BIOHYGAS Phase 1 Final Report

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Introduction and overview

The BIOHYGAS technology used in this project is a two stage biohydrogen/biomethane anaerobic digestion system which has the potential to increase energy recovery from sewage biosolids by up to 37% compared with single stage digestion alone. The University of South Wales (USW) has already published work demonstrating this effect using flour milling co-product, and pelletised grass, in 100L scale digesters.

In stage one of the BIOHYGAS process, anaerobic digestion is used to produce hydrogen directly from sewage biosolids, this stage has a lower volume and faster hydraulic retention time than a conventional anaerobic digester requiring a smaller digester and less energy input for heating which has the potential to result in lower CAPEX and OPEX costs. In stage two, the output from the hydrogen digester is fed to a conventional methane digester; however, due to the effect of the first stage methane yields are hypothesised to be up to 37% higher can be obtained. Methane from this system can be sent to a steam methane reformer (SMR) for conversion to fuel cell grade hydrogen and the carbon dioxide which is co-produced in both digestion stages can be used in food packaging.

In Phase one of the study, USW utilised its 100l pilot scale anaerobic digester in conjunction with smaller scale batch fermentations, to optimise the BIOHYGAS process for use with the specific sewage biosolids being produced by our collaborators Dwr Cymru Welsh Water (DCWW). The objective of these fermentation experiments was to produce a set of performance data for the BIOHYGAS process using the sewage biosolids to be treated in Phase 2 of the project. This information, along with USW's existing research on the BIOHYGAS process will inform both the design of a full scale demonstration plant in phase 2, as well as allowing lifecycle analysis to take place alongside an evaluation of commercial opportunities and deployment options within DCWW. The information can also be used to understand how BIOHYGAS can be integrated with downstream SMR and carbon capture and utilisation (CCUS).

Science Underpinning the Proposal

In this section a description of the experimental work carried out together with key scientific findings and their evaluation is presented. In summary, three main phases of work were carried out in the 6 months of this project. An initial series of fermentation experiments were carried out using thermally hydrolysed sewage biosolids (THSB) obtained from a wastewater treatment plant that will be referred to as Site A However after several attempts to demonstrate a viable first stage anaerobic digestion of this material it was decided that non thermally hydrolysed material from a different wastewater treatment plant referred to as Site B be investigated as an alternative. In phase 2 of the study a series of batch fermentations were carried out comparing biosolids from Site B with those from Site A and it was concluded that a viable first stage digestion could be carried out using Site B Biosolids. In the third phase of work a 100l pilot scale fermenter was used to trial a first stage fermentation of Site B biosolids and the output from this fermenter then evaluated for its methane production potential. Each of these phases of work is presented in detail below.

Phase 1 – Fermentation of Thermally Hydrolysed Solids from Site A

Four separate fermentation experiments were carried out to establish the suitability of THSB as a substrate for hydrogen production. In each experimental phase, all bioreactors were inoculated with digestate from Site A, heat treated at 110°C for 15 minutes to inhibit methanogens and returned to room temperature prior to being introduced to USW's laboratory based pilot

bioreactors at 5% of their working volume. The temperature of all bioreactors was maintained at 35°C throughout all experimental phases.

In the first experimental phase, THSB was normalised to 5% total solids and used to fill a 93 L working volume bioreactor along with inoculum and 1% v/v antifoam. Continual feeding of 5% TS THS plus 1% v/v antifoam began after 24 hours in batch mode, at a rate of 5.16 kg per hour. This equated to an 18-hour hydraulic retention time (HRT).

In response to zero hydrogen production in this initial fermentation experiment, it was hypothesised that THSB maybe inherently inhibitory to hydrogen producing microorganisms. During the second fermentation experiment this hypothesis was tested by adding 1% m/m (fresh weight) sucrose to the bioreactor at start up. All other parameters were kept as per the first experiment.

In both fermentation experiments samples were taken from the feedstock and bioreactor for offline data measurements. Analyses were carried out for total and volatile solids and volatile fatty acids (VFAs) and COD.

Phase 1 Results

During the first fermentation experiment, no hydrogen gas production was observed. The lack of any hydrogen production during the first experimental phase, despite being seeded with methanogen-inhibited inoculum suggests that THSB is either biologically unavailable or inhibitory to hydrogen-producing microorganisms in isolation.

During the second experimental phase, where the bioreactor was initially dosed with sucrose, hydrogen production can be observed after approximately 10 hours (Figure 1). Subsequent spikes in hydrogen and carbon dioxide production occurred following further dosing of sucrose once hydrogen partial pressures had fallen below detectable levels in the bioreactor headspace (Figure 1). This data suggests that there is nothing inherently inhibitory about THSB as a feedstock from which to produce hydrogen (at least when simple sugars are used as a substrate), however coupled with the data from the first experimental phase it appears that THSB is biologically unavailable for microbial conversion to hydrogen in isolation.

In addition, no significant reduction in the COD of the THSB feedstock following its fermentation in the bioreactor was observed. The data show a mean reduction of 0.02% from 23.98 gCOD in the feedstock, and 23.97 gCOD, which cannot be considered a significant difference. This however is reflective of the apparent inactivity of the bioreactor between sucrose doses and consistent with the hypothesis that THSB alone is not a suitable substrate for hydrogen production. Some studies also indicate low levels of COD utilisation in THSB compared with raw sludge, suggesting a lower substrate biodegradability which those studies attribute to inhibitory compounds such as melanin and melanoidin being produced during the Maillard reaction in THSB (Zhang et al., 2019).



Figure 1. Hydrogen and carbon dioxide flow rates during the second experimental phase. Letters A, B, C and D indicate the times at which sucrose was added to the liquid phase of bioreactor.

