Anaerobic Digestion using Mini Reactors.

Validation and optimization of analytical instruments for anaerobic bioprocesses

BScEng Thesis in Chemical Engineering

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Sammanfattning

I vattenreningsverk används anaerob nedbrytning för biologisk stabilisering av slamvattnet. I den anaeroba nedbrytningen skapas metan och koldioxid med andra ord biogas. Biogasen tas tillvara och kan användas som grön energikälla, men produktionen är inte alltid pålitlig i vilka mängder biogas som produceras. För att optimera och utveckla biogasproduktionen används småskaliga biogasreaktorer i laboratoriemiljöer. Fram tills idag så är det främst semikontinuerliga reaktorer som kommer till användning som behöver manuell matning och dataavläsning varje dag. VIVAB i Varberg har investerat i automatiska reaktorer som ska förenkla laborativa undersökningar med mindre spill, automatisk avläsning och ta bort det vardagliga manuella momentet. Målet med arbetet under den här rapporten, har varit att möjliggöra en ersättning av de semikontinuerliga reaktorerna med de optimerade automatiska reaktorerna. Vid uppstart av de automatiska reaktorerna uppkom flera problem, som upptäcktes via höga värden på de organiska fettsyrorna och låg biogasproduktion. Optimering av reaktorutformningen skedde genom följande korrigeringar. Identifiering och eliminering av gasläckor, utbyte av delar av utrustningen och utformning av ett rör som möjliggör utflöde från reaktorn. Reaktorerna var sammankopplade med en PLC controller som via en skärm tillät enkla förändringar av reaktorerna vad gäller matning, temperatur, mixning och kalibrering av gasmätare och flödespump. Resultat från reaktorerna sparades i realtid i PLC controller som registrerade mätdata från reaktorerna varje gång gasmätaren läste av gasproduktion. Gasmätaren uppvisade i några fall fel resultat då gasmätningsutrustningen fastnat och inte läste av biogasproduktionen. För att undvika utelämning av resultat sammankopplades ytterligare en gasmätare, µ-Flow som kunde påvisa uteblivna data och bidra till en pålitlig kalibrering av gasmätaren. De automatiska reaktorerna fungerade efter optimeringen mer eller mindre självgående med undantag för matning av slam var 4–5 dag. Detta innebär en stor minskning i arbetsbelastning vid laborationer och analyser vid optimering och utveckling av biogasproduktion som i efterhand kan appliceras på storskalig biogasproduktion.

Abstract

In wastewater treatment, anaerobic digestion is used in the biological treatment to stabilize the volatile solids. In anaerobic digestion, methane and carbon dioxide are produced, also known as biogas. The biogas is utilized as a green energy source, but production is not always reliable in the quantities of biogas produced. To optimize and develop biogas production, small-scale biogas reactors are used in laboratory environments. Until today it is mainly semi-continuous reactors that have been used for these trials. They need manual feed every day. VIVAB in Varberg has purchased automatic reactors to simplify laboratory investigations with smaller spills, automatic data logging and removal of the everyday manual tasks. The goal of this work has been to enable the replacement of the semi-continuous reactors with the optimized automatic reactors. At the start-up of the automatic reactors, several problems arose, which were detected by irregular values of the organic fatty acids and low biogas production. Optimization took place in the form of sealing of gas leaks, replacement of parts of the equipment and construction of a pipe enabling effluent from the reactor. The reactors were connected to the PLC controller, which via a screen allowed simple changes in feed, temperature, mixing and calibration of gas measurement and feed beam. Results from the reactors were logged in real time in the PLC controller, which logged all possible data from the reactors, each time the gas meter registered gas production. In some cases, errors occurred when the gas measuring equipment got stuck and did not log the amount of biogas produced. To avoid omission of results, another gas meter, μ-Flow, was linked in between the reactor and the gas flow meter to detect missing data and contribute to a reliable calibration of the gas meter. After optimization, the reactors operated self-sufficiently except for sludge feeding every 4-5 days. This contributes to a reduction in workload during development and optimization of biogas production processes.

Acknowledgements

Table of content

Abbrevations

AD	Anaerobic digestion
AnMBR	Anaerobic membrane bioreactors
AT	Anaero Thechnology
CSTR	Continuous stirred tank reactors
OFMSW	Organic fraction of municipal solid waste
OHPA	Obligate hydrogen-producing acetogen
OLR	Organic loading rate
TS	Total solids
STP	Standard conditions for temperature and pressure
VFA	Volatile fatty acids
VS	Volatile solids
WWTP	Wastewater treatment plant

1. Introduction

The environmental issues today are growing, and fossil fuels are a big part of the pollution worldwide. Fossil fuels is used as the predominant source of power around the world and beside the accumulation of greenhouse gas it is also a cause of smog, hazes and acid rain (Seider et al. 2009). As the environmental issues have become more apparent the search for sequestration of carbon dioxide rather than the release of it has become more urgent and as the price of oil quadrupled in three years before 2009 the demand of renewable green resources increased (Seider et al. 2009). During the last year the oil price has however decreased majorly which have had its impact on the green resources. (DN. 2020) Solutions for green energy to keep up with the low price of oil; it becomes cheaper to produce, or the government put higher taxes on the non-renewable resources.

Biomass is used as an energy source around the world and is used for 10% of the energy consumption in the world. When using the term biomass, it includes all biologically produced matter (Seadi et al 2013). The biogas production is under development and for that, laboratories need to be well equipped. An automatization of the anaerobic digestion in laboratory scale is a way towards easier studies, and for that to become realized, the equipment needs to be optimized.

1.1. Wastewater treatment

To produce biogas there are different raw materials that could be used for the process. Wastewater is a resource that is accessible in all cities that has a wastewater treatment for conversion to e.g. drinking water or process water. In the main capital of Sweden (Stockholm) 335 000 m³ wastewater gets collected and treated every 24 hours (Stockholm vatten och avfall, 2020). Organic and inorganic compounds contained in soluble, insoluble liquid, colloidal and particulate forms are part of wastewater (Seider et al., 2009). These compounds are perfect food for the microorganisms in the anaerobic digestion and this is used for biological treatment of wastewater. In the anaerobic process anaerobic membrane bioreactors (AnMBRs) are usually used for the biological treatment. The organic matter is converted to stable end products, which can be separated by physical treatments and generate biogas (Mannucci and Lubello, 2020).

In the wastewater plants (WWTPs) sewage water gets treated and cleaned so the water is safe and uncontaminated to go back into nature. The most common way to stabilize the sludge volatile solids (VS) is by anaerobic digestion which produces methane. The components of the sewage sludge are not predictable as it is different in separate regions and it usually also varies within the region. The complexity of predicting the composition of sewage sludge is a problem in producing an even biogas production (Erdirencelebi and Baygan, 2019). Sewage sludge usually contains dry solids between 5% and 15%. With a substrate with a large amount of dry solids it is hard to pump the substrate and it is not advantageous for the microorganisms because they get a less accessible supply of the substrate. In that case liquid can be added and make the substrate more rendered for the process (Shurner and Jarvis. 2010).

The sludge is usually separated into three types, primary sludge, secondary and tertiary sludge. The primary is the sludge from mechanical wastewater treatment stage, secondary from biological treatment stage and tertiary is from third purification stage, e.g. chemical precipitation (Schnell et al. 2020).

1.2.   Biogas production

The biogas production is done by an anaerobic digestion (AD). The AD is made from contains various organic compounds usually with a high content of sugar, starch, proteins or fats, which are easily decomposed in the AD in the absent of oxygen (Seadi et al 2013). The AD is a bioprocess where several groups of microorganisms are involved in the following fermentative biochemical reactions: Hydrolysis, Acidogenesis, Acetogenesis and Methanogenesis. Together the reactions result in biogas primarily containing methane and carbon dioxide (Mudhoo. 2012).

Hydrolysis is the first step in the AD where aerobic and anaerobic microorganisms degrade cellulosic substrates by hydrolytic microorganisms. So, there will be both microorganisms that can be facultative anaerobes and microorganisms that tolerate anaerobic conditions. Within the process the cellulose and proteins get broken down to monomers (Moran, 2018).

Acidogenesis is the second step, where the monomers are converted to volatile fatty acids (VFAs) by facultative and obligate anaerobic bacteria through fermentation. The fermentation creates these VFAs, water and carbon dioxide from the monomers produced in the first step, hydrolysis (Moran, 2018). If the process contains too many VFAs the buffer capacity of the digester can decrease and this could lead to a pH drop and it is therefore necessary that the next step in the AD process where the VFAs is consumed happens quickly (Deublein and Steinhauser, 2011).

Acetogenesis is the third step where the fatty acids are converted to acetic acid, carbon dioxide and hydrogen by oxidation with acetogenic microorganisms (Moran, 2018). There are two classes of bacteria that produce acetate, hydrolytic and obligate hydrogen-producing acetogen (OHPA) bacteria. The hydrolytic bacteria can produce acetate in the presence of hydrogen that they produce themselves. The OHPA however can only produce acetate if the hydrogen first is removed. The hydrogen pressure will therefore have an important impact on the amount of acetate that gets produced (Aresta et al 2015).

Methanogenesis is the final step of the AD as the methanogenic bacteria convert the acetate to biogas (carbon dioxide and methane). These bacteria are obligate anaerobes which means that oxygen is poison to the organism (Moran, 2018). The production of biogas is not only based on the conversion from acetate, the methanation phase also produces methane by conversion of carbon dioxide and hydrogen. This process is complex and is dependent on the role of the metal enzymes. The last step, the production of biogas is made 70 % by the conversion of acetate and 30 % of hydrogen and carbon dioxide. If the process would only be produced by acetate the product composition would be 50 % methane and 50 % carbon dioxide. The methanation phase generates an increased composition of 65 % methane and 35 % carbon dioxide by using the carbon dioxide for methane production. This has been shown to be dependent on the nutritional requirements of the methanogenic bacteria (Aresta et al 2015).
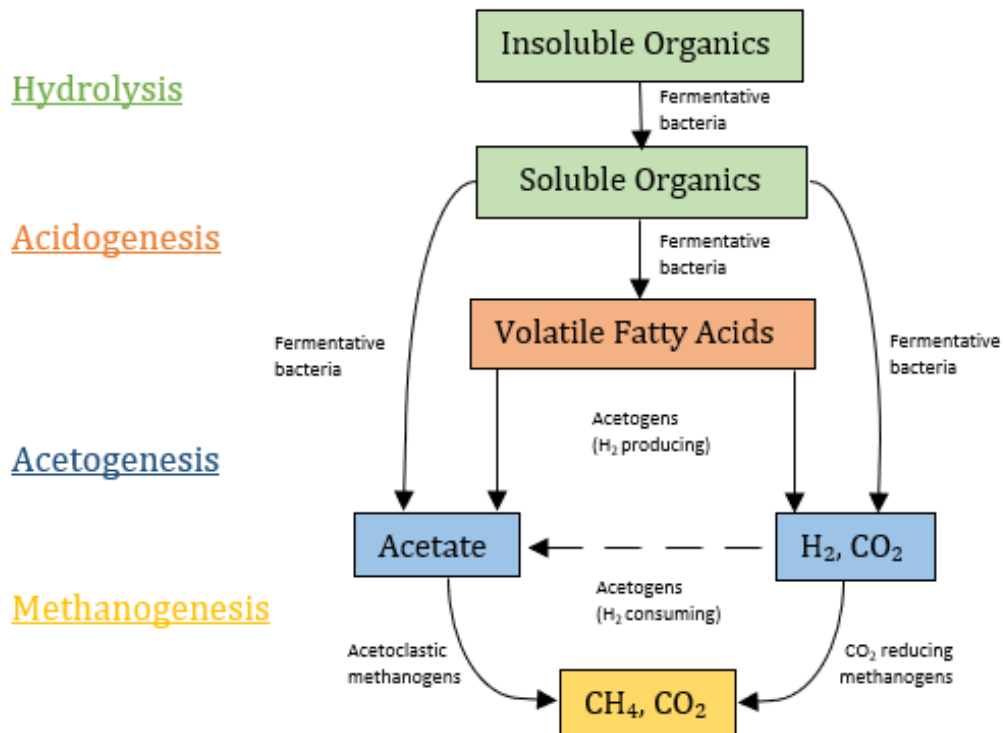
Figure 1. The Anaerobic digestion process. (Ostema, 2004) (Dutton, 2018)

1.3.   The importance of operating parameters

In an AD process it is important that the operating parameters are correct so the bacteria can thrive. There are different bacteria working in the AD and distributed among them there are different processes as mentioned. The processes have their own velocity but to maintain a working velocity for the total process it is important that the subprocess stay in the relation: $k_{hydrolysis} < k_{acidogenesis} < k_{methanogenesis}$. (Aresta et al. 2015).

1.3.1.   pH

The pH is to be stable during the operation, bacteria in the methanogens are sensitive towards a change in the pH. The range that the bacteria can work in is between 6.5-8.5. If the pH extends outside the range the bacteria will stop producing methane and there will be a process failure (Kumar and Samadder. 2020). The acidogenic phase can cause an increase of VFA which leads to a decrease of pH. In the methanogenesis, the bacteria eat the acids to produce the methane. Thereafter the pH increases again and methanogenesis is therefore the rate limiting step. If the analyses from the process show a high value of VFA it can be a sign that the methanogenic bacteria do not function properly (Mudhoo. 2012). To maintain the pH in the process the VFA/alkalinity ratio is observed and is recommended to stay in between 0.07-0.08. Some researchers make other claims, e.g. that a ratio above 0.35 indicates process failure. The VS ratio should therefore be observed so when an irregular VS ratio is discovered, it is possible to take action (Habagil. 2009).

1.3.2. Temperature

The temperature has impact of the key gases in the solution (CO_2, NH_3, and H_2S) by affecting the solubility of the liquid gases (Aresta, Dibenedetto and Dumeignil. 2015). The temperature has the largest influence on the methanogenesis part of the AD because the temperature controls the degradability of the organic material. The AD process is divided into three thermophilic temperature ranges: psychrophilic, mesophilic and thermophilic ($\sim$20 C^o, $\sim$35 C^o and $\sim$55 C^o). The AD process operates mostly in the mesophilic range which could be argued as the temperature where the process is most stable (Kumar and Samadder. 2020).

