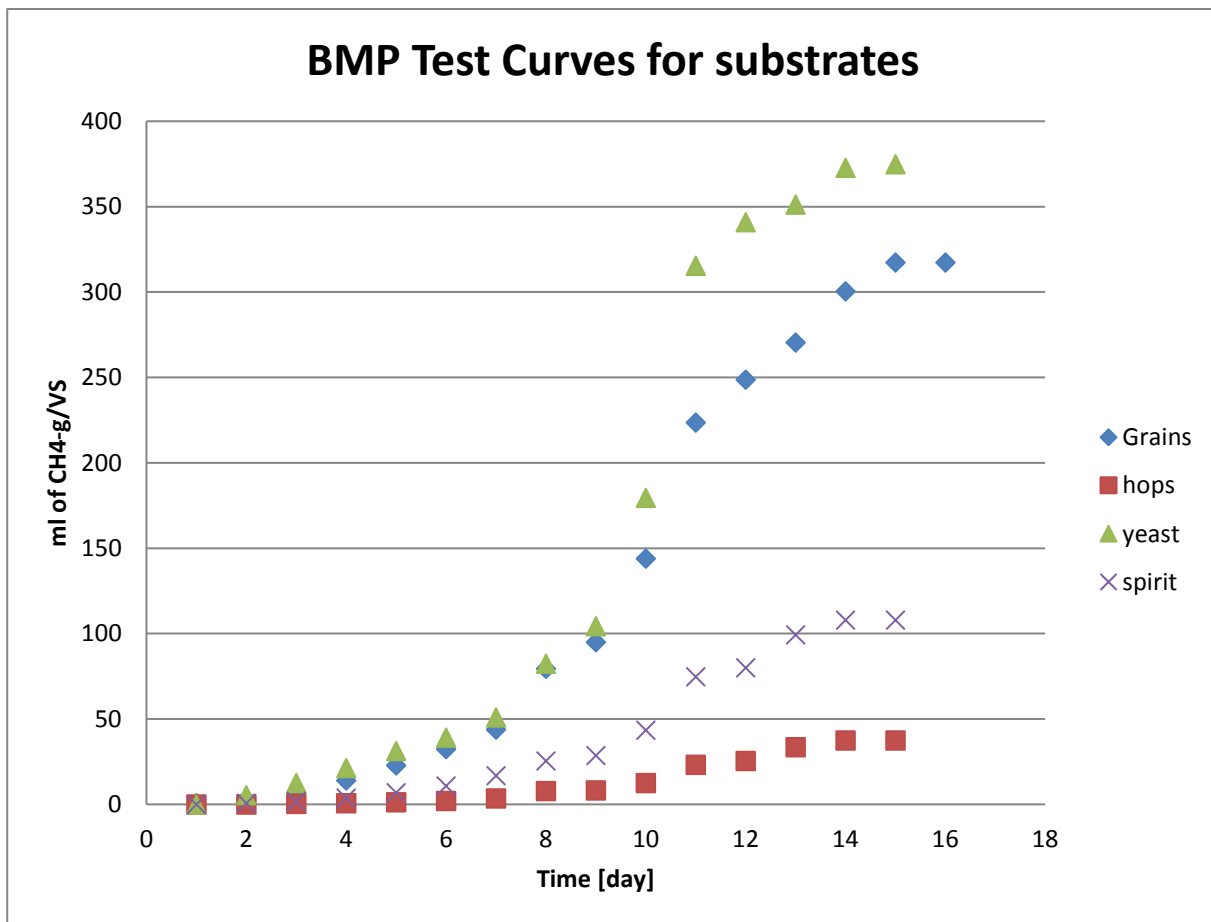


Figure 5-1 BMP test curves

In the BMP curves showed in Figure 5-1 the yeast and the grains and the yeast presented the higher potential to produce biomethane. The spirit waste and the hops showed a lower potential in this determination. It was also noticed that after day 12 the bottles started to stabilize meaning that the feed was consumed by the microbial communities. The main objective of this test was to know if the substrates were able to produce biogas

in anaerobic conditions. This allowed to have a clear idea on how much biomethane could be obtained from each substrate.

Once the cumulative methane curves were obtained it was possible to use the modified Gompertz equation [94]. This equation gives cumulative biogas production from batch digesters assuming that biogas production is a function of bacterial growth. Research has found that this equation gives the best fitting while treating organic wastes and food waste [6, 95, 96]. The modified Gompertz equation is given by

$$M = P * e - \exp \left[\left(R * \frac{e}{P} \right) * (L - x) \right] \quad (5.2)$$

where:

M Cumulative biogas production, l/(g VS) at any time t

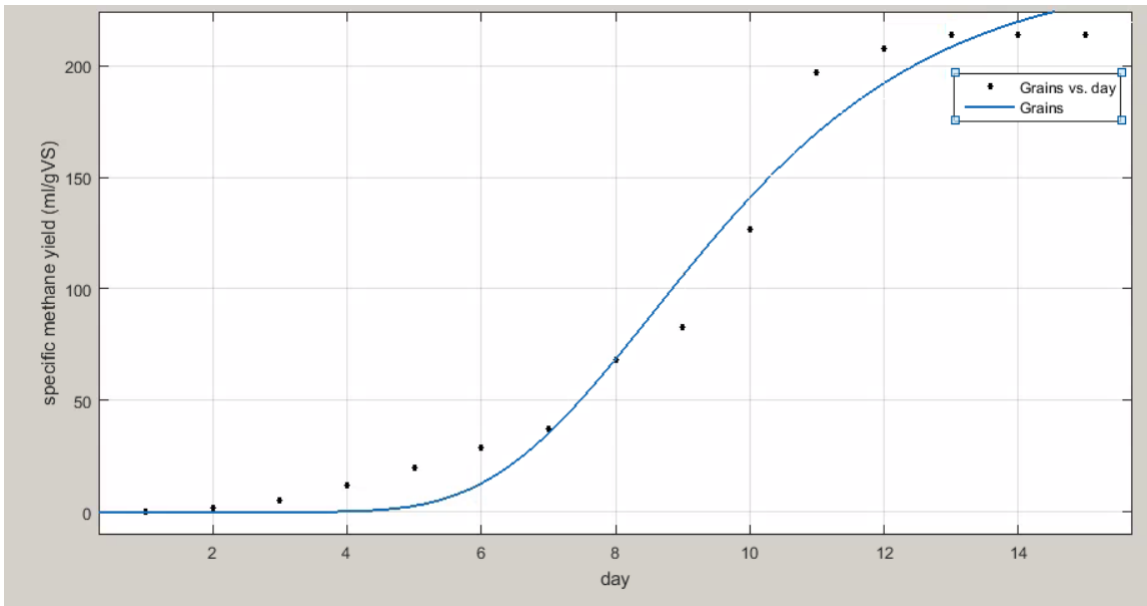
P Biogas yield potential, l/(g VS)

R Maximum biogas production rate, l/(g VS d)

λ Duration of lag phase,

d (days) t Time at which cumulative biogas production M is calculated, d

The gas data was corrected for temperature and pressure variations and reported at standard conditions STP(0°C and 1 atm). Furthermore specific methane potential of the grains with the modified Gompertz curve fitted using matlab were determined and shown below.



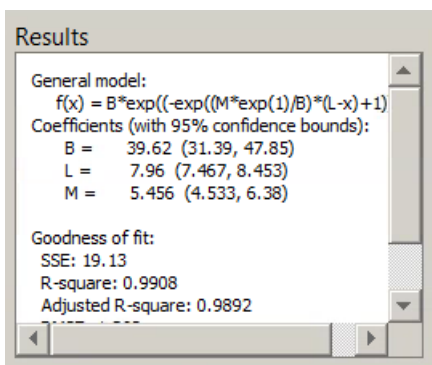
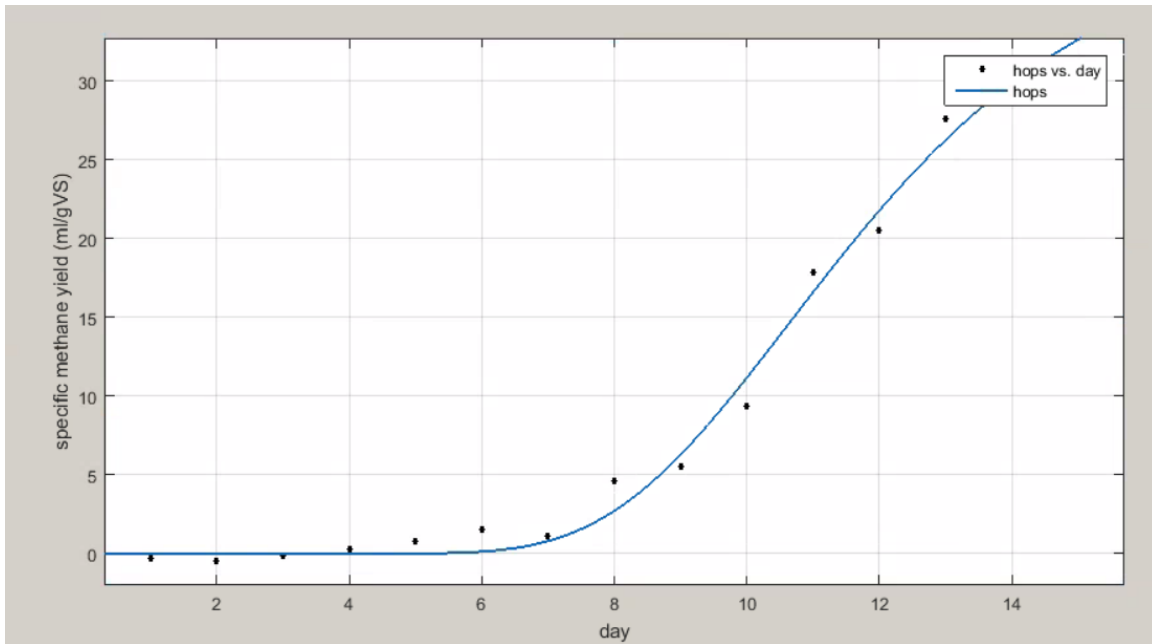
Results

General model:
 $f(x) = B * \exp(-\exp((R * \exp(1)/B) * (L-x) + 1))$

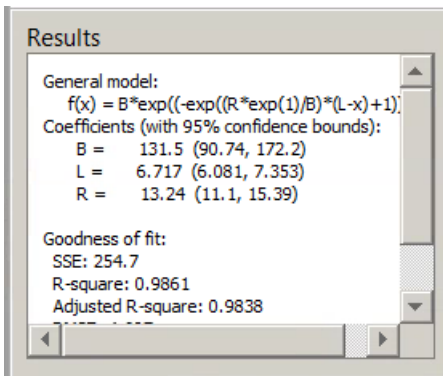
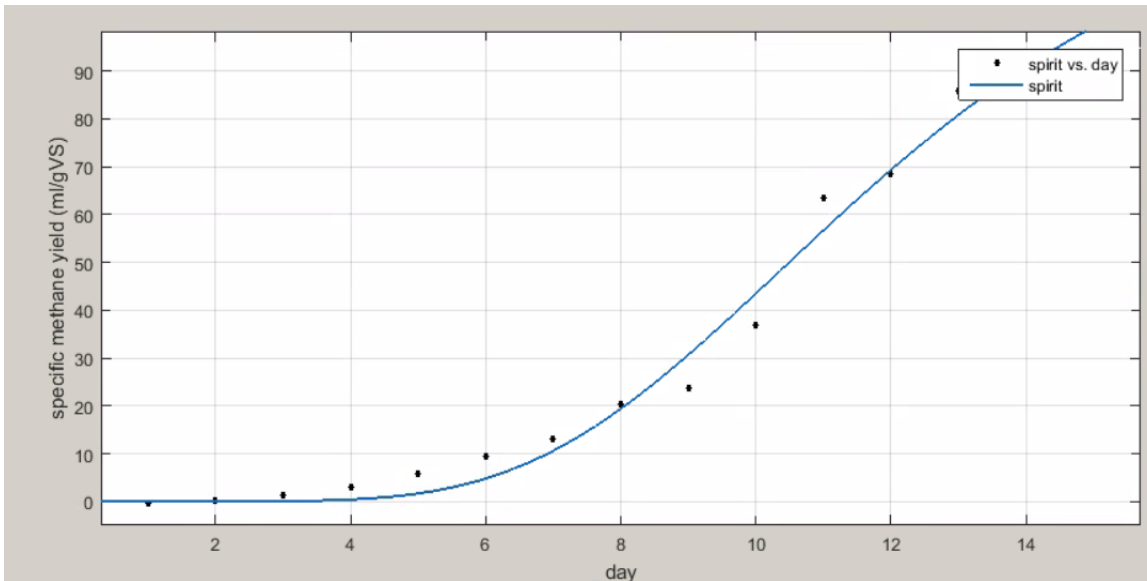
Coefficients (with 95% confidence bounds):
 B = 243.2 (200.8, 285.6)
 L = 6.187 (5.311, 7.063)
 R = 37.65 (26.19, 49.12)

Goodness of fit:
 SSE: 2673
 R-square: 0.9763
 Adjusted R-square: 0.9724
 RMSE: 14.93

(a) Grains



(b)Hops



(c)

Figure 5-2 Specific methane potential of the grains with the modified Gompertz curve fitted using matlab

The same method was used for the other substrates. It can be seen that the most suitable substrates are the grains and the yeast, having both a significantly higher Bio methane potential than the hops.

Table 5-7 BMP for the different substrates

	B ₀ (ml of CH ₄ /gVS)
Grains	331.56
Hops	39.62
Yeast	312.16
Spirit	131.5

5.5 Semi continuous experiment results

In addition to the batch tests a semi continuous experiment was also completed as described in Chapter 4. This was run in order to determine the extent of biodegradability and bio methane potential of the substrates in an operation closer to a real anaerobic digestion plant. The gas volume production was collected daily in gas bags and measured using a large syringe (SGE, Australia). The experiment was run for a period of four full 25 days HRT's (100 days). The gas data was corrected for temperature and pressure variations and reported at standard conditions STP(0°C and 1 atm). The data presented corresponds to the 3rd HRT where the system reached steady state.

Gas volume

As it can be seen in Figure 5.3 after the 2nd HRT the daily gas production of the reactors becomes steady. It can be noticed that the grains at mesophilic and thermophilic conditions produced more gas in a more consistently way than the hops. The reactor with the hops at thermophilic condition presented a failure. This was noticed within the first HRT by recording a very low bio gas production and other parameters such as pH and volatile solids. This reactor was reseeded and started again. Also another experiment was run in two mesophilic reactors. One being fed with a mixture of 90% grains and 10% hops, which is a very similar mixture of the actual waste produced by the brewery. Finally, another reactor was fed with the yeast waste.

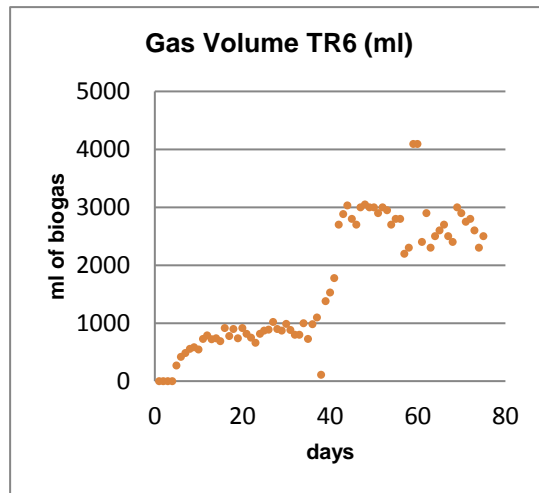
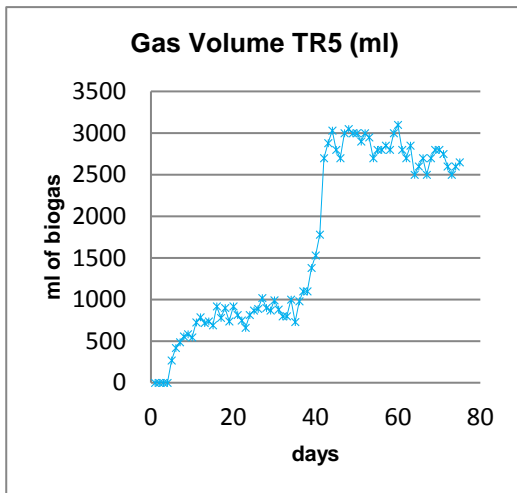
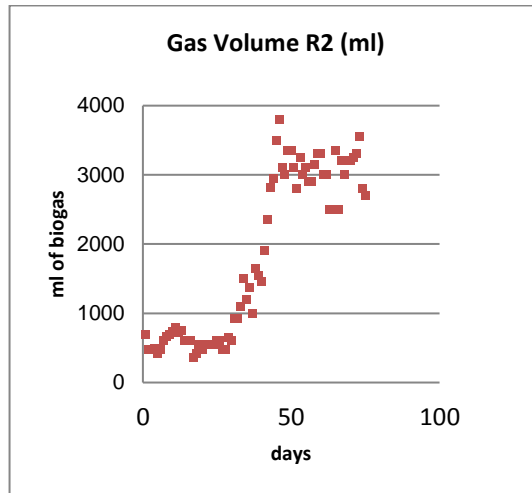
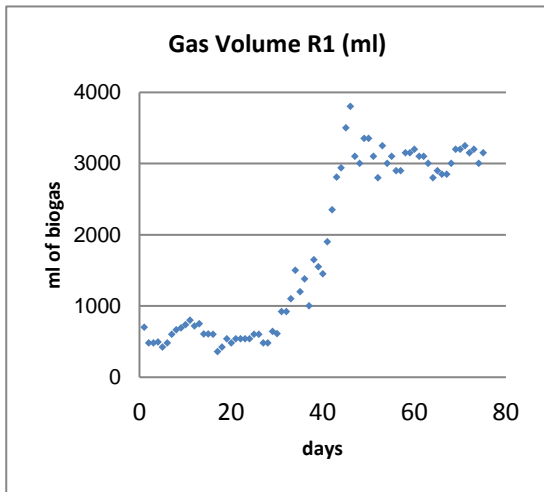


Figure 5-3 Biogas production for each reactor during the experiment.

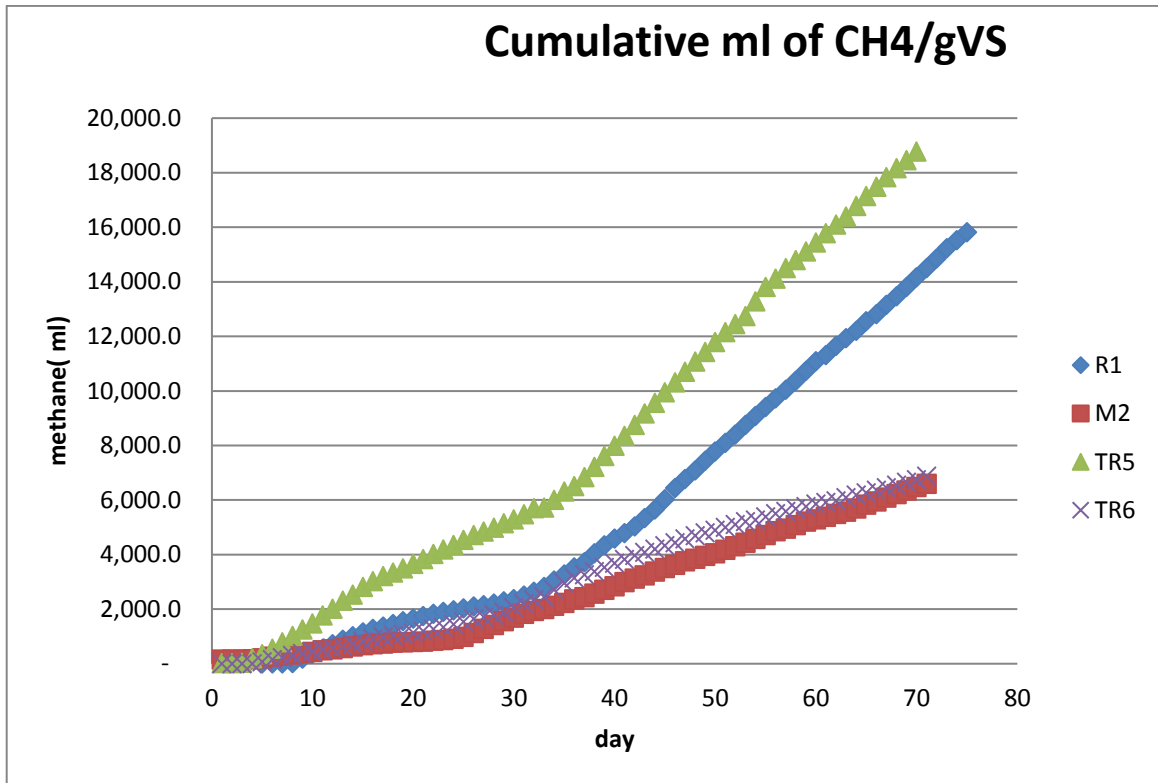


Figure 5-4 Cumulative methane production for the reactors.

In Figure 5.4, it can be noticed that the overall cumulative methane yields during the length of the experiment. The thermophilic reactor fed with grains presented the highest yield, followed by also a reactor working with grains as feed but operating at mesophilic conditions. On the other hand, there is no significant difference for the reactors working with hops. Neither at mesophilic nor thermophilic temperatures and both of them show a lower methane yield.

pH of the reactors

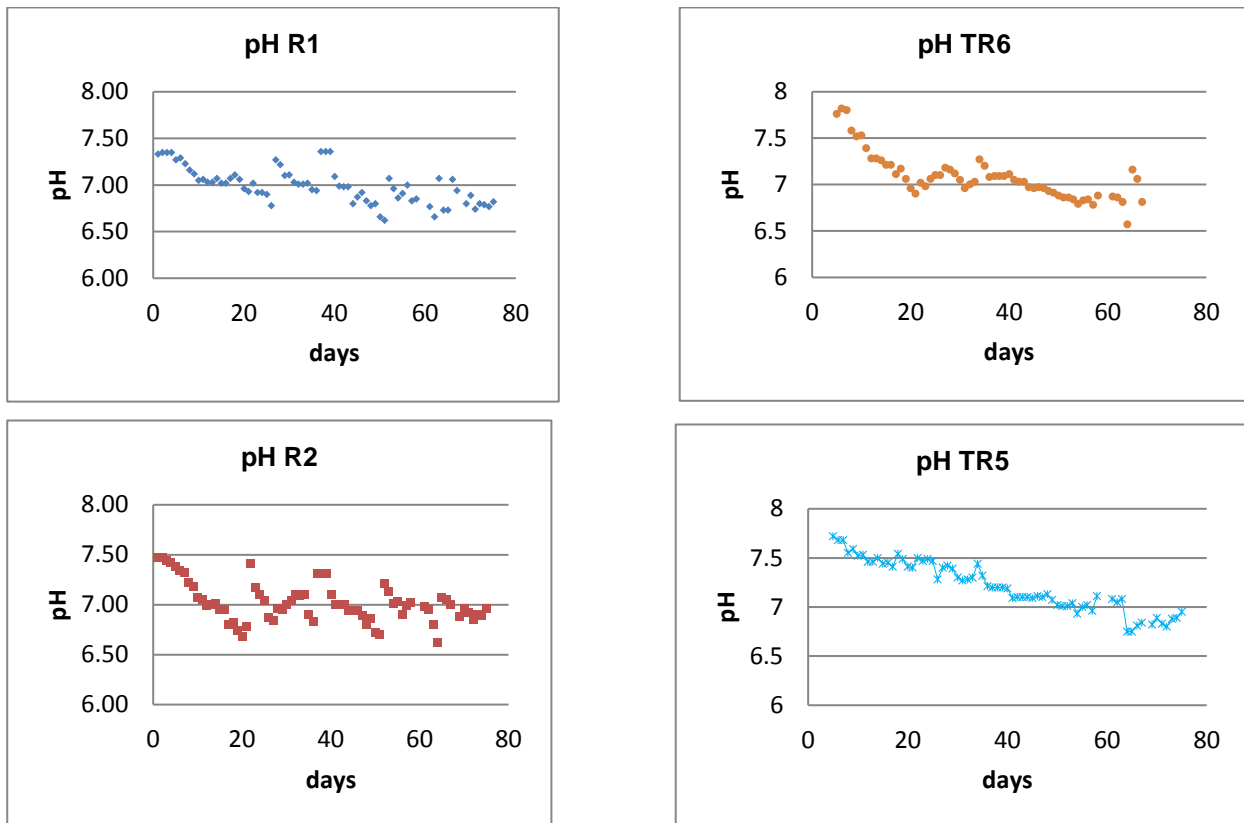


Figure 5-5 pH of the reactors during the experiment.

