TECHNOLOGY DEVELOPMENT FOR THE THERMAL WET OXIDATION OF MUNICIPAL BIOSOLIDS.

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ABSTRACT

The Rotorua District Council in conjunction with Scion has recently established a wet oxidation pilot plant, situated at the Rotorua waste water treatment plant. The technology seeks to achieve a substantial solids reduction from waste through a thermal deconstruction process which breaks down the physical structure of material and segregates the solids into useable process outputs, such as readily degradable carbon feedstocks. In this paper, we report the basis for the selection and design of the pilot plant facility, outlining the laboratory scale experiments used to prove the concept for thermal deconstruction of municipal biosolids. Challenges encountered in the establishment of the pilot plant were discussed.

Keywords

pilot plant, thermal deconstruction, laboratory scale, wet oxidation, design

1 INTRODUCTION

Scion in partnership with Rotorua District Council (RDC) developed a technology platform, known as Waste2Gold, with the ultimate goal to reduce organic solid waste sent to landfill. The technology is based on solids deconstruction concept: using heat, pressure and oxidant to convert organic wastes into (1) readily degradable organic feedstocks, and (2) a by-product containing nutrients and metal salts. The approach is aimed at controlling the deconstruction process to yield useful intermediaries and substrates for downstream bioconversion, rather than complete breakdown to CO_2 and water.

In addition to the substantial reductions in transportation and landfill costs achieved by solids reduction (>\$700K/yr for RDC sewage biosolids alone), the process outputs could improve overall economic efficiency by offering a number of viable, added-value opportunities like:

- Replacement of existing ethanol supplementation (currently \$380K/yr) at the RDC Wastewater Treatment Plant (WWTP) with the biodegradable organic byproduct.
- Energy recovery from the processed organic wastes; as heat, methane or electricity.
- Feed stocks for novel industrial biotechnology applications, such as production of biodegradable plastics and liquid biofuels.

Recently, Scion and the RDC successfully commissioned a thermal oxidation pilot plant situated at the RDC wastewater treatment plant. The main objective of establishing the hydrothermal pilot plant is to demonstrate the development of the laboratory proof-of-concept, up to a larger scale with more industrial relevance. As a result of experience gained with the pilot plant, the decision to proceed with the full scale plant project will be based on a proven process, and on a more reliable economic estimate. This paper outlines basis for the selection and design of the pilot plant facility, outlining the scale up challenges encountered throughout the stage developments of the pilot plant.

2. PROCESS SELECTION AND DESIGN

Process selection bench scale tests were carried out in 200 ml Berghoff and 600 ml Parr reactor reactors (Figure 1) in order to select the best process option for thermal deconstruction suitable for scale up to a pilot plant operation. Three hydrothermal processes; thermal hydrolysis, wet oxidation and 2–stage hydrothermal were investigated.



Figure 1.Bench scale reactors used for process selection and design.

During the thermal hydrolysis experiments, the Berghoff reactor was pressurised with nitrogen at ambient temperature to 1 MPa and the temperature increased to 140 °C. For the wet oxidation experiment, the reactor was pressurised at ambient temperature with pure oxygen to 1 MPa and the temperature increased to 220 °C. The oxygen pressure was in excess of the stoichiometric chemical oxygen demand requirements calculated using the ideal gas law. In both cases the reactants were allowed to react for 120 minutes after the reaction temperature was achieved. The two–stage experimental work was done in sequence. The first stage involved pressurising the reactor with N₂ to 1 MPa under ambient conditions. The temperature was increased to 220 °C and contents allowed to react for 120 minutes. After the time set was achieved, the reactor headspace was discharged to vapour pressure of water value, assuming minimum temperature drop occurred during headspace discharging. Chemical analytical results showed that wet oxidation was the most promising option for thermal deconstruction of municipal biosolids. Table 1 shows the results obtained from the three hydrothermal process investigated to establish the process concept.

Table 1. Bench scale process selection results

Process	Solids Destruction		Product carbon	
	TSS	VSS	Carbon	Acetic Acid
	(%)	(%)	(kg/t DS)	(kg/t DS)
Thermal Hydrolysis	22%	23%	166	1
Wet Oxidation	89%	97%	155	38
Two Stage	81%	90%	205	18

Data in Table 1 shows that wet oxidation achieved 89% solids destruction compared to 81% and 22% for the two stage process and thermal hydrolysis respectively. Acetic acid was the most dominant VFA recovered. Studies by other authors (Khan et al., 1999; Wu et al., 1999; Jin et al., 2005, 2008; Shanableh, 2005; Mucha and Zarzycki, 2008; Chung et al., 2009,) have mentioned that acetic acid is highly biodegradable and can be substituted as carbon source for denitrification and phosphorous removal. A higher acetic acid yield of 38 kg/t dry solids was achieved through oxidation compared to the other processes. Therefore, no significant advantages were identified for the thermal hydrolysis and 2 stage process (Aggrey et al., 2011) and hence wet oxidation was chosen as the process that could achieve the Waste 2 Gold primary objectives.

As a result, factorial designed wet oxidation experiments were conducted in the Parr reactor at temperatures between 180 - 260 °C, oxygen partial pressure of 2 - 4 MPa and 3 - 6 % dry solids content in order to define the optimum operating envelope of the process. The key results obtained showed that:

- A maximum TSS destruction of 90% was achieved at 220 °C with no further increase at 260 C and no apparent effect of feed solids concentration.
- A maximum was obtained for acetic acid production at 220 °C with lower concentrations achieved at higher and lower temperatures.
- Optimum yields were obtained after 2 hours of operation at 220 °C.



Figure 2. Results showing the transformation of biosolids feed (cyclinder 1) at different temperatures from 180, 220 and 260 °C.