1.3.3. C/N ratio

The substrate that is used in the AD process needs to contain specific matter for the bacteria to thrive. Two of these matters that needs to be present are carbon and nitrogen that are energy sources and fundamental nutrients. The ratio between the carbon and nitrogen (C/N) indicates how well the bacteria will operate in the substrate. If the C/N ratio is too high the operation risk becoming acidic due to fast degradation in the acidogenesis phase which result in instability in the following process. Because the excess carbon content, the microorganisms has more carbon to consume and will create a slower degradation process (Kumar and Samadder. 2020). If the C/N ratio is low, it shows an excess in ammonia nitrogen which inhibits the anaerobic process. The preferable C/N ratio is most optimal between 15 and 25 but it has also been reported in literature that the process is able to work between 10 to 30. To be certain the bacteria is working ideally, it is preferred to maintain in the optimal ratio (Shurner and Jarvis. 2010).

1.3.4. Organic loading rate

The amount of organic matter is directly linked to the amount of produced biogas and to maintain a continuous amount the organic loading rate (OLR) is calculated and maintained. To calculate the organic matter the dry solids (DS) and volatile solids (VS) are measured in the substrate. The DS is the dry content in the substrate, what is left after liquid evaporation. The VS is the organic component of the DS. From the knowledge of the DS and VS the organic loading rate can be maintained by mixing different substrates (Shurner and Jarvis. 2010). The OLR is as mentioned the amount of organic matter fed during a period and it is determined for a defined reactor volume, commonly expressed in laboratory scale as *g L^{-1} day^{-1}* and in full-scale as *kg m^{-3} day^{-1}* (Habagil, 2019).

1.3.5. Internal mixing

An internal mixing in the reactors provide more equal temperature, nutrients and substrate in the whole reactor. The mixer should be continuous with a motor for best result, and to reduce sedimentation in the bottom and foaming in the top. The hydrolytic microorganisms need to have contact with a variety of molecules and the mixing provides a variation of contact between the organism and the substrate (Shurner and Jarvis. 2010). The mixing should be careful but enough, a sufficient mixing will increase the biogas production as it presents the microorganism with new food and prevents accumulation of the biogas produced (Deublein and Steinhauser, 2011).

1.4. Automatically fed digester system

The anaerobic digestion in this report is done with the automatically fed digester system *Lobster-Max-i* and *Ray/Ray-i* from Anaero Technology (AT). Feeding optimization has earlier mainly been done by manual feeding procedures. In contrast, the equipment from AT uses the automatic Autofed CSTR system to provide a closed system from the feed to the digestate

collection to minimize spills, leaks and provide a flexibility during the operation (Blanco and Patil, 2020). The system enables a precise feedstock that reduce system losses for laboratory study and with its specific measurements make the research applicable to full-scale AD-plants.

The Lobster Max-i is working in a homogeneous temperature and the reactors have their own beam allowing different feeding rates, mixing and substrates to be operating at the same time. All the sets have a gas flow meter that measures the flow in real-time and the gas can be collected. The process pipework has non-return valves that ensure a constant volume without ingress of air.

The automatic feeding system is connected to a PLC controller that allows easy monitoring and also logs the data from the reactor as a tumble occur in the gas flow meter. A tumble occurs when enough gas amount has produced and been collected underneath the tumble-lid. The tumble lid will thereafter release the gas captured underneath the tumble lid and that will cause a tumble. This provides more exact data than the manual data collection that was needed for semi-continuous reactors. In semi-continuous reactors gas production was collected once a day which only provides an average rate.

This information is collected from the operating manual for the Lobster Max-i (Blanco and Patil, 2020).

2. Purpose

The scope of this work was to evaluate the equipment Autofed CSTR from Anero Technology and to investigate the comparability between the continuous and the semi-continuous reactors.

VIVAB has for several years carried out biogas experiments with the aim of optimizing the process in their biogas plants and evaluating various organic substrates with semi-continuous biogas reactors at laboratory scale, purchased from Bioprocess Control AB. Recently, the company has acquired new advanced equipment from Anaero Technology with the intention of conducting continuous anaerobic digestions in the future. These reactors will replace the older semi-continuous reactors previously purchased from Bioprocess Control. The equipment consists of two 5-liter reactors as well as six 2-liter reactors, continuous feed arm and on-line methane flow measurement. Since the equipment has not been in continuous operation before, it needs to be tested, evaluated and optimized for future experimental activities. The goals of this work were to:

- Evaluate the equipment from Anaero Technology and propose optimization measures to increase the performance of the new equipment.

- Write instructions for handling the equipment when performing new experiments in the future.

- Startup the equipment and the process and to record any problems that arise with the equipment during the experiment and document this in a report in English.

2.1. Limitations

The projects duration time was limited to ten weeks, the length of a study period. One trial run for biogas reactors requires more than 22 days. The reactors were placed three reactors at one

feed bench. When arranging the feeding settings, the setup only allows the same setting for the three reactors that are connected.

The project was done during the COVID-19 pandemic which gave limitations in time and allowance of presence in the laboratory.

3. Materials and Methods

The substrates used for the AD process were sludge from wastewater.

The sludge was collected from the municipality of Varberg in Sweden, Getteröverket wastewater treatment (WWTP) that collects sludge from households and food industries. For this examination mixed sludge is used, which is a mixture of the sludge that comes from the biological treatment and mechanical treatment, ca 30/70. The VS of the sludge got determined in a lab by analysis technique SS-EN 12880 and by the knowledge that the VS/TS is stable in the municipality, the VS was known by the calculations of TS. The TS of the substrate vary more and was therefore examined every time new substrate was collected by a Halogen moisture analyzer HG63, and by knowledge of the TS the substrate was diluted to the OLR of 1 g L^{-1} d.

3.1. Semi-continuous reactors

The manually operated semi-continuous reactors were Continuous Stirred-Tank Reactors (CSTR), two 5-litres volume tanks. These were from Bioprocess control AB, Sweden, designed to act as a full-scale fermentation reactor in a laboratory that allow foam observation through the glass.

The tank reactors were opened and filled with 5 liters sludge collected from Getteröverket. The sludge was stirred before poring, to get a representative sample. The reactors where sealed and where connected from a port on the reactor by a tygon tube to a flow measuring device(μ-Flow). To be sure that there were not any leaks a gas warning device EX-TEC snooper 4 from Sewering got used. Were leakage was found clips where used to tighten or the tygon tube was changed for a tube with a smaller diameter. The CSTR reactors have two different 14 mm ports where one is used for feeding, attached to a feeding funnel and one is an outlet of the digested sludge. These ports got attached with a silicon stopper to avoid leakage of gas during the operation. During the operation the ports were closed, and the gas was forced into the 10 mm port and the biogas got registered in the μ-Flow (Habagil et al. 2020).

A daily feed was done after Total solids (TS) was measured and together with the knowledge of organic volatile solids (VS), the organic loading rate (OLR) was maintained at 1 g VS L^{-1} d. The substrate was then diluted with water and added to the 14 mm port. When the port was full, sludge was taken out from the tank with a syringe connected to a tube that was inserted into the outlet and more substrate could be added. This process was continued until all the substrate had been added and then the lids were placed back, and the gas outlet was opened.

3.2. μ-Flow

The μ-Flow is an equipment for small flow measurements with high accuracy and precision that was used to create a linear measurement for the semi-continuous batch reactors. It was monitored continuously with an online instrument. With the instrument ultra-low gas flow is detected and compensation is made for normalization of volume measurement and gas flow rate at one atm and zero degrees Celsius (Bioprocess control, 2020)(Keucken et al, 2018).

3.3.   Lobster-Max-i and Ray/Ray-i

The semi-continuous reactors Lobster-Max-i and Ray/Ray-i was used for the AD operation, two 5-litres reactors(Ray) and six 2-litres(Lobster) reactors were to be prepared for the optimization. The reactors feeder syringe were filled with 5 liter sludge in Ray and 2 liter sludge in the Lobster reactors collected from Getteröverket. The reactors then got monitored by a touchscreen PLC controller where the start time was set after the tumble calibration, and residence time was set to 22 hours. From the feeder syringe, a piston delivers the feed to the digester for a chosen frequency. The feeder syringe is set dependent on the residence time. The internal mixing on and temperature was set to 37,5 degrees Celsius. When starting the reactors, internal mixing was enabled. The feeder syringe got pushed to feed the reactor with a motor that was fastened with a copper-thread. To move the feeder syringe, the copper-thread was withdrawn and by turning the wheel, moving of the feeder beam was possible.
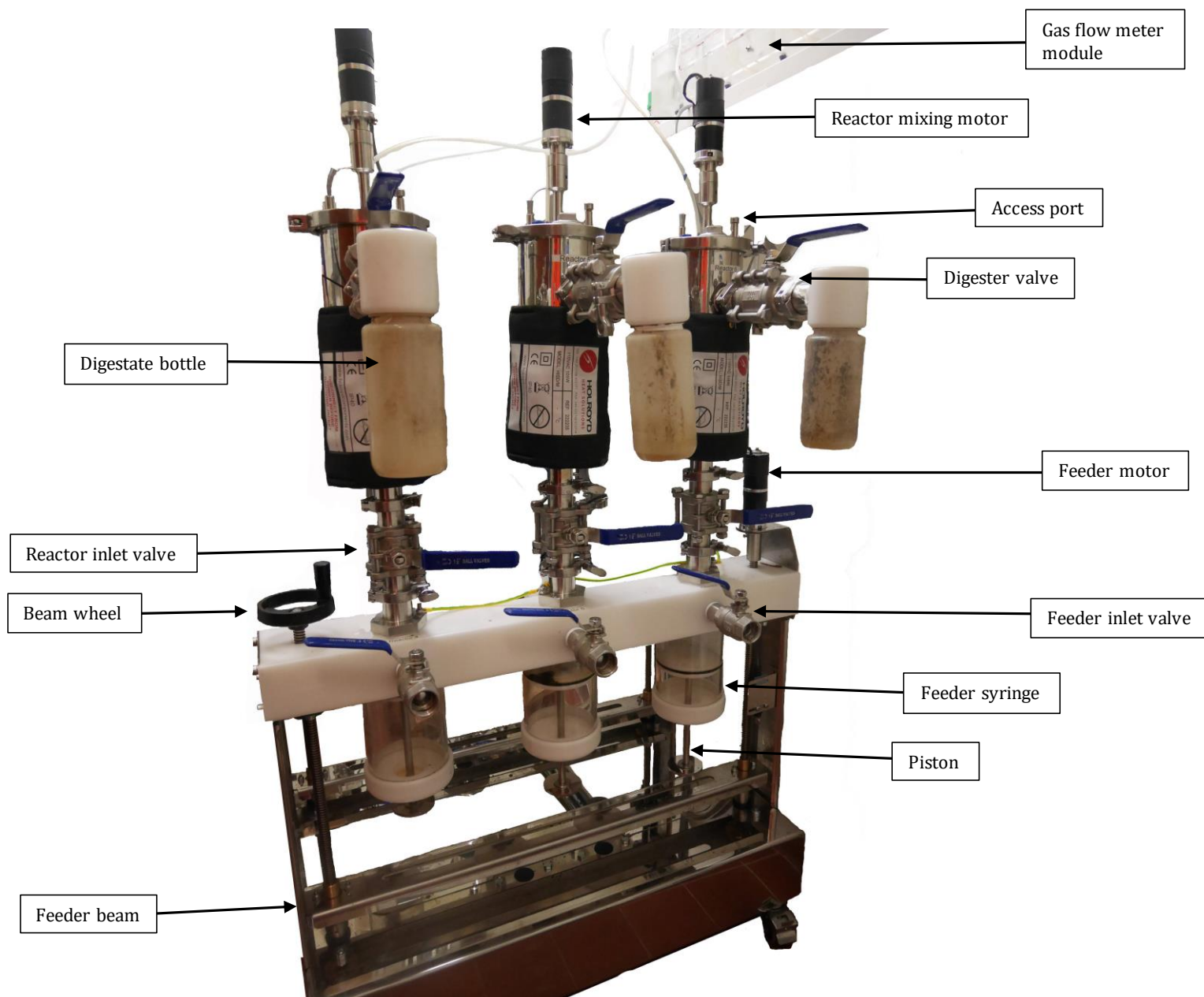
Figure 2. Reactor 2:4, 2:5 and 2:6 on one feed bench connected to a gas flow module.

Before filling the feed-syringe the TS of the new substrates were measured. For five of the reactors (2:1, 2:2, 2:3, 5:1, 5:2) the substrate then got diluted so that the OLR was 1 g VS/L day. Three of the reactors (2:4, 2:5, 2:6) got fed by the undiluted substrate and water separately with a 100 ml syringe connected to a tube that got pressed down into the access port on the top of the reactor. The water addition was made every day.

3.3.1. Feed procedures

When filling the feed-syringe, a check was done first that the reactor inlet valve was shut, and then the Beam wheel was moved to get the beam downwhile the pistons stayed up. The easiest way to fit the copper-thread to fasten the motor was to lower the beam to the bottom and place the thread before filling the syringe. The fresh feed was then fed in the top up syringe, which is

constructed the same way as the feeder syringe. To eliminate air in the top up syringe, the air got pressured out before fitting it to the syringe top up valve. While keeping the valve shut, the top syringe gently got pressed to drive the last air out before tightening the top up syringe. When the air is eliminated, the feeder inlet valve is opened, and the feed got pushed into the feeder syringe. Sludge was pushed in until the syringe piston rod engaged with the beam. The syringe top up valve was then closed and the digester valve opened to able the beam feeding into the reactor. No mixing in the feeders was implemented. This was repeated for each feeder when they were needed to be refilled.

3.3.2. Measurement of gas production

The amount of gas was measured by a gas flow meter (Anaero Technology). It measures the flow in real-time. It also measures the barometric pressure and temperature and converts the volumetric to STP automatically. The measurement is made by counting "tumbles". A tumble occurs when a certain amount of gas is inserted, and the gas is released. The tumble is logged by the PLC controller.

The gas flow meter contains six Perspex tumbling buckets where the spare space is provided for filling liquid in the gas flow meter. It needs to be the same pressure over all for comparable data. When using the gas flow meter, it is necessary to avoid:

- Sagging in gas lines, which can accumulate condensation and increase head pressure in reactors affected.

- Only connecting some Tedlar gas sampling bag. Connection of all cells to gas bags if possible, to avoid pressure differentials between cells.

- To place the reactor higher than the gas flow meter. Ideally the gas flow meter is higher.

- Kinks in gas lines.

- Cooling the reactors because it will create vacuum and draw water from the gas flow meter into the reactor and loose data.