The pH was closely monitored as it is an indicative of the good operation of the reactors [97]. In this case due to the nature of the substrates the operation of the reactors was run at lower pH's than for other substrates, such as cattle or pig slurry. As seen in Figure 5.5, it can be noticed that for the four reactors the slope is negative. In some cases like reactor R2 it is very pronounced during the first 20 days. In order to keep the pH above acceptable levels (6.5) 2 g of sodium bicarbonate were added. This can be observed in the peaks for reactors R1 and R2. Out of the four reactors, TR5 did not need the pH to be adjusted. Nevertheless it can be seen that it decreased every HRT but it was always above 6.6 this was also noticed in TR6 even though its operation was not as stable.

Total VFA's and detailed VFA's

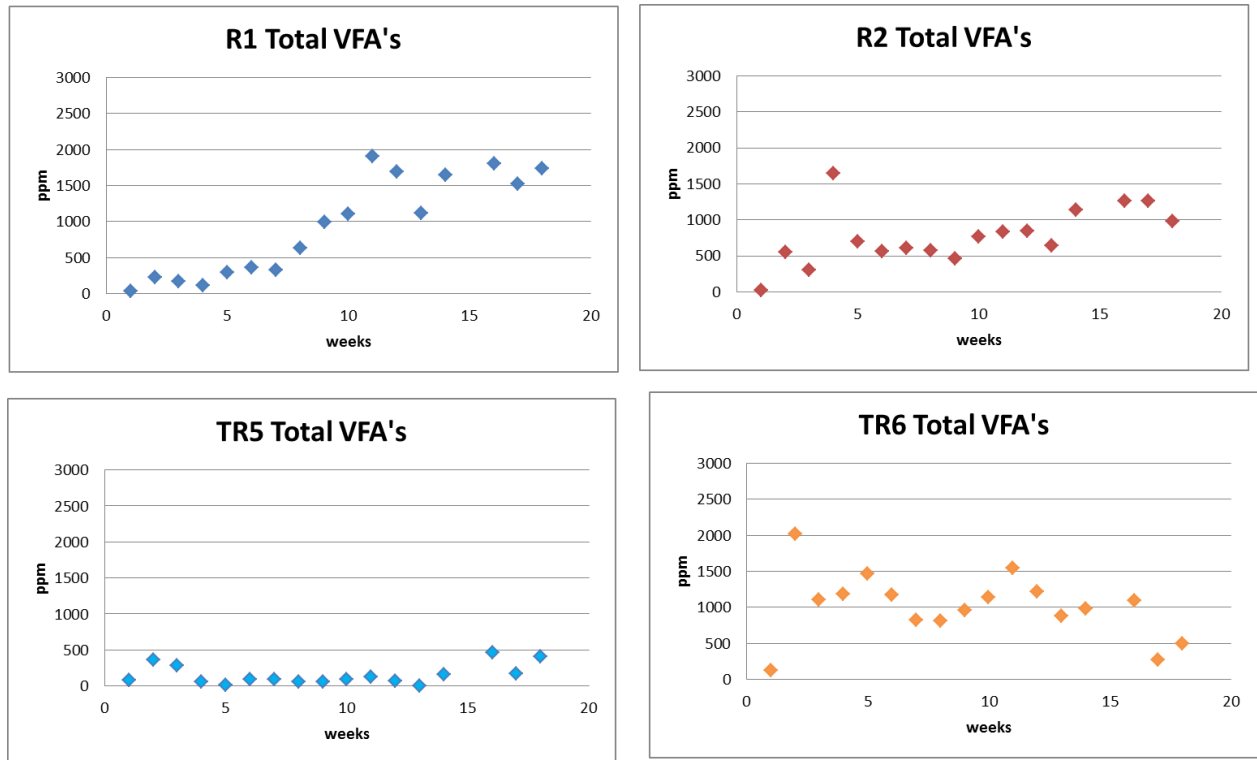


Figure 5-6 Total Volatile Fatty Acids accumulation

The total VFA's were monitored weekly and the profile is shown in Figure 5-6. It can be noticed that inverse to the pH for R1 an accumulation of VFA's is present. A positive slope is also appreciated for R2 but is less pronounced and presents a lower average concentration. TR5 presents the lowest concentration of VFA's which can be related to a better operation. While TR6 has some accumulation the concentration of VFA's is closer to the mesophilic reactors. The influence of the VFA's in the performance of the reactors has been studied and it is known that an accumulation of them will represent problems for the operation as pointed by [98].

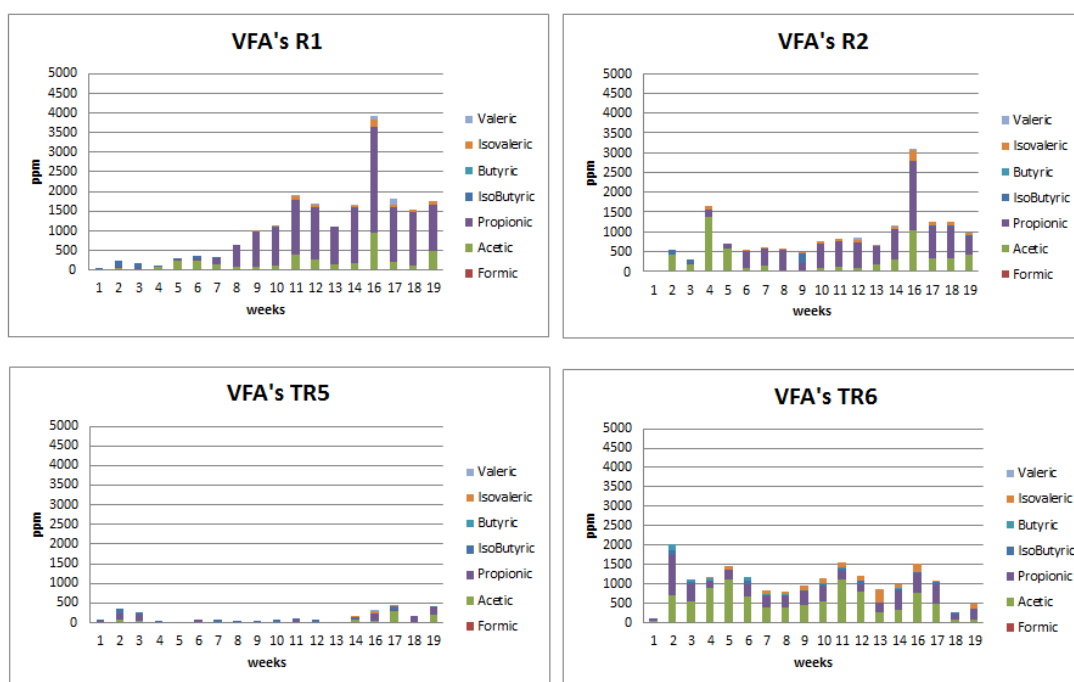


Figure 5-7 Volatile Fatty Acids profile

The VFA's results were then studied in more detail. This was done to see the profile and composition of them in each reactor, as shown in Figure 5.7. It was noticed that for R1, the main volatile fatty acid present was the propionic accounting for a 80% of the Total VFA's. A similar composition was observed for R2 after week 5 when the propionic acid also accounted for the largest proportion of TVFA's with an average of 70%. Meanwhile in TR6 the acetic acid was present in a higher proportion. Little occurrence of large chain VFAs, butyric, iso-butiric, Valeric and isovaleric where present in any of the reactors. The composition for TTR5 was mainly of acetic acid and propionic acid in a 1:1.6 ratio.

Total Alkalinity was also measured from week four, and it is shown in Table 5.7. It was noticed that the changes in the VFA's were reflecting very fast in the pH. Because of that, this measurement was done to know the buffer capacity of the reactors. This was done following the standard method and the values are reported in mg/l of calcium carbonate.

Table 5-8 Total Alkalinity

week	Total Alkalinity (mg/l of calcium carbonate)			
	R1	R2	TR5	TR6
4	8,000.00	7,683.00	1,352.00	24,500.00
5	5,586.67	5,290.00	7,546.10	12,119.10
6	7,585.00	7,045.00	5,125.00	9,505.00
7	1,350.00	5,085.00	2,785.00	5,645.00
8	5,280.00	5,085.00	2,405.00	4,745.00
9	2,480.00	2,340.00	1,040.00	1,400.00

Moreover, a two-point titration was then performed in a further analysis in order to find the partial alkalinity. Then, the difference between Total Alkalinity (TA) and Partial Alkalinity (PA) is the so called Intermediate Alkalinity (IA) and is related to VFA presence [99]. A titration to a pH of 5.7 gives the Partial Alkalinity this volume was recorded and then the titration continued until the 4.3 pH point for the Total Alkalinity.

$$\alpha = \frac{IA}{PA} < 0.3 \quad (5.3)$$

It is suggested by [100] that the ratio between the intermediate and the partial alkalinity to be < 0.3.

Table 5-9 Ratio of Intermedia Alkalinity and Partial Alkalinity IA/PA

	IA/PA		
R1	1.43	1.14	1.63
R2	1.09	1.12	1.52
TR5	0.86	0.71	0.63
TR6	1.50	0.61	0.87

From Table 5-9, it can be seen that the four reactors are operating at higher ratios than the recommended. The TR5 reactor showed the lowest ratio, even though still double than the recommended.

Total Solids and Volatile Solids

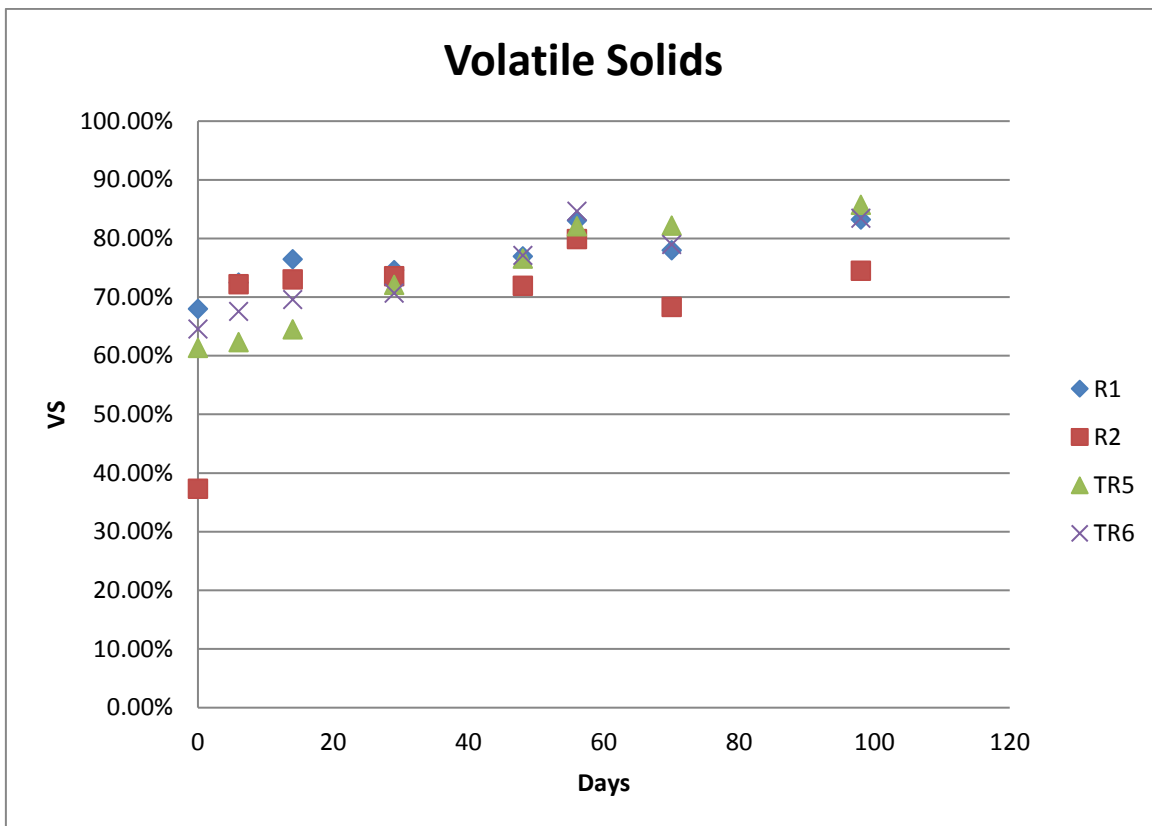


Figure 5-8 Weekly Volatile Solids accumulation.

Figure 5.8 shows the volatile solids through the experiment. It can be noticed that the four reactors reached a stable operation. This means that the Organic loading rate was within the limits. As mentioned before, due to availability of equipment two more 5 litres reactors were run in addition to the previous four. This run was not made in parallel, as the operation of these reactors started 30 days after the first four.

The objective for running these two extra reactors was to study a mixture of the wastes in a similar way as it is produced by the brewery: 90 % of grains and 10 % of hops. A second reactor was fed with yeast since it is another waste stream produced by the process. The characterisation and set up of these reactors was identical as the previous four. The determination of the TS and VS was done in order to determine the amount to be feed. The organic loading rate was also kept at 5gVS with a hydraulic retention time of 25 days. Because of time restriction it was not possible to run these reactors as long as the previous four. Nevertheless it was possible to reach the third HRT and analyse the results.

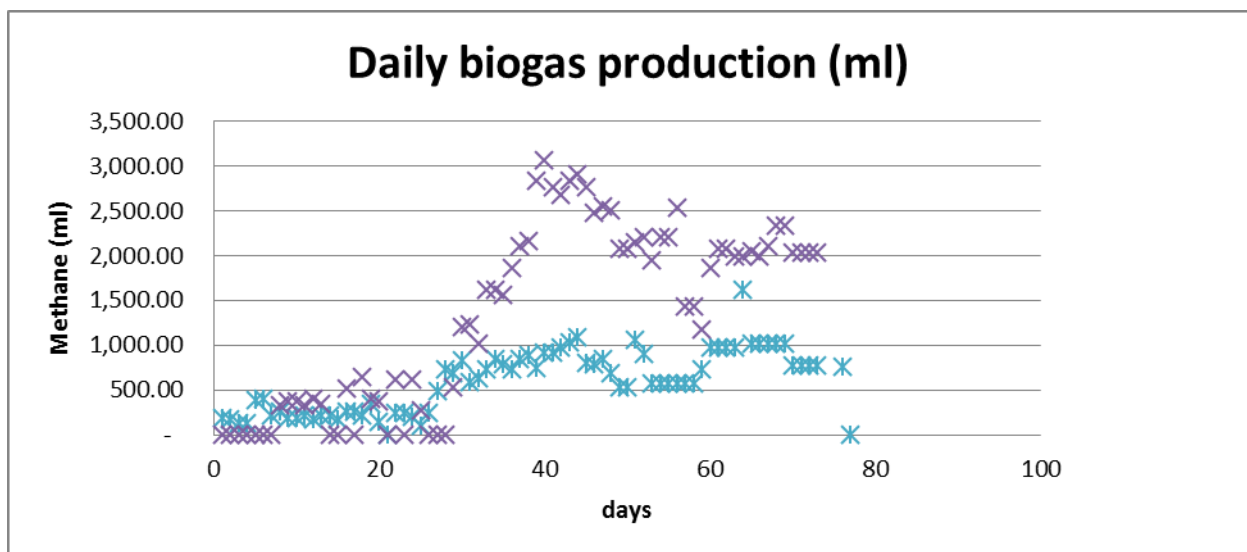


Figure 5-9 Daily biogas production for reactors R3 and R4.

In the figure 5-9 it can be noticed that the reactor 4 operating with yeast as feed had a higher biogas production. It can also be seen that after the first HRT it presented an increase in the production which was later stable. The reactor fed with the mix of grains and hops presented a slower start up. But it also reached a steady state operation by the third HRT, although producing less biogas.

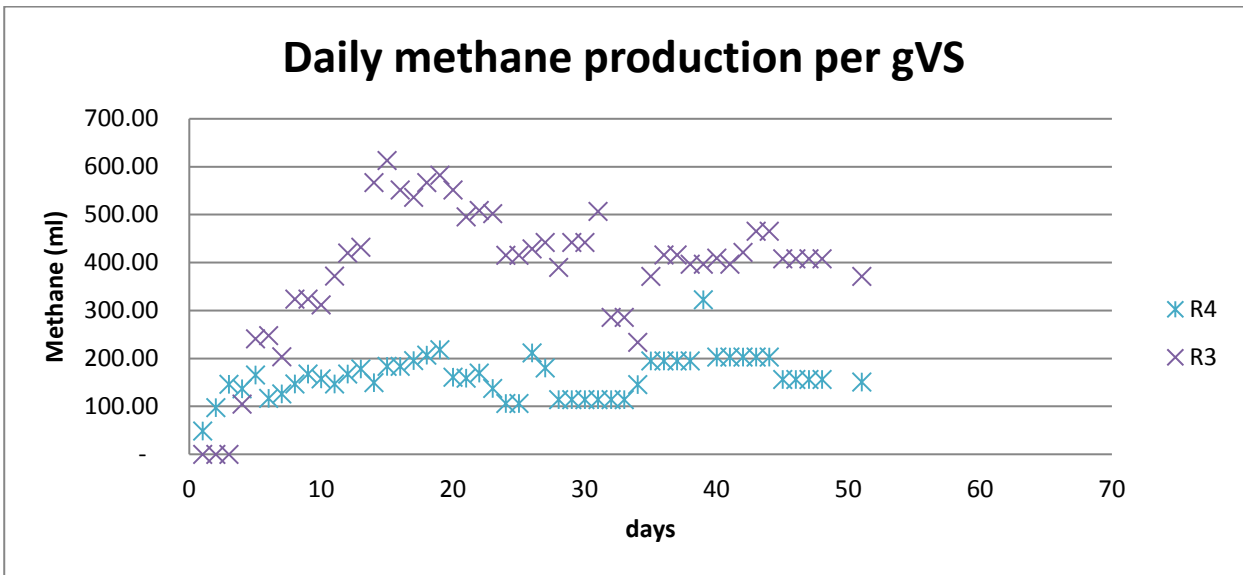


Figure 5-10 Daily volume of methane production for the reactors.

Figure 5-10 shows the daily methane production through the experiment. The methane production showed a similar behaviour than the biogas generation. It was observed that Reactor 4 was the one presenting consistently higher methane content, due to a higher concentration of it in the biogas and a higher production of biogas.

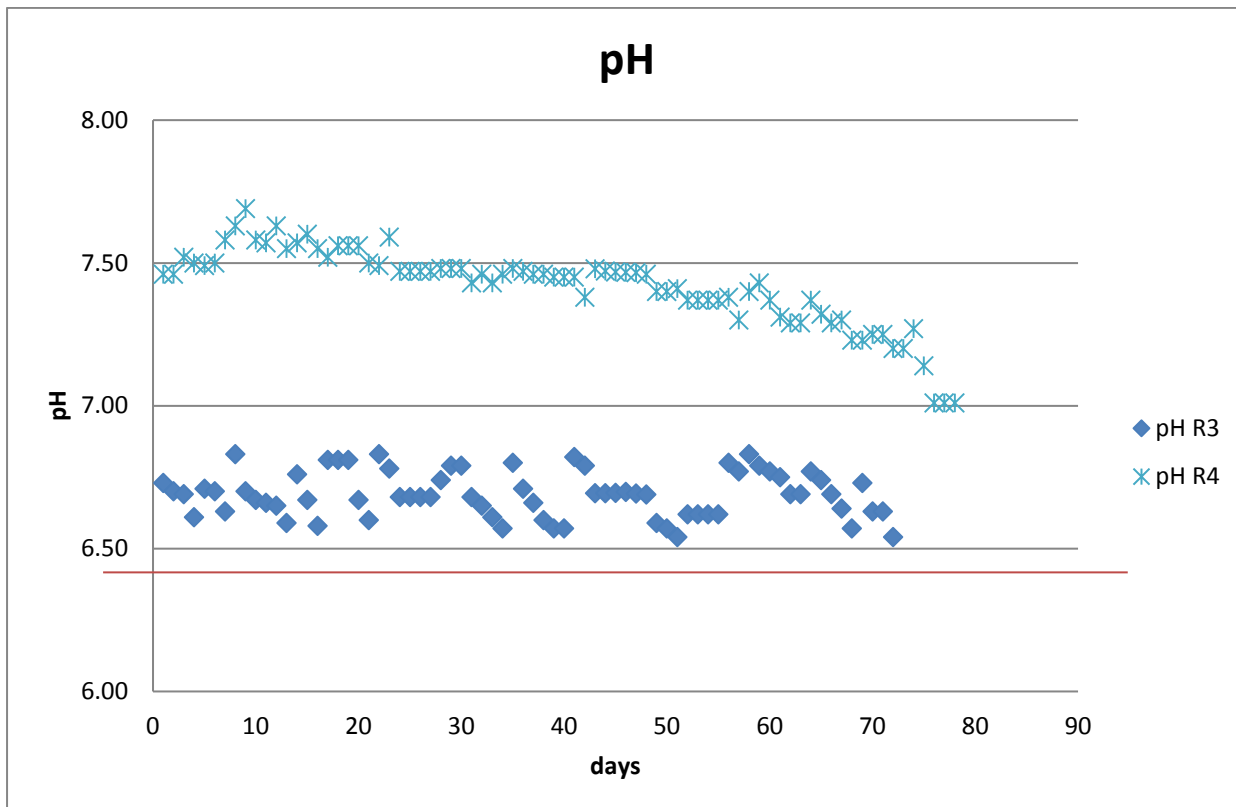


Figure 5-11 Reactors 1 and 4 ph

The pH was measured in a daily basis for these two reactors as well. The pH for reactor 3 (R3) was dropping as it can be observed in the figure 5-11. Because of that it was necessary to adjust the pH with sodium carbonate as done with the previous reactors. Overall it operated closer to the lower limit for most part of the run. Reactor 4 did not require pH adjust but over the time it presented a decrease in the pH. But still it operated at higher values than all the other reactors, including R1, R2, TR5 and TR6. This could be because of its composition and the fact that it presented a lower VFA's content and accumulation during the process.

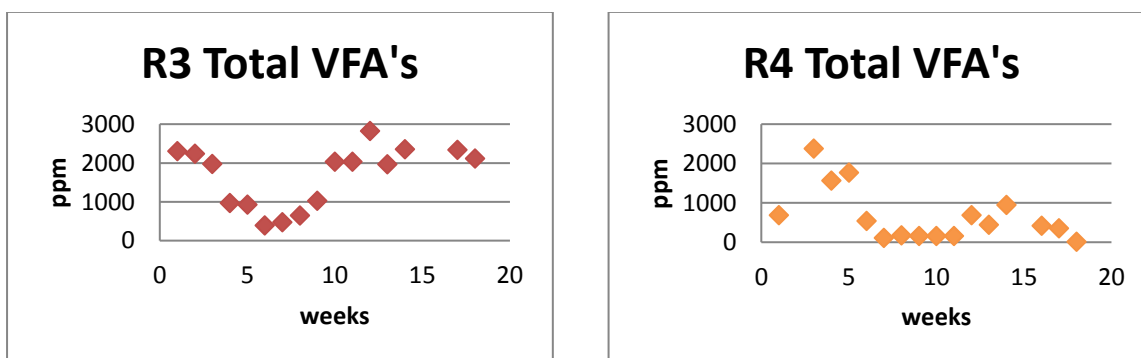


Figure 5-12 Total VFA's accumulation through the experiment.