Compositional analysis showed insignificant total and volatile solids utilisation during the second fermentation experiment. The feedstock and bioreactor effluent had total solids values of 4.88% and 4.83%, respectively, and the volatile solids values were 4.17% in the feedstock and 4.05% in the effluent. This is consistent with the COD data and demonstrates little to no fermentative activity within the bioreactor. There was some reduction in mean total carbohydrate from 26.5 g L⁻¹ in the feedstock to 17.7 g L⁻¹ in the bioreactor effluent. This, compared with poor COD and solids utilisation, suggests that simpler substrates are more biologically available to hydrogen-producing bacteria and is consistent with other bodies of work, which similarly report that when employing short HRTs, biological hydrogen production favours less complex substrates as feedstock. Small amounts of VFA were produced at a rate of 55.58 mg day⁻¹. Proportions of VFAs were found to be 30% acetic acid,47% butyric acid, and 22% propionic acid. It should be noted that some if not all of this acid may have derived from the sucrose added to the bioreactor rather than the result of any THSB being utilised.

Phase 2 Batch Scale fermentation comparing Site A THSB and Site B PSB

In phase 2 batch scale fermentation experimental runs were carried out at batch scale to compare the suitability of THSB from Site A and primary sewage biosolids (PSB) from Site B WWTP and/or VFA production. In each experimental run, batch reactor system capable of performing 15 1I batch fermentations in parallel was used to test several variables in triplicate. In each 1L fermenter, feedstock was normalised to 3% total solids and each fermenter was inoculated with conventional AD effluent which was heat-treated at 110°C for 20 minutes and added at 5% of their working volume. All bioreactors were continuously stirred and the temperature was maintained at 35°C throughout both experimental phases. Gas flow data was logged throughout and samples were taken at the start and end of each experiment for future VFA analysis. During this run, THSB and PSB were fermented, both with and without the addition of 10g/L sucrose.

Phase 2 Results

During this experimental phase, no gas production was observed in the reactors fed with THSB (no line is shown on the graph in figure 2 therefore). Those fed with THSB and 10 g/L sucrose produced gas for the first 10 hours only (Figure 2) indicating that once the sucrose had been used up, THSB



alone was biologically unavailable as a substrate. These results are consistent with previous experiments and suggest that THSB is unfavourable for microbial conversion to biohydrogen.

Figure 2. Comparison of fermentability of THSB and PSB in batch scale first stage digestion experiments.

There was little difference between data for PSB alone compared with PSB and 10 g/L sucrose. All PS bioreactors produced a greater amount of gas than those fed with THS and the gas flow was maintained for longer during the experiment, suggesting that PSB is a more biologically amenable substrate. Compositional analysis shows that VFA production in the PSB fed fermenters was considerably higher than that measured in the THSB fed bioreactors, in particular acetic acid which increased by a factor of 3.8 from 255 mg/l to 972 mg/l.

Phase 2 results clearly indicate that PSB from Site B would be a more suitable candidate for the BIOHYGAS process and so pilot scale fermentation of this material was carried out in phase 3 of the fermentation experiments.

Phase 3 Pilot scale fermentation of Site B PSB and Batch methane potential testing

Results from the previous phases have suggested that PSB is a more favourable feedstock than THSB for use in the BIOHYGAS process. To test this hypothesis further, a 100 L scale experiment was carried out using PSB collected from Site B WWTW. Conventional AD effluent, which was heattreated at 110°C for 20 minutes, was used as inoculum and added at 5% of the working volume. The reactor was left in batch mode for 24 h, after which continuous feeding of PSB began at an 18 h HRT. Gas flow and composition data was continuously logged and samples were taken at 24 h intervals for VFA analysis using headspace gas chromatography.

The second fermentation experiment in this phase aimed to compare the biomethane potential of the effluent and the feedstock from the first experiment. The aim of this experiment was to determine whether PSB that undergoes a 2-stage AD process, consisting of an acidogenic step at a short HRT followed by a standard methanogenic step, is able to produce a higher methane yield

than PSB undergoing single-stage methanogenic AD. For these BMP tests, a 15-capacity 1 L batch reactor system was used to test 5 variables in triplicate. Conventional AD effluent was used as the inoculum and each reactor had a 3:1 inoculum:substrate VS ratio. Cellulose reactors were used as positive controls and inoculum-only reactors were used as blank controls. The remaining reactors were fed with effluent from the pilot scale experiment in this phase or PSB from Site B.

Phase 3 Results

Throughout the pilot scale fermentation, no hydrogen was detected but VFA analysis indicated an average total VFA production of 1296mg/L per day. In particular, the production of acetic acid more than doubled over the course of 3 HRTs (Figure 1). These results demonstrate the presence of acidogenic activity within the reactor but suggest that any hydrogen produced was consumed instantly. This could be due to hydrogen-consuming microbial processes being more dominant, such as homoacetogenesis and the propionic acid production pathway.

The data for the second experimental phase shows little difference in gas flow rate between those reactors fed with the output from the pilot scale (first stage) fermentation and those fed with untreated PSB. In fact the reactors fed with PSB showed higher cumulative methane production during these fermentations (Figure 3). The corresponding BMP yields were 385.63 ml_{biogas}/g_{vs} for fermenters fed with the output from the pilot scale fermenter, and 438.32 ml_{biogas}/g_{vs} for those fed with PSB.



Figure 3. Comparison of methane production production from PSB feed, stage 1 digestate and inoculum at batch scale.

Carbon life cycle assessment of technology

Reducing energy and carbon emissions are critical for the success of the process, and so the life cycle energy and carbon impact was calculated. This was done with a mixed approach using anticipatory/prospective LCA coupled with primary data from the experiments and scale up projections.