Before starting the measurement, the flow meters needed to be calibrated. For preparation of the gas flow meter, flow hoses were connected to the gas inlet at the bottom of the gas flow meter and connected to a bottle with water. From the bottle a hose was connected to the reactors' gas outlet port. The gas then went through the water before coming to the gas flow meter. When the operation only should measure the amount of methane, the bottle's content would change to sodium hydroxide to eliminate carbon dioxide. The gas flow meter was then filled with water until liquid dropped from the spill point. Calibration was thereafter provided by levelling the gas flow meter with a spirit level. When calibration was done the spill-port was changed to up position. A 150 ml calibration syringe was connected by a hose to gently introduce air to cause a tumble. Six tumbles where made for the calibration of the 5 liters reactors and the milliliters for six tumbles of each reactor got recorded. The value is the quantity of biogas that the buckets could measure per tumble. This was done by following formula:

$$\frac{ml}{tumble} = \frac{Start\ point - End\ point}{N^\underline{o}\ of\ tumbles}$$

When the calibration values where done the calibrated ml/tumble form Table 1 was entered in the PLC software. The amount of gas per tumble then gets registered.

Table 1. Calibration of gas measurement

Reactor	Start point [*ml*]	End point [*ml*]	№ of tumbles	*ml/tumble*
R5:1 (5 liter)	137	24	6	19
R5:2 (5 liter)	149	33	6	19,3
R2:1 (2 liter)	147	29	5	23,6
R2:2 (2 liter)	146	28	5	23,6
R2:3 (2 liter)	133	15	5	23,6
R2:4 (2 liter)	147	27	5	24
R2:5 (2 liter)	123	2	5	24,2
R2:6 (2 liter)	139	16	5	24,6

The data logging and monitoring was collected from the PLC controller and copied to an USB stick. The data was transferred into a pivot diagram to compare the result between the reactors.

Gas hose from a reactor to bottles

Bottles filled with water that the gas go through

6 gas flow meters connected to the reactors

Figure 3. Gas flow meter connected to the reactors.

3.4.   Analytical methods

To investigate the status of the bacteria, samples were taken for determination of the TS, VFA, pH, Alkalinity and VS.

Samples were taken from the reactors and the analyses were made in an accredited laboratory using following methods:

pH: SS-EN ISO 10523
VS: SS-EN 12880
TS: SS-EN 12879
Alkalinity: Svenska medicinal styrelse nr 122
VFA: LCK 365

The tests were made once a week with samples from different reactors. Which reactors that got examined was chosen by the data collected since the last sampling, those who showed the most off numbers from the rest of the reactors were examined. The reactors that are of the same size

and are fed with the same OLR could be expected to give similar result if the gas measurement is consistent.

3.4.1   VFA test (LCK 365)

VFA tests were made after demand, if there were uncertainties observed in the biogas production. A high amount of VFA could be, as mentioned in in the section about pH, early signs of the bacteria not feeling so well.

Sludge was taken out from the reactors that was to be analyzed, a syringe connected to a tube was lowered in the access port of the automatic reactors and ca 200 ml sludge was removed into a vial. From the semi-continuous reactors, the sludge that was taken out of the reactors when refilling was saved, and ca 200 ml was stored.

A dilution was made because of the sludge itself is often too dark to get a proper result out of the spectrometer. The vial was shaken well, and 50 g was poured into a 100 ml volumetric flask and filled to 100 ml with water. The volumetric flask was shaken, and 40 ml was poured into a centrifuge tube. The tubes were centrifuged at 8500 rpm for 11 minutes. After 11 minutes the clear phase was filtered through a filter paper.

The cuvette system LCK 365 from Hach-Lange was used by first pipetting 0.4 ml of solution A and 0.4 ml of the filtered sludge sample. Two cuvettes of every sample were prepared, one blank and one for analysis. The cuvettes were thereafter mixed gently and put in the dry thermostat at 100 ^{o}C for 10 minutes and then cooled to room temperature for 30 minutes. When the cuvettes had cooled to room temperature 0.4 ml from solution B, 0.4 ml of solution C and 2 ml of solution D were added to the non blanc cuvette. They were gently mixed, put into a DR6000 Hash-Lange spectrophotometer and the VFA value was noted.

4. Result and Discussion

The aim of the report was to examine the automatic reactors from Anaero Technology by comparing them to the already optimized semi continuous reactors from Bioprocess control. For comparison the reactors needed to have the same operation factors and they needed go get set up at the same time.

4.1. Troubleshooting during startup of automatic reactor trials

The automatic reactors were set up and filled with sludge and the PLC controller logged the information from the reactors, but some changes were added to the original method.

To get the feed motor running, an overview of the feeder motor was made. The copper thread is there to prevent the motor from overloading and the copper thread will break before the motor does. In an earlier trial, the motor overloaded so the copper thread broke and got stuck. To get the motor going again it was disassembled, cleansed and put together. The disassemble of the motor is very time consuming. Therefore, if the motor overloads many times during an operation, it will interfere with the trial and increase the workload. To prevent workload only 2 of 4 screws where put back in case the motor would overload. The rest of the screws where arranged after the reactors been running with one loading rate for 2 weeks. Because of earlier overload the feeding rate was chosen to be at a more frequent paste. The overload could have happened as a result of dried substrate in the reactor inlet valve and a feed with shorter amount of time in between was concluded as a possible solution.
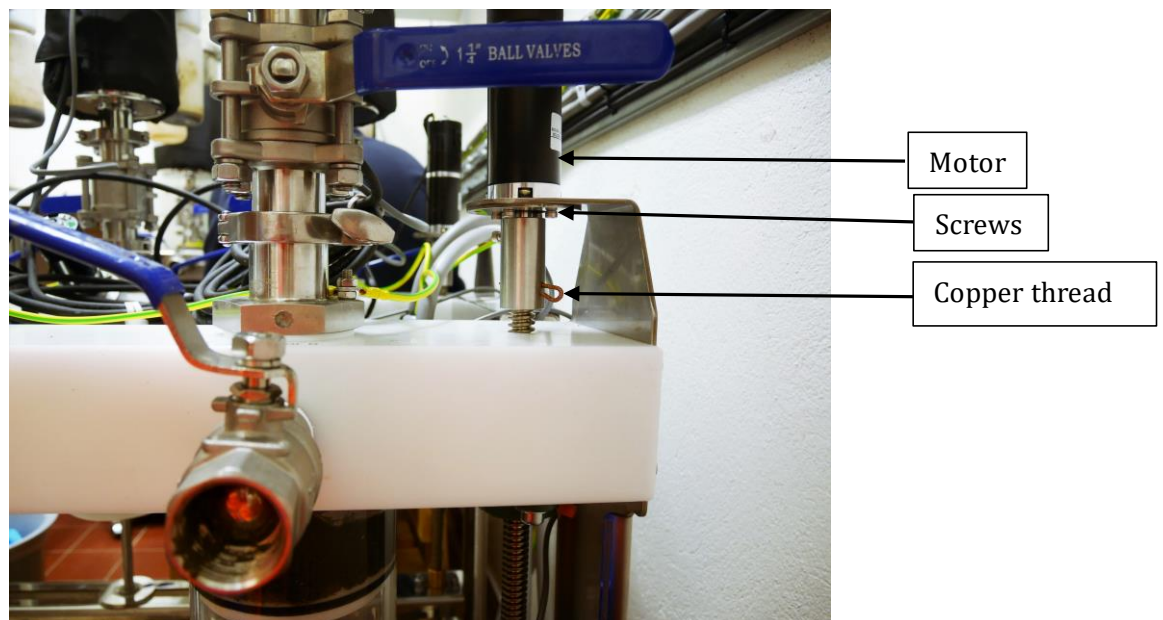
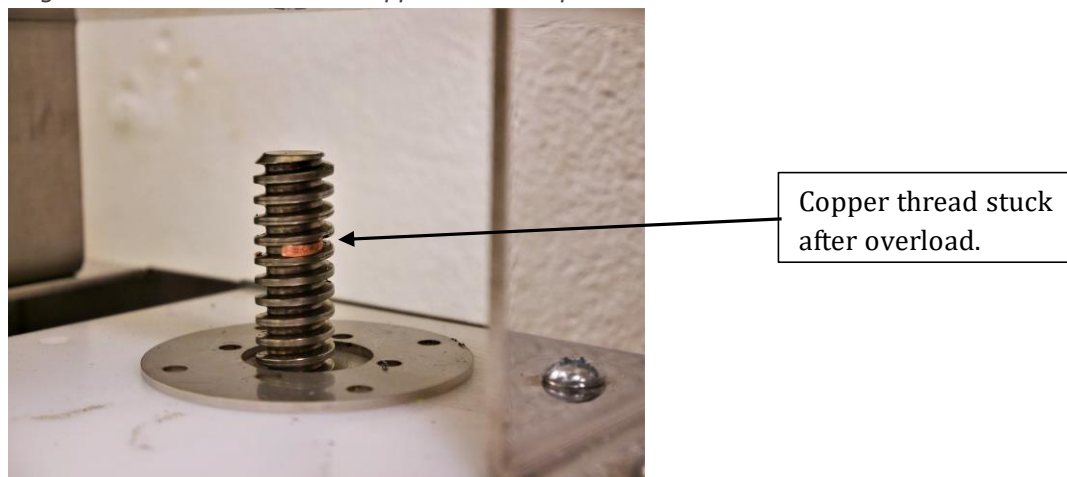*Figure 4. Feeder motor with copper thread in place.*

Copper thread stuck after overload.

Figure 5. Shaft after dissembled the motor

After the reactors have operated for two days the gas measurement had yet not given any result. This could have happened because of a gas leak. That was investigated with a gas warning device EX-TEC snooper 4 from Sewering. The lid of the bottle where the gas-tube go into water was showing gas leakage and the lids thereafter were mediately changed to newer ones from Bioprocess control. A gas leak was also found by the outer digestate valve, so a decision was made for the valve to be closed during the operation except for emptying of the reactors. When the outer digestate valve was closed it provides fresh outgoing sludge when the handle is opened. Therefor having a closed digestate valve enables fresh sampling.

At day 4, the substrate in the feeder syringe to the R:2 reactor was sedimented, see Figure 6. The feeder mixer was then enabled and set to a period of 20 seconds before every feed. After one more week R:5 also showed sedimentation in the feeder syringe so the mixing was started on all of the reactors, set to 40 seconds.

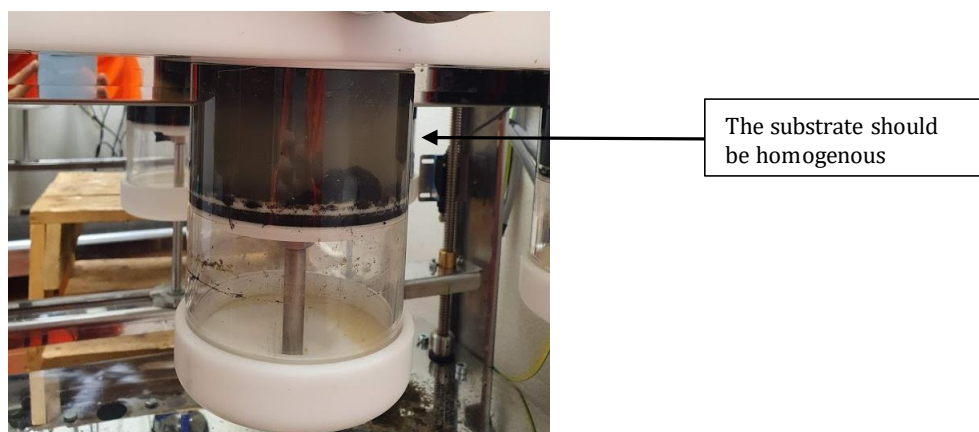
The substrate should be homogenous

Figure 6. Feeder syringe with sedimented sludge

At day 6, the Lobster Max-i PLC controllers did not log any information and the reason turned out to be that the SD card (digital memory card) stopped to work. To compensate for the loss of

data, an addition of manual gas measurement was made in the same way as for the semi continuous reactors. A new card was purchased, and the data could be collected again, but the manual measurement was continued if further problems would occur.

The outlet of the sludge did not work as expected. When the digester valve was opened only water-like liquid slowly poured out containing no sludge, which was a problem. The same amount of sludge that is fed into the reactor must also be removed. The volume of sludge that the reactors contain should always be the same, 5 liter or 2 liter dependent on reactor volume. Opening up the digestate valve on R2:4 showed that no sludge was stuck in the valve itself, so blocked valves could not explain the problem.

After 14 days a small amount of sludge had been obtained on some days by opening the digester valve every day. The 14:th day the sludge had filled the gas hoses from the reactor 5:1 and 5:2 because of the reactors being overfull. The valve was then opened as all the days before, but this time sludge poured out from all the reactors. It seemed as a siphon was created when the sludge started to flow. The reactors poured sludge from the digester valve until they all were done, and the digester valve was closed again to not waste any biogas. The hose, bottles and gas flow meter were cleaned and the gas flow meter also was calibrated again.
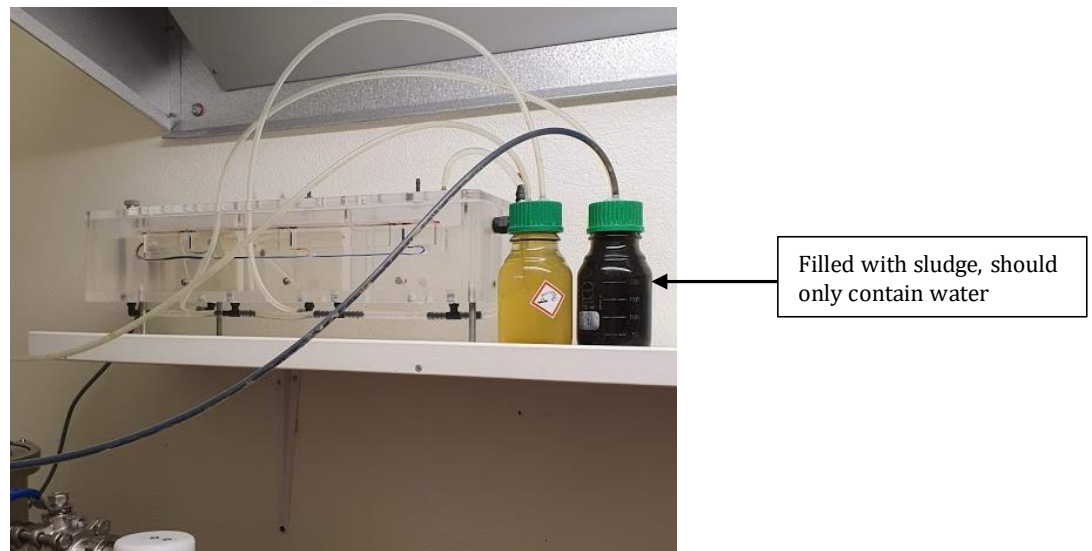

Filled with sludge, should only contain water

Figure 7. Gas flow meter with gas hoses from the reactor and water bottles filled with sludge from the reactor.