The VFAs for reactor R3 were higher through all the run. This can be also noticed in the constant drop of the pH. Whereas for R4 the VFA's were much lower and they became stable and low for the rest of the run, as can be seen in Figure 5.12 and 5.13.

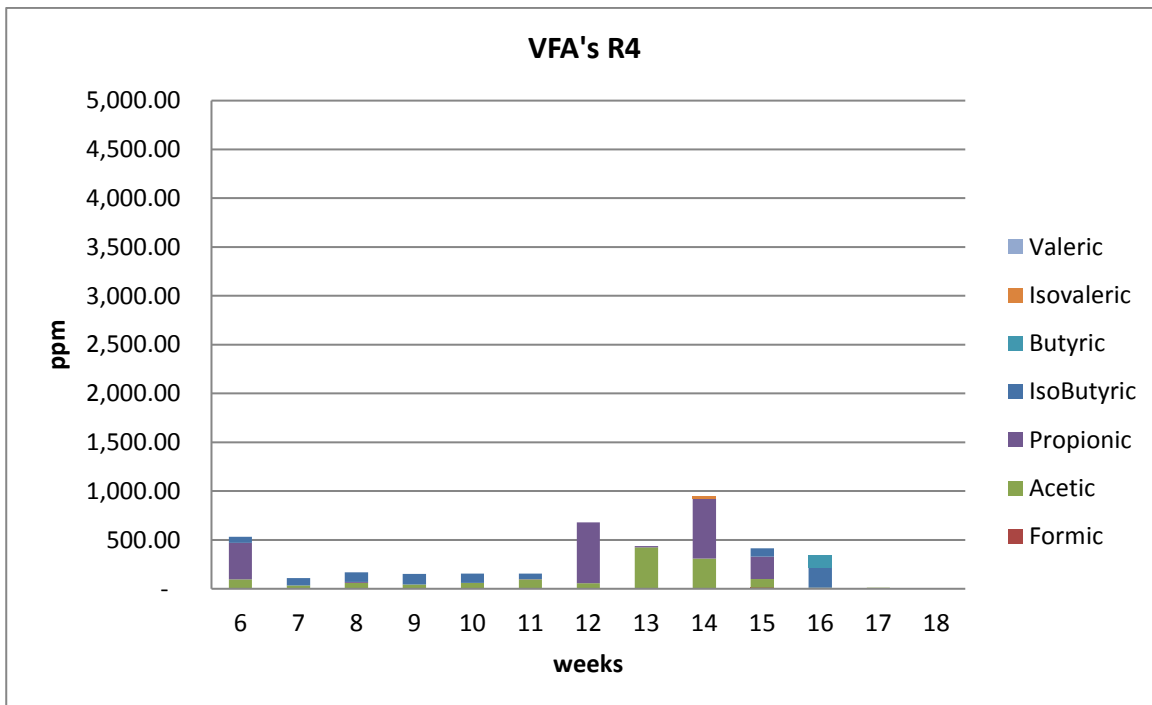
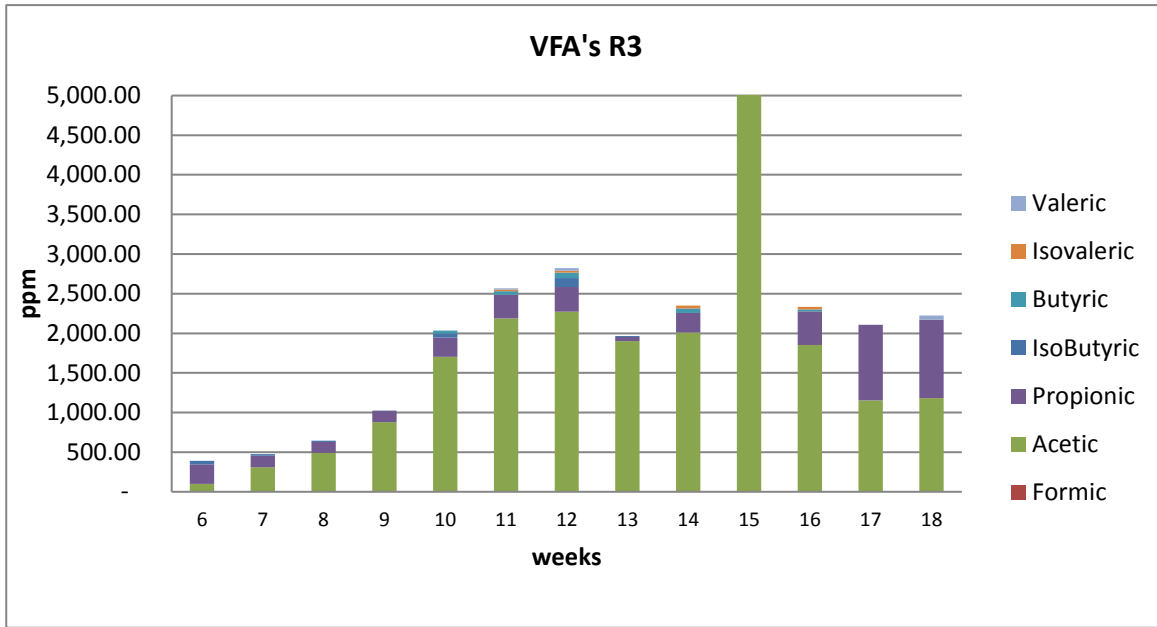


Figure 5-13 Detail for the VFA's composition of the reactors.

3rd HRT results and analysis

From the previous results it was noticed that during the 3rd HRT the four reactors reached steady state. Once this happened a more detailed analysis was carried out in order to compare the substrates working at mesophilic vs thermophilic temperatures.

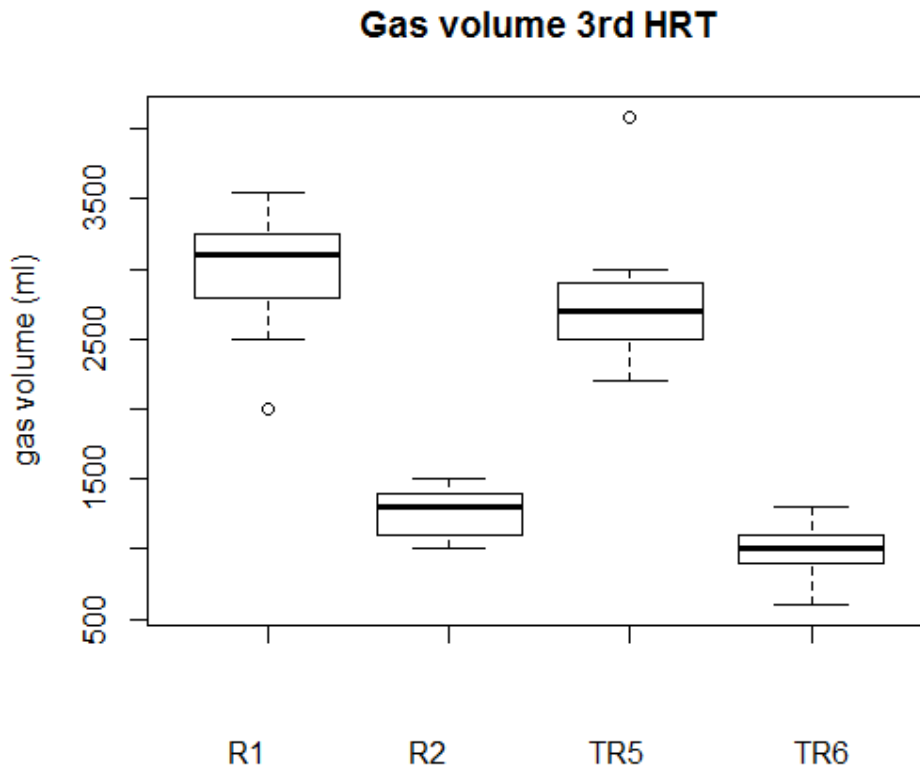


Figure 5-14 Biogas volume production in the 3rd HRT

In the figure 5.14 it can be seen that the reactors working with grains as feed were performing better than those with hops. In both cases the reactors working at mesophilic conditions for grains and hops presented a higher biogas production rate than those operating at thermophilic conditions.

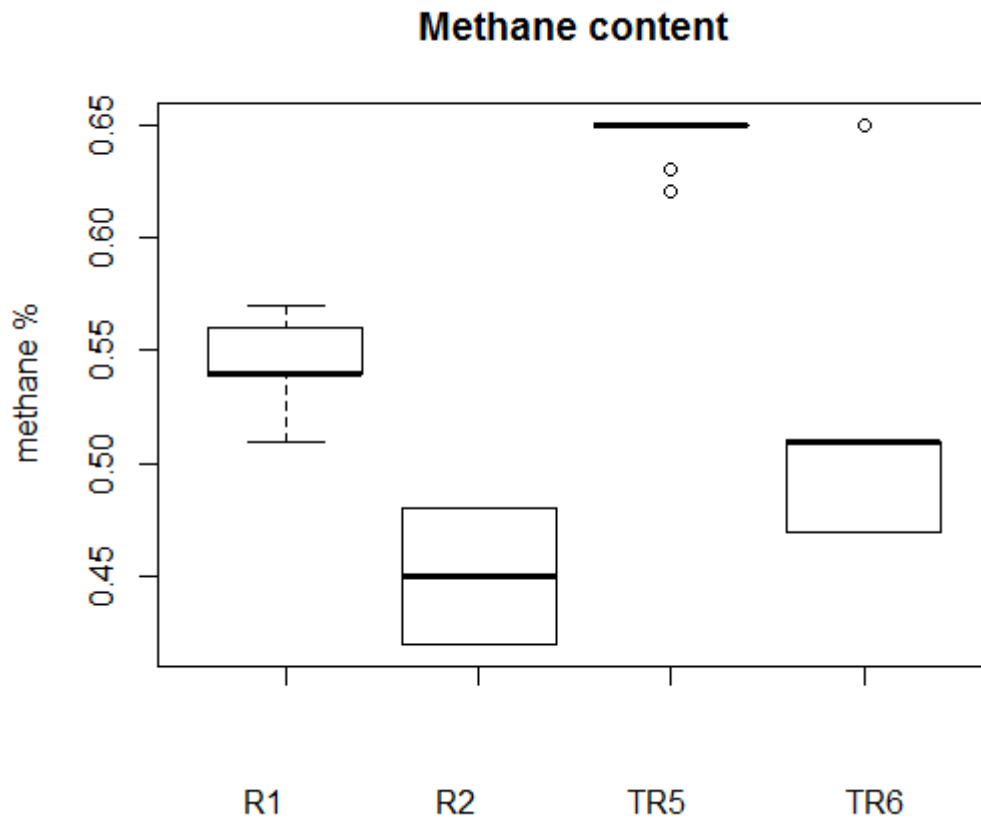


Figure 5-15 Methane content in the biogas in the 3rd HRT.

As mentioned in Chapter 4, the methane content was monitored 3-4 times a week for each reactor. In Figure 5.15 the methane content can be observed. This analysis was made in triplicate for each gas bag and the average was recorded. Then a weekly average was calculated and reported. Once again the two reactors working with grains showed higher methane content in the biogas. But then for this parameter the reactors working at thermophilic conditions presented higher values of methane than the ones working at 35 °C.

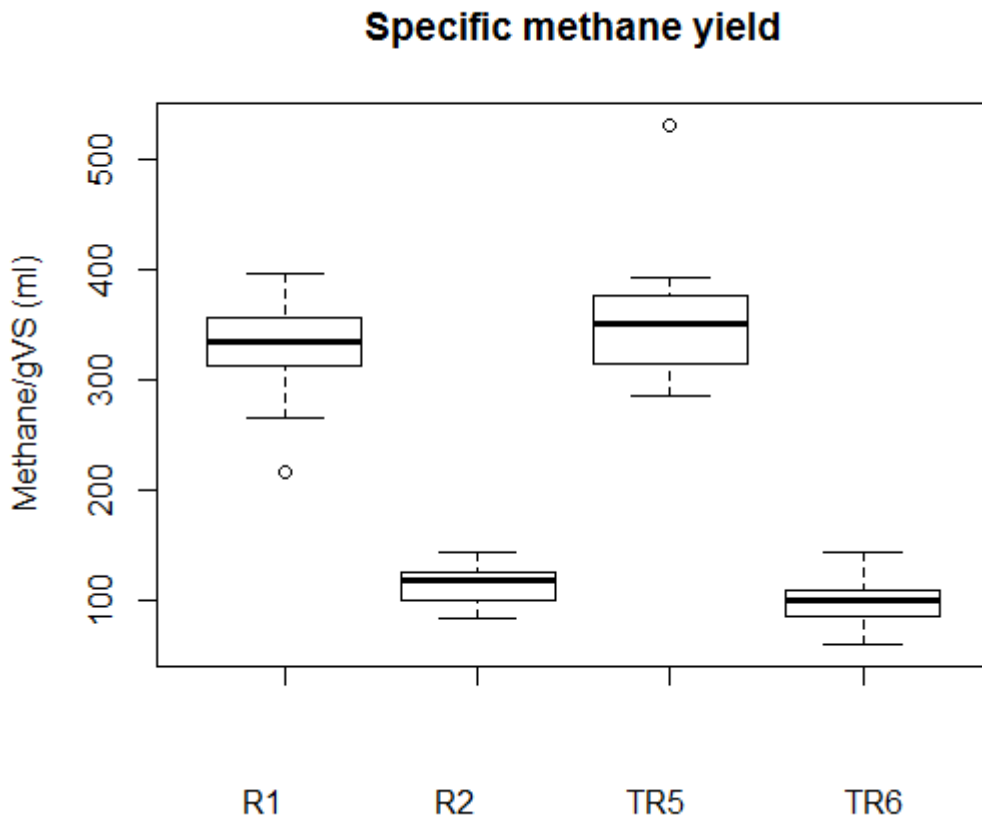


Figure 5-16 Specific methane yield production in the 3rd HRT.

As it can be seen in Figure 5.16, with the previous data it was possible to calculate the specific methane yield for each reactor. This was determined by multiplying the biogas production by the methane content and divided by the VS added. In the figure above it is noticeable that the two reactors working with grains generate a higher SMY. The reactor operating at thermophilic conditions has a higher SMY than the one operating at mesophilic.

A t-test was carried on in order to determine if there is a significant difference in the reactors operating at different temperatures. In the test the null hypothesis is that the two means are equal, and the alternative is that they are not. This test was performed using the environment R. R is a language for statistical computing and graphics developed at Bell Laboratories by John Chambers and colleagues.

R provides a wide variety of statistical (linear and nonlinear modelling, classical statistical tests, time-series analysis, etc.) and graphical techniques. (www.r-project.org).

The results from the test are shown below. A Welch Two Sample t-test, with N = 25 samples and confidence interval of 95% was run using the software. The formula for the test is:

$$t = \frac{X_1 - X_2}{\sqrt{\frac{S_1^2}{N_1} + \frac{S_2^2}{N_2}}}$$

Where:

X is the sample mean

S sample variance

N sample size

The results obtained from R are shown below:

t = -2.0931, df = 40.824, p-value = 0.0426

where:

t= t value

df=degree of freedom

Alternative hypothesis: true difference in means is not equal to 0, 95 percent confidence interval:

-60.125672 -1.071128

Sample estimates:

mean of R1 mean of TR5

326.1044 356.7028

Meanwhile the p-value is < 0.05 the null hypothesis which is that the two means are equal can be rejected with moderate evidence since $0.01 < p < 0.05$ for this case.

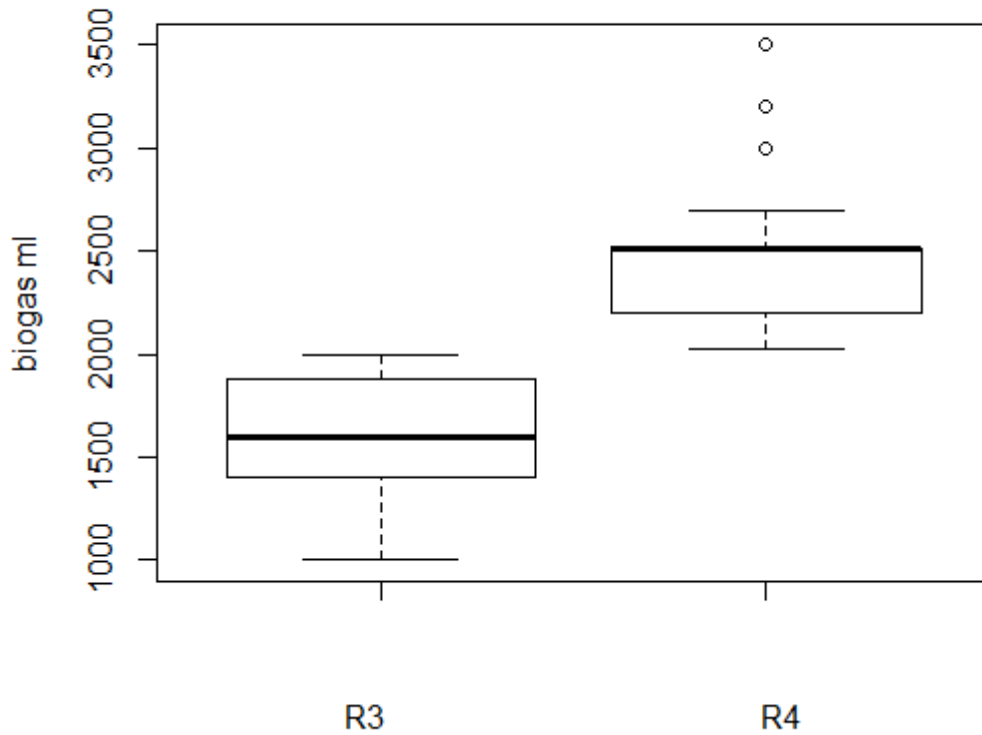


Figure 5-17 Box-plot for the biogas production in the 3rd HRT for R3 and R4.

The reactor fed with yeast presented a higher volume of biogas production in comparison with the 90/10 mixture, as seen in Figure 5.17. This is according to findings about the positive impact of the yeast. It has been reported by [101] finding that yeast improves the digestibility and methane production of the waste water from a brewery due to its addition of microbial communities. Furthermore [102] also found that yeast helps in the fermentative breakdown of sugars and lipids from food wastes.

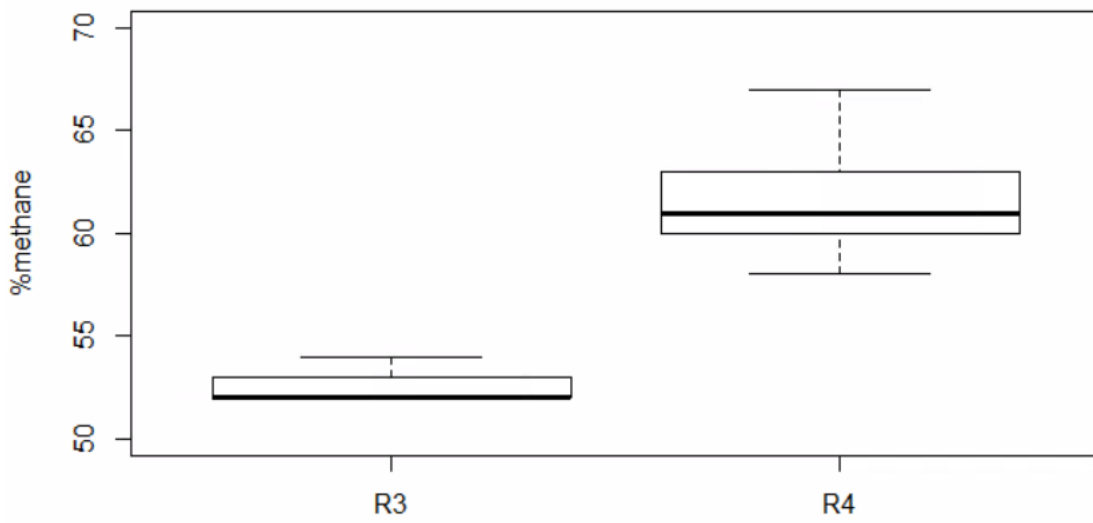


Figure 5-18 Box-plot for the methane composition of the biogas production in the 3rd HRT for R3 and R4

In terms of methane content Reactor 4 had a higher value too. The analysis was done in triplicates just as with the other four reactors, as shown in Figure 5.18.

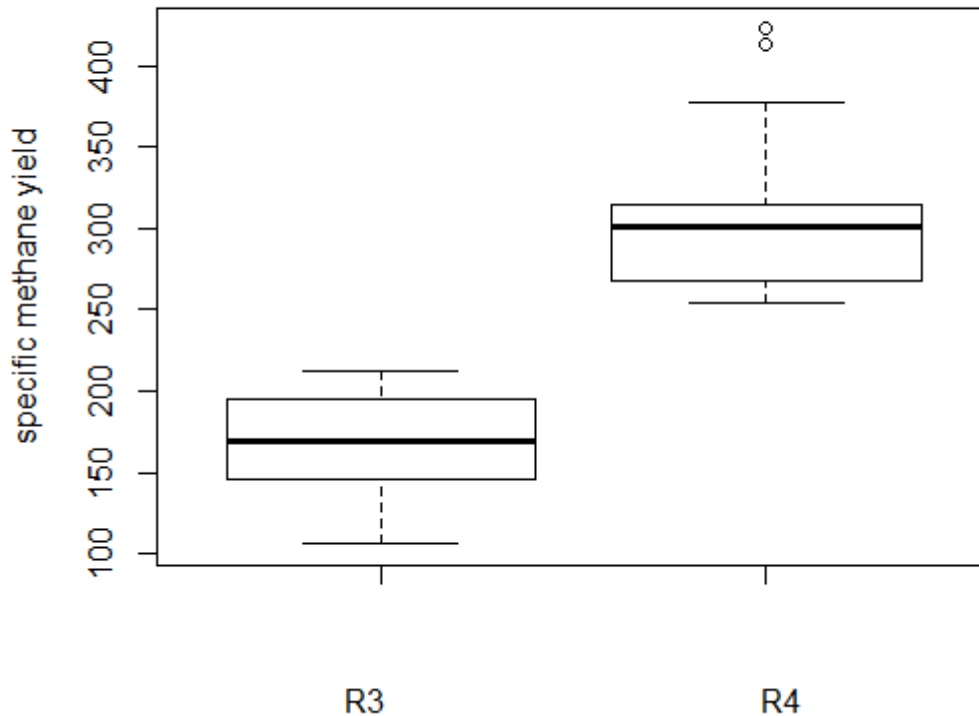


Figure 5-19 Box-plot for the specific methane production for R3 and R4 in the 3rd HRT

The specific methane yield for Reactor 4 is 306 ml/gVS. While for Reactor 3 it was 169.5 ml for each g of VS added. These reactors were only run at mesophilic conditions. But as it can be seen from Figure 5.19, the yeast has a good potential of biogas production and in the future would be worth to run it at thermophilic conditions to see if these figure improves.

There was an important variable to monitor during the experiment. Due to the nature of the samples the operation ran at lower pH, and even close to the lower limit (6.6). In some cases a dose of 2 g of Sodium Carbonate was required to stabilize the pH and allow the reactors to operate in better conditions. Volatile Fatty Acids were monitored weekly and it can be noticed a close relation with the pH. Where high peaks of VFA's can be observed it was reflected a lower pH for that period of time. Also is important to notice that during the run the reactors running at low VFA's were the ones which presented the better yields. Figures 5-7 and 5-13 give a profile of the VFA's showing different concentrations for

substrates operating at mesophilic and thermophilic conditions. It would be recommendable to have a closer look to this profiles and the ratio among the different volatile fatty acids. As is can have a significant impact in the operation of the continuous stirred tank reactors.