LCA can be undertaken in a consequential or attributional manner. Attributional LCA (aLCA) is historically how all LCAs were performed and explore only the data associated with the particular process or system under examination. Consequential (cLCA) takes a wider system approach and can explore the consequences of adopting a particular system or process. aLCA has fewer uncertainties and can give good data for energy and GHG balances etc. and is often used for product and system improvements internally. cLCA is often used for policy and wider decision making. This means that we can use LCA to understand the impacts of a particular process or system, and that we can look at the wider (more uncertain) impacts of the consequences of the changes associated in the wider systems as a result of the new process. Optimally these two methods would use different input data with aLCA using average data and cLCA using marginal data.

In this instance, we have explored the impacts of the actual system under development (aLCA) using an anticipatory/emerging technology approach, and considered the avoided impacts of alternative processes in places.

Attributional LCA Process model

The energy and carbon calculations are based on the stages shown in Figure 4. Energy balance and material flows from the flows are calculated based on primary data from project partners and from literature and existing LCA data from similar process stages in other projects.





Products of the BioHygas process

The BioHygas strategy will result in the production of two main products: hydrogen and digestate, which is the material that is left over following the digestion. When properly processed, it can be used as fertilizer and can be directly applied in land and incorporated into soil to improve soil characteristics.

Green House Gases emissions of BioHygas

Assuming a H_2 production of 12.8 kg/day and the utilisation of grid electricity to power the process the energy (kWh) that is required for each unitary process is shown in Figure 2. This shows a total process energy of 388.23 kWh. Considering the calorific value of hydrogen at 33.34 kWh/ kg the potential energy to be obtained from the reaction of the hydrogen is 426.7 kWh. Therefore, with the experimental data the net energy gain of the BioHygas process is 38.47kWh.

Net energy = Potential energy of the reaction - energy of the process

$= 426.7 \, kWh - 388.23 \, kWh$



Net energy = $38.47 \, kWh$

Figure 1. Energy balance in kwh for the BioHygas system in unitary process.

Using the current UK Government GHG Conversion Factors for the grid electricity used, the embodied CO₂e for the BioHygas process is shown in Table 1.

 Table 1. CO2 emissions from the BioHygas strategy using grid electricity for the process.

Source	kg CO ₂ e of	kg CO_2e of CH_4	kg CO_2e of N_2O	Total kg CO₂e	Tonnes CO₂e per
Juice	CO₂ per unit	per unit	per unit	per unit	year
UK electricity	74.233	0.31	0.53	75.076	27.40

Therefore, the total CO₂e output of the BioHygas strategy would be as follows,

$$Total CO_2 BioHygas = \frac{Total CO_2}{Total H_2} = \frac{75.076 kg CO_2}{12.8 kg H_2}$$

$$Total CO_2 BioHygas = 5.86 kg CO_2 / kg H_2$$

The value of 5.86kg CO2e/kg H2 is within the lower (ie more desirable) range when compared with literature on the GHG impact of comparative methods for producing H₂. In addition, this value does not include reductions for using renewable only electricity, which would bring this value down substantially as well. This could be reduced naturally over time as the grid changes, or with specific renewable technology at site.

Consequential modelling: Pasteurisation

There are several critical advantages to producing hydrogen in the proposed manner which would not be modelled through an attributional approach. These are system advantages such as avoided impacts elsewhere. In this situation we have not modelled the consequential impacts in great detail at this stage – but are using the attributional study to help identify areas where avoided impacts or consequences would have additional impacts and benefits.

Consequential impacts of onsite pasteurisation

One of the major concerns of anaerobic digestion is the correct handling and disposal of the digestate sludge, which has a high concentration of pathogens but also the potential to be transformed into fertilizer due to the high content of nutrients such as nitrogen, phosphorus, and potassium, among others.

The digestate that is currently produced on site is transported to a larger DCWW operated Treatment Works (Site C), where it is treated with using thermal hydrolysis, followed by anaerobic digestion and dewaterting. The inclusion of this additional process will alleviate the requirement to transport the digestate for further treatment.

The distance from Site B to site C is 97 km. Assuming a 100% diesel HGV transport the total CO_2e released from the transportation of the digestate to Site C is approximately 100kg CO_{2e} per trip.

The implementation of an onsite pasteurisation represent the reduction of this carbon emission as it would treat and transform the sludge that is produced from the fermentation, allowing the reutilisation of the digestate. Exact details of the impact of the Site C processing has not been available within the time period, but will clearly include a further impact including increasing available throughput capacity for the site.

Consequential Impacts – use of outputs

The hydrogen and other products produced can and will be used in various ways which will likely change based on economic drivers. However, there are some which have been mooted by the industrial partners as likely options.

The use of CO2 for food storage

A coproduct of the system is CO₂, which has been identified as an opportunity by the company for use in food storage. The life cycle impact of this process is complex, but assuming CO₂ is a continued requirement for some food storage and within the food industry a direct swap for biogenic based carbon would (according to current IPCC modelling) reduce its emission impact to zero.

Hydrogen vs Diesel for transport

One clear area where localised impacts can be felt will be the potential for substituting associated diesel transport with hydrogen. Recent research suggests that the GHG emissions associated with hydrogen vehicles are a) substantially lower than diesel in most cases (not grey H_2) and are dominated by the hydrogen supply chain production method. As is shown here, the BioHygas GHG are in the lowest sector of H_2 production impact and therefore can have the highest impact.

Further work and limitations

The LCA and energy and carbon balance work has been restricted by the amount of empirical data available and the recruitment of researchers within the time period. Never the less, simple aLCA

(carbon and energy) data for the BioHygas proposal has been calculated. This can be refined and updated over time to show where the clear areas for wider system benefits can be adopted. Prioritisation of use between these sectors will require some additional scenario modelling.