The overflow from the reactors happened again with a week in between. The need of emptying the reactors was discovered due to low biogas production, that also occurred the day before the first time the reactors where overfull.

When comparing the gas production from the automatic reactors, a difference between them was observed. They should produce the same amount due to same volume, content and feed. Therefore, one µ-Flow was exchanged with the bottle connected to R5:1 to compare the gas production results. This was done for one reactor for 2-3 days before starting the same procedure for another reactor. The checkup was made for R5:1, R5:2, R2:3 and R2:5. The new logged gas production was calculated to *ml/tumble* and the average value put into the PLC controller. The calculation was done by following formula:

$$\frac{Gas\ amount\ from\ \text{μ-Flow}}{Gas\ amount\ from\ PLC\ controller} * set\ ml/tumble = New\ ml/tumble$$

The gas leak detector was used again to check if the 5 liters reactors where leaking gas. The check was made because they did not show as high gas production values as the 2 liters when comparing them by ml biogas produced per day and reactor volume. All the reactors still had leakage in between the digestate valve and the digestate bottle. This had been solved by closing the digestate valve and only opening when emptying the overflow sludge. Because of problem with the sludge outlet, changes were needed to make the reactors work. The 5 liters reactors got sealed with silicone grease where the leakage was found, see Figure 8. When there was no more leakage the digester valves were opened so sludge would pour out as the feeder pushed in the new sludge.
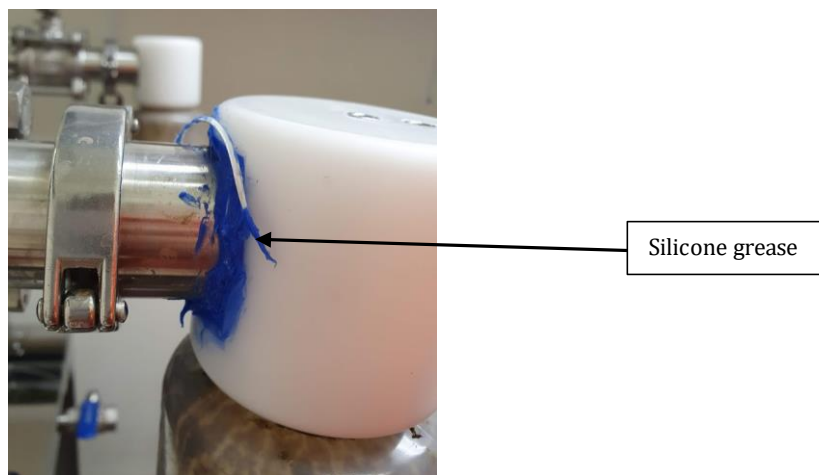

Figure 8. Digester valve and digester bottle with silicone in between to isolate.

The sludge did not pore out as wanted so the digestate valve was removed to eliminate the distance that the sludge needed to pass to get to the bottle. The distance was believed to cause a halt because of the slow rate, the sludge could then dry and create a block. The bottle was fitted directly against the reactor to see if that could be the problem.

Because none of the solutions tried fixed the outlet sludge problem a pipe was made that went from the middle of the reactor and connected to the digester valve. The pipe was put there so the sludge that got pushed out from the reactor was not only foam. The pipe diameter was the biggest possible that fitted in between the wall of the reactor and the mixing module.

Figure 9. Pipe made for the outlet of the sludge.

The pipe created a pressure in the digestate bottle, so no sludge got out. When unscrewing one of the screws in the top and released the pressure sludge got out. At first the right amount got out through the pipe but eventually a siphon was created, and all sludge that was above the pipe outlet was emptied.

After several trials of different solutions new equipment from AT was procured and mounted on the reactors, the new material is shown in Figure 10. The gasket was installed to cover gas leaks from the thread in the bottles. The smaller reactor inlet valve is to create a higher pressure from feeding the sludge and with that cause a greater push of the sludge out from the reactor. The digestate valve is shorter and bent for a shorter flow with less friction. The digestate bottle is thereafter placed lower and makes it easier for the sludge to pour out.

Figure 10. New equipment purchased to solve outlet sludge problem.

The first trial was made for three days. R2:4, R2:5 and R 2:6 were all equipped with the new Digestate valve but only R2:4 got the smaller reactor valve installed. After three days the reactor 2:4 worked but not the R2:5 and R2:6 (Figure 11) These were emptied so the smaller reactor inlet valve could be mounted on them.

Figure 11. 2 liters reactors 4,5 and 6 with new equipment

After 24 hours the new equipment did not work on R2:5 and R2:6 so to examine if using a pipe would work with the new equipment, two pipes with different lengths where made, see Figure 12. The smaller pipe was put in R2:4 and the longer in R2:5. The reactors 2:1, 2:2, 2:3 were still going with the original equipment and R2:6 had the new purchased materials but was working without a pipe.
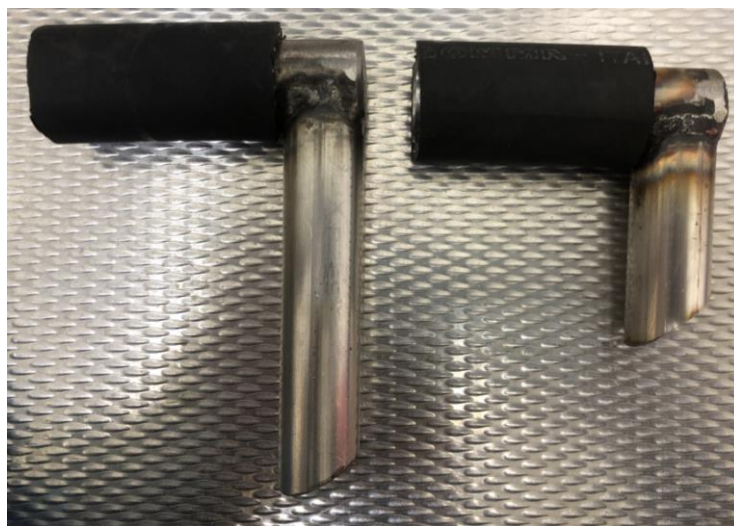

Figure 12. Pipes made at the WWTP, longer put in R2:5, shorter in R2:4.

4.2. Result from changes of the automatic reactors

One of the goals of this study was to follow up the equipment from Anaero Technology and record problems that would arise. Following changes were made during the operation:

- Gas leaks were detected and eliminated by change of equipment and packing with silicone.
- Calibration of the gas flow meter with μ-Flow.
- Connected the μ-Flow to the gas flow meter.
- Cleansed the motor and copper thread, if overload would occur only a few screws were replaced.
- The copper thread should be placed when the beam is down and before loading the feeding syringe. This makes the feed procedure quicker.
- Mixing in the feeding syringe must be turned on. Otherwise there is a high risk of sedimentation.
- The SD card was replaced in the Lobster Max-i PLC controller due to old SD card stopped working.
- Changed the length of digestate valve.
- Constructed a pipe within the reactor connected to the digestate valve to facilitate removal of sludge from the reactor.
- Replaced the reactor inlet valve.
- Replaced the digestate valve.
- Mounted gasket for the digestate bottle.

The reactors did not work ideally from the beginning which resulted in several changes and tests to get them working. In Figure 13 the biogas production is presented per day and the modifications are pinned to the time of change. Error events are also pinned to the same chart, and by comparing and analyzing the patterns the following conclusions are made.
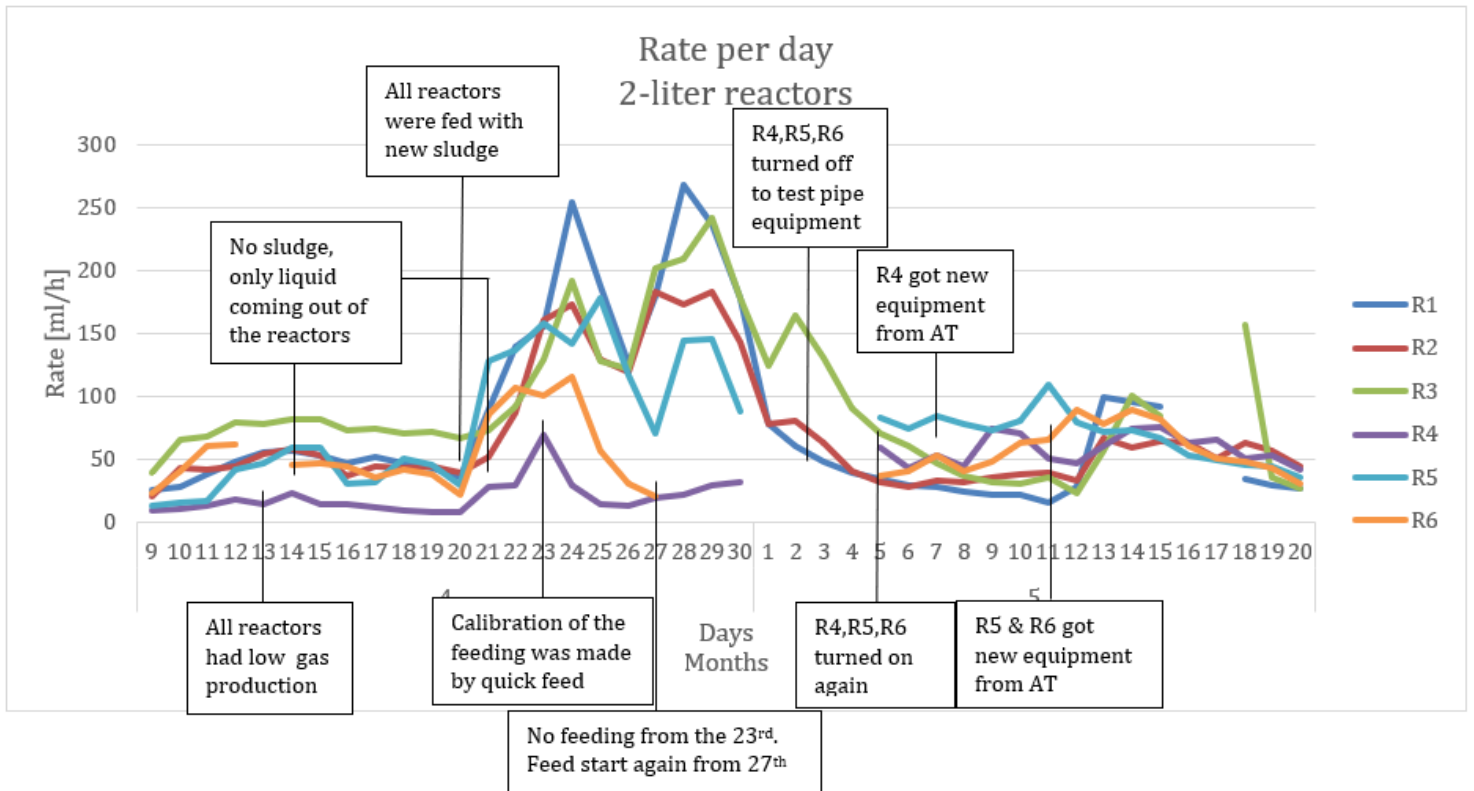
Figure 13. 2-liter reactors, the rate of biogas with events and changes pinned to the date.

Figure 13 displays changes in the biogas production together with practical changes. As seen for the 13 April the biogas production decreased. This was with high probability connected to the sludge that should have been replenished but was still in the reactor. When the reactors the day after were emptied of that sludge the production increased again. Because of the trouble of removing sludge from the reactors, low biogas production occurred within seven days.

Changes were made to solve the outlet problem. The outlet was investigated for gas leaks and sealed with silicone grease, see Figure 8. The outlet was shortened to eliminate problems caused by a slow outlet rate. A pipe was monitored to prevent the foam in the top of the sludge being the problem, see Figure 9. When none of these solutions worked the equipment was upgraded by a tighter inlet valve, an improved gasket and a shorter outlet digestate valve (see Figure 10).

During 21 April to 1 May there were occasions of irregular high biogas production. The increase was caused by the following events: Refilling the feeder syringe after the syringe being emptied, happening the 20 April. The reactor feed was recalibrated by feeding the reactors and measuring the millimeters pressed during the feeding period. The effect of the feed was a higher OLR and thereafter a higher biogas production. The feeders were turned off after the calibration to equalize the feed and therefore on the 24 to 27 of April the biogas production decreased. When starting up the feeders the biogas production increased before it turned to regular production rate.

19

4.3. Final product of the automatic reactors

One aim of the project was to evaluate and optimize the equipment from Anaero Technology. Because the equipment did not work properly from the beginning, the focus became to make the reactors work to require the least amount of manual work.

The moderated reactors and their new operating factors are presented in Table 2.

Table 2. Reactors final operating factors.

Nr	Reactors	Volume	New equipment from AT	Mixing in syringe	Pipes for outlet	Diluted substrate	Self-sufficient
2:1	Lobster Max i automatic	2 liters	NO	On 40 sec	NO	YES	NO
2:2	Lobster Max i automatic	2 liters	NO	On 40 sec	NO	YES	NO
2:3	Lobster Max i automatic	2 liters	NO	On 40 sec	NO	YES	NO
2:4	Lobster Max i automatic	2 liters	YES	On 40 sec	YES	YES	YES
2:5	Lobster Max i automatic	2 liters	YES	On 40 sec	YES	YES	YES
2:6	Lobster Max i automatic	2 liters	YES	On 40 sec	NO	YES	NO
5:1	Ray Max i automatic	5 liters	NO	On 40 sec	NO	YES	NO
5:2	Ray Max i automatic	5 liters	NO	On 40 sec	NO	YES	NO
5:7	Semi - continuous	5 liters	-*	-	-	YES	NO
5:8	Semi-continuous	5 liters	-	-	-	YES	NO

* The section with (-) indicates that the option is not possible.

4.3.1 Feeder motor
Three of the reactors were fed with different density and different feed-rate to examine if the feeding motor would overload. The motor did not overload at any point during the experiment, when working with TS in between 2.0 to 4.7%.