In this section of the study the experimental values have high importance. One of the crucial measures was the methane content. In order to get reliable values a calibration curve was done before every session at the chromatograph. The procedure followed was to inject in triplicate different amounts of a known standard concentration of gas (Scientific technical gases, UK) in order to obtain the calibration curve. All the curves used in the experiment had a value for R^2 of minimum 0.98 in order to ensure good accuracy. The samples were measured using a similar procedure, injecting in triplicate each one. In every session the same syringe was utilised and the parameters of the equipment were kept constant all the time. The standard deviation and standard error were measured through all the determinations. In the table 5-10 an example for the 3rd HRT is shown. The full measurements can be found in the appendix. Typos corrected

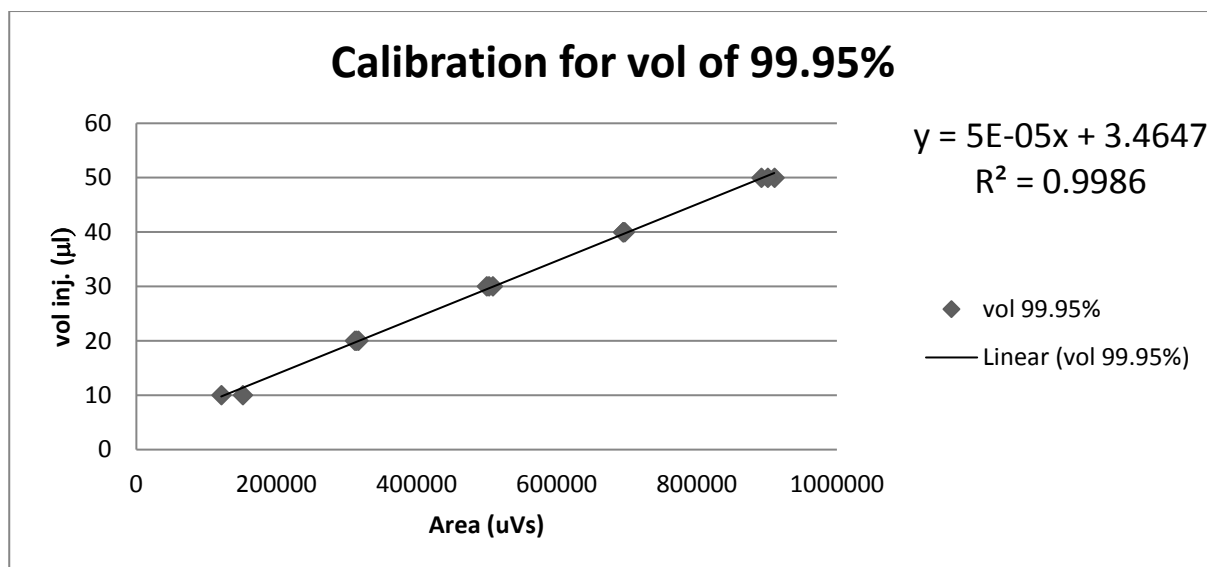


Figure 5-20 Example of calibration for methane determination

Table 5-10 Example of methane measurements in the chromatograph

no. Inj.	date	Reactor	Retention time (min)	Area (uVs)	volume from equation	% of methane	average	std dev	st error
1	25-Feb-15	R1	10.442	430298.8	24.980	50%			
2	25-Feb-15	R1	10.64	446484.2	25.789	52%			
3	25-Feb-15	R1	10.823	438412.3	25.385	51%	51%	1%	0.27%
1	25-Feb-15	R2	11.162	372230.0	22.076	44%			
2	25-Feb-15	R2	11.322	371337.3	22.032	44%			
3	25-Feb-15	R2	11.488	369856.6	21.958	44%	44%	0%	0.04%
1	25-Feb-15	RT1	13.17	540771.0	30.503	61%			
2	25-Feb-15	RT1	13.362	537707.0	30.350	61%			
3	25-Feb-15	RT1	13.552	545522.8	30.741	61%	61%	0%	0.13%
1	25-Feb-15	RT2	13.835	447103.9	25.820	52%			
2	25-Feb-15	RT2	14.065	439149.1	25.422	51%			
3	25-Feb-15	RT2	14.283	428535.3	24.891	50%	51%	1%	0.31%
1	2-Mar-15	R1	10.227	445928.2	29.778	60%			
2	2-Mar-15	R1	10.437	437640.3	29.281	59%			
3	2-Mar-15	R1	10.628	445311.3	29.741	59%	59%	1%	0.18%
1	2-Mar-15	R2	11.107	395467.9	26.750	54%			
2	2-Mar-15	R2	11.308	427872.4	28.695	57%			
3	2-Mar-15	R2	11.503	408908.7	27.557	55%	55%	2%	0.65%
1	2-Mar-15	RT1	13.15	532901.7	34.996	70%			
2	2-Mar-15	RT1	13.362	530774.2	34.869	70%			
3	2-Mar-15	RT1	13.547	535205.1	35.135	70%	70%	0%	0.09%
1	2-Mar-15	RT2	13.878	412137.3	27.750	56%			
2	2-Mar-15	RT2	14.078	402242.3	27.157	54%			
3	2-Mar-15	RT2	14.295	406286.5	27.399	55%	55%	1%	0.20%
1	18-Mar-15	R1	34.362	402456.1	31.995	64%			
2	18-Mar-15	R1	34.51	424550.3	33.321	67%			
3	18-Mar-15	R1	34.658	437252.6	34.083	68%	66%	2%	0.70%
1	18-Mar-15	R2	31.442	326400.8	27.432	55%			
2	18-Mar-15	R2	31.61	353208.4	29.040	58%			
3	18-Mar-15	R2	31.785	354378.5	29.110	58%	57%	2%	0.63%
1	18-Mar-15	RT1	33.118	499986.8	37.847	76%			
2	18-Mar-15	RT1	33.272	518051.0	38.931	78%			
3	18-Mar-15	RT1	33.412	525225.6	39.361	79%	77%	2%	0.52%
1	18-Mar-15	RT2	33.778	178658.8	18.567	37%			
2	18-Mar-15	RT2	33.953	163134.8	17.636	35%			
3	18-Mar-15	RT2	34.1	161513.1	17.538	35%	36%	1%	0.38%

Table 5-11 Specific methane yield for each substrate.

	Specific methane yield (ml/gVS)		
	theoretical	BMP	Reactors
Grains	535.76	331.56	326.11
Hops	501.38	39.62	92.95
Yeast	481.45	312.16	306.00
Spirit	516.49	131.50	N/A

Summary of the performance of the reactors are shown in Table 5.10.

Table 5-12 Summary of the performance of the reactors

	R1	R2	TR5	TR6
Biogas production (l/d)	3.00	1.23	2.69	0.99
CH ₄ content (%)	55%	43%	65%	46%
CH ₄ production (l/d)	1.63	0.46	1.73	0.47
Specific biogas production (l/gVS)	0.60	0.24	0.53	0.19
Specific CH ₄ production (ml/gVS)	326.11	92.95	356.15	93.73
pH	6.70	6.70	6.83	6.98
TVFA's (mg/l)	1569.48	967.41	196.48	904.46
TS %	1.37%	1.47%	0.86%	1.41%
VS %	73.65%	80.28%	81.63%	81.03%
TAN (mg/l)	308	420	-	196
TKN (mg/l)	840	448	336	1680

The four reactors have reached steady state during the 3rd HRT. Gas production of the reactors fed with grains was significantly higher in both cases (mesophilic and thermophilic). The reactor R1 had the highest volume of biogas produced but the methane content was lower than the reactor operating at thermophilic conditions. The reactor TR5, which was the one producing the highest volume of methane also showed the best yield in terms of methane produced per gVS fed. Studies comparing both conditions have found similar results, being the thermophilic temperatures the ones showing higher biogas production [64, 66, 103, 104]. Whereas for the reactors fed with hops the biogas production and methane content is similar. The higher values of methane per gVS are achieved by reactors working with grains as feed. This was also noticed in the batch experiment which can lead to an initial conclusion that the grains are more suitable for anaerobic digestion than the hops. This can be studied in more detail as by the characterization done it was not possible to detect any differences in lignocellulose and cellulose content which affects the digestion. Studies of the characterisation and feasibility of spent grains have been done by other researchers. In those studies the spent grains are considered as the mixture of the waste as reported by [105] and [106]. A detailed characterisation of each of the waste grains would be recommended for the future in order to confirm which one is more suitable for anaerobic digestion.

The thermophilic reactor TR5 is the reactor working with the lowest total VFA's showing that it can possibly process a higher OLR. Similar behaviours were found by [63] while comparing the anaerobic digestion of sugar beet. Contrary to R1 which has a high ppm of VFA's . The performance of the other two reactors was also monitored and reported as the previous ones. Both reactors reached steady state at the 3rd HRT and those values are presented.

With the data obtained it is possible to perform an energy balance for the anaerobic digestion system. This was carried out following the methodology in Chapter 4. From the experimental results it was concluded that the grains presented a better performance at both conditions yielding a higher methane amount in the biogas. Therefore the analysis of the digester presented is considering the grains as feed and a comparison of the two conditions.

The thermal energy requirements were given by loss from walls and roof to the ambient. The heat required to bring the system to the digester temperature, 35 °C for mesophilic and 55 °C for thermophilic. Considering an insulated concrete wall for the construction of the digester which has a heat transfer coefficient of .6-.8 WR² °C, the calculations were done, as shown in Table 5-12. Since the production plant is in the UK the annual energy requirements were also considered.

Table 5-13 Energy requirements to run the Anaerobic digesters at both temperature conditions.

	Mesophilic Reactor(35 °C)	Thermophilic reactor(55 °C)	
Brewers Waste			
Amount of brewers waste	1500	1500	
Water content	0.63	0.63	
Total Solids	0.25	0.25	
volatile solids	0.954	0.954	
kg of VS of grains	357.75	357.75	
Slurry			
Amount of slurry	4500	4500	
Water content	0.78	0.78	
required water content	0.85	0.85	
Additional Water	4300	4300	
total volume	10300	10300	
Inlet temperature	15	15	
temperature desired	35	55	
cp Water	4.2	4.2	
Heat demand			
Overall Heat demand	MJ	361,200.00	722,400.00
thermal power	kWh	101,136.00	202,272.00

5.6 Discussions

Interpretation of the BMP assay results is of supreme importance. There is concern regarding the suitability of using the results of the BMP assay to predict the performance of semi- or continuous-flow or even a commercial size system. In this determination the substrate remains in contact with the sludge during the whole period of time. This allows microorganisms, enzymes, intermediate products, and final products to accumulate within the system. This sometimes can lead to an altered balance especially if intermediate products, such as volatile fatty acids (VFA's) particularly acetic and propionic reach its maximum. Then other metabolic reactions could be inhibited. BMP tests are a good parameter to see how biodegradable a substrate is, but definitely a semi continuous or a continuous experiment would be closer to a commercial anaerobic digestion.

5.7 Summary

In this chapter the results of both experiments were presented. First a BMP assay was carried out, in order to determine the feasibility of the substrates. Once having finished the BMP a semi-continuous daily fed system was operated. It consisted of a set of 5 L continuous stirred tank reactors (CSTR's). It is relevant to notice that the grains presented a better performance than the other substrates in both experiments. Running at thermophilic condition of 55 °C the grains presented a better performance in the semi continuous reactors. This could be further studied as it gives the opportunity to either increase the OLR or have shorter HRT's, giving an advantage against the mesophilic conditions. As it has been presented in Chapter 3, there is waste heat available that could be used to run a thermophilic digester without incurring in extra demand for heat.

Chapter 6. Modelling and simulation

6.1 Introduction

In this Chapter the modelling set up for the two study cases is presented. In order to complete the simulations an energy audit was first carried out in both sites. This allowed the collection of relevant data as well as the opportunity to have a full understanding of the operations which were very useful to construct the simulation.

6.2 Modelling strategy

6.2.1 ECLIPSE software

ECLIPSE software was utilised for the simulation of the cases. ECLIPSE was developed for the European Commission and has been used by the Northern Ireland Centre for Energy Research and Technology at the University of Ulster since 1986. It is a personal-computer-based package containing all of the program modules necessary to complete rapid and reliable step-by-step technical, environmental and economic evaluations of chemical and allied processes. It uses generic chemical engineering equations and formulae including high-accuracy steam-water thermodynamics package for steam cycle analysis.

The main procedures in the simulation of any process using the software are:

- 1) Completing the compound database with all the elements and compounds involved in the process.
- 2) Preparation of the process flow diagram with all the equipment and streams to be utilised.
- 3) Inputting the required process conditions to the process

4) Finally it gives the opportunity to perform an utility analysis

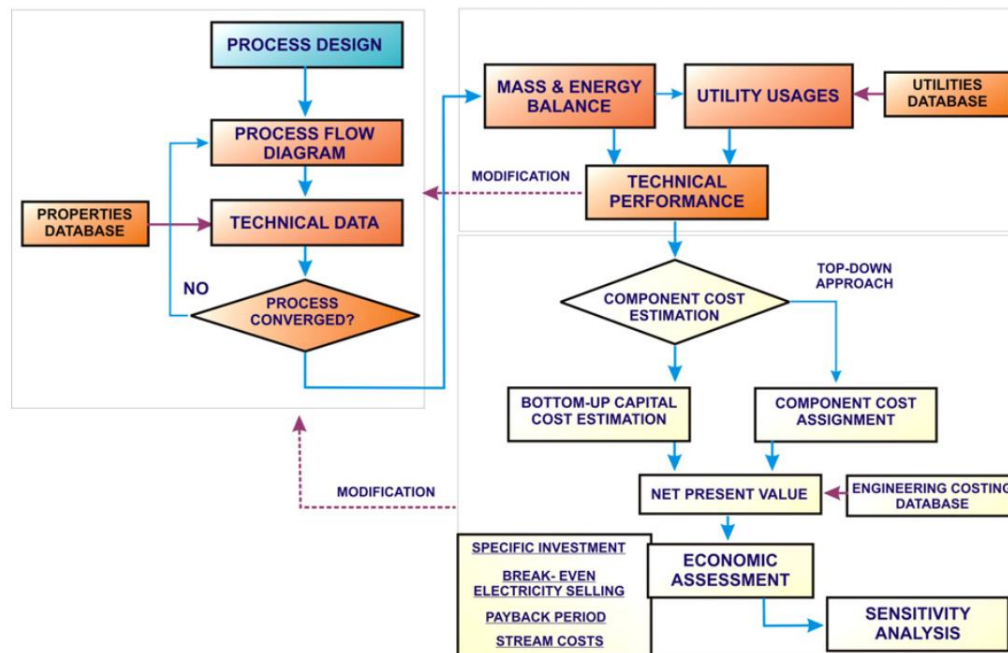


Figure 6-1 ECLIPSE process model and simulation flow diagram [107].

Once all the data is in the system, then the calculation of the mass and energy balance can take place. ECLIPSE also has the option of calculating the utilities usage as well as the economic analysis. ECLIPSE has been successfully used in previous studies involving biomass utilization and combustion among other studies of carbon capture as presented by [107-109]. In those studies the reliability of the software has been positive.

6.2.1.1 Compound data base

As stated before, the first procedure in ECLIPSE software simulation is the gathering of the necessary data for the compounds that will be used in the simulation. In order to run successfully ECLIPSE software requires all the mandatory input variables to be completed in the compound database. Some of these data already existed in the compound data base used. Whereas those that were not on the compound database were included and completed through the experimental characterizations and literature review.

The four grains feed stocks and their respective distillers grains (DGS) data from the spirit plant and the brewery were entered using these compound names CORN, RICE, SORGHUM and ST-RICE. The screen view of the compound database for the spirit production and biogas simulations are shown in table as it can be seen in Figure 6.2. The composition and values such as Critical Temperature, Critical Pressure, Acentric Factor, Heat of formation and combustion among others are all necessary.

The screenshot shows a software window titled "Modify the Compound (Type 2 and 3)" with a close button (X) in the top right corner. The window contains several input fields for compound data:

- Compound ID: CH4
- Molecular Weight: 16.04
- Compound Type: Standard State Gas
- Tc: -82.55
- Pc: 46
- Number of Elements: 2
- Acentric Factor: 0.008
- Symbol 1: H, Weight (%age): 25.13
- Symbol 2: C, Weight (%age): 74.87
- Symbol 3: (empty), Weight (%age): 0
- Symbol 4: (empty), Weight (%age): 0
- Symbol 5: (empty), Weight (%age): 0
- Symbol 6: (empty), Weight (%age): 0
- Heat of Formation (kJ/kgmole): -74901.8
- G. E. of Formation (kJ/kgmole): -50869.6
- Cp=a+bxt+cxt (2)+dxt (3) (kJ/kg mole Deg K)
 - a: 19.248
 - b: 5.213
 - c: 1.198
 - d: -11.316
- Valid Temperature Range (C): 0 To 3000
- Buttons: Cancel (with X icon), Apply (with checkmark icon)

Figure 6-2 Compound data base entry for methane

6.2.1.2 Process flow diagram

All the different streams and modules must be properly connected together. Every module must have at least one incoming and one outgoing stream for the simulation to run successfully. In the order to cover all the scenarios a process flow diagram was drawn for each case to ensure complete analysis of the system. The Process Flow Diagrams (PFDs) were built from the notes taken during the energy audits and the diagrams provided by the companies, that can be found in Appendix A and B. The process flow diagram for the spirit production is showed in Figure 6.3.

The PFD used here was drawn to provide basic method for the production of alcoholic rice spirit in an ethanol industry. The modules used included a Fixed Flow, to control the amount of stream entering the process. Mixers, to combine different streams. Tanks/Vessels, to simulate cooking/steaming process. Heat exchangers, to simulate the heating and cooling stages and Chemical Reaction, for the fermentation process.

The actual process is carried out in a batch operation, but ECLIPSE software can only simulate continuous processes. In order to address this issue the amount of material that is added at each stage was divided by the length of the operation. This means that for the first step which takes 4 hours the total amount was divided by the time and then inputted to the software in kg per second as required. The other parameters like temperatures andn pressure were kept the same.

The process begins with the terminal start streams for inputting the grains consisting on Sorghum 1420kg, Corn 80kg, Stick-Rice 300kg and Rice 200kg for the GRAINS. One more stream for the WATER and injection of STEAM was connected to the cook tank. This was linked to the air cooling. Then it was connected to the fermenter where yeast is added for the fermentation and is formerly distilled. Finally it is followed by a separation of the bio waste and extraction of the raw spirit.

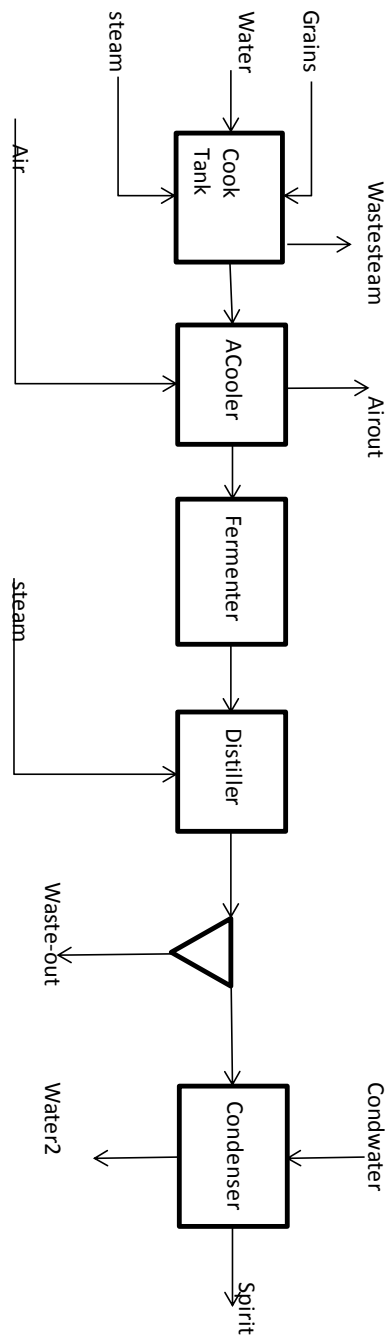


Figure 6-3 Process flow diagram for the spirits plant

6.2.1.3 Technical Data Input and Mass and Energy Balance

In order to specify the behaviour of all the modules the correct information of the technical data must be stated and inputted accurately. The technical data was added based on the

information collected from the spirit production plant in the Energy Audit. Mass and energy balance calculations were carried out for the different systems. This was done after specification and addition of the data to the feed streams. The modules that required initialisation were checked to ensure that they are consistent with the compound database. Figure 6.4 shows the Mass and Energy balance screen with the stream description of the spirit production site in ECLIPSE.

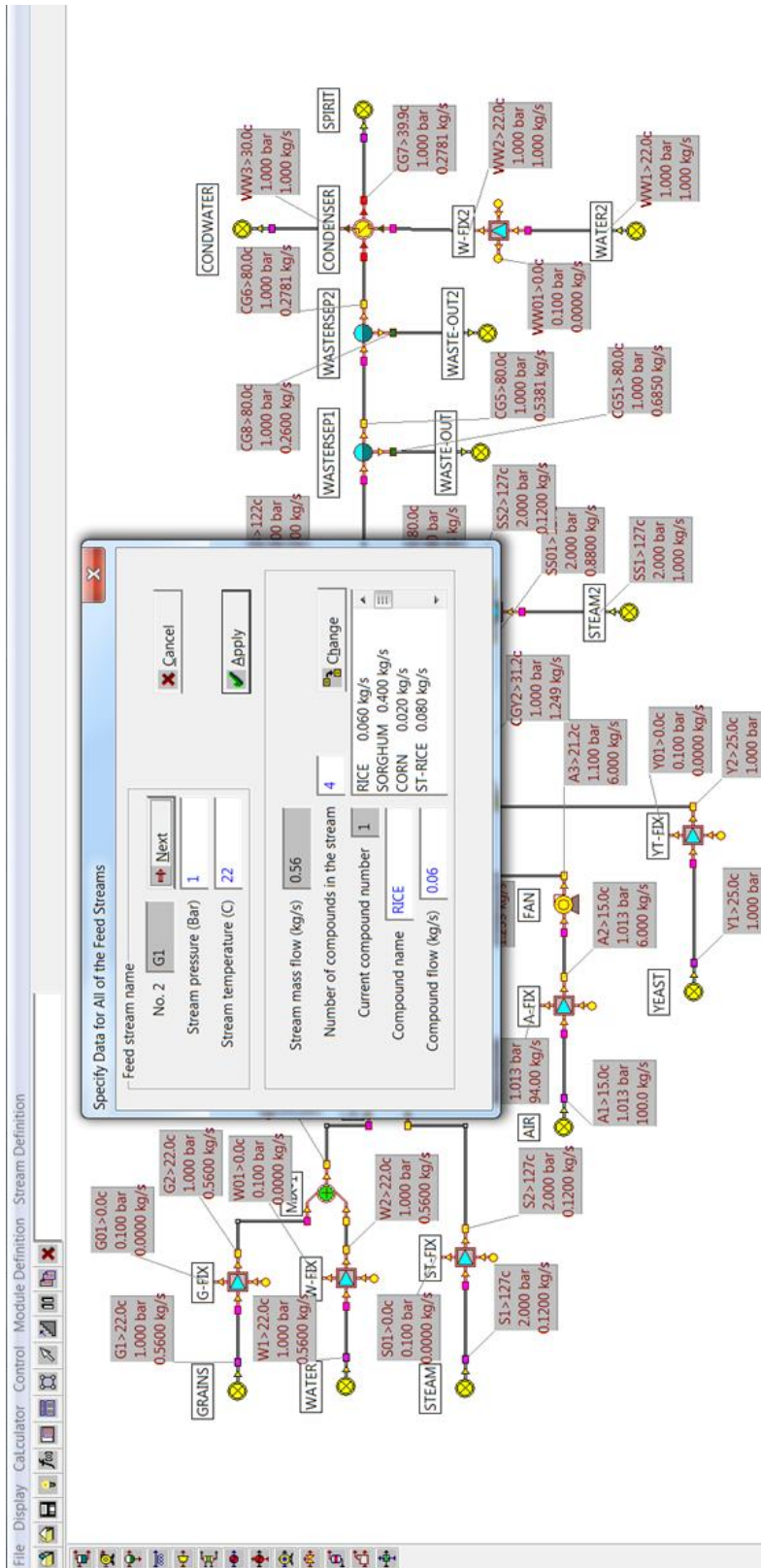


Figure 6-4 Mass and energy balance screen from the spirit plant.