A detailed engineering design for a demonstration project

Overview

The task of the project was to evaluate the potential of using an abundant substrate (sewage Sludge) to produce hydrogen as a fuel using a high-rate bacterial fermentation (the dark – fermentation route). Downstream options could be enabled in the process including steam reforming of methane to hydrogen and the extraction of valuable Volatile Fatty Acids. The process would be modelled by the University of South Wales using a continuously-fed laboratory digester and a set of batch digesters.

The source of the sewage sludge substrate and potential site for the full-scale plant was the Site A water treatment plant belonging to Dwr Cymru Welsh Water (DCWW). The role of AD Ingenuity LLP in the project was to:

- Produce an outline design concept
- Carry out a survey of existing assets and determine their suitability.
- Produce a process flow philosophy
- Produce a process control philosophy
- Produce energy, mass balance and process calculations

To provide that initial information to the project in written format and assist Marches Biogas to

- Produce the detailed process design
- Produce the detailed mechanical design
- Produce the detailed electrical design
- Perform process safety (DSEAR and HAZOP) reviews
- Bring the concept to design development

Initial Works at Site A

The target site was initially Site A, a Welsh Water facility that is processing sewage sludge using the thermal hydrolysis. The site is well known to AD Ingenuity and initial data sets provided a list of benefits of the thermal hydrolysis process. There was a greater conversion of solids to gas than in simpler single stage mesophilic digesters, the final effluent was pasteurised and met every regulation in this regard. The disadvantage of the process at Site A was the energy gained by extra solids reduction to biogas did not compensate for the large amounts of energy required to raise the relatively thin sludge to a pressure of 6 Bar (160 deg. C). Any extra energy that could be found from hydrogen would be a bonus. DCWW was keen to participate.

Site A was surveyed by AD Ingenuity and while the University of South Wales was generating data on Site A thermally hydrolysed sludge in the laboratory, detailed plans for the main site were made by AD Ingenuity based on the results of two prior papers issued by the University of South Wales (Massanet Nicolau et al 2015, 2013). The plans thus created included in detail the design concept that would take a side stream of the Site A thermally hydrolysed sludge, treat it for 18 hours in a dark fermentation for hydrogen production (BIOHYGAS) and then take the digestate from the BIOHYGAS tank as a feedstock for a conventional single stage methane-respiring anaerobic digester. Process flow philosophy was designed, controls selected and mass and energy balances and process calculations made. A risk analysis was undertaken. These are detailed in the appendices with this summary (collated as Appendix 1).



Site A THSB initial findings

The initial results that came back from the USW laboratory trials implied that while the methods used in the 2015 and 2013 papers were essentially identical to the laboratory trials on thermally hydrolysed Sludge, the latter was unsuitable for H₂ production. Site A was abandoned as a potential site on the grounds that its thermally hydrolysed sludge substrate was incompatible with H₂ production. A source of non- thermally hydrolysed sludge was required if the project was to continue.

The Primary Outline Design Phase For BIOHYGAS At Site B SSMAD

Dwr Cymru Welsh Water (DCWW) operate single stage conventional mesophilic anaerobic digestion (SSMAD) plants at Site B. The resulting digested sludge from Site B is transported to and reprocessed at one of the AAD (Thermal hydrolysis) plants to comply with regulations. It was decided by the project partners that the thickened sludge produced at Site B would be far more suited to the BIOHYGAS System. After discussions with DCWW regarding the change of site and very positive feedback from DCWW a meeting was held between the partners at Site B to introduce the project to the Site B operational staff. The Single Stage Mesophilic Anaerobic Digester (SSMAD) facility at Site B, like Site A, has some redundant equipment that was initially deemed suitable for reuse or conversion within the BIOHYGAS System and the decision was jointly made by all partners to change the focus of the Design Work Packages from Site A AAD Facility to Site B SSMAD Facility. This put a time pressure on the deliverables for the project however, the site was known to AD Ingenuity and the groundwork had been done on the process flows so the challenge was accepted, and the target site changed to the Site B facility. In the meantime, the University of South Wales was able to set up two sets of experiments on the Site B thickened sludge, a dark fermentation for H₂ and Methane production in the continuous digester and a batch fermentation to compare the substrate from Site B with control substrates.

Engineering uncertainties at Site B accommodated by Stage-Gate Approach

In the initial detailed examination of the equipment at Site B and the processes currently in use to export sludge for pasteurisation, AD Ingenuity proposed a Stage Gate process to a full-scale implementation. This approach recognised the very large potential benefit of pasteurising the sludge on the Site B site which would save on local management costs, transport costs for the sludge and further processing costs in a thermal hydrolysis plant. However, this would best be justified if the energy savings from the H₂ production could be demonstrated. Thus, the three stages of development for the site were defined as in table 2 below:

STAGE	FLOW FRACTION	BIOHYGAS TANKS	METHANE DIGESTERS	PASTEURISATION
1	HALF	1	1	NONE
2	HALF	1	1	YES
3	FULL	2	2	YES

Table 2. Stages of site development.



Site B Specific Process Calculations and Design Concept

A complete set of process calculations as listed in the appendices was made for the Site B sludge using the 2013 and 2015 results as before while waiting for the results from the laboratory to arrive. A design concept for each of the stages as listed above was developed and can be found in the appendices (Appendix 1).

Initial Site B Specific Project Risks as Determined By AD Ingenuity

- Delay on results of trial from USW to feed into the process design package
- BIOHYGAS tank suitability for feedstocks and H₂ production/storage
- The method of short-term storage of H_2 and other gases from the BIOHYGAS tank, whether it is a fixed roof/ double membrane or bell over water.
- Fine bubble diffuser requirement and the suitability of a compressor for redispersion of H₂ tank gas into subsequent digesters
- Suitability of existing pumps and pipework at elevated dry matter content and low pH range.
- The design must ensure that the site can meet its obligation to treat the required daily tonnage of dry solids.
- If the BIOHYGAS system stops the current process flow would need to be reinstated quickly and safely to ensure sludge processing continues.
- Impacts of odour created by the BIOHYGAS and Pasteurisation processes and the mitigation thereof.