4.3.2 Feeding
The feeding was made in two different ways. R2:1, R2:2, R2:3, R5:1 and R5:2 was fed with mixed sludge into the feeding syringe where the OLR where 1 g VS/L day. R2:4, R2:5 and R2:6 were fed with sludge without dilution into the feeding syringe and separately fed with water through the access port every day to get OLR 1 g VS/L day. Feeding the reactor with water separately is more similar to the semi-continuous reactors. The different feeding procedures did, however, not cause any specific differences in the biogas production, see Figure 13. Feeding the syringe with diluted sludge did not need daily manual feed and therefor the reactors with the manual water feed was changed to mixed sludge after 20 days. It is as shown possible to feed water manually every day instead of diluting the sludge, so if an experiment requires manual feeding it is

possible. However, mixing the sludge into the determined OLR will make the feeding process more self-sufficient and less time consuming.

4.3.3 Syringe mixing

At startup all the syringe mixings were turned off. The mixing is constructed so when it is turned on by the PLC controller the prefeed mixing is set to the time requested. After 4 days of running the reactors, the syringe connected to R2:2 contained sedimented substrate (Figure 6). The syringe mixing was turned on to 20 seconds of stirring before feed. Sedimentation kept occurring in the reactors, and the syringe mixing was turned on in all syringes working with mixing of 40 seconds before the syringe feed the reactor. The syringe mixing provided the reactor with a stirred and homogeneous substrate. A minimum of 40 second mixing was necessary when using sludge with TS between 2.0 to 4.7 %.

4.3.4 Analytical result

Analytical tests were made to anticipate the bacteria's behavior and biogas production. The results from the analytical tests are gathered in Table 3.

During the first week R5:1 and R5:2 showed high numbers of VFA which is a warning sign of an impending failure of the digestion. The first test showed a VFA/alkalinity ratio of 0.087 in R5:1. If the VFA remained high, the methanogenic bacteria could have stopped working and the sludge in the reactor would need to be exchanged. The 2-liter reactors and the semi-continuous reactors showed low ratio and where not looked at closer. Due to the high ratio in R5:1 two more VFA tests were made using the same Alkalinity, to ensure if the reactors should be emptied. The second test showed an even higher value of VFA which was alarming, but the VFA however did not stay abnormal, which led to the operation of the reactors being continued with the same content.

Table 3. Result from analytical tests

Reactors	Date of analysis	pH	Total Solids (TS) [%]	Alkalinity [ml/l]	Volatile Fatty Acids (VFA) [ml/l]	VFA/ Alkalinity
R2:1	20-04-08	7.71	2.10	6934	162	0.023
	20-05-06	7.45	2.42	5354	157	0.029
R2:2	20-04-15	7.58	2.50	6096	185	0.030
	20-04-27	7.51	2.55	5486	206	0.038
	20-05-19	7.39	1.23	4572	148	0.024
R2:3	20-04-15	7.49	2.40	6629	248	0.037
	20-04-22	7.39	2.61	5334	220	0.041
	20-05-06	7.54	1.47	4724	139	0.029
R2:4	20-04-08	7.89	3.18	7506	248	0.033
	20-04-15	7.64	2.14	6096	176	0.029
	20-04-22	7.54	1.60	5334	159	0.030
	20-05-19	7.50	1.75	5639	148	0.026
R2:5	20-04-27	7.62	3.25	7353	310	0.042
R2:6	20-04-08	7.74	3.35	7849	270	0.034
	20-05-19	7.33	0.24	7010	154	0.022
R5:1	20-04-08	7.70	3.34	8306	720	0.087
	20-04-09			8306	1174	0.141
	20-04-10			8306	509	0.061
	20-04-15	7.58	3.34	7391	522	0.071
	20-04-22	7.42	2.71	6629	222	0.033
	20-04-27	7.65	2.61	6401	240	0.037
	20-05-06	7.39	2.41	5486	188	0.034
	20-05-19	7.34	2.24	5182	196	0.038
R5:2	20-04-09			8306	1648	0.198
	20-04-10			8306	465	0.056
	20-04-22	7.42	2.47	6096	186	0.030
	20-04-27	7.66	2.45	6096	214	0.035
	20-05-06	7.51	2.17	5410	200	0.037
	20-05-19	7.35	2.13	5182	172	0.033
R5:7	20-04-08	7.63	2.80	5067	194	0.029
	20-04-22	7.50	2.77	3734	165	0.034
	20-05-06	7.34	2.10	3277	144	0.038
R5:8	20-04-15	7.50	2.56	5563	189	0.34
	20-04-27	7.52	2.76	4801	170	0.035

4.3.5   New equipment

From the start-up the outlet digester valve did not work as it was expected to perform. The flow of sludge did not exit through the digestate valve which caused errors in the biogas formation. The final reactors that were able to operate as the Ray/Ray-i and Lobster Max-i were designed to do were R2:4 and 2:5, after the following changes.

A pipe was constructed to go from the inlet of the digestate valve that ended in the middle of the sludge in the reactor. The sludge did not come out with the pipe installed until it was combined with new inlet and digestate valves and a new gasket from AT (Figure 10). Then the right amount of sludge was removed when squeezing the bottle. The new smaller reactor inlet valve created an increased speed of sludge into the reactor. The gasket was placed to decrease gas leaks. The digestate valve had less gas leaks, was shorter than the original valve and was to facilitate attachment of the digestate bottle. The digestate bottle was also placed lower than the first bottle.

The new equipment from AT was mounted on R2:4, R2:5 and R2:6. However the pipe was only installed in R2:4 and R2:5 to examine if the new material from AT would make the digestate valve work as expected. When observing the sludge exiting from the reactors the R2:4 and R2:5 had a thick sludge pouring out and R2:6 had a transparent liquid coming out. To confirm the theory, that the sludge pouring out when squeezing the R2:6 digestate bottle was incorrect, a TS analysis was made from the digestate bottle sludge from R2:4 and R2:6. The value presented for R2:6 did indeed maintain irregular low amount of TS that confirms an error in using the reactors without a pipe (Table 3).

The reactors were finally able to operate self-sufficiently by changing the reactor inlet valve, adding a digestate bottle gasket, changing the digestate valve and installing a pipe connected to the outlet digester valve. Then the right amount of sludge poured out in the digestate bottle when squeezing it. The outgoing sludge is therefore representative for the reactor contents, which is necessary when taking samples for analysis.

4.4. Gas measurement

The gas measurement by the gas flow meters provided by Anaero Technology was very useful with its real time logging. The data could be displayed by the minutes, hours or months. Other parameters were also logged, such as temperature, volume amount of the sludge and the pressure of the gas. The data where easily accessed through an USB-port and managed in Excel.

A problem that occurred with the gas flow meter was that it easily got stuck and therefore did not register the true amount of biogas. When the tumble got stuck the PLC controller logged either extremely high numbers of biogas or amounts near zero. If the stuck tumble was discovered early the average true data could still be collected. If the tumble did not get identified there would be only false data during that time.

After running for 8 weeks, one part of the gas flow meter showed signs of corrosion. That could possibly be repaired but is another problem that does not occur with the µ-Flow.

The µ-Flow is more reliable considering it does not get stuck and create errors. On the other hand, the µ-Flow does not have online logging, the data is collected manually and for that, someone need to be present. The µ-Flow does not need to be calibrated and therefore one cause of error is avoided.

The µ-Flow was installed to measure the gas production, the result is assembled in Table 4. The amount of produced gas calculated by the microflow was put into *ml/tumble* and used as calibration values to put in the PLC controller.

Table 4. Calibration using µ-Flow.

Reactor	Calibrated "ml/tumble"	µ-Flow gas amount	Gas flow meter gas amount	Calculated "ml/tumble"	New average "ml/tumble"
5:1	25.2	6529	3700	44.47	-
5:1	-"-	291	182	42.29	42.38
5:1	42.4	386	388	42.18	-
5:1	-"-	2645	2763	40.59	-
5:1	-"-	3618	3754	40.86	-
5:1	-"-	2538	2602	41.56	41.63
5:2	25.2	325	195	42.00	-
5:2	-"-	3184	1097	44.81	-
5:2	-"-	3316	1762	47.43	44.99
2:3	23.6	301	324	21.92	-
2:3	-"-	692	861	18.97	-
2:3	-"-	196	238	19.44	20.11
2:5	24.2	1273	1510	20.40	-
2:5	-"-	3240	3657	21.44	-
2:5	-"-	840	1024	19.85	20.56

Table 4 shows significant differences between the first calibrated *ml/tumble* and the final *ml/tumble* calculated and confirmed by the µ-Flow. Therefore, the data collected from the R5:1 and R5:2 got changed from *25,2 ml/tumble* to *41,63 ml/tumble* (R5:1) and *44,99 ml/tumble* (R5:2). In Figure 14, R5:1 and R5:2 have modified data based on Table 4.

The difference in the calibration shows an error in the calibration and that additional confirmation of the calibration could be needed.

To get the benefits of both equipment the µ-Flow could always be used to calibrate the gas flow meter by linking the µ-Flow before the gas flow meter, so the gas goes to both apparatuses. To minimize the tumble errors the µ-Flow would still be linked, so if the tumble would gotten stuck, there would still be data from the time during an error. The data would then be collected manually and replace the false data.

4.5.   Comparison of the semi-continuous and the automatic reactors

Laboratory studies of biogas production at VIVAB in Varberg has earlier been done with semi continuous reactors. The equipment is well known and reliable, the obstacle has been that it requires manual feeding. The automatic reactors from AT was purchased so a manual daily feeding shouldn't be necessary. For the semi-continuous reactors to be replaceable by the automatic reactors they need to perform equally.
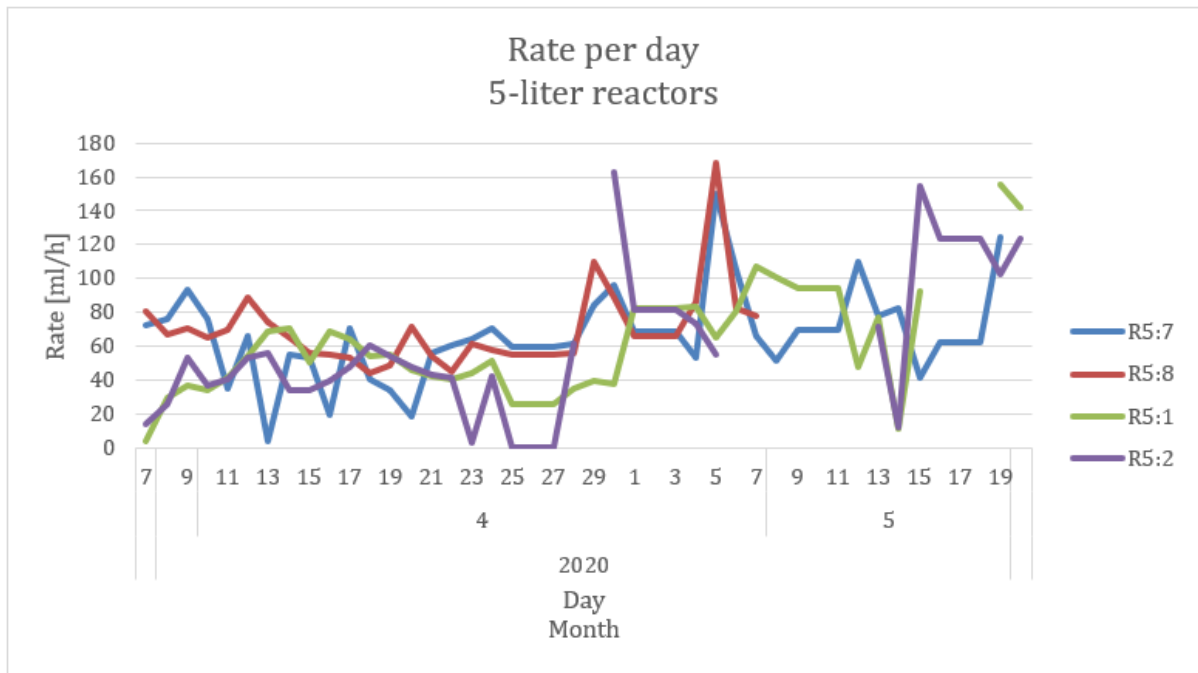
Figure 14. The biogas production rate of the 5-liter reactors, based on manually collected data.

The reactors show a quite uneven biogas production. The semi-continuous reactors were fed once a day from the start-up. Beginning from the 25:th the reactors where fed once a day except for the weekend and were therefor fed double amount Friday and double amount Monday. When the anaerobe bacteria get fed fresh sludge, there will be an increase of biogas production. The feed will therefore occur irregularly, and the discontinuity when feeding the double amount, will increase the variation in the biogas production.

The lack of data from the automatic reactors (R5:1 & R5:2) is a result from errors with the tumbles in the gas measurement, as mentioned in the section 4.3. The reactors did not work properly from the beginning, gas leaks were found and modification of the reactors were made.

Based on the knowledge that errors and gas leaks occurred, Figure 14 shows four reactors with similar biogas production. A conclusion that the reactors perform equally is therefore possible to make.

4.6.   PLC software

The software allowed a real-time data collection that gave multiple analysis options. The PLC software save data from the reactors the second the software register biogas from the gas flow meter module. The data is saved as a csv file that can be taken out from the controller by an USB and opened in an excel sheet.

To analyze the data the A column is collected and is changed from text to columns, the whole sheet collected, and a pivot diagram is made. In the pivot diagram the factors of choice are selected to make the selected diagram. Figure 15 show the average rate production of biogas over each day for the 5 liters reactors.
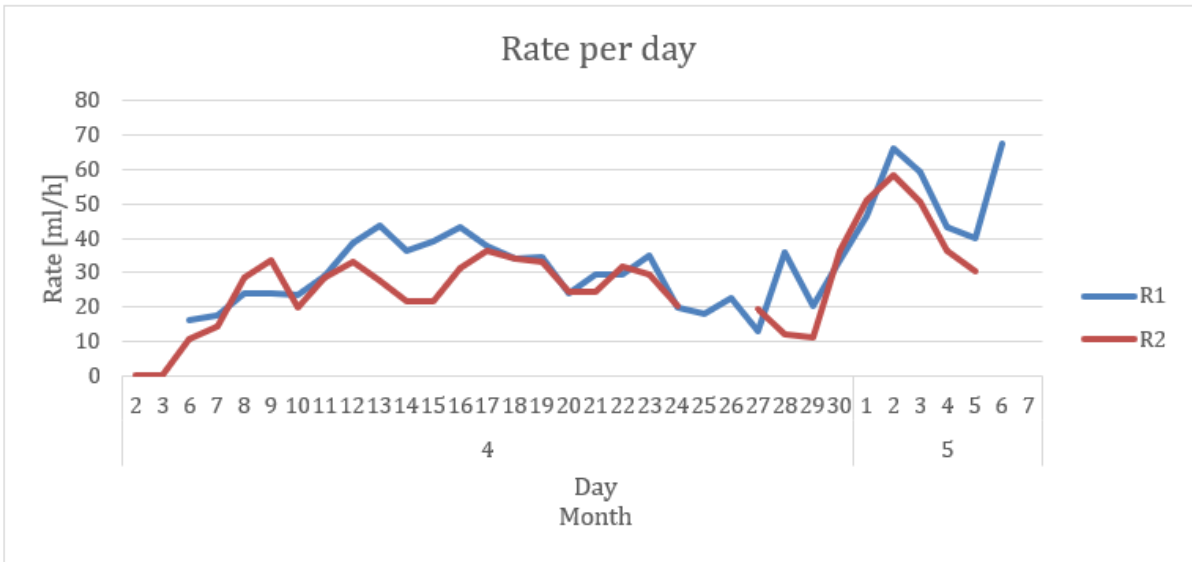
25

Figure 15. The rate of produced biogas for the reactors R5:1 and R5:2.