6.3 Study cases plant scenarios

6.3.1 Modelling of spirit plant waste treatment scenarios

For the combustion of the waste grains from the spirit production plant in China three scenarios were simulated and compared: Current situation using 100% of coal as fuel for the boiler, mixing 50% of the bio waste generated with Coal for co-firing, and the use of 100% of the dried bio waste for direct combustion. The conditions for the simulation were taken from the data obtained from the energy audit. The characteristics of the waste were determined in the laboratory as mentioned in Chapter 4. Once the production process was modelled the next step was to include the stage of drying and co-firing the bio waste as shown in the Figure 6-5.

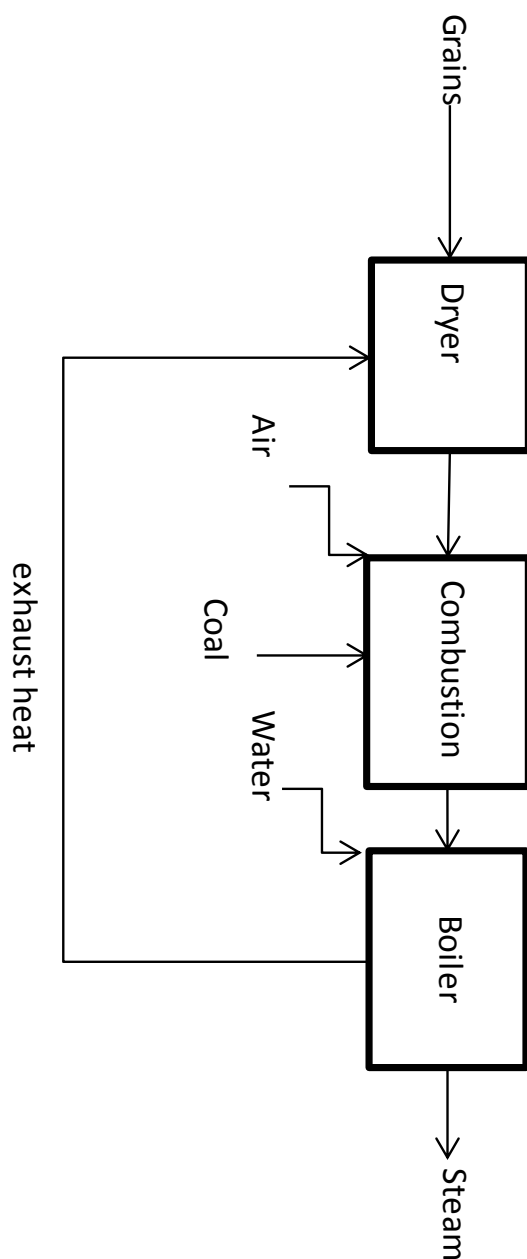


Figure 6-5 Co-firing of bio waste from spirit production process flow diagram

As it can be seen in the PFD different scenarios were taken in account to explore the potential of the bio waste to be fed to a boiler for steam generation. The waste heat from the combustion is used for the pre treatment of the wet spent grains as a source of heat for the drying step. The possibility of using the spent grains as feed to anaerobic digestion was also analysed. That possibility was based solely in the batch experiments. It was not

possible to run reactors with the spirit plant waste since the amount of sample collected was not enough. Also a simulation using Eclipse was performed to represent this scenario. Figure 6.6 shows the anaerobic digestion coupled system to treat the waste from spirit production process flow diagram. Figure 6.7 is the separated anaerobic digestion system to treat the waste from spirit production process flow diagram.

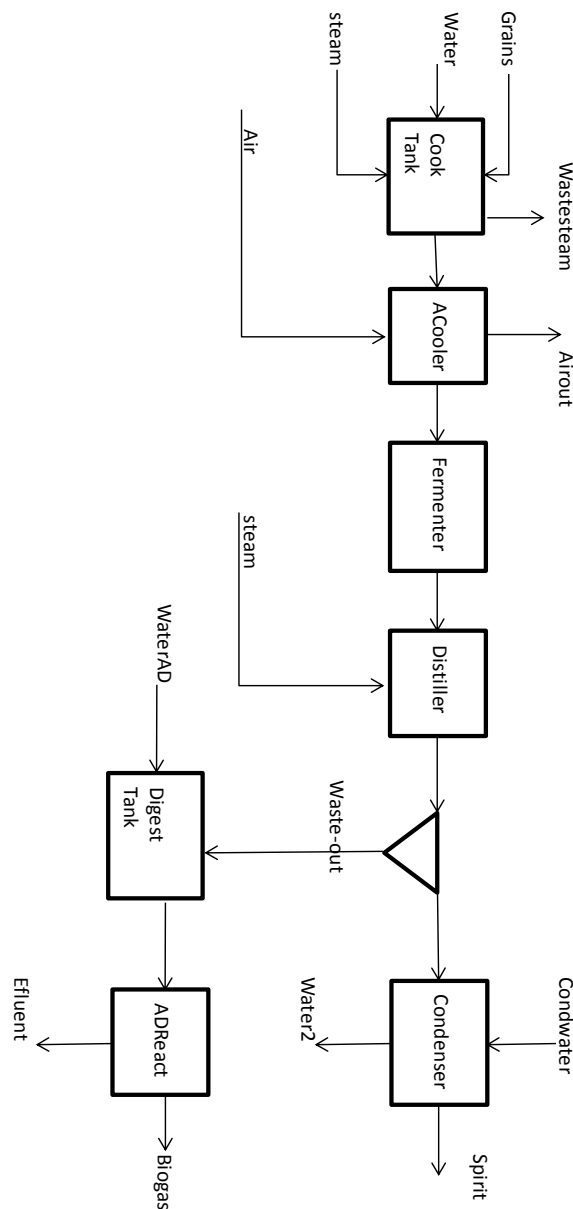


Figure 6-6 Anaerobic digestion coupled system to treat the waste from spirit production process flow

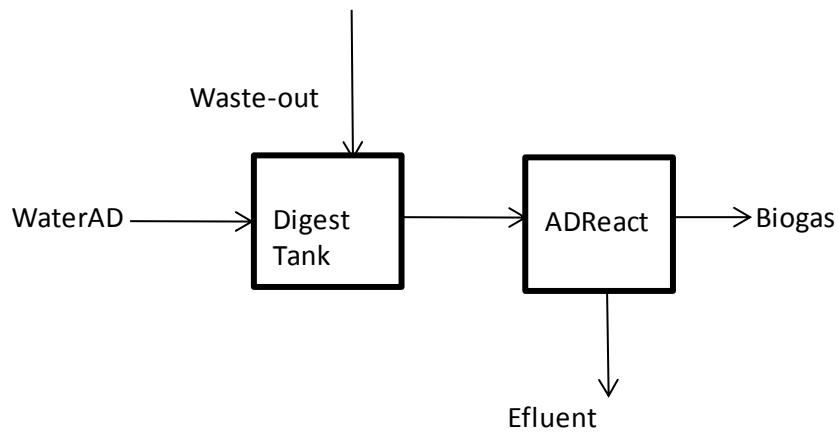


Figure 6-7 Anaerobic digestion system to treat the waste from spirit production process flow diagram.

6.3.2 Modelling of the brewery

Following the same methodology as the spirit plant, a model of the brewery was first built and tested at the given conditions when the audit was carried out. The first step was to build a process flow diagram using the modules available in Eclipse to represent the brewing process. In the same manner as for the spirit plant the modules used were Fixed Flow, Mixers, Tanks/Vessels and a Chemical Reaction to represent the fermentation process, as shown in Figure 6.8. This model was then compared with the data obtained during the energy audit and the further calculations in order to validate the modelling software.

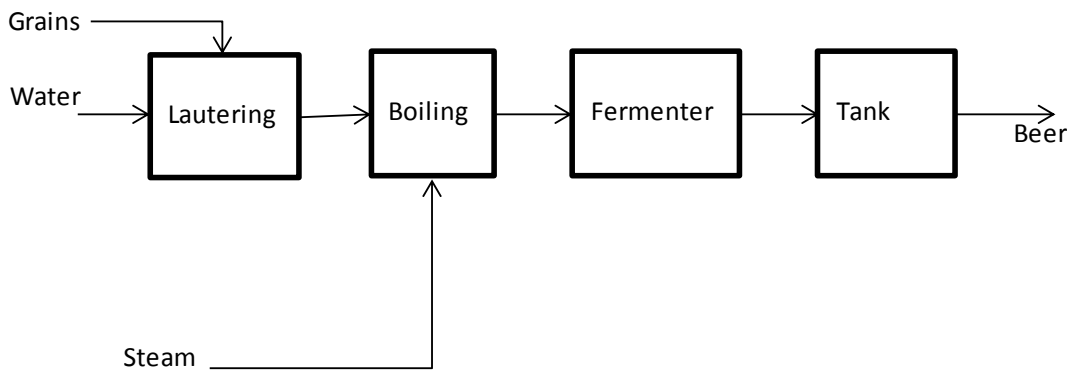


Figure 6-8 PFD of the brewery

From the energy audit it was also possible to determine the amount of waste that is generated in a daily basis and the fraction of that waste that is biodegradable and could be used for an Anaerobic Digestion reactor. Table 6-1 shows the waste produced by the brewery.

Table 6-1 Waste produced by the brewery

	Grains	Hops
	1500 kg/day	150 kg/day
TS	24.58%	16.36%
VS	96.17%	95.71%

A Continuous Stirred Tank Reactor (CSTR) Anaerobic Digester was proposed to be installed in order to produce the biogas as fuel for the industry. This was made using the results obtained from the experimental work. It was possible to determine theoretically and experimentally the biogas production for the waste and the methane yield per kgVS of the spent grains. Then a simulation using this biogas as fuel for a CHP system was completed according to the amount that is generated.

There were mainly two conditions studied in the experimental work. One of them assumes a mesophilic anaerobic digestion process at 35 °C and the other one operating at thermophilic conditions of 55°C. Each of them showed different methane yield performances, as shown in Table 6-2. With this data a simulation was carried on to determine which one would lead to a better performance in terms of energy usage. From the experimental work it was possible to identify the grains as the most suitable feed and the most available in terms of volume.

Table 6-2 Biogas production and methane content from the experimental work.

	R1	R2	TTR5	TTR6
biogas production (l)	3.00	1.23	2.69	0.99
CH ₄ content (%)	55%	43%	65%	46%
CH ₄ production (l)	1.63	0.46	1.73	0.47
Specific CH ₄ production (ml/gVS)	326.11	92.95	356.15	93.73

The scenarios were modelled using the information presented in the Table 6-1. Based on the experimental results and knowing the amount of waste available. It was possible to estimate the biogas that could be produced in an anaerobic digester at the given conditions.

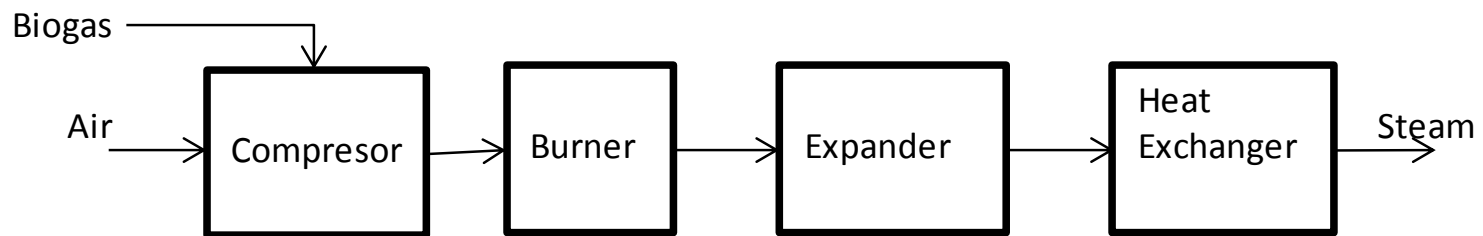


Figure 6-9 Model for a CHP system for the brewery using the biogas produced.

The simulation of the Combined Heat and Power plant fuelled by the biogas was done following the same steps as the previous simulations. First the process flow diagram was drawn and all the components were specified, as shown in Figure 6.9. It is assumed that the heat waste from the expander is recovered and re used within the system. The amount and quality of the biogas showed to be a good option to be used as a fuel for the same process. This biogas would be able to cover up to 65 % of the current demand.

6.4 Summary

In this chapter the simulation of the different processes and scenarios was presented. A simulation of both plants was done for each one. It was possible to replicate the main operations in to ECLIPSE. It was also possible to create scenarios where the bio wastes consisting of spent grains were reused in order to produce fuel for the operation. In the case of the spirit plant a pre treatment of the grains consisting on a drying step to reduce the moisture of the grains was considered. The heat for the drying could be supplied by one of the waste heat streams of the process. Then, the dried grains could be either mix with the coal or even burned in the boilers in order to generate the steam required for the process. For the brewery it was also possible to model the process with good accuracy. Then it was possible to explore scenarios where biogas produced in an anaerobic digestion unit could be used feed a CHP system. All the process flow diagrams were prepared and the compound data base was completed with the necessary data in order to run the simulations.

Chapter 7. Modelling results and discussions

7.1 Introduction

In this chapter the results from the modelling are presented. The description includes the adaptations that were made in order to fit the simulations into ECLIPSE and the comparison with the actual data that was obtained and measured in the plant visits. The simulation of the scenarios proposed in this study are also shown and discussed. One of them is the combustion of the waste in the boilers to provide steam while the other scenario involves anaerobic digestion of the organic waste. For this purpose only the simulation of a combined heat and power (CHP) plant is presented as the results from the digestion of the wastes were determined in an experimental way.

7.2 Results and discussion of spirit plant modelling

The approach for the spirit production plant was to model the possibility of using the spent grains as feed for a boiler to combust or co combust with coal which is currently the fuel being used. Mass and energy balances were performed using the data gathered during the energy audit. In the next figure a schematic form of the raw materials and the energy in each step is shown.

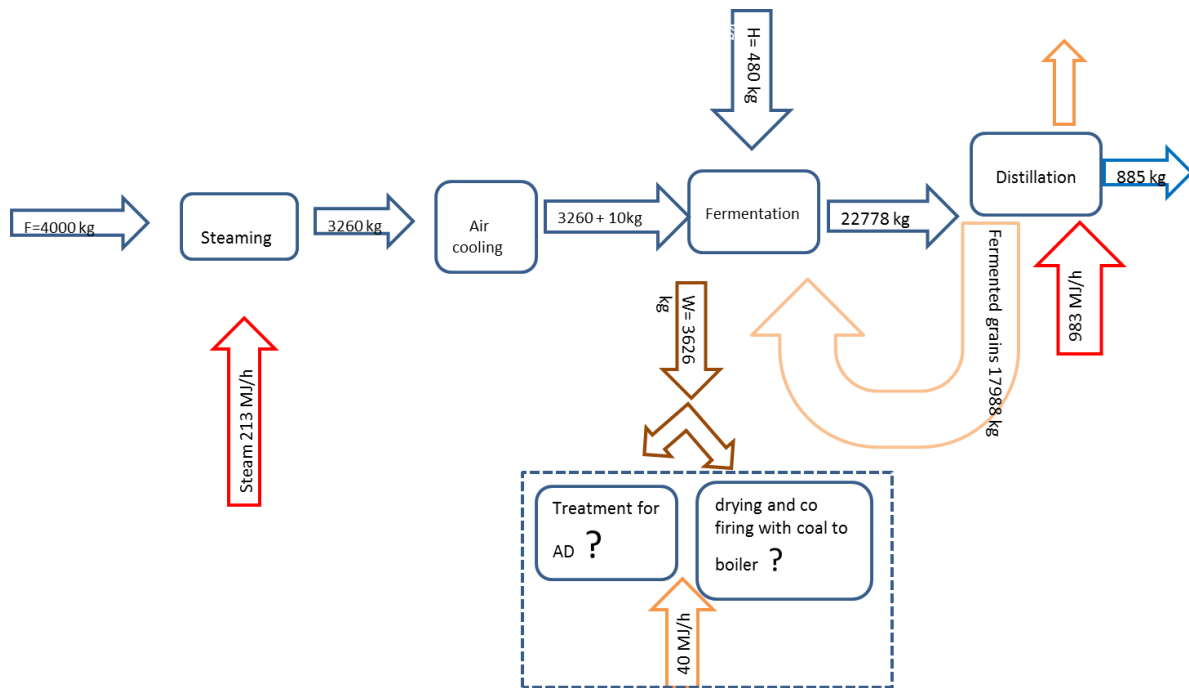


Figure 7-1 Schematic process of one unit of spirit production

First a simulation of the process was also performed as mentioned in chapter 6. This was used to validate the simulation software against the data measured in the site, as the results obtained from it were similar to the data collected and calculated during the audit.

7.2.1 Considerations for the spirit plant simulation

The production process of the spirit plant consists of a batch operation having mainly all of their operations done manually. ECLIPSE, as many other simulation packages does not have a batch platform. In order to adapt the process into a simulation environment, it is assumed that the production is in a continuous process basis, as seen in Table 7.1.

Table 7-1 Feed stream flow rates

Feed stream	mass (kg)	kg/s for 4 hour process
Water	2000.00	0.14
Rice	500.00	0.03
Grains		
Corn	80.00	0.01
Sorghum	1420.00	0.10
Total	4000.00	0.28

The feed stream consisted of the initial mix of grains; sorghum, rice and corn with the addition of 2000 kg of water for a period that lasts 4 hours. These amounts were calculated in kilogram per second (kg/s) considering the time of the process in order to specify the feed streams in ECLIPSE. The same procedure was done to the other operations.

Table 7-2 Stream conditions

Process	Stream name	compounds	flow rate (kg/s)	process temp (°C)	process pressure (bar)
Steaming	G1	rice husks	0.03	22	1
		sorghum	0.1	22	1
		corn	0.01	22	1
		water	0.14	22	1
		water	0.11	127	2
Air cooling	G-W	rice husks	0.03	102	1
		sorghum	0.1	102	1
		corn	0.01	102	1
		air	0.32	15	1
		water	0.06	22	1
Fermentation	CG2	rice husks	0.0005	22	1
		sorghum	0.0024	22	1
		corn	0.0002	22	1
		water	0.1545	22	1
		ethanol	0.1047	22	1
Distillation/condensation	CG6	oxygen	0.0477	22	1
		water	0.0541	80	1
		ethanol	0.1047	80	1

The results from this simulation using the values showed aboved are presented in the next table:

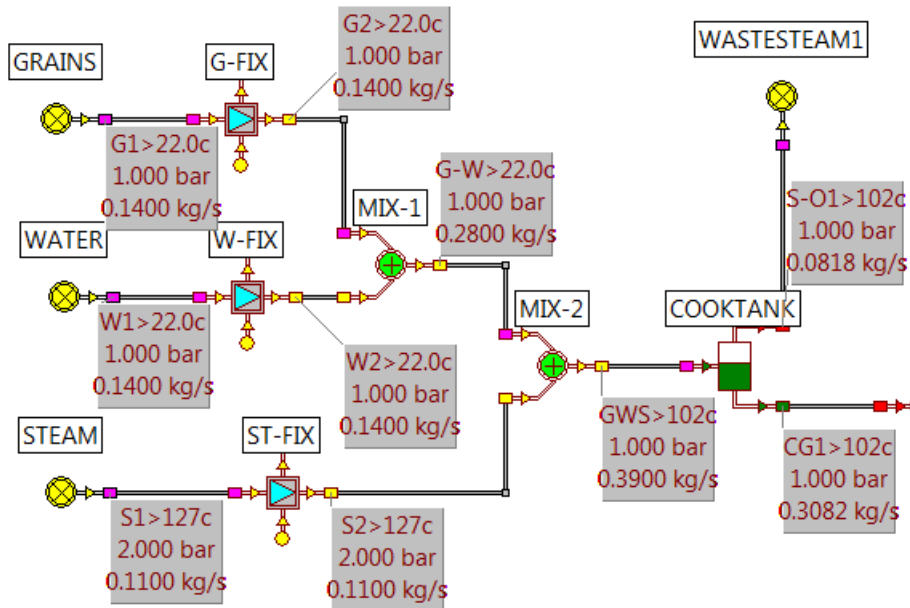


Figure 7-2 Feed streams for the spirit process.

In the Figure 7.2, it can be seen how the production processes are represented in the ECLPSE simulation programme. The feed stream named grains, consisting of the mix of the grains used for the production is mixed with the steam at the operation conditions and flow rates that were given in the audit. Then it is fed into a vessel named COOKTANK to simulate the steaming process. The same was done for the additional operations. The times measured during the process were considered for each step and finally the operation process for a batch was simulated.

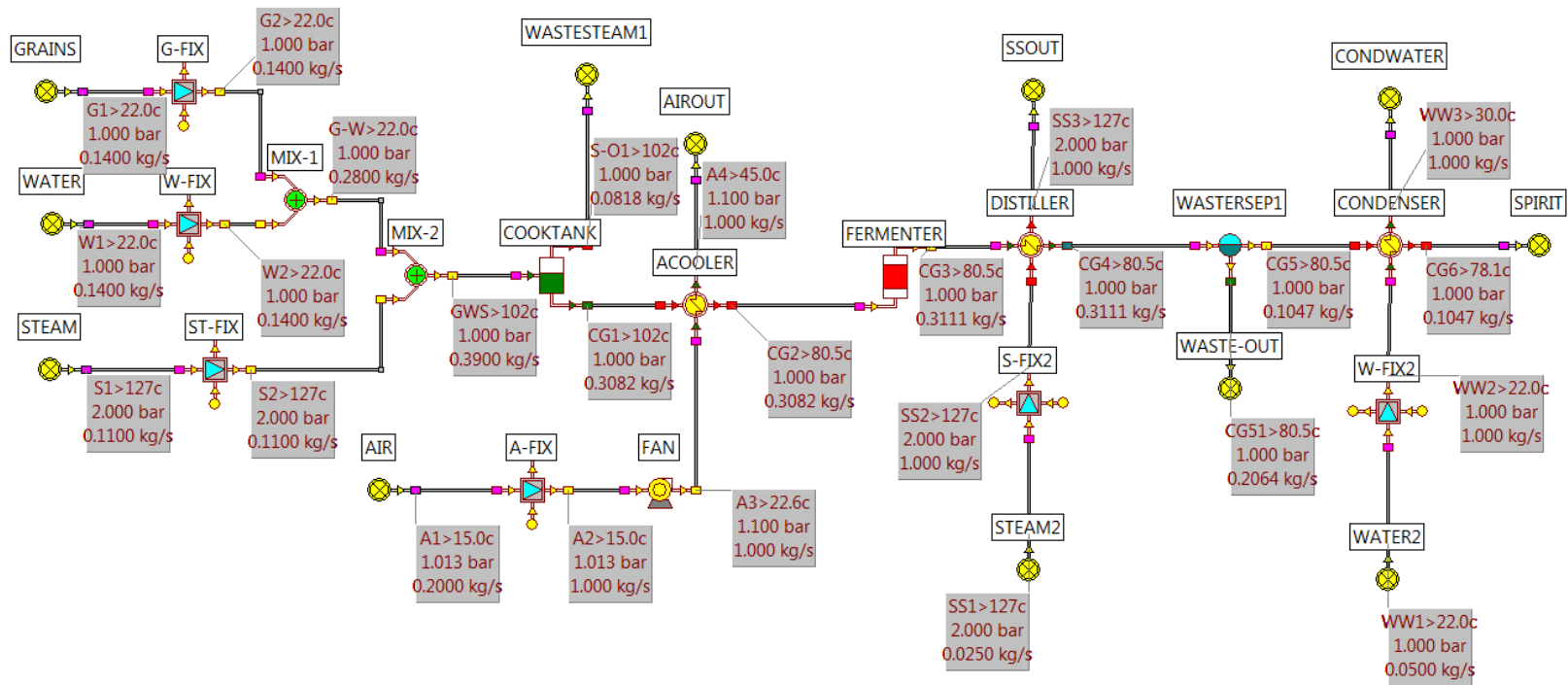


Figure 7-3 ECLIPSE simulation of one spirit production unit.