University of South Wales Results Indicate High VFA and Inhibition of H₂ Production

The results from the laboratory testing of the Site B sludge arrived on 4th January. The key performance indicators of the cumulative gas generation from the continuous digesters indicated that the raw feedstock from Site B produced more gas than the output of the first stage BIOHYGAS fermenter. AD Ingenuity revised the process flow diagrams, mass balance and process calculations to reflect the USW results and established the following conclusions:

- BIOHYGAS digestate processed in a mesophilic digester will produce about 62% methane possibly enhanced by fats within the sewage.
- The Hydrogen generation is not detectable
- Single Stage AD produces 271 m³ methane per tonne of organic dry matter, this is very much in line with the methane production of the thickened sludge from Site B as analysed independently by AD Ingenuity.
- Two stage BIOHYGAS digestate produces 234 m³ methane per tonne of organic dry matter
- The uplift of biomethane production by using BIOHYGAS is minus 15.5%
- 3,174 mg/l of total VFA is created in the BIOHYGAS fermentation
- The volatile COD attributed to this VFA is 0.386 tonne-COD per day and if digested in a digester with suitable acetoclastic bacterial population would produce ((386*11*0.83)/24) = c.146 kWth of thermal energy continuously. Alternatively;
- O.52 Tonnes per day of total VFA is produced with a value of about a **quarter of a million pounds per year** on extraction and refining.

These results and the analysis is illustrated in ADI-300-070-C BIOHYGAS MASS BALANCE CALULATIONS TO REFLECT THE FINAL USW ANALYSIS. (Figures of note are highlighted in yellow blocks) (Appendix 1)

High level design for BIOHYGAS System 1st stage digester

Following the switch from Site A to Site B AD Ingenuity have updated the process mass balance calculations and shared with Marches Biogas. These have then been fed into Marches design and form the basis of the model produced.

On site biosolids flow design / Deliverable – D2.2 – Flow scheme for biosolids incorporating H_2 Digester

The Flow Design has been generated by AD Ingenuity in the form of block diagrams and flow mass balances. The Flow design required redevelopment and remodelling following the project move from DCWW Site A to DCWW Site B. This flow scheme was finalised by Marches Biogas to suit the existing assets at DCWW Site B. Modification of the damaged Enzymatic Hydrolysis process vessel was also identified for conversion to a dedicated gas holder for the BioHyGas process (Phase 1). The requirement of which was led by the need for a gas 'buffer' prior to injection into the gas mixing system, should any downstream process requirements dictate that the biogas generated by the BioHyGas process cannot be utilised via the digester. The possible requirement for a dedicated SGB (surplus gas burner) for the BIOHYGAS gas holder was identified - should the maximum BIOHYGAS biogas storage capacity be reached before utilisation can recommence - and investigated. The design for the post-digester pasteurisation process has also been finalised and is included with the flow schema, adjacent to the BioHyGas process, utilising the remaining redundant enzymatic

hydrolysis infrastructure whilst providing flexibility to the overall BioHyGas and downstream AD processes.

Operational parameters Feeding rates, pH, Temperature and HRT finalised for 1st stage digester

The determination of the actual operating parameters was to be ultimately dictated by the outcome(s) of USW's findings from WP1. In order to prevent any delay to deliverable deadlines, operational parameters were led by the findings from academic literature provided by USW (Massanet-Nicolau et al. - 2013 and 2015). These operational parameters have remained in place for the purpose of design due to the low H₂ production results established in WP1.

Telemetry and data collection: Parameters, data collection points, frequency and informatics solution designed

The preferred position of the inline VFA analysis equipment was discussed with USW and adequate valves/connections were included within D2.5. Suitable gas analysers for Hydrogen analysis, inline pH meters for pH control are being investigated should the project reach Phase 2. Process safety equipment (pressure relief, ultimate pressure relief and foam mitigation) have been specified within the HAZOP exercise. Control system and possible incorporation of existing telemetry (where appropriate) have been investigated.

BIOHYGAS process detailed design

A 3D scanner was utilised to more accurately review and survey the layout of existing assets and infrastructure. Utilising this method provided time-saving benefit to the project, particularly in light of the disparity between available site drawings and existing layout. The 3D renders generated from the 3D surveying exercise fed into the detailed design process, ensuring that the detailed design was generated from an accurate 'starting point' rather than potentially out-of-date/unrepresentative site drawings. The drawing listed below include various isometric views of the rendered 3D model generated and indicate the detailed design encompassing the revised equipment. These are included within the appendices (Collated in Appendix 2).

- MB202-2726-M-1007 A BioHyGas Stage 3 Site Layout
- MB202-2726-M-1008 A BioHyGas Tanks Plan and Elevations
- MB202-2726-M-1009 A BioHyGas Tanks Southeast Isometric View
- MB202-2726-M-1010 A
 BioHyGas Tanks Northwest Isometric View
- MB202-2726-M-1011 A BioHyGas Tanks Northeast Isometric View
- MB202-2726-M-1012 A
 BioHyGas Tanks Southwest Isometric View

Following completion of the design the proposed system was then subject to a HAZOP (Hazard and Operability Study) to identify Hazards within the present design, confirm if safeguards exist and make recommendations for consideration by the design team where residual risk remains. A copy of the system HAZOP is included within the appendices along with the following drawings where the nodes have been identified (Collated in Appendix 3):

- AD1300-091-A NODED
- AD1300-092-A NODED

Costing of BioHyGas System : CAPEX and OPEX over timespan and beyond determined

The CAPEX has been estimated following the input of the 3D scan renders into AutoCAD Plant 3D to accurately model pump positions and valving etc. Table 4. below details the equipment included within each stage of the project and table 5 indicates CAPEX associated with these stages.