When using the PLC software, the data that is false is available to delete and the true data is still saved, and an average rate is still assessable. When using manual data logging for example once a day, if there is one error on that day it will ruin the data for that whole day, see Figure 17. To get a readable chart, the days with errors need to be excluded and that will reduce result, see Figure 18.
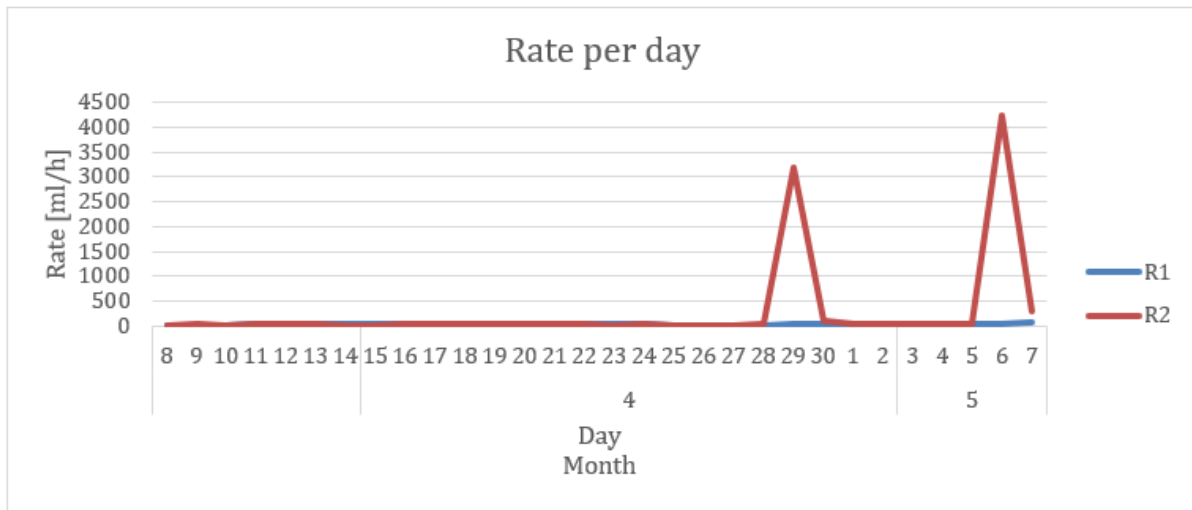

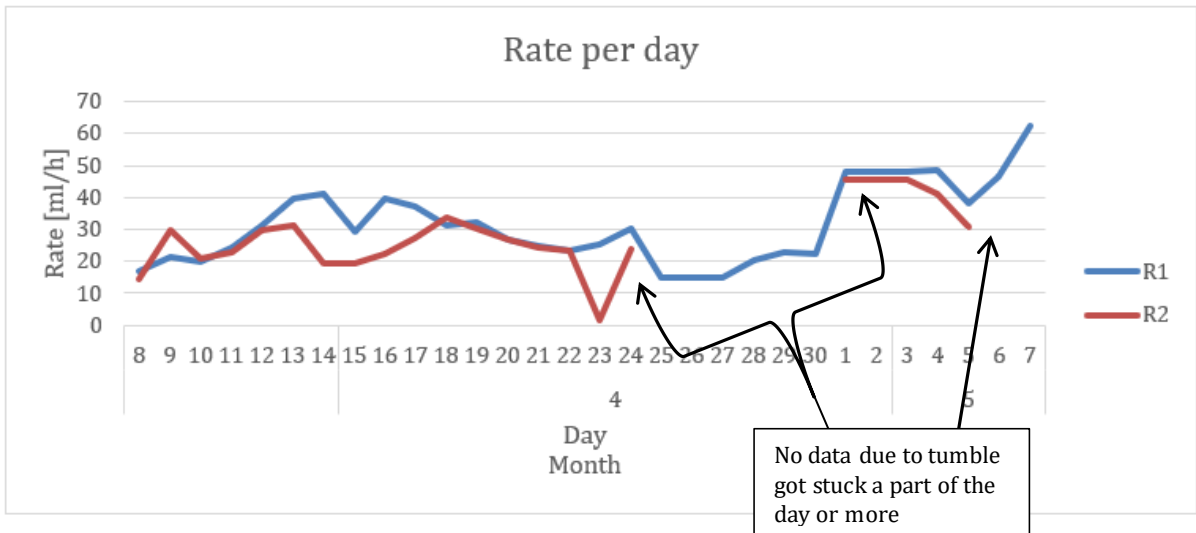*Figure 17. The manually logged data starting from test day 8.*

Figure 18. The manually logged data starting from test day 8 with the data errors deleted.

The two reactors Ray/Ray-i and Lobster Max-i gave individual data and to compare the reactors their data where merged and compared by their reactor volume. They got compared by their volumetric gas production (Figure 19).
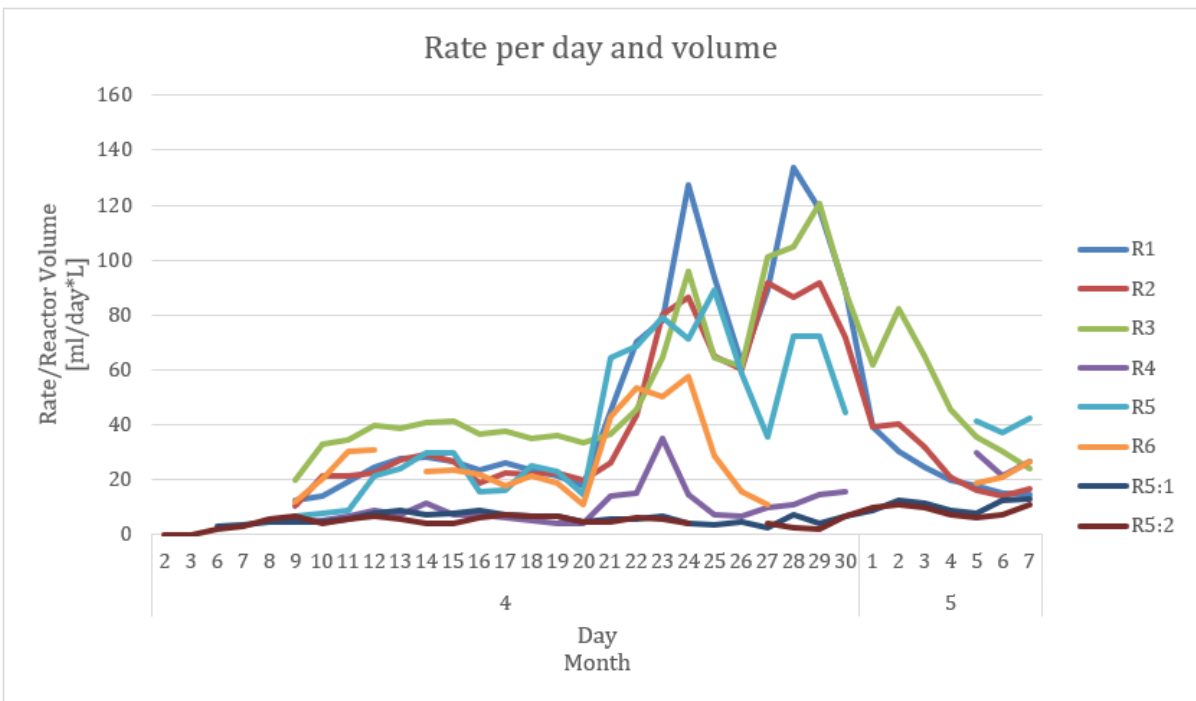

Figure 19. Merged chart of the Ray/Ray-i reactors and Lobster Max-i reactors rate/reactor volume.

27

5. Conclusion

The Anaero Technology reactors Ray/Ray-i and Lobster Max-i were optimized to replace the semi-continuous reactors. This was possible by changing the reactor inlet valve, adding a digestate pot gasket, changing the digester valve, placing the digestate bottle lower, and installing a pipe connected to the outlet digester valve. The automatic reactors will thereby not only function equally to the semi-continuous but also minimize workload.

For further improvement digestate bottles with volume markings should be exchanged for the present bottles. A bottle with volume markings will make it easier to check that the right amount of sludge has poured out.

The PLC controller does real-time logging and gives extensive data. As an addition to the standard gas flow meter μ-Flow gas flow meter should always be connected, so if the tumble would get stuck, there would still be measurements available. The data would then be collected manually and replace the false data. The μ-Flow will also work as confirmation of the gas flow meter measurements.

To operate with the automatic reactors the only manual processes are needed is:

- Filling the reactors at startup.
- Refilling the feed syringes each 4-5 days.
- Squeezing of the digestate bottles to get the sludge out.
- Controlling the function of the feeder beam once a month.
- Collecting the biogas production data.

To perform new experiments with the AT equipment, instructions are gathered in a manual that is based on this report. The manual is included in this report as an appendix.

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Manual for Ray/Ray-i and Lobster Max-i automatic reactors

Startup

Gas flow meter module

Reactor mixing motor

Access port

Digester valve

Digestate bottle

Feeder motor

Reactor inlet valve

Beam wheel

Feeder inlet valve
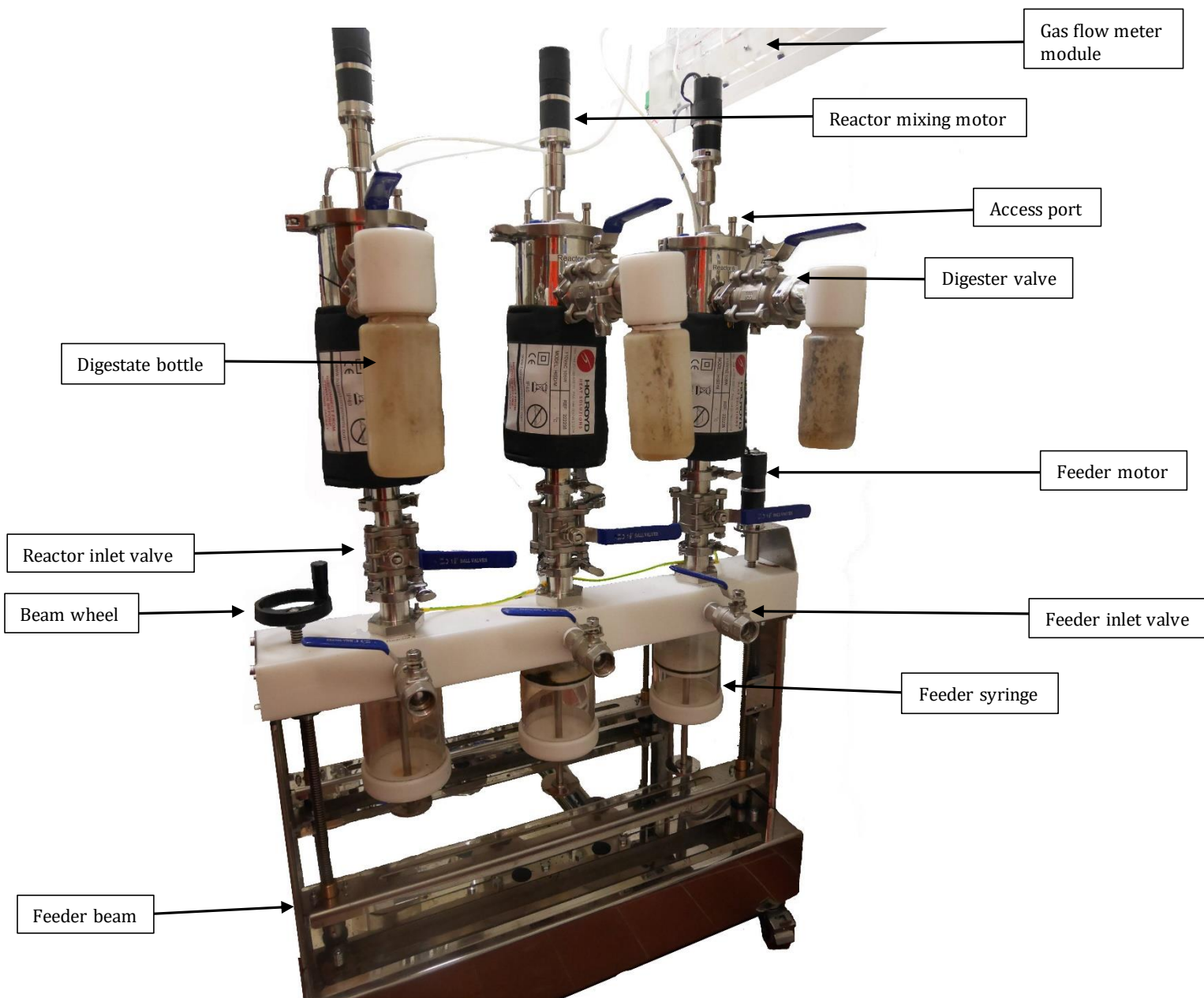
Feeder syringe

Feeder beam

Figure 1. Reactor 2:4, 2:5 and 2:6 on one feed bench connected to a gas flow module.