From the simulation it was possible to obtain similar values for the spirit production and composition. These values were compared with the ones provided by the company. It was possible to know that the basic parameters of the waste characterization in ECLIPSE can give accurate results. This also applies for the process operations and their specification in the model.

Table 7-3 Stream results from spirit simulation.

stream name	G-w	CG1	CG2	CG3	CG5	CG6	
	inlet steaming	outlet steaming	outlet cooler	outlet fermenter	outlet distiller	outlet solid removal	outlet condenser
Phase(%)							
solid	50.0	45.4	45.4	0.9			
liquid	50.0	54.7	54.7	70.0	100.0	100.0	
gas	-	-	-	29.0	-	-	
pressure (Bar)	1.1	1.1	1.1	1.1	1.1	1.1	
Temperature (°C)	15.0	101.6	80.5	80.5	80.5	27.0	
quantity (kg)	4000.0	4320.0	4320.0	1080.0	686.9	686.9	
flow rate(kg/s)	0.3	0.3	0.3	0.3	0.2	0.15	
Composition(%)							
grains	50.0	45.0	45.0	0.9	-	-	
water	50.0	55.0	55.0	49.7	65.0	32.0	
ethanol	-	-	-	33.7	35.0	68.0	
Oxygen	-	-	-	15.3	-	-	
Nitrogen	-	-	-	0.5	-	-	

In the table 7-3 the stream highlighted named CG6 represents the final product when it is collected from the condenser which is the rice spirit. This stream shows that the ECLIPSE simulation was able to produce a very similar result of the actual production in terms of flow rate, 0.159 kg/s (572 kg/h) and the composition of ethanol and water.

The simulation results showed that the spirit produced was 571 kg/h. This is only about 12% different from the figures obtained during the visit which were 600 kg/h (with a distillation process of 65 minutes). This could be because of differences between the batches or a lower conversion rate in the fermentation. Meanwhile for the stream composition it is observed that the ethanol content only differs in 4 % from the data reported. The wastes generated after the fermentation process are 3 616 kg for every

batch. That is 83 600 kg/day of wet distillers grains with. 40 % of moisture. These organic wastes can be used as a renewable fuel to replace the fossil fuel (coal) in the boilers to generate steam for the production processes.

Table 7-4 Spirit stream final composition results from the simulation.

Product from simulation	Stream name	compounds	flow rate kg/batch	composition	data from the plant	composition
Spirit	CG6	water ethanol	571.68	34% 66%	650-700	30% 70%

7.2.2 Results of wastes to heat

Once the simulation of the process was done, then knowing the energy usage and the amount of waste that is generated three scenarios were explored. The scenarios that were simulated and compared are: Current situation using 100% of coal, mixing 50% of the bio waste with Coal for co-firing, use 100% of the bio waste for direct combustion. ECLIPSE was used to perform the simulations, for the simulations using the spent grains it was necessary to include a drying step in order to remove the water prior entering the burner. This step was simulated using the waste heat from the process, and then is fed in to the boiler.

Table 7-5 Elemental analysis fro the coal and the dried spent grains.

Fuel	Coal	Spent Grains
Composition (%)		
Carbon	79.59	49.25
Hydrogen	4.59	7.27
Nitrogen	0.66	5.88
Oxygen	2.66	37.21
HHV	33.15	19.60

On the table above the composition of both substrates studied are presented. It can be noticed that the carbon content represents the biggest difference with the coal presenting a composition of 79.59 % while the spent grains from the plant has 49.25 %. This can also be noticed in the HHV where the spent grains have a lower value in comparison from the coal.

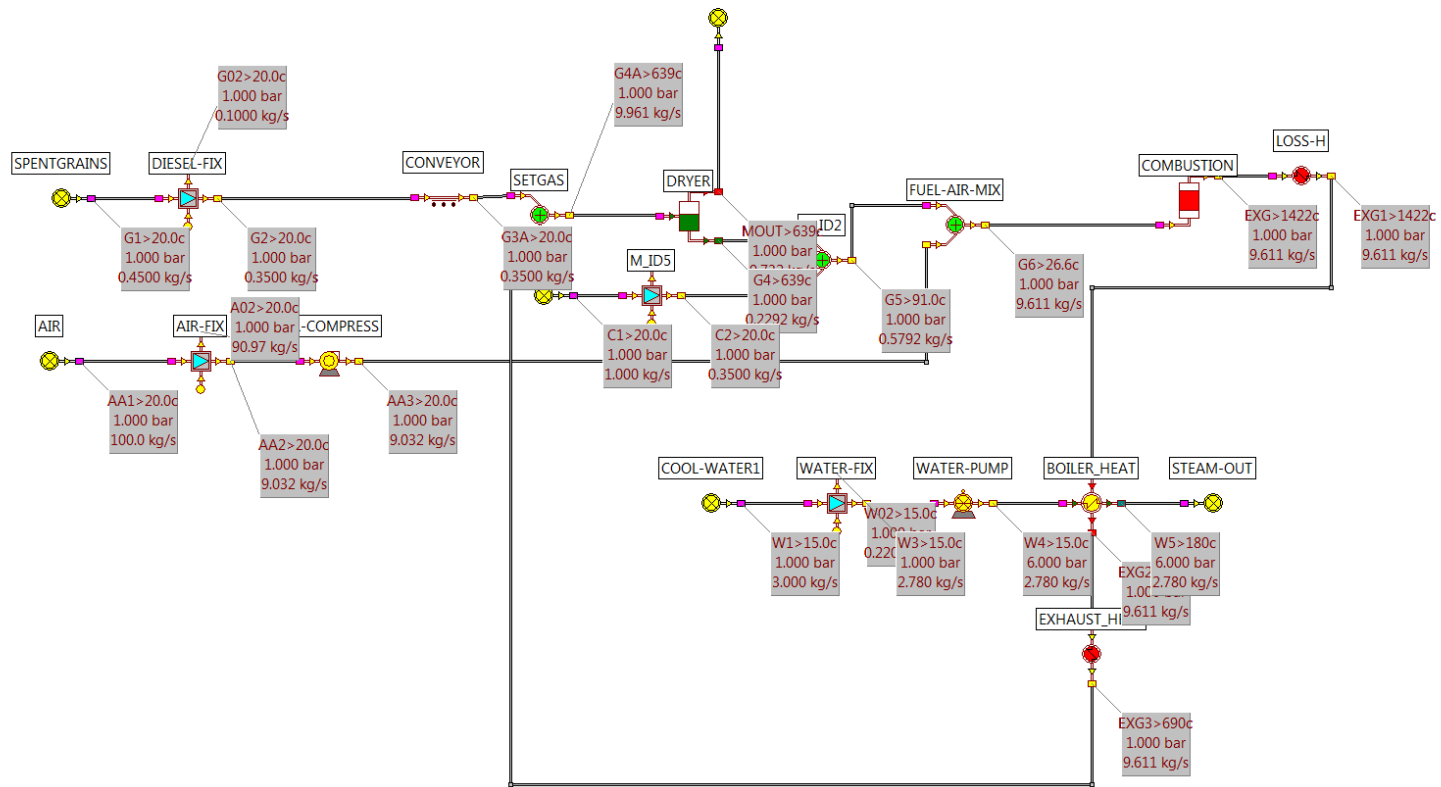


Figure 7-4 Co-firing simulation of spirit waste.

The runs were performed and it was shown that by using solely the dried spent grains the thermal energy requirement was covered, leading to a CO₂ emission reduction.

Table 7-6 Results from the spirit study case simulation

Fuel	Case 1	Case 2	Case 3
Co-firing ratio	0	50 %	100% SG
Fuel input (kg/s)	0.7	0.81	0.92
Coal input (kg/s)	0.7	0.35	0
Total thermal input, kW (HHV)	18 188	18 188	18 188
Gross heat production (steam, kWth)	7972	7972	7972
Boiler efficiency% (HHV)	43.8	43.8	43.8
Temperature	1750	1226	1036
CO ₂ (g/kWh heat)	977.2	800.2	597
CO ₂ (g/kWh heat) (excluding biomass)	977.2	488.6	0
CO ₂ reduction (g/kWhheat) (Relative to the base case)	0	488.6	977.2

Table 7-6 shows a summary of the results from the simulation. This was set to generate the steam to supply the process operations. The base case considered was the current

situation where coal is used. Taking the value of the heating value it was possible to determine the gross heat production which is the actual heat produced by boilers (kWth). That value was set and then it was possible to determine the fuel input necessary to generate that amount of energy in combination with the spent grains for case 2 and for solely the spent grains in the case 3. The three different scenarios can provide the amount of heat needed to produce the steam with the difference for case 2 and 3 on the temperature. It is also important to notice that in this simulation the use of the same installation is considered but improvements on this could increase the efficiency. The table also shows the advances in reducing CO₂ emission in relation to the amount of fuel used and heat produced. It is in this aspect where replacing the coal can bring environmental advantages by reducing the emission in 977.2 g/kWh heat.

The simulations showed that the combustion of the dried spent grains can produce enough energy to generate 2.78 kg/s which is 10 080 kg/h of steam for the process. This figure is very close to the actual values that has been used, 10 000 kg/h. Likewise, co firing the bio waste with coal can be a possibility with both cases showing the reductions of CO₂ emissions by 50% (for half bio-waste) and 100% (for 100% bio waste) respectively. Also the heat from the exhaust gas from the boiler can be used to dry the grains. Therefore no more energy is needed. This will lead to a possible reduction of the fossil fuel consumption. A positive impact in the economic performance of the production could also be achieved, as the amount of fuel to be purchased can be reduced by half or even eliminated.

Both cases of direct combustion of the spent grains and co firing them with coal showed that are suitable to use for the steam generation for the process. The reduction of CO₂ emissions can be achieved, especially using only the bio waste as fuel. This also will lead to a self-sufficient process without the direct dependence on fossil fuels. Further research can be performed in order to evaluate the possibility of electricity generation by a CHP system, for example as well as a full techno-economic analysis.

7.3 Results and discussion of micro - brewery modelling

7.3.1 Brewing process

A similar approach was taken for the brewery. But in this case the scenarios explored involved anaerobic digestion rather than direct combustion or co combustion, due to the nature of the waste. It was possible to simulate a Combined Heat and Power system (CHP) with the results obtained from the experiments. That using the biogas produced could provide thermal energy and electricity to the brewing process

Firstly the brewing process was modelled in ECLIPSE.

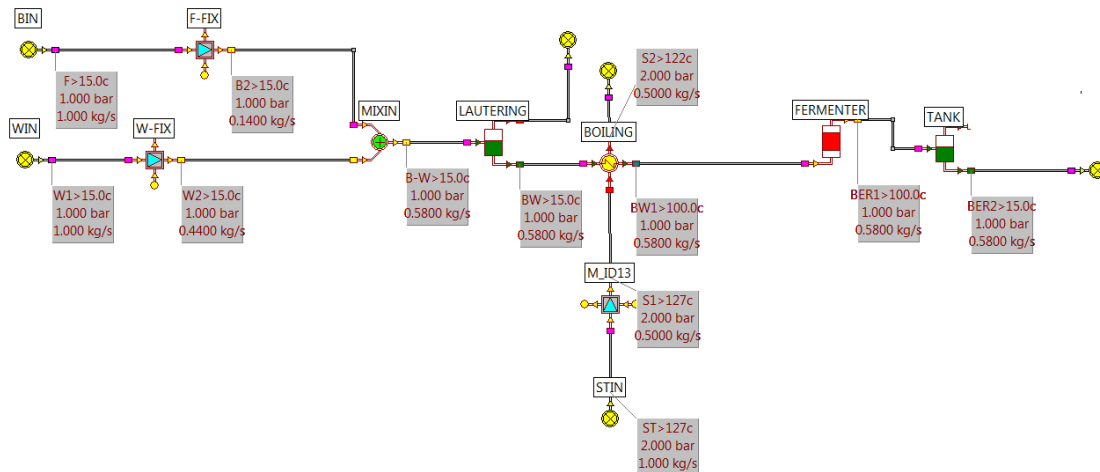


Figure 7-5 Simulation of brewery process.

7.3.2 Biogas used as fuel for boiler to generate steam

The steam that is generated at the site comes from the boiler using diesel as a fuel. In this study the use of biogas as fuel in order to produce steam has been explored. As seen

in Chapter 5, biogas with content of 55% and 65 % (v/v) for mesophilic and thermophilic reactors can be obtained from the waste generated in the process.

The amount of biogas generated is highly dependent on the waste generation. The data from the last 5 years was analysed and used as a reference for this study case. But the estimations from the company are that the production will increase in the next years, which means more bio waste will be produced. With the data obtained at the moment it was possible to perform the simulations. It was noticed that in both scenarios production of steam at 2.5 bar was achieved which could be used later for the process. The biogas that could be generated from the anaerobic digestion was used.

As described in Chapter 5, it was possible to determine experimentally the amount of biogas that can be generated using the wastes from the brewery. This figure accounts for 0.6 L/gVS-day for the mesophilic condition and 0.54 L/gVS-day at thermophilic. Having the yield of biogas per gVS fed daily and the amount of waste grains available. This figure represents around 1, 600 kg per day and has a total solids and volatile solids value of 26% and 96% respectively. This means that around 400 kg of VS a day are available to feed into an anaerobic digester. From this it can be calculated that up to 210 m³/day of biogas can be generated if operated at mesophilic conditions and 188 m³/day at thermophilic. With the later having a higher calorific value due to the higher methane content, 65 (v/v%) against 55(v/v %) for the mesophilic. This will represent a 10 % increase in the energy content of the biogas. With these values a scenario where the biogas is used only as fuel to the boiler was simulated. In the first case the production of only steam is considered. The simulation was run with the input of the biogas generated at both conditions as showed in the table below.

Table 7-7 Summary of the biogas produced in reactors R1 and TR5

	R1	TR5
biogas production (l)	3.00375	2.697
CH ₄ content (%)	55%	65%
CH ₄ production (l)	1.63	1.73
Specific biogas production (l/gVS)	0.60	0.54

The scenario where the biogas is only used as a fuel to a boiler for steam production was simulated using the ECLIPSE. The operations that correspond to the process included a mixer for the air and the biogas, a compressor and a chemical reactor where the combustion reaction took place. In the figure below the model can be observed.

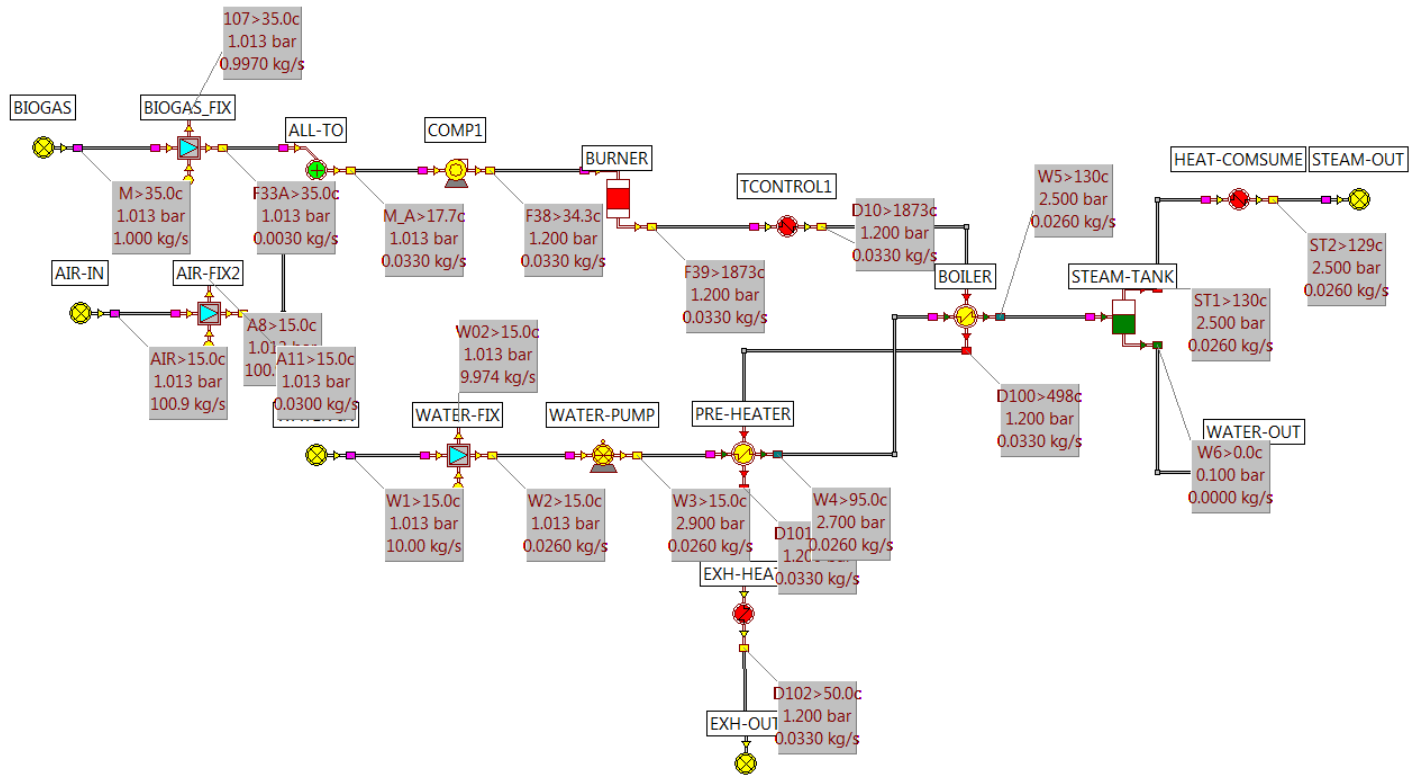


Figure 7-6 Simulation of steam production using bio gas as fuel.

The relevant modules used for this simulation were the PUMPS, a PRE-HEATER and the BURNER. These modules were specified according to the data available of the feeds in order to explore the maximum amount of heat that could be generated after using the biogas as fuel. One stream that was looked closely was the STR6 which represents the steam generated in a boiler. This could be used later for the process replacing part of the steam used that is generated in the boiler.

In order to compare both scenarios the same process flow diagram of the model was used. Then the feed stream was described according to the scenario. The mix of biogas for the mesophilic run was 30 % methane and 70 % carbon dioxide (by mass) as shown in the next figure.

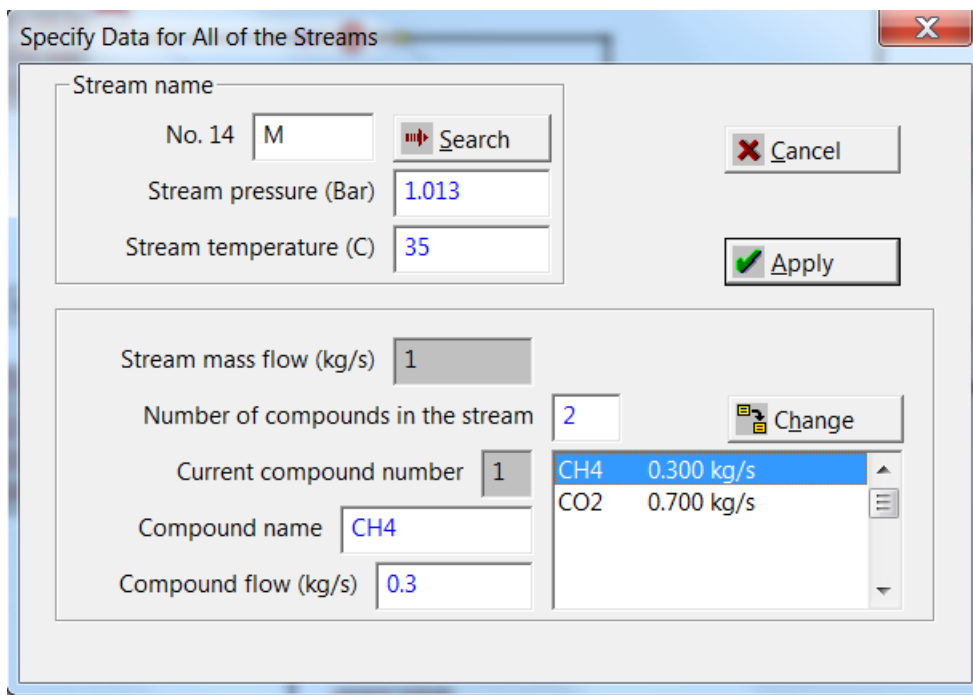


Figure 7-7 Screenshot from the input of the biogas as fuel from the mesophilic production.

The flow rate was also fixed according to the experimental results and using the module BIOGAS-FIX. The rate produced that was converted into ECLIPSE flow rate units was 0.03 kg/s for this case. Whereas for the same simulation but using the biogas produced at 55 °C the feed stream was adjusted to the conditions mentioned above. An adjustment

to the corresponding flow rate was also necessary which resulted slightly lower than the mesophilic being 0.028 kg/s.

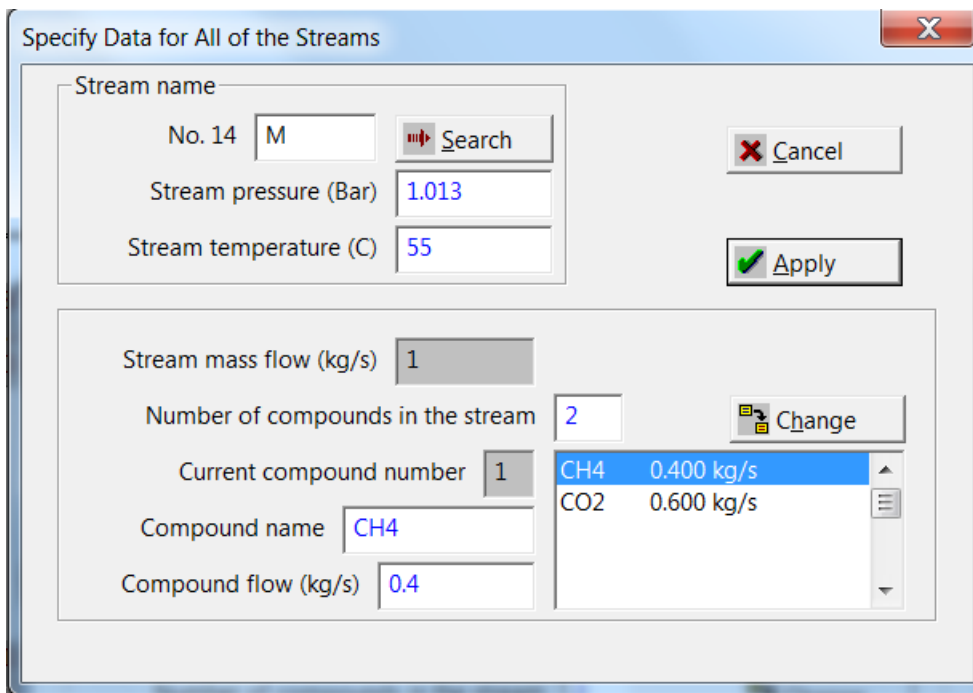


Figure 7-8 Screenshot from the input of the biogas as fuel from the thermophilic production.