Equipment scope for proposed Site B site	Stage 1	Stage 2	Stage 3
BioHyGas proposed equipment			
Thickened sludge holding tank			
Existing thickened sludge tank new discharge pumps,	\checkmark	\checkmark	\checkmark
duty/standby complete with necessary non-return valves,			
isolation valves, temperature transmitter and pressure			
transmitters before and after each pump for protection			
Stainless steel pump main from thickened sludge discharge			
pumps to BioHyGas tank No 1			
BioHyGas tank No 1			
Modification of existing tank to suit requirements of			
BioHyGas tank No 1			
BioHyGas tank No 1, complete with heat exchanger, slurry	\checkmark	\checkmark	\checkmark
recirculation pump, liquid relief valve, gas phase dual acting			
pressure and vacuum safety protection valves and all			
necessary instrumentation			
BioHyGas tank No 1 discharge pumps, duty/standby	\checkmark	\checkmark	\checkmark
complete with necessary non-return valves, isolation valves,			
temperature transmitter and pressure transmitters before			
and after each pump for protection			
Slurry return pipework from BioHyGas tanks through to	\checkmark	\checkmark	\checkmark
existing primary digester No 1 recirculation/feed pipework			
Gas mixing system – refurbish existing gas mixing system to	\checkmark	\checkmark	\checkmark
ensure adequate gas mixing of BioHyGas tank(s)			
BioHyGas bell over water gas holder			
Partial demolition of existing damaged tank and made good			
to suit floating roof gas holder			
Design and installation of a bespoke bell over water floating	\checkmark	\checkmark	
gas holder roof, incorporating level and pressure			
measurement and gas phase dual acting pressure and			
vacuum safety protection valves			
BioHyGas off gas return pipework to existing Primary	\checkmark	\checkmark	\checkmark
Digester No 1 gas mixing system manifold			
Hot water circuit – extend existing hot water circuit to	\checkmark	\checkmark	\checkmark
provide hot water feed and return to BioHyGas heat			
exchanger			
BioHyGas tank No 2			

Tahle 4	Fauinment sco	one for develo	nment of Site B	Site in Demon	stration Phase
IUDIC 4.	Lyuipinent see	ιρε τοι μενείο	prine ne oj sile d	SILE III DEIIIOII	strution i nuse.

Demolition of existing damaged tank and replace with new			\checkmark
to create BioHyGas tank No 2			
BioHyGas tank No 2, complete with heat exchanger, slurry			\checkmark
recirculation pump, liquid relief valve, gas phase dual acting			
pressure and vacuum safety protection valves and all			
necessary instrumentation			
BioHyGas tank No 2 discharge pumps, duty/standby			\checkmark
complete with necessary non-return valves, isolation valves,			
temperature transmitter and pressure transmitters before			
and after each pump for protection. Including additional			
pipework to connect to discharge back to primary digester 1			
recirculation/feed.			
Pasteurisation System			
Primary Digester No 1 new discharge pumps, duty/standby		\checkmark	\checkmark
complete with necessary non-return valves, isolation valves,			
temperature transmitter and pressure transmitters before			
and after each pump for protection			
Pre-pasteurisation buffer tank			
Modification of existing tank to suit requirements of pre-		\checkmark	\checkmark
pasteurisation buffer tank			
Pre-pasteurisation buffer tank complete with liquid relief		\checkmark	\checkmark
valve, gas phase dual acting pressure and vacuum safety			
protection valves and all necessary instrumentation			
Pre-pasteurisation buffer tank discharge pumps,		\checkmark	
duty/standby complete with necessary non-return valves,			
isolation valves, temperature transmitter and pressure			
transmitters before and after each pump for protection			
Slurry transfer pipework from buffer tank to scavenge heat		\checkmark	\checkmark
exchanger and to pasteuriser tank(s)			
Gas mixing system – refurbish existing gas mixing system to		\checkmark	\checkmark
ensure adequate gas mixing of tank			
Pasteurisation tank No 1			
Modification of existing tank to suit requirements of		\checkmark	\checkmark
pasteurisation tank No 1			·
Pasteuriser tank No 1, complete with heat exchanger, slurry		\checkmark	\checkmark
recirculation pump, liquid relief valve, gas phase dual acting		·	·
pressure and vacuum safety protection valves and all			
necessary instrumentation			
Pasteurisation tank No 1 discharge pumps. dutv/standbv	1	 ✓ 	
complete with necessary non-return valves. isolation valves.			
temperature transmitter and pressure transmitters before			
and after each pump for protection			
Slurry transfer pipework from pasteuriser to post-	1	\checkmark	
pasteuriser recovery tank			
Gas mixing system – refurbish existing gas mixing system to	1	\checkmark	
ensure adequate gas mixing of tank			
	1	1	

Post Pasteurisation recovery tank		
Modification of existing tank to suit requirements of	\checkmark	\checkmark
recovery tank		
Recovery tank, complete with scavenge heat exchanger,	\checkmark	\checkmark
slurry recirculation pump, liquid relief valve, gas phase dual		
acting pressure and vacuum safety protection valves and all		
necessary instrumentation		
Recovery tank discharge pumps, duty/standby complete	\checkmark	\checkmark
with necessary non-return valves, isolation valves,		
temperature transmitter and pressure transmitters before		
and after each pump for protection		
Slurry transfer pipework from recovery tank through to	\checkmark	\checkmark
existing post digester storage tank No 1		
Gas mixing system – refurbish existing gas mixing system to	 	\checkmark
ensure adequate gas mixing of tank		
Pasteurisation tank No 2		
Modification of existing tank to suit requirements of		\checkmark
pasteurisation tank No 2		
Pasteuriser tank No 2, complete with heat exchanger, slurry		\checkmark
recirculation pump, liquid relief valve, gas phase dual acting		
pressure and vacuum safety protection valves and all		
necessary instrumentation		
Pasteurisation tank No 2 discharge pumps, duty/standby		\checkmark
complete with necessary non-return valves, isolation valves,		
temperature transmitter and pressure transmitters before		
and after each pump for protection		
Slurry transfer pipework from pasteuriser to post-		\checkmark
pasteuriser recovery tank		
Gas mixing system – refurbish existing gas mixing system to		\checkmark
ensure adequate gas mixing of tank		
Pasteuriser off gas return pipework to existing double	 	\checkmark
membrane gas holder		
Hot water circuit – extend existing hot water circuit to	\checkmark	\checkmark
provide hot water feed and return to pasteuriser heat		
exchanger(s)		