Before starting the reactors, the syringe needs to be filled but before that the motor should use a checkup. There is a copper thread, which function is to prevent the motor from overloading. The copper thread will break before the motor does. To prevent the motor from overloading the motors should therefore be disassembled, cleansed and put together. In case of overload only 2 of 4 screws is put back, the rest of the screws can be arranged after the reactors been running with one loading rate for 2 weeks.
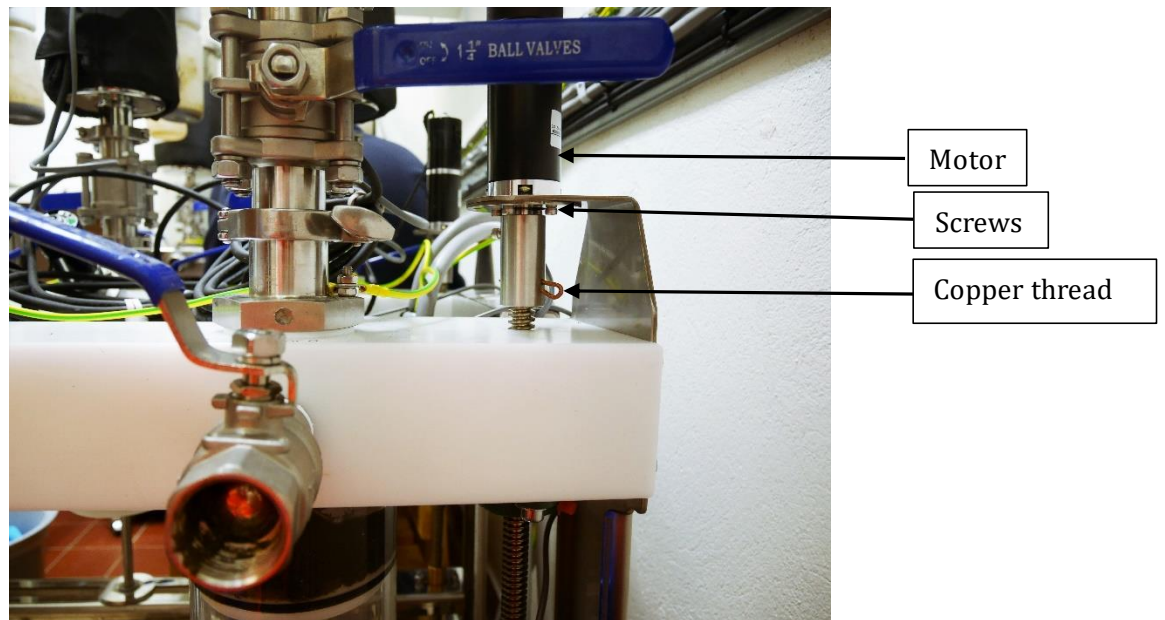

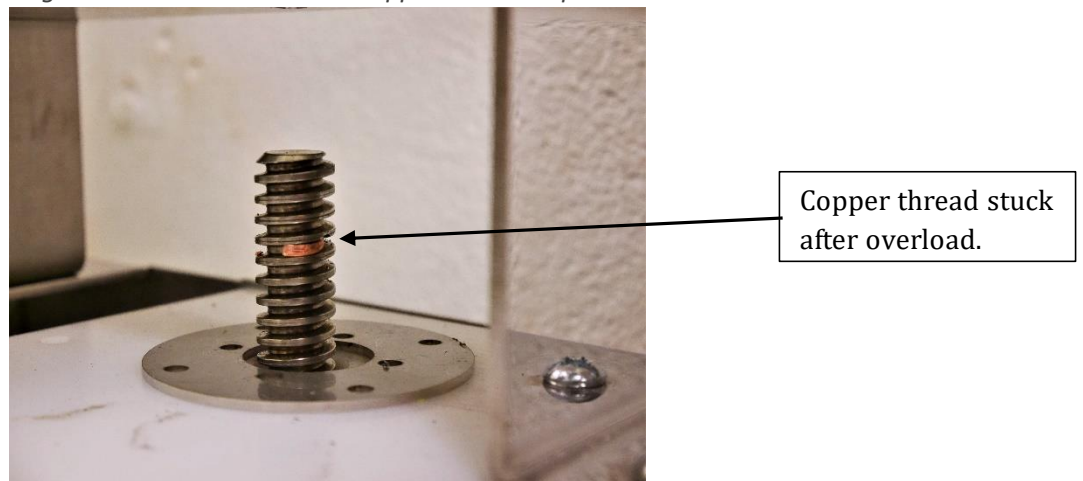*Figure 2. Feeder motor with copper thread in place.*

Figure 3. Shaft after dissembled the motor

Before feeding the reactors, the new substrates TS needs to be measured. It is possible to dilute the sludge before filling the feeding syringe or it can be done manually every day by filling the reactors with water through the access port.

When feeding the feed-syringe, a check needs to be done first that the reactor inlet valve is shut, and then the wheel is moved to get the beam down and the pistons stays up. The easiest way to fit the copper-thread to fasten the motor, is to lower the beam to the bottom and place the thread before filling the syringe. The fresh feed is then fed in the top up syringe, so the air

bubbles get removed before fitting it to the syringe top up valve. When the valve is still shut the top syringe gently is pressed to drive air out before tighten the fitting and open the syringe top up valve, the feed is pushed into the feeder until the syringe piston rod is engaged with the beam and the feeder is re-loaded and the syringe top up valve was closed and the digester valve opened. The data for the reactor trials are collected and put into a table, see section PLC software.

Emptying the reactor of outgoing sludge is made by squeezing the digestate bottles. The amount of sludge going out should be the same amount that has been put into the reactor. A check needs to be done to confirm the right amount of sludge before emptying or preparing the sludge for analyze tests.

The amount of gas is measured by a gas flow meter that measures the flow in real-time together with the barometric pressure and temperature and converts reads to STP automatically. Before starting the measurement, the flow meters need to be calibrated. For preparation of the gas flow hoses is connected to the gas inlet at the bottom and connected to a bottle with water. From the bottle a hose is connected to the reactors' gas outlet port, the gas then will go through the water before it comes to the gas flow measurement. When the operation only wants to measure the amount of methane, the bottles content is to be change to sodium hydroxide to eliminate the Carbon dioxide. The gas flow meter is then filled until liquid drops from the spill point, then calibration is provided by level the gas flow meter with a spirit level. The spill port is changed to up position and a 150 ml calibration syringe is connected and gently introduce air to cause a tumble. Six tumbles were made for the calibration of the 5 liters reactors and the milliliters for six tumbles of each reactor got recorded, the value is the quantity of biogas that the buckets could measure per tumble. This was done by following formula:

$$\frac{ml}{tumble} = \frac{Start\ point - End\ point}{N^{o}\ of\ tumbles}$$

When the calibration values where done the calibrated ml/tumble form Table 1 was entered in the PLC software.

Table 1. Calibration of gas measurement

Reactor	Start point [*ml*]	End point [*ml*]	Nº of tumbles	*ml/tumble*
Example	139	16	5	24,6

The data logging and monitoring is collected from the PLC controller and copied to an USB stick. The data was transferred into a pivot diagram to compare the result between the reactors.

Gas hose from a reactor to bottles

Bottles filled with water that the gas go through

6 gas flow meters connected to the reactors

Figure 1. Gas flow meter connected to the reactors.

Because of possible tumble errors, the bottles can be replaced with μ-Flow to make one more calibration. To minimize the tumble errors during the operation the μ-Flow would still be linked, so if the tumble would gotten stuck, there would still be data from the time during an error. The data would then be collected manually and replace the false data. Following formula show how to make the calibration calculation.

$$\frac{Gas\ amount\ from\ \mu - Flow}{Gas\ amount\ from\ PLC\ controller} * set\ ml/tumble = New\ ml/tumble$$

Table 3. Calibration using μ-Flow.

Reactor	Calibrated "ml/tumble"	μ-Flow gas amount	Gas flow meter gas amount	Calculated "ml/tumble"	New average "ml/tumble"
Example	25,2	6529	3700	44,47	-
Example	-"-	291	182	42,29	42,38

Operating factors

Operating factors are chosen in the PLC Controller, shown in Figure 4 and Figure 5. The calibrated gas flow meters result is put as the tumbler volume, as the reactors produce gas, the number of tumbles, total gas and flow rate will be presented at the display.

When a reactor startup is to be made, create a new data file by writing a new number after *Data file*. The data that will be collected by the PLC controller and saved in that file. Data from earlier operations will therefore not be mixed with the new one. When collecting the data with a USB the new file can be transferred and analyzed in excel. The start time is typed in under *Run start time.*

The heater is possible to switch of but to get the selected temperature in the reactor it should be put on *Enabled.* The target temperature is to be put in and the actual temperature in the reactors is presented.

The Ancillary system controls the mixing inside the syringe. This is preferable to use, the substance in the syringe can otherwise sediment and the feeding will not be homogeneous. Based on latest operation a minimum of 40 seconds of mixing is preferable to get a homogeneous substance.
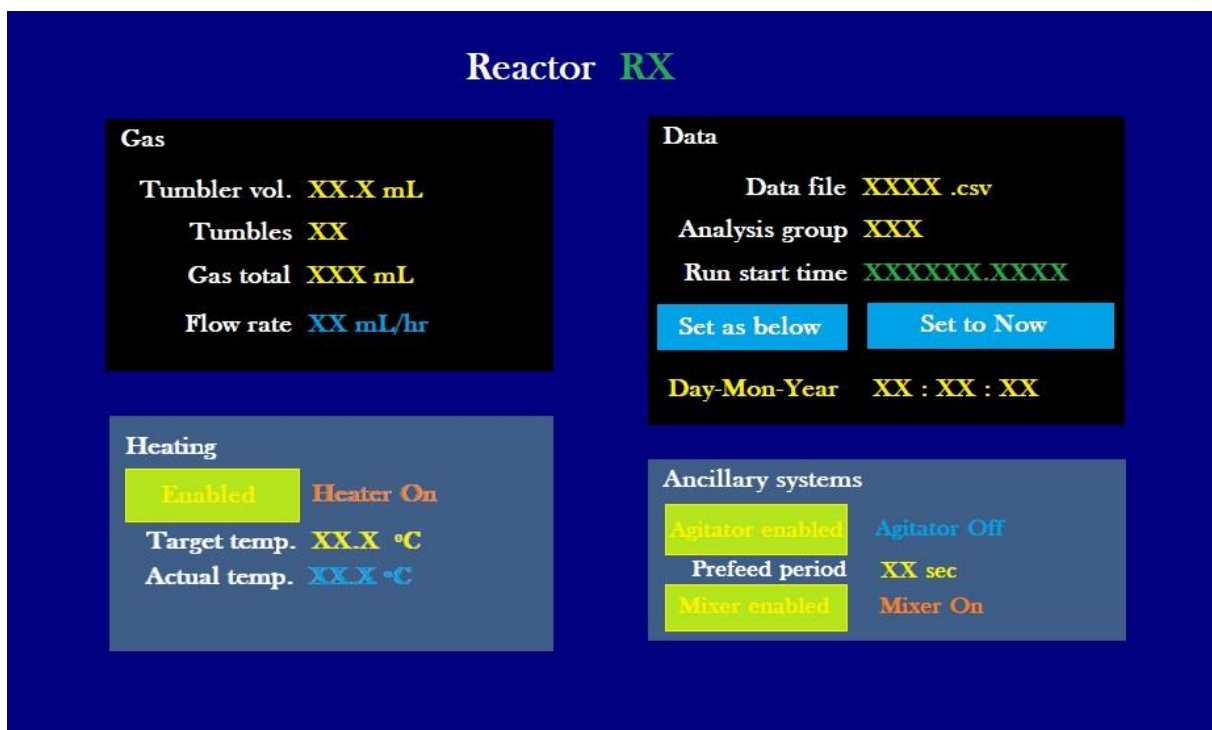

Figure 4. Display of Reactor options

In the display of the feeder the operating options of the feeder is to be put in. The orange text is the factors that is changeable, and the green text will be based on the orange numbers. The *Feed cycle period* is how often the feed is to be made and the *Feed time per cycle* decides how quick the feed is to be made. The numbers will decide the period of residence and will also have an impact on the feed motor. The feed motor can be overloaded and the feed cycle period and feed time per cycle should therefore be monitored with the motor in mind. When monitoring new operation observe Figure 4 first.

The Beam speed is to be calculated in the beginning of the operation. The millimeters that the beam have moved will be divided by the amount of hours that have passed. A control of the beam is to be made once a month, to make sure it is correct. If a change of beam speed has occurred, a new beam calibration is needed.

When the feeder is operating the *Feeder disabled* button will change. When it stands on the display *Feed motor idle* it means that the motor for the syringe feeder is turned on. If there will be an overload the text *Beam OK* will change. To know when the next beam feed will come the Feed timer is to be enabled. The lines in the middle will show how far it is until the next feed.
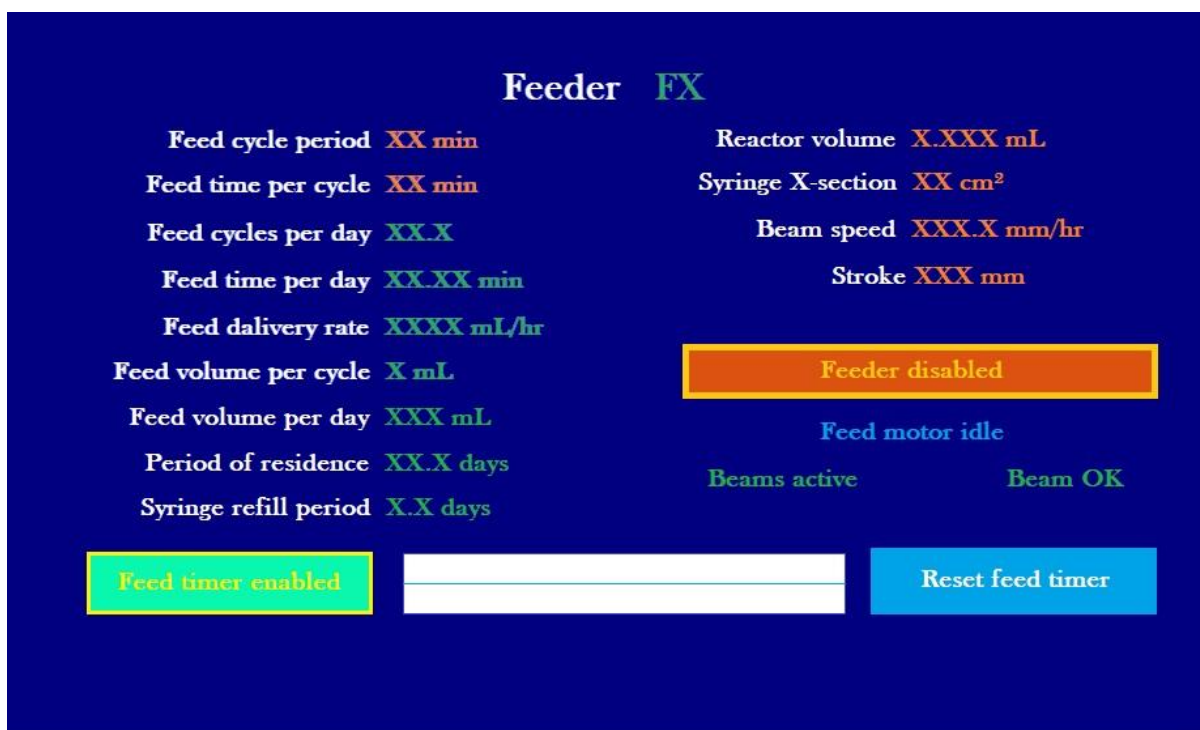