In the following table, the data for the steam generated in kg per hour are presented.

Table 7-8 Steam produced when using the biogas from the two different scenarios as fuel.

steam generated at 2.5 bar		
Mesophilic	65	kg/h
Thermophilic	76	kg/h

The second scenario produced up to 16% more steam because of the higher content of methane. Although the flow rate was lower, representing 209.9 MJ/h and 243 MJ/h respectively. This signifies higher energy content available for the process in the same ratio.

7.3.3 Biogas used as fuel for CHP to generate power and heat

One more scenario was simulated. The inclusion of a power generation system was considered. This system included an expander in order to produce electricity forming a CHP system. Yet again, the feed stream of the biogas was the one changed depending on which scenario was being studied, mesophilic or thermophilic. The expander feed is the exhaust gases from the combustion of the biogas. Then the gas expanded with still high temperature, 811 °C is used to heat water and produce steam to the process.

Table 7-9 Electricity output for both cases

	Case 1	Case 2
	Power consumption kW	
Compressor	16.63	16.63
pump	0.105	0.105
Expander	-41.9	-46.8
	-25.165	-30.065

Once again, the second case using the biogas produced from the thermophilic digestion had a better performance in electricity generation for about 20%. This represents that it is able to provide up to 30 kW of load to the process.

Table 7-10 Steam flow generated in the CHP system.

	steam flow	
Mesophilic	46	kg/h
Thermophilic	55	kg/h

In terms of steam generation a similar behaviour is observed, being the case 2 the one producing around 22% more of steam which will represent 42 kW for the process.

7.3.4 Comparison of boiler with CHP using biogas

The biogas produced under the two conditions was compared. Table 7-9 shows that for both cases the biogas produced at thermophilic conditions produces more steam per hour. This represents around 17 % higher production. Although when used in a CHP this amount reduces significantly due to the cause that a proportion of the steam is used to produce electricity which is one of the advantages of the CHP.

Table 7-11 Steam comparison between the boiler and the CHP system.

	Boiler	CHP	
	steam generated at 2.5 bar		
Mesophilic	65	46	kg/h
Thermophilic	76	54	kg/h

The anaerobic digester energy balance was done using the equations described in Chapter 3. The energy balance was done for each of the conditions studied in this study. Then it was compared with the waste heat available from the process. With the data obtained it is possible to perform an energy balance for the anaerobic digestion system. This was carried out following the methodology in Chapter 4. From the experimental results it was concluded that the grains presented a better performance at both conditions yielding a higher methane amount in the biogas. Therefore, the analysis of the digester presented is considering the grains as feed and the comparison of the two conditions.

The thermal energy requirements were given by loss from walls and roof to the ambient at the average temperature during the year which is 15 °C. The heat required to bring the system to the digester temperature, 35 °C for mesophilic and 55 °C for thermophilic. An insulated concrete wall for the construction of the digester was considered, which has a heat transfer coefficient in the range of 0.6-0.8 W/m² °C. The calculations were done considering this figure. Since the production plant is in the UK the annual energy requirements were also considered based on the annual temperatures of the area.

Table 7-12 Energy balance for the Anaerobic digester

		Mesophilic Reactor	Thermophilic reactor
Brewers Waste			
Amount of			
brewers waste	kg	1600	1600
Water content		0.75	0.75
Total Solids		0.25	0.25
volatile solids		0.96	0.96
kg of VS of grains		360	360
Slurry			
Amount of slurry		4500	4500
Water content		0.78	0.78
required water			
content		0.85	0.85
Additional			
Water		3100	3100
total volume		9100	9100
Inlet temperature		15	15
temperature			
desired		35	55
cp Water		4.2	4.2
Heat demand			
Overall Heat			
demand	kJ	260,400.0	520,800.00
	kJ/h	10,850.00	21,700.00
thermal power	kWh	72.91	145.82

As it was expected a thermophilic operation requires more energy to run because of a higher temperature is required. In Table 7-10 it can be seen that this accounts for a total thermal power requirement of 145.82 kWh. However the mesophilic operation only necessitates 72.91 kWh. Nevertheless, as mentioned in Chapter 3, after doing the energy balances heat losses were identified and they could potentially provide the thermal energy for the anaerobic digestion process.

The major heat loss in the process can be found at the Copper while boiling the wort. In this process approximately 100 kg of wort is lost through evaporation and at the moment it is not recovered. This waste heat could be used to provide the energy necessary to run the anaerobic digestion at both conditions. This means that the extra amount of energy required for the thermophilic operation could come from the waste heat from the process. This will mean a better operation in terms of energy use, as the improvement in using the thermophilic system will not represent any additional energy demand.

Table 7-13 Energy losses in the copper for the brewing process.

Copper	kJ	kWh	kW	kJ/h
Heat loss from sides		0.75	0.75	
Energy loss in phase change	226,000.00	62.78	22.83	188,333.33
Heat loss from sides during boiling		1.11	0.63	

The possibility of using this biogas as fuel will permit the reduction of fossil fuels. As for case 1 where only steam is generated in both cases there is enough thermal energy available.

7.4 Conclusions

After the execution of the experimental work with the results obtained it was possible to explore scenarios for both cases in order to use the wastes that are generated and currently given away. This work helps to understand that within the industrial sector a sustainable industry could be achieved. This can be relevant especially in the food and drink industry due to the nature of their processes. It may be the case that not all of them will be able to cut the use of fossil fuels, but by reutilizing the waste can significantly improve their energy usage. It was also possible to show that the bio waste could be revalorized as a fuel. Finally the waste heat that is generated during the process and is usually considered as low grade (below 150 °C) could be utilized to run a Waste to Energy technology.

Chapter 8. Conclusions and recommendations

8.1 Conclusions

This work was carried out in an integral way. It was possible to do some field research with visits and audits to production processes as well as sample collection. It was also possible to do the characterisation of the samples and perform experimental work with them in laboratory scale reactors to determine the feasibility of the biogas production. And finally perform simulations based on the data obtained during the audits and the experiments. From the objectives proposed for the study it was possible to perform:

- Field study and energy audits of the case studies; The two sites were visited and audited in the appropriate levels according to the information that was available.
- Evaluation of the energy requirements for the processes by computational simulation applying the principle of energy and mass balance- Simulations using ECLIPSE software were run and validated in order to evaluate the use of the waste as fuel.
- Laboratory experimental tests: Two sets of experiments were run in batch and with continuous stirred tank reactors in the University in order to find the potential of the wastes from the brewery to produce biogas.
- Simulation of potential technologies to utilise the waste heat in these selected process industries to maximize the usage of energy; Two waste to energy technologies were explored and simulated. The co-firing for the spirit case showed potential to provide energy for the whole process. While anaerobic digestion was studied as an alternative for the brewery covering as much as 60 % of the total thermal energy requirement.

This led to the conclusion that with the current amount of waste generated in the spirit plant in China which at full capacity generates 86 tons/day is possible to generate enough steam by incineration to provide thermal energy to the production process. This can

replace the 9, 291 tons a year which is the current amount of coal that is being used as fuel for the boilers.

Integrating an anaerobic digestion process into the production for the microbrewery could be a good option to increase the energy efficiency in the process. The biogas generated could cover up to 60 % of the energy requirements of the production. This will lead to a more sustainable operation as 1,500 kg of grains and 130 kg of spent hops are discarded in a daily basis. From those wastes up to 350kg of VS for the grains and 25 kg of VS are actually biodegradable and at the moment are treated as waste. Currently they are thrown away but it was demonstrated that they could act as fuel. Two conditions for the anaerobic digestion were studied for this study case. Mesophilic condition with temperate at 35 °C and thermophilic under 55 °C. It was found that at thermophilic conditions the grains could produce biogas with higher content of methane than the mesophilic, 65% and 55% v/v therefore a higher calorific value. In order to achieve the higher operation temperature for the thermophilic process, waste heat could be re used and that way no extra energy is needed to run the digestion system.

In this study a medium scale spirit plant situated in China and a microbrewery in the North East of England were reviewed. Based on the data obtained, energy and mass balances were performed to find the sources of thermal energy. The potential cost effective reduction, and recovery and assess potential uses to lower the fossil fuel consumption and CO₂ emission.

Current and existing research has been mainly focused in either bio waste conversion (e. g. anaerobic digestion)[110]. With most of it focused on the biochemistry and microbiology of the process which will lead to a higher yields in the process. On the other hand the waste heat recovery has been mainly focused on thermodynamic cycles including Organic Rankine Cycles and Heat pumps to recover the heat[13, 111]. In this processes as in the majority of the Food and Drink Industry the temperature of heat requirements are not high. Temperatures of 150 °C are already considered high for the process. Steaming or evaporating/distillation operations are widely used, meaning that the waste heat is of low “quality” and not having many uses. This research looks into the recovery of this low-grade heat to run waste to energy technologies, in order to improve the overall energy consumption in the process.

8.2 Recommendations for future work

This work allowed exploring different options in terms of waste management and low grade heat recovery. Even though a run of experiments was performed during the study it was noticed that there could be improvements in the system. This could maximise the biogas quality and production leading to an even better and more sustainable operation. Understanding the relation between the temperature and the biogas would lead to a better use of both, the waste heat and the biomass. Also, exploring a combination of different wastes could lead to a better operation of the anaerobic digesters. During the experiments two extra reactors were set to run with a mix of waste streams from the brewery showing also good potential to generate biogas. This approach should be also explored as it will contribute to the waste management and can also potential increase the yield of biogas produced. Different combinations can be explored according to the current production. The yeast waste also showed high potential to produce biogas on it's own. Combining either grains or yeast with the hops could improve the performance of them and reduce the overall waste produced by the process.

During the project other waste to energy technologies were considered. One of them was combustion, which is a simple way of reducing and re using the wastes. But there are more technologies that could be studied in order to improve the industrial operation and energy consumption. Doing so, could certainly lead to a more sustainable process. That will be reflected in economic benefits for the industry and in environmental remunerations.

The use of waste to energy technologies has been proved as a potential way of revalorizing organic wastes. Several configurations have been explored but there are still opportunities in combining these technologies in order to maximise the waste reduction and energy generation. In this work the simulations where used to calculate the amount of heat and electricity that could be used from the bio waste studied. This approach is helpful but in the future, optimization models could also be implemented for technologies like anaerobic digestion. This would give a better understanding of the dynamics of the process and the interactions of the different wastes and the microbial communities to produce high quality biogas.

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Apendix A

Preliminary Energy audit for the brewery.

Preliminary Energy Audit

for

Wylam Brewery Ltd.

Wylam, United Kingdom



Prepared by

[Eric Siqueiros]

Report Date: [13 Feb 2016]

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Introduction

Site Visit

Organisation Name: Wylam Brewery Ltd

Site Name & Address: Wylam Brewery Ltd
South Houghton Farm
Heddon on the Wall
Northumberland
NE15 0EZ

Buildings included: Brew hall 1, Brew hall 2, Brew hall 3, Refrigeration room, washing room, warehouse, office.

Dates of Visits: [19 Feb 14]

Energy Advisor: Eric J. Siqueiros

Vist hosted: Mr. John Boyle

Objective

The objective of this Preliminary Energy Audit, to assess the viability of implementing an energy efficiency upgrade of the facility using energy from the biowaste generated and waste heat streams.

This objective will be achieved by:

Identifying a suitable energy performance indicator for existing and target energy use to quantify the potential for energy savings. This also helps assess the impact of the energy conservation measures in achieving this potential and provide a sense-check of calculations.

Identifying a suite of measures, including savings and implementation budget, which together are of sufficient scale and combined payback to create a financially viable project suitable for implementation as a single package of works. Where appropriate, non-energy savings, such as water or maintenance, will also be quantified.

Identifying essential client requirements to be incorporated in the works (such as replacement of windows). Savings and implementation budget figures will be provided.

Identifying other benefits, including renewal of plant which has reached end of life or resolution of comfort issues. These may need to be quantified.

Identifying additional metering and recording requirements, including any environmental conditions that are likely to be required for a baseline should the measurement and verification of savings be necessary. The associated installation budget will be included.

Identify any potential technical, financial or other risks to the project as currently defined.

Description of Site & Scope of Assessment

Wylam brewery is a micro-brewery based in the Northumberland area of the United Kingdom, producing both lagers and ales. The facility currently produces around 340 barrels or 55760 litres per month.

The brewery is located on a farm whose buildings date back to the late 19th century. A refurbishment of the facility was carried out in 2006 which saw the installation of insulation to all roof spaces and the addition of the cold storage area. The building is constructed from stone which is approximately 500 mm thick and is not currently insulated. The building also consists of a pitched slate roof insulated with 100 mm of polystyrene insulation material. Furthermore all of the rooms, except a section of the brew hall housing the copper and mash tanks, consist of suspended ceilings. The ceiling tiles are 12 mm thick and are made from compressed fibre. This is then insulated with 50 mm of rock wool.

Building Name	Floor Area (specify if Gross Internal or Total Useful Floor Area)	Year of Construction (for applicable building regulations)	Comments
Brew Hall (1,2,3)	90.1	Late 19th century	Refurbishment carried out 2006
office	59.7	Late 19th century	Refurbishment carried out 2006
Bottling room	22.3	Late 19th century	Refurbishment carried out 2006

This assessment includes the following network utilities:

Electricity

Natural gas

Energy Consumption

Annual Consumption

. Wylam's Brewery annual energy consumption of fuel oil is 10 m³ per year.

Main Energy Consumers

The main energy consumers at the site that have been quantified for this assessment are summarised in Tables 2 & 3 below.

Table 2: Summary of Primary Electrical Energy Consumers

Electrical Energy Consumer	kWh/day	Comments
boiler	17.8	Fuel oil futon boiler
pumps	8.75	
Heating	91	
Cooling	53	
Office equipment	75	

Table 3: Summary of Primary Thermal Energy Consumers

Thermal Energy Consumer	kWh/day	Comments
Boiler	311	
Heating water tank	242	
Boiling wort	214	

Process

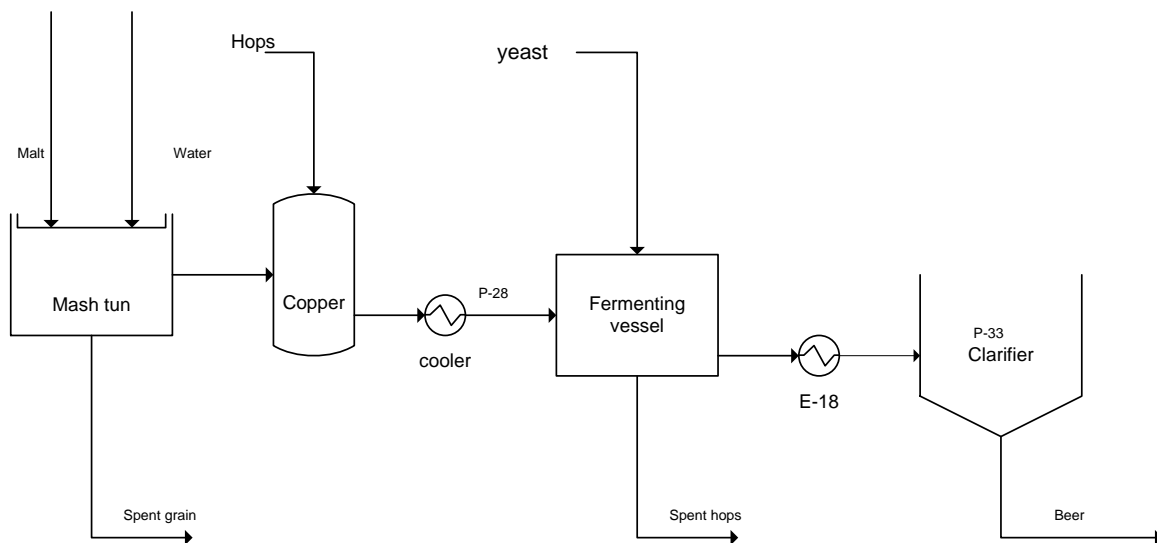
The first step is mashing, which takes place in a stainless steel tank where approximately 1600 L of water previously heated to 70 °C is mixed with 500 kg of malted barley. This process breaks down the starch into simple fermentable sugars to form the mash. It is critical to keep the temperature at 65.5 ± 1 °C during this period in order to achieve the breakdown of the starch and this is achieved by adding more hot water heated with steam.

Next step is boiling, wort is transferred from the mashing tank to the copper. At this stage the spent grain are then scraped out of the tank and discarded. At the moment is given to local farmer where it is later used as animal feed. Once in the copper, the wort is heated up to a 100 °C and brought to boil after the addition of 50 kg of aromatic hops. Approximately 100 L of the wort evaporates to the environment during this time via an externally vented flue.

Wort is then transferred to the fermentation vessels and it's cooled down by cooling water in a heat exchanger. Yeast is then added to the fermentation vessel(s) and the liquid is maintained at 21 °C for the fermentation process to occur. After the copper has cooled, the hops are scraped out and discarded, receiving the same treatment as the grains.

Once the beer has fermented a chiller is used to bring the temperature down to 11 °C to stop the fermentation process. The liquid is then transferred to conditioning tanks in a

cold room held at 10 °C. There it is stored for a number of days, depending of the beer produced, to let the yeast settle and the beer to mature and stabilise. From there the beer is barrelled and stored in a refrigeration room (storage room) for a short period before it is transported.



Process mapping

In order to map the process data was collected during the visit using the following equipment:

Testo 435 Multifunction measuring instrument

Water-proof surface probe with wended measurement tip for smooth surfaces, TC type K

Vane probe, 60 mm diameter, telescopic to max. 910 mm

Pressure dew point probe for measurements in compressed air systems

Lux probe, probe for measuring luminous intensity

Fast-action surface probe with sprung thermocouple strip.

Flir Infrared handheld camera

Operation	Temperature (oC)	Comments
Mashing	70	This operations last 1 hour
Boiling	100	
Fermentation	21	
Cooling	11	
Conditioning	10	

Recommendations for the system

While conducting site surveys of the brewing facility, many opportunities to save energy were identified.

Waste heat from the hot liqueour tank lid

A new, sealed lid which does not allow steam to leak from the tank should be applied. Moreover, a method of recovering the waste heat from the water vapour that is given off during this process could be adopted. A vapour condenser, which condenses the water from a gas to a liquid, could be utilised. The heat from the water vapour would be recovered and the condensed water could be fed back into the HLT, thus reducing the brewery's energy and water consumption. The recovered heat could be used to produce hot water for other uses within the brewery or for space heating.

Heat recovery in wort boiling

The boiling of wort within the CT is the largest demand of thermal energy within the brewery, requiring an average of 214 kWh each day. Therefore, it seems a logical solution to attempt to recover some of the waste heat from this process.

Heat recovery from boiler flue

Another waste heat stream to consider is the flue from the boiler as it can be recovered in order to use it in further stages or to pre heat the water entering the boiler

Insulation of all pipes

Insulation of pipes through the site will minimize the amount of heat that it lost to the ambient, and it is recommended as a good practice.

Insulation of the stone walls

Just like the pipes improving the insulation of the walls for the rooms of the site will minimize the amount of heat that it lost to the ambient, and it is recommended as a good practice.

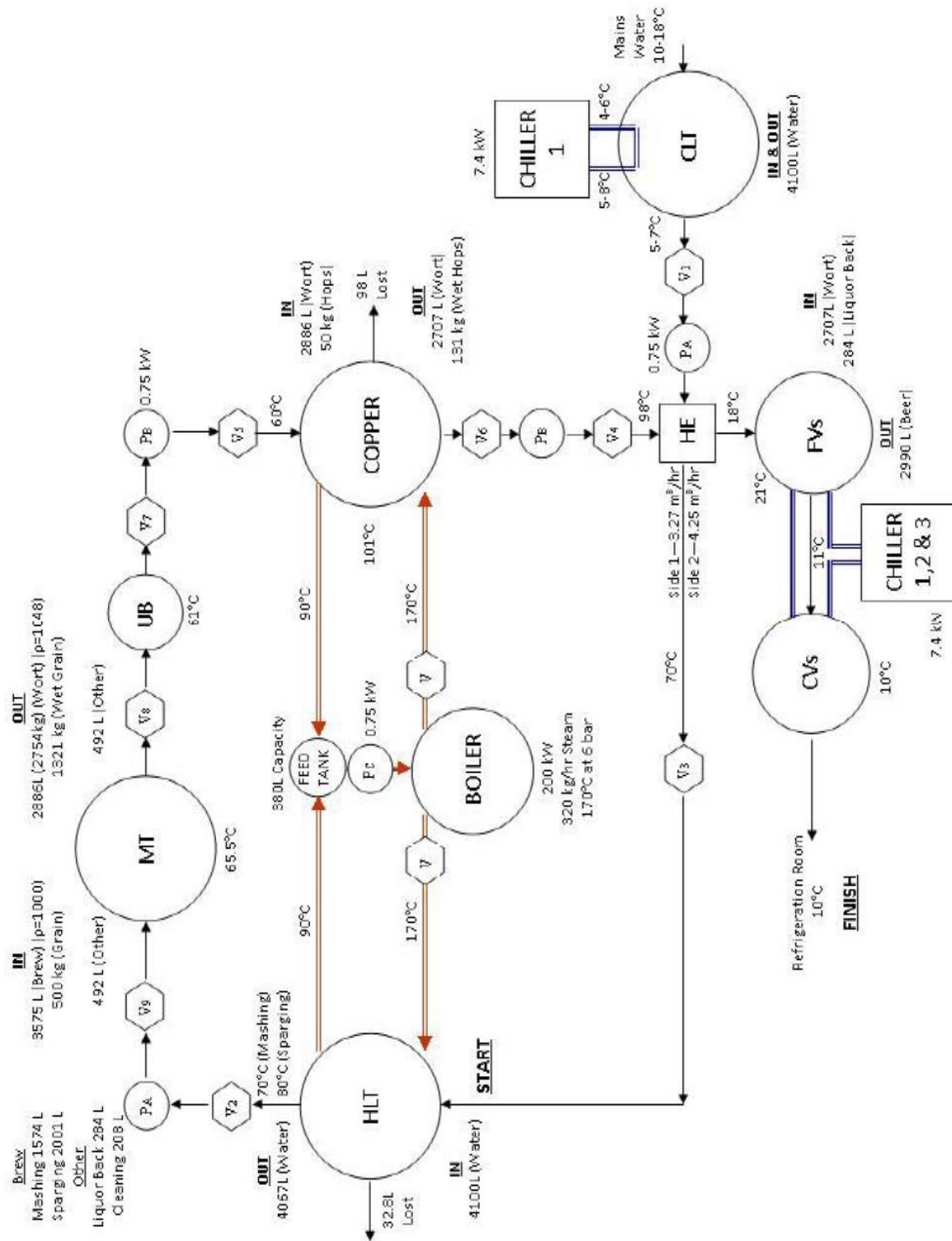


Figure A-1 Brewery layout

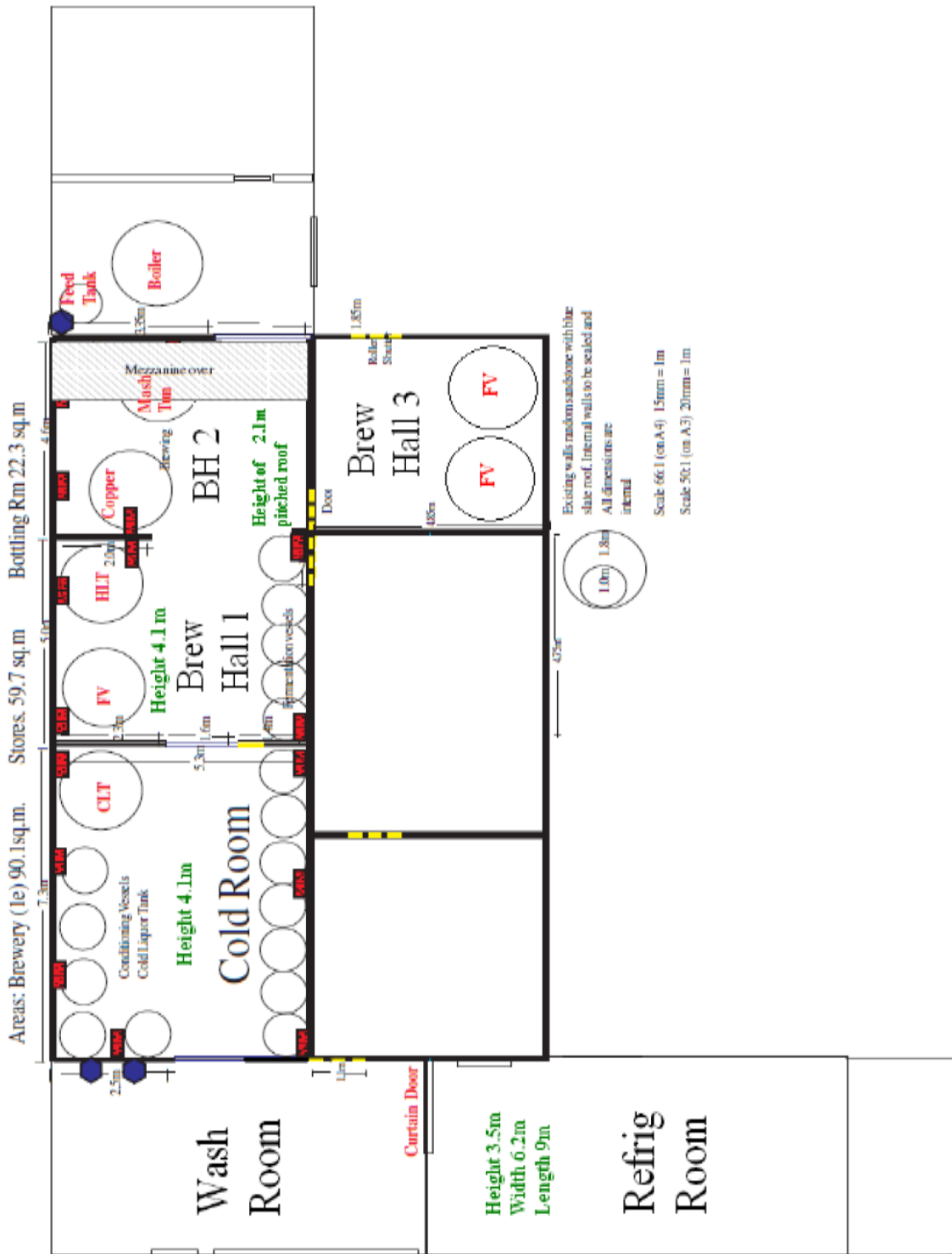


Figure A-2 Brewery production layout

Appendix B

Preliminary Energy audit of the spirit plant

Preliminary Energy Audit

for

JIU GUI JIU Ltd
Hunan, China



Prepared by
[Eric Siqueiros]