Table 5. Pricing summary forDemonstration phase.	Stage 1	Stage 2	Stage 3
Staff for design, construction and commissioning	£119,460.00	£192,720.00	£192,720.00
Civil bases	£24,000.00	£45,600.00	£58,200.00
Mechanical pipework installation	£397,476.00	£876,288.00	£993,624.00
Electrical & instruments	£258,078.00	£420,066.00	£476,790.00
Drives & equipment	£121,800.00	£280,800.00	£394,200.00
Tanks	£126,000.00	£198,000.00	£360,000.00
Plant hire, welfare and expenses	£24,000.00	£37,080.00	£37,080.00
Total cost	£1,070,814.00	£2,050,554.00	£2,512,614.00
BioHyGas tanks included	1	1	2
Pasteuriser tanks included	0	1	2

Testing and innovation in demonstration project

As has already been discussed, the results of fermentation experiments carried out during the Phase 1 feasibility study did not show the necessary increase in methane production which could result in greater hydrogen productivity if the methane were to be converted to hydrogen through SMR. Consequently progression from phase one to the demonstration phase of the project (Phase 2) is not being sought. In light of this only a brief outline of the testing and innovation work envisioned for this phase is given here.

Phase 2 research was intended to take place in 2 synergistic phases. The main phase of work was to be a full scale demonstration of the BIOHYGAS process at Site B Wastewater Treatment Plant. As has been discussed in the section on engineering and design, this would utilise currently redundant, onsite treatment tanks located at Site B as the first stage of the 2 stage BIOHYGAS process and one of Site B 's two Anaerobic Digesters as the second stage. This would have been the first demonstration of a two stage fermentation process for producing hydrogen from sewage biosolids anywhere in the world and would have been as significant development for the treatment of wastewater biosolids.

The full scale demonstration of the BIOHYGAS concept would have been supported by continued fermentation experiments at pilot scale using USWs unique 100l lab scale fermenter for the continuous fermentation of high solids substrates. This would have been expanded with additional manually fed continuous fermenters to simulate the second stage of the BIOHYGAS process. Supporting the full scale demonstration process with lab pilot scale fermentation experiments would enable a wider range of parameters to be evaluated with the most successful of these then deployed at full scale.

Both the full scale plant and the pilot scale fermenters would have been equipped to continuously monitor key variables during fermentation including the production rate and composition of biogas, particularly with respect to hydrogen and methane production. Phase 1 results showed that the production of VFAs was a key variable for understanding the performance of the phase one fermenter. To augment our capability in this area, USW would have sought to deploy its innovative VFASense technology to both the full scale and pilot scale fermentation systems. VFASense is a novel, real time VFA measurement technology which has already successfully been used to

continuously measure VFA production in a full scale anaerobic digester in partnership with DCWW at Site A WWTP.

To understand in greater depth, the potential mechanisms of action which underpin the BIOHYGAS effect USW intended to leverage ongoing collaborations with colleagues at Imperial College to conduct detail genomic and metabolomic analysis of the two fermentation stages of the BIOHYGAS system, comparing them with the microbial activity present in a conventional anaerobic digester. These analyses would allow us to test the theory posited in our previously published research that increases in methane yields could be attributed to an inhibition of competing microbial flora in both fermentation stages.

Although the BIOHYGAS process will not now be progressing to Phase 2, the means to undertake much of this testing and evaluation will be sought through participation in other research projects as appropriate. In addition these techniques will also prove useful in understanding why additional methane was not observed from sewage biosolids as it was for other substrates.

Commercialisation plan informed by information gained during Phase 1

It was initially proposed to utilise the Site A Advanced Digestion facility for the BioHyGas phase 2 project. Part of the BioHyGas project scope was to confirm the suitability for this through substrate testing. This identified different characteristics between the sewage sludge processed through the Thermal Hydrolysis plant at Site A, and the feedstock used in the 100 litre digesters in the laboratory. This finding steered the project to find suitable alternative digestion sites which do not use the Thermal Hydrolysis process for pasteurisation prior to the mesophilic digestion. This identified E Site B.

Site B WwTW has a mothballed Enhanced Enzymatic Hydrolysis plant on site which was previously operated up to 2010 and now could be re-purposed for two-stage digestion. Hydrogen would be produced in the Enhanced Enzymatic Hydrolysis plant, as a first stage digestion, and methane from secondary digesting using existing digesters. As Site B has two primary digesters one would be a 'control' digester with the second as the 'experiment' digester to understand any variation in digester performance and biogas production. The existing Combined Heat and Power engines on site would be capable of processing the increased volumes of biogas being produced.

It is intended, should the project progress to phase 2, that Welsh Water's Digester Safety group would consider any process safety implications of new processes. This would ensure the existing Dangerous Substances and Explosive Atmospheres Regulations management on the site would be adequate or identify additional 'zoning' requirements.