Figure 5. Display of Feeder options

Operation with TS between 2,0 and 4,7 have not overloaded the motor with following preferences:

Table 4. Statistics over successful feeding operation

Feed cycle period	55 min	60 min	60 min	55 min	60 min
Feed time per day	0,14 min	0,90 min	0,22 min	0,13 min	0,58 min

PLC Controller

To analyze the data the A column is collected and is changed from text to columns, the whole sheet collected, and a pivot diagram is made. In the pivot diagram the factors of choice are selected to make the selected diagram. Figure 13 show the average rate production of biogas over each day for the 5 liters reactors.
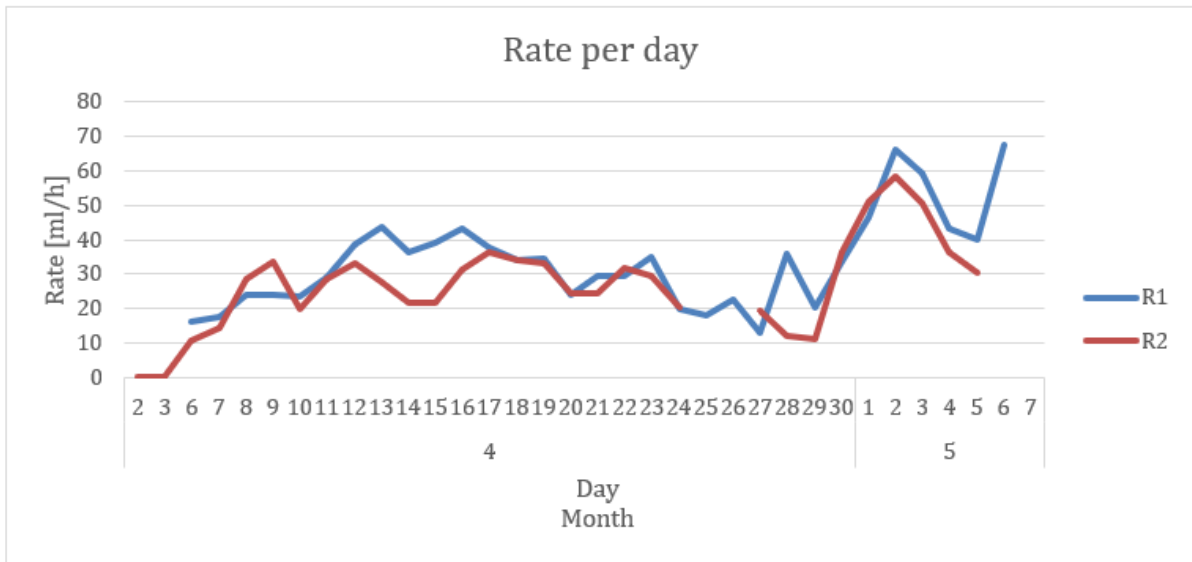

Figure 13. The rate of produced biogas for the reactors R5:1 and R5:2.

During the examination time the gas flow meter showed errors a couple time where the tumble got stuck in the middle which were collected as biogas production by the software. This resulted in false data and problematic read of the graphs, see Figure 14.
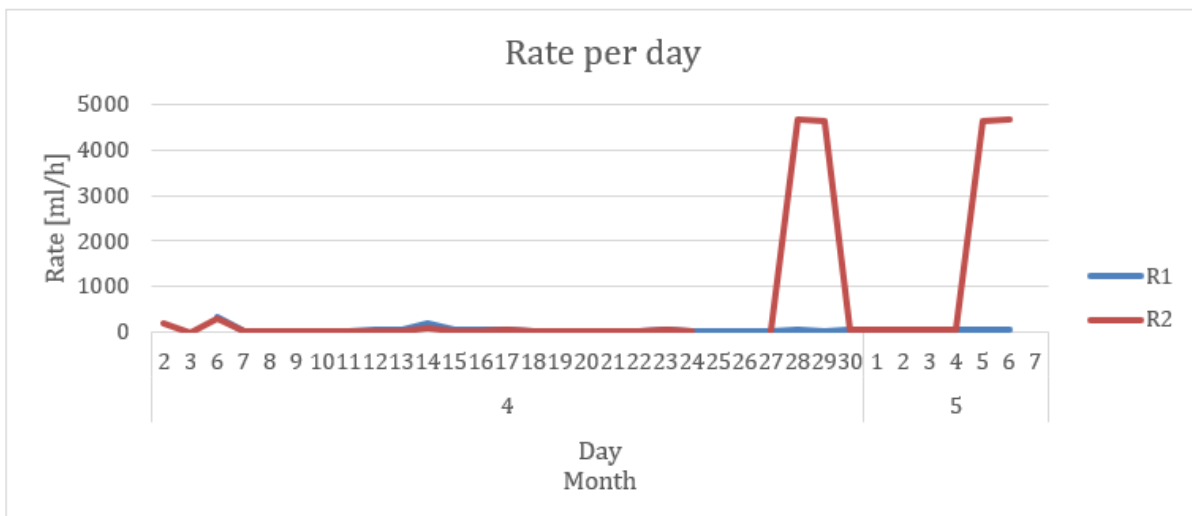

Figure 14. The rate of produced biogas for the reactors R5:1 and R5:2 including false data.

To delete the false data an exclude column is made in the sheet. A filter is put on the "*g-rate*" label if that is the data that is wanted, the filter is *g-rate*>100. For this to work the data in the

column need to be put to numbers, otherwise the whole column is marked and replace all the dots to commas by using the replace feature in find. The number 100 got chosen by the fact that the reactors do not seem to produce biogas in a rate above 100 ml/h. When the filter was added the *Exclude* column was filled with 1. In the pivot diagram the *Exclude* label is put in the Filter section and the elements chosen for *Exclude* should exclude 1. Then the graph only shows an average rate from the day only including true data, seen in Figure 13.

When using the PLC software, the data that is false is available to delete and the true data is still saved, and an average rate is still assessable. When using manual data logging for example once a day, if there is one error on that day it will ruin the data for that whole day, see Figure 15. To get a readable chart the days with errors need to be excluded and that will reduce result, see Figure 16.
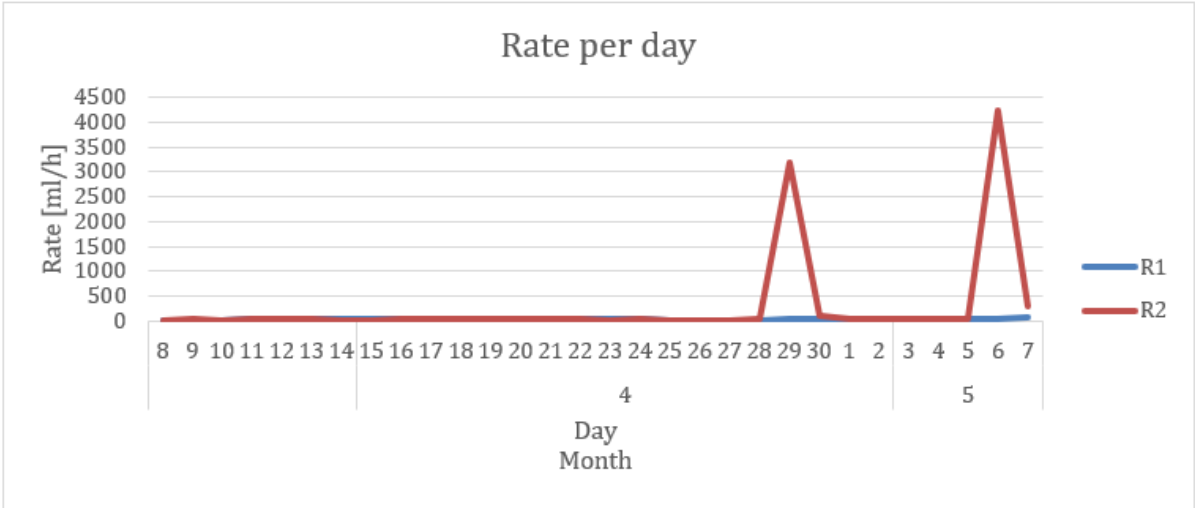

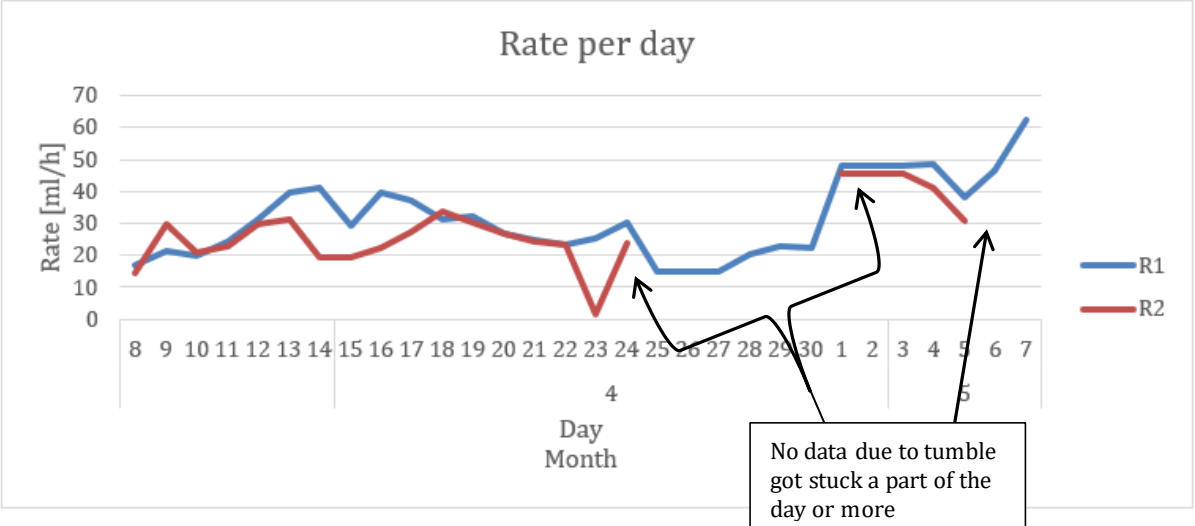*Figure 15. The manually logged data starting from test day 8.*

Figure 16. The manually logged data starting from test day 8 with the data errors deleted.

39

The two reactor Ray/Ray-i and Lobster Max-i gave individual data and to compare the reactors their data where merged and compared by their reactor volume.

The merged chart is done in a new sheet where the data from both reactors gets pasted in, underneath the other with a blanc line in between. The columns AG-AM is a bit different and is excessive and a differ in content between the charts, therefor it's deleted. To not mistake the 5-liter reactors for the 2-liters the Label column is selected for the 5-liter data and by using "find and replace", mark R1 and replace with R5:1. The same thing is done for R2 to R5:2.

The rate needs to be represented by their reactor volume and a new column is made where the data from column *g-rate* is divided by the reactor volume. This is done in the first row and then the formula is multiplied through the whole column. When both reactors have their rate compared by their reactor volume a pivot chart is made and the *g-rate/reactor volume* is put in the value section against the *e-month* and *e-day* in the axes category. The exclude column is used in this chart as well. The result is shown in Figure 17.
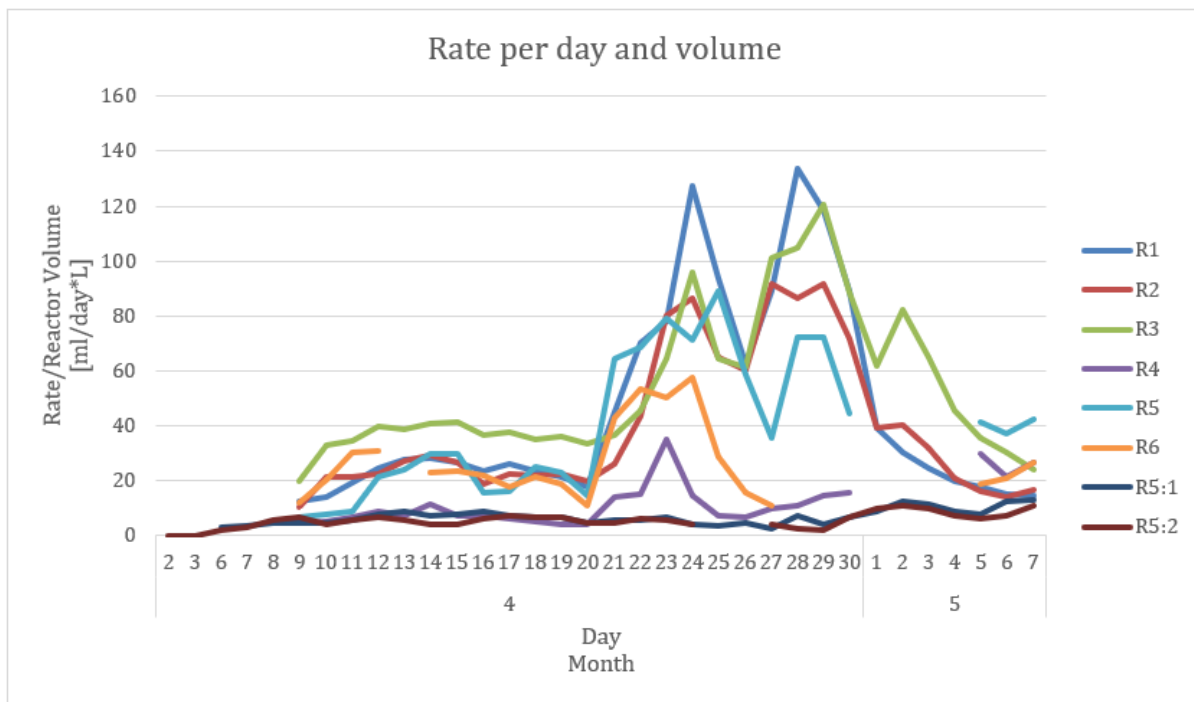

Figure 17. Merged chart of the Ray/Ray-i reactors and Lobster Max-i reactors rate/reactor volume.