Report Date: [13 Sep 2012]

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Introduction

Site Visit

Organisation

JIU GUI JIU Ltd

Name:

Site Name &

JIU GUI JIU Ltd

Address:

ZHENG WU YING JISHOU CITY HUNAN

Buildings included:

Production lines, warehouse, office.

Dates of Visits:

[19 Sep 12]

Energy Advisor:

Eric J. Siqueiros

Visit Hosted by:

Mr. Chen

Objective

The objective of this Preliminary Energy Audit, to assess the viability of implementing an energy efficiency upgrade of the facility using energy from the biowaste generated and waste heat streams.

Description of Site & Scope of Assessment

The Spirit plant is located in the province of Hunan, at Jishou city. It has 2000 employees and an annual turnover of 164, 455 \$/year. The total annual production is 8,900 tons where the rice spirit is the main product. The company production process is a non-continuous batch, having 2 shifts of seven hours a day with total working days through the year of 218.

The production process used is a traditional technique. This is important as there is great concern that any changes during the process may affect the quality of the product which is among the top priorities from the business point of view. The challenge that this represents is big because even though there is interest in improving the energy management in the site, there are limited opportunities within the process as the priority is to achieve the desired final quality of the spirit. There is no cold generation in the plant and for the moment a heat recovery system for the effluent gases from the boiler has not been considered. Currently the inclusion of renewable energies in the process has not been explored, being the coal the only source of fuel for the thermal energy.

8 workshops with 4 production lines each.

Each line has 2 Distillers, an air cooling unit, and 120 fermentation pools.

There are 38 working groups of 12 people each.

This assessment includes the following network utilities:

- Electricity
- Coal

Energy Consumption

Annual Consumption

. The Jiu jiu plants annual energy consumption of fuel oil is 10 m3 per year.

Energy consumption		1	2
Fuels used	-	Coal	Electricity
annual consumption	unit	Tonnes	MWh
	units/year	9291	4480
	MWh / year (LCV)	1170666	4480
fuel price	€/kWh LCV	101.2	0.1049
annual energy cost	€/year	1,012,000	469952

Main Energy Consumers

The main energy consumers at the site that have been quantified for this assessment are summarised in Tables below.

Equipment for heat generation		
Decriptive data		1
Short name of equipment	-	Boiler
Manufacturer	-	
Year of manufacturing or/and installation	-	
Model	-	
Type of equipment	-	
Number of units of the same type	-	3

Technical data		
Nominal power (heat or cold, output)	kW	10 tonnes/hour
Fuel type	-	Coal

Table 2: Summary of Primary Electrical Energy Consumers

Electrical Energy Consumer	kWh/day	Comments
boiler	17.8	Manually operated coal boilers
Cooling	53	
Office equipment	75	

Table 3: Summary of Primary Thermal Energy Consumers

Thermal Energy Consumer	kWh/day	Comments
Boiler	311	
Steaming	242	
Distillation	214	

Process

The first step is the steaming or cooking of the grains. In this step 2000 kg of raw materials containing the grains; sorghum, rice and corn in a 75:25:5 % ratio are poured into an open tank. Then, 2000 kg of water is added at ambient temperature (22 oC) and 360 kg of steam at 2.5 bars and 127 oC pass through the bottom of the container for four hours (90 kg/h) (correction #19, mass flow rate and temperature of steam). The container is a cylindrical concrete open tank with no insulation, causing that all of the steam not absorbed by the material to be lost to the ambient at approximately 100 oC.. There is no waste generation in this first stage.

The next step is to manually put the steamed grains into another pot where air is used for cooling it down to 22 oC. Then distillers yeast is added so the saccharification can begin, and this last for 48 hours.

After that, the fermentation step takes place and it last for 60 days. It is done at ambient temperature, and the grains are covered with a layer of wet clay. In this stage there is a generation of waste of 3, 600 kg comprising wet grains.

Material input for the fermentation:

3260 kg of grains cooled down

480 kg rice husks

10 kg distiller yeast

17, 988 kg of fermented grains discarded from previous batches.

The final step is the distillation. The fermented grains are mixed with husks and manually transferred to the distiller, which is an uninsulated stainless steel tank. The distillation occurs by adding steam to the material, as in the first step, the steam flow is controlled manually to get 80 oC inside the distiller and the ethanol is evaporated. Cooling water is used in the condenser at 18 oC. Material input for the distillation:

22,778 kg fermented grains

The energy input is of 1,620 kg of steam at 2 bars which represents 983 MJ/h and the waste generated 17,988 kg wet distillers grains to re use in next batches.

Once the distillation is finished the final step is to bottle. In the figure below a process flow diagram of the operation is shown.

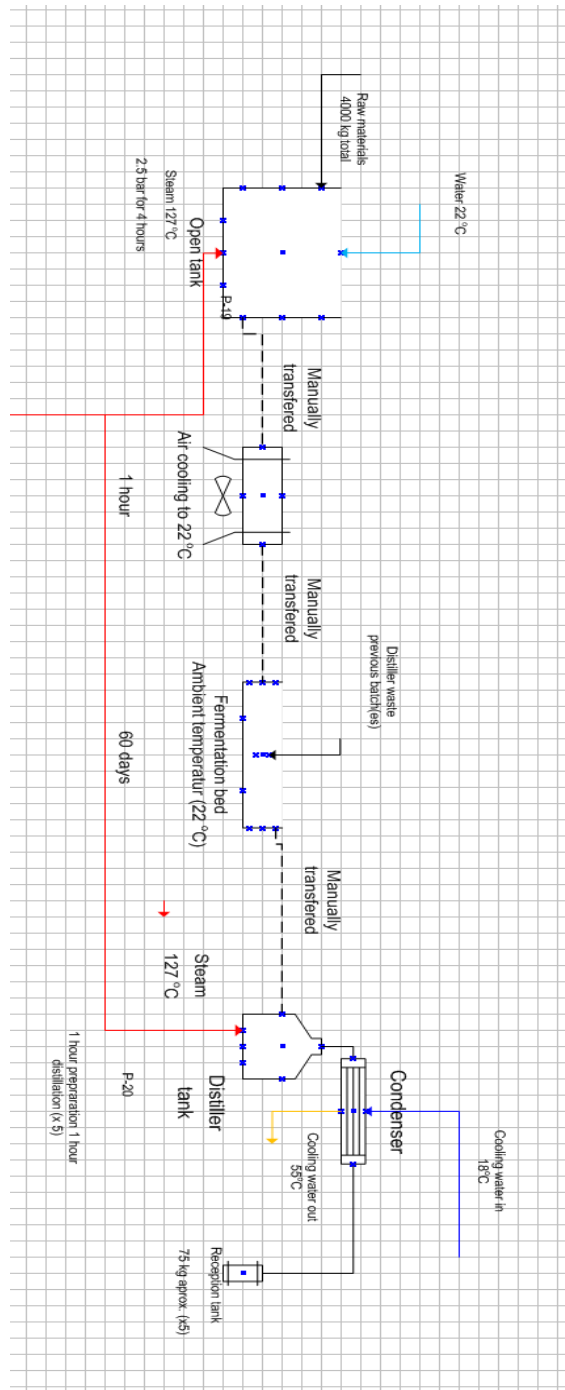
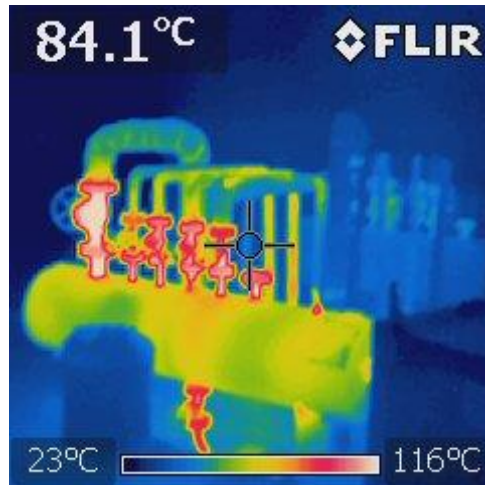
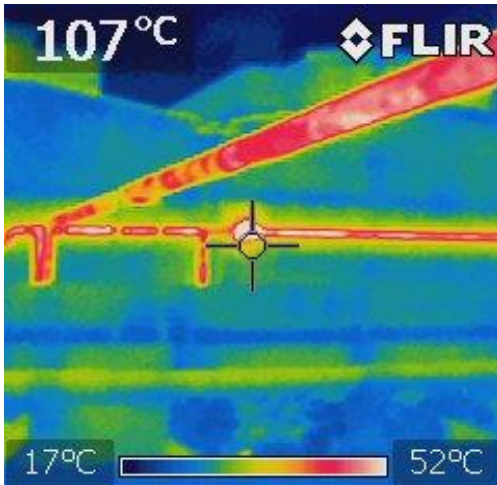


Figure B-1 Process flow diagram for the spirit production

Process mapping

The process mapping was done during the walk through visit. In order to gather all the relevant data from the process measurements were taken as required using the following equipment:

- Testo 435 Multifunction measuring instrument
- Water-proof surface probe with wended measurement tip for smooth surfaces, TC type K
- Vane probe, 60 mm diameter, telescopic to max. 910 mm
- Pressure dew point probe for measurements in compressed air systems
- Lux probe, probe for measuring luminous intensity
- Fast-action surface probe with sprung thermocouple strip.
- Flir Infrared handheld camera



Operation	Temperature (°C)	Comments
Steaming	120	This operations last 1 hour
Cooling	20	This operations last 4-6 hours
Fermentation	20	This operation takes 60 days
Distillation	85	The distillation is done in 1 hour approx.
Condenser	15	

Recommendations for the system

While conducting site surveys of the brewing facility, many opportunities to save energy were identified.

Waste heat from the steaming tank

The steaming tanks are open through the whole operation leading to a steam lost. Covering the tanks could be a solution but also considering recovering the heat available from the steam.

Heat recovery from boiler flue

There are three boilers in the site to produce the steam for the process each of them represents a considerable waste heat stream. Recovering the heat from the flue from the boiler could lead to significant improvement on the energy efficiency.

Insulation of all pipes

Insulation of pipes through the site will minimize the amount of heat that it lost to the ambient, which in many cases due to the size of the plant can be long sections and it is recommended as a good practice.

Apendix C

		A	B	C	D	E	B-A		
		crucible (g)	crucible + sample (g)	dried crucible(g)	dried crucible (g)	dried crucible (g)	g of sample		
	ID	at 550 24 h at °C 15 at 550 °C 104 °C min 5 min					% of TS	%VS	
Grains	1	44.5793	45.5797	44.8306	44.5915	44.5899	1.0004	25%	95%
	2	42.4505	43.4615	42.7096	42.4619	42.4602	1.0110	26%	96%
	3	53.1105	54.1150	53.3615	53.1219	53.1143	1.0045	25%	95%
							25%	95%	
Hops	4	24.0292	25.0391	24.1588	24.0328	24.0305	1.0099	13%	97%
	5	24.0134	25.0457	24.1307	24.0187	24.0135	1.0323	11%	95%
	6	13.7930	14.7945	13.9183	13.7976	13.7969	1.0015	13%	96%
3 ml							12%	96%	
3 ml							10%	84%	
Yeast I	1	44.5815	47.1888	44.9216	44.6162	44.6136	2.6073	13%	91%
	2	46.4860	49.5736	46.8838	46.5256	46.5200	3.0876	13%	91%
	3	21.6334	24.6076	22.0240	21.6831	21.6757	2.9742	13%	89%
5 ml							2.8897	13%	90%

Appendix D

Figure B-1 Quantities for the batch test of the substrates and the sludge

	TS %	VS%	for 0.2 g per bottle		X 3	
Grains *	25.25%	95.40%	0.8304	g	2.491258	g
Hops *	12.24%	96.34%	1.6966	g	5.089744	g
Yeast	13.02%	90.40%	1.6993	ml	5.097836	ml
Spirit	90.98%	8.30%	0.2397	g	0.719185	g

			for 0.6 g per bottle		X 3	
Sludge 35 °C	2.89%	67.68%	30.7012	ml	92.1036	ml
Sludge 45 °C	2.61%	68.01%	33.7393	ml	101.2178	ml
Sludge 55 °C	1.95%	45.82%	67.0259	ml	201.0776	ml

Apendix E

Figure B-1 Batch test conditions for calculations

Day	Date	Time	Gas Volume (ml)	Gas temperature (K)	Air pressure (mbar)	Pressure in gas phase	Vapour pressure of the water
0	11-Jun	11:00	60	308	1023	1013.25	24.9312
1	12-Jun	11:00	60	308	1012	1013.25	24.9312
2	13-Jun	11:00	60	308	1010	1013.25	24.9312
3	14-Jun	11:00	60	308	1017	1013.25	24.9312
4	15-Jun	11:00	60	308	1018	1013.25	24.9312
5	16-Jun	11:00	60	308	1024	1013.25	24.9312
6	17-Jun	11:00	60	308	1019	1013.25	24.9312
7	18-Jun	11:00	60	308	1019	1013.25	24.9312
8	19-Jun	11:00	60	308	1021	1013.25	24.9312
9	20-Jun	11:00	60	308	1019	1013.25	24.9312
10	23-Jun	11:00	60	308	1009	1013.25	24.9312
11	24-Jun	11:00	60	308	1010	1013.25	24.9312
13	25-Jun	11:00	60	308	1011	1013.25	24.9312
14	26-Jun	11:00	140	308	1012	1013.25	24.9312
15	28-Jun	11:00	65	308	1013	1013.25	24.9312
16	1-Jul	11:00	60	308	1015	1013.25	24.9312
17	3-Jul	11:00	60	308	1015	1013.25	24.9312
18	4-Jul	12:00	60	308	1015	1013.25	24.9312

Appendix F

Figure D-1 Gas injections calibration using the GC and Atlas software.

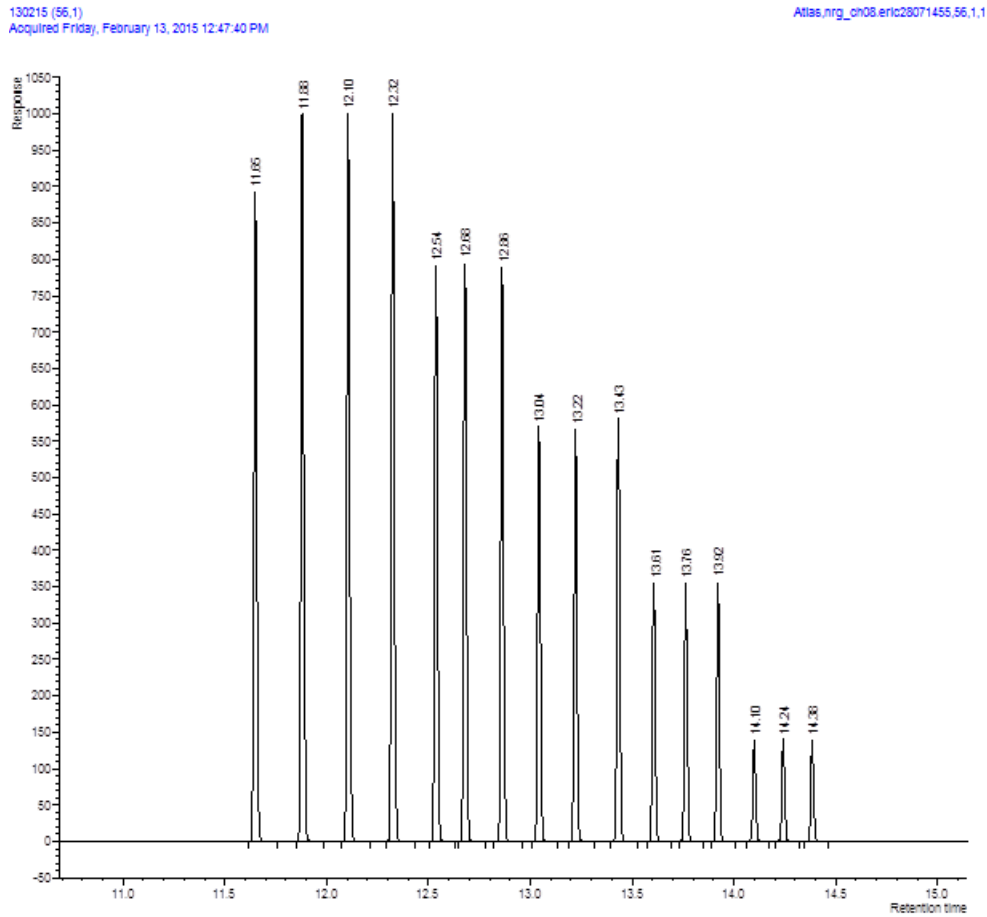


Figure D-2 Calibration injections and reactors injections, both in triplicate using the GC and Atlas software.

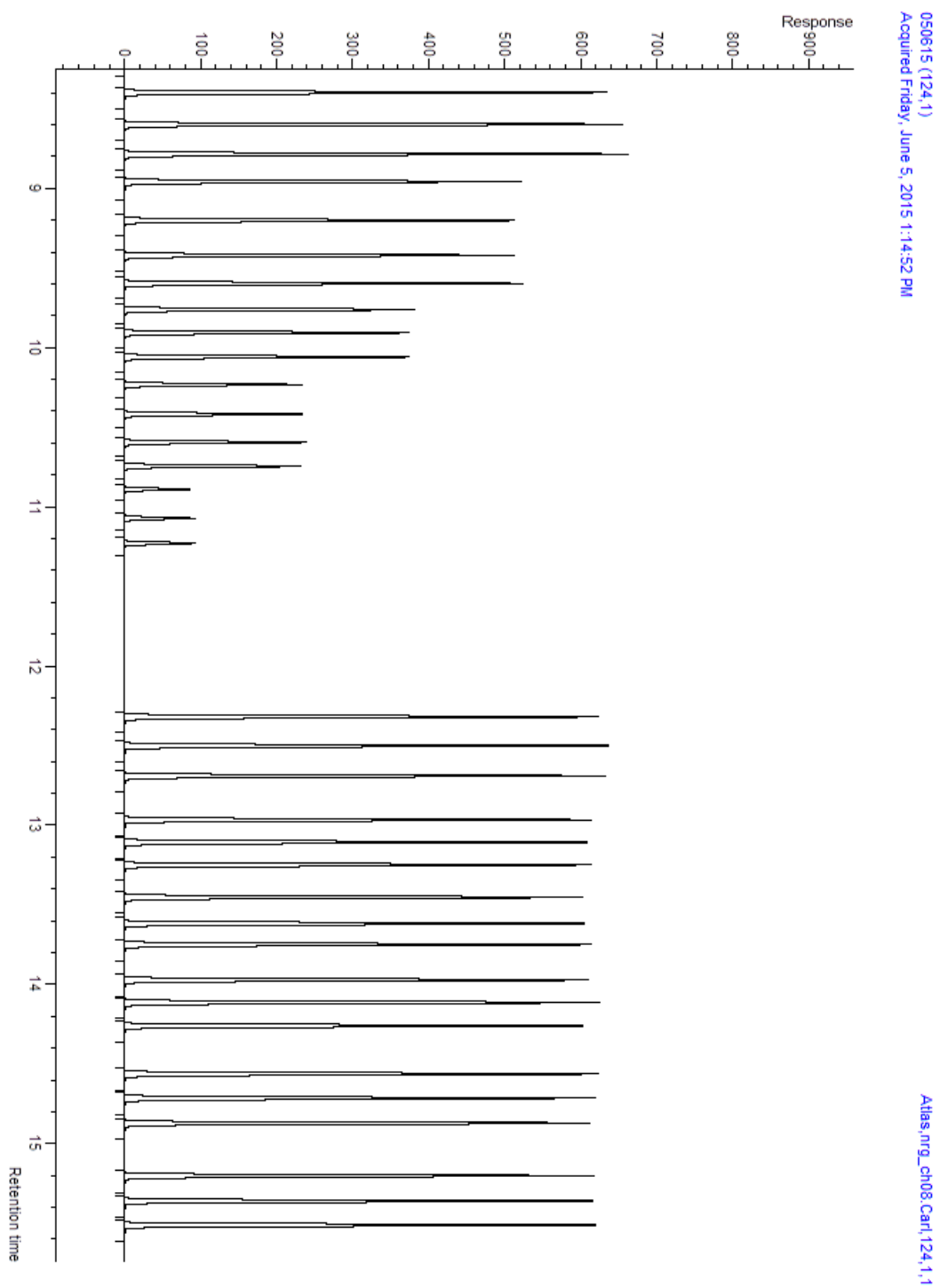


Figure D-3 Calibration curve for gas cilinder with 99.95% of methane.