Due to the competitive nature of securing land bank to recycle the digestate from sewage operations, Welsh Water's approach would be to process any mesophilic conventionally digested sludge to undergo subsequent pasteurisation. The proposals for Site B 's digestate is to haul the digested sludge to the Site A site for Thermal Hydrolysis pasteurisation and subsequent digestion to pasteurise before recycling to land. This method of operating is common across the water sector, as Environmental Permitting obligations mean 'enhanced' digestate i.e. pasteurised digestate is necessary to secure landbank for recycling. Because of this, any BioHyGas facilities that may be developed are not considered an environmental compliance risk due to subsequent pasteurisation and digestion at an Advanced Digestion facility, in Welsh Water's case for BioHyGas, Site A. This environmental driver has led to the Water Sector centralising Advanced Digestion facilities and either operating 'de-watering' or 'satellite digestion' facilities that essentially produce feedstock for an Advanced Digestion site. This would make Site B a good candidate for further research as it represents a typical operating model of digesting sewage sludges, in some cases brought in from other assets, and then de-watering for onward haulage to an Advanced Digestion facility. This process is inherently demanding on logistics, requiring fleets of Heavy Goods Vehicles to ensure the environmental requirements of the digestate are met, which is itself an avenue for the potential Hydrogen production.

There are two aspects to the commercialisation of phase 2, the utilisation and/or sale of the produced Hydrogen and the opportunity for commercial sale of Carbon Dioxide. These two activities are inherently linked as a Hydrogen production facility can benefit the environment in two ways;

- Produce a fuel that can displace fossil fuels without the flue/tailpipe emissions of CO₂ and accompanying particulate emissions;
- Provide a consistent stream of (biogenic) carbon dioxide that can displace other fossil fuel derived carbon dioxide products such as in beverages;

Welsh Water is actively exploring ways to capture the biogenic CO₂ from its biomethane production location in North Wales, to produce food grade CO₂. The market has been engaged and shown interest in the proposed end-of-waste product and plans to send an official request to tender out to parties soon. Work in this area has identified that there is a growing market for biogenic carbon dioxide, particularly if it can be 'cleaned' to a food grade standard such as EIGA 70/17. This is the most stringent standard for carbon dioxide intended for human consumption and would therefore allow the biogenic carbon dioxide to be used commercially. If this can be achieved there are potentially multiple market opportunities for this product. Engagement to date identifies end-users, brokerage firms, and carbon dioxide suppliers may all be 'Customers' for biogenic carbon dioxide, which is captured as a by-product of producing Hydrogen. This technological and commercial opportunity allows for the displacement of fossil fuel derived carbon dioxide (e.g. from fertiliser plants) contributing to carbon emission savings more widely than just on-site at the sewage digestion facility.

The carbon dioxide market is highly volatile at present, meaning a specific price is difficult to gauge, however there is consensus in the market that carbon capture technologies have a rate of return that would allow for self-financing. This allows negative carbon dioxide emissions to be associated with Hydrogen production, in addition to the fossil fuels displaced directly by the Hydrogen, leading to environmental benefits as well as possibly avoiding carbon emission payments for businesses.

The Biohygas project links closely with Welsh Water's HyValue project and the Business, Energy and Industrial Strategy department's funded H₂Juice project. HyValue aims to convert the biogas in to high quality hydrogen suitable for use in Fuel Cell Electric Vehicles. The HyValue project is funded through the Ofwat Innovation fund in preparation the Front-End Engineering & Design work. The HyValue project identified a number of potential hydrogen off-takers such as Emergency Services and Public Transport, in addition to the self-supply opportunity to replace the Water Sector's Heavy goods vehicles with hydrogen fuelled vehicles. This would allow the Water Sector to avoid diesel costs and carbon emissions from self-supply of hydrogen, and the sector will continue to operate advanced digestion assets and centralised sludge haulage logistics for many years, making the hydrogen production identified from BioHyGas as a possible contributor for re-fuelling at satellite sites.

The industrial heat market has also been explored, through the H2Juice project. This project aims to produce hydrogen from biogas (using the same technology as HyValue) and supply directly into an industrial process and demonstrate the distribution of hydrogen through a dedicated pipeline between the producer and the industrial end user. With impending changes to the Gas Safety and Management Regulations to facilitate the adoption of hydrogen fuel into the gas grid, there will be many opportunities for digestion facilities (typically located close to urban centres) to provide energy intensive industrial applications in the urban area with hydrogen commercially.

Summary

The experiments detailed above represent six months of feasibility testing of the BIOHYGAS process applied to sewage biosolids from two different waste water treatment plants. The hypothesis was that we would see increase yields of methane from a two stage fermentation of sewage biosolids, as we had already demonstrated with both flour milling co-product and pelletized grass (Massanet-Nicolau et al., 2015, 2013). However the permutations and experimentation we have been able to conduct within the time scale of this project has failed to replicate this effect. Thermally hydrolysed biosolids did not seem to be amenable to a low HRT first stage fermentation necessitating a switch to primary sewage biosolids from a different treatment plant. Primary sewage biosolids did result in the production of VFAs in the pilot scale first stage fermentation which is of great interest to researchers here at the University of South Wales as it validates much of our parallel research into producing VFAs as a valuable platform chemical using our BIOACE technology which we are also pursuing with Welsh Water. The conclusion remains however, that on the basis of these data, progression to phase two which involves a full scale deployment of the BIOHYGAS process does not seem practical without more research at pilot scale.

USW will continue to seek further funding opportunities to develop the BIOHYGAS concept for sewage biosolids and build on the positive data which did emerge from this phase one study. USW believes that further pilot scale testing and systematic optimisation of the first fermentation stage still offers the potential to reproduce the large methane (and by extension, hydrogen) yield increases observed in other substrates, and that subsequently, the advanced anaerobic digestion of sewage biosolids can play a pivotal role in meeting the UK's 2030 decarbonisation targets.

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