In addition to investigating the operating envelope for the pilot plant design, the following aspects were also investigated to aid in the development of the process flowsheet.

- Kinetic analysis
- Impact of different types of oxidants (O2, air, hydrogen peroxide)
- Impact of continuous oxidant supply.

3 PILOT PLANT DEVELOPMENT

A formal tender process was conducted from a selection of suitably qualified process plant designers and fabricators. Engineering contracts were awarded for the detailed engineering design, process control and fabrication of the pilot plant. A local engineering consultant firm, Allan Estcourt Limited was awarded the detailed engineering design contract. A series of regular meetings were held during the detailed design process, where each aspect of the plant was evaluated. The consultant was responsible for:

- Equipment sizing and selection.
- Material of construction selection.
- Instrument control systems,
- Specifications and drawings for all equipment (process, mechanical, structural, electrical, piping and instrument).
- Plant layout.

• Contacting the vendors for scheduling of critical equipment and materials.

The detailed engineering design of the pilot plant was effected with flexibility built into the process. Flexibility is basic to the pilot plant concept, because one of the main objectives of the pilot plant is to check a process that has been established at laboratory scale. The ultimate goal was to find a balance between proposing a plant;

- that is sufficiently scaled up from laboratory scale;
- with highly flexible operational capability;
- able to meet the initial process objectives without over-complexity of design specifications (with the resultant cost burden).

Figure 3 shows the process flow developed for the hydrothermal wet oxidation pilot plant, with Figure 4 showing the plant onsite at the RDC waste water treatment plant.



Figure 3. Hydrothermal process flow diagram.

The heart of the process is the wet oxidation reactor (Reactor 1), where biosolids content ranging from 3 % - 6% and oxidant (air/O₂) are fed under three modes of operation; batch, semi-batch and continuous operation. The heat of reaction is supplied by a heat exchanger incorporated in the slurry recycle loop which also has the dual

purpose of mixing the reactants and providing reactor temperature control. Mixing in the reactor is also enhanced by a gas recirculation loop which ensures a high oxygen transfer in the reactor to enhance reaction kinetics. Major components of the pilot plant include:

- Sludge conveyor belt, designed to take solids from the existing belt press to the feed tank;
- 500L feed tank, capable of automatic dilution of sludge biosolids to desired dry weight content;
- Two 200L pressure vessels, designed to operate up to 260 °C and 70 bar total pressure. These can be operated in series for two stage operation;
- Off-gas analysis will be via a primary O₂ sensor, but samples will also be sent to more advanced off-gas analysis using Scion's proprietary TOGA system;
- Off-gas scrubber for volatiles removal prior to discharge;
- Controlled cooling through heat exchangers and depressurisation into a 500L discharge tank;
- LPG fired thermal oil heating plant.

An external consultant, Control Synergy Ltd was contracted to assist in the development of the process control philosophy. The Scion engineering team defined the process functional description, which the consultant used in writing software program and setting up the control of instruments and equipment for the safe operation of the pilot plant.





A prime consideration in the design and operation of the pilot plant is safety. Because one of the major objectives of the pilot plant will be to define operational limits during the process evaluation, the design needs to

incorporate redundant safety systems and eventually enable the overall system to fail safely. As a result, two major Hazards and Operability (HAZOP) studies were carried out during the detailed design phase and the second, during plant fabrication. The first study was an in-house initiated study led by the consultant Design Engineer, which focussed more on the identification of perceived operational hazards and influence on the final design of the pilot plant. The second study was a more comprehensive study, where an external HAZOP expert from Safety Solutions Limited (New Plymouth) was contracted to facilitate the process. The HAZOP study identified modifications that were implemented to reduce risk and operability problems. 130 study items involving 7 nodes generated 68 action items which were all addressed prior to process commissioning of the pilot plant.

The overriding hazards associated with this operation are the high temperature (~260 °C), high pressure (~70 barg) and the use of pure oxygen which in itself is not flammable but will react violently if it comes into contact with any organic material such as grease or oil. The HAZOP study recommended that Scion adapted the Permit to Work system, which the RDC uses to manage all non routine and procedurised work in order to deal with hazards associated with the process. It was also recommended to implement a control of change procedure (change orders) to ensure hazards associated with the change have been identified, and the change has been approved/authorised by a competent person.

A Hamilton based engineering firm, Longveld Engineering Limited, was awarded the construction contract. A series of meetings with the fabricator, engineering consultant and Scion were regularly held in order to effect and approve change orders (control of change) which were generated as a result of deviations from the planned scope of work. After fabrication, individual components were tested for functionality and their ability to meet operational conditions (pre-commissioning), for example leak checking of the pressure reactor and pipework, calibrating, dry running of pumps and service connection testing.

4 PROCESS COMMISSIONING

Process commissioning work with municipal bio solids was carried out under batch operating conditions. To comply with one of the action plans identified during HAZOP study, which asserted the need to develop a cautious approach to operation start up and manage possible runaway reactions, very low biosolids content was reacted with air in excess to the stoichiometric requirements. Two trials were successfully completed using

biosolids contents ranging from 0.5% to 1.5 % at 160°C and 220 °C respectively. Figure 5 (a) – (c) shows the transformation of biosolids components achieved during the second trial run against time using 1.5% solids and 220 °C operating temperature.



Figure 5 (a) showing solids deconstruction through TSS, VSS and COD removal, (b) total VFAs and acetic acid yield, (c) soluble nutrient behaviour during the second trial run.

Data in Figure 5 revealed the following key results:

- A maximum of 72 % to 73 %, TSS removal and VSS removal was achieved respectively during the second trial run.
- COD removal ranged between 58–68 % of the biosolids input.
- Total VFA yield of 139 kg/t dry solids input was achieved. The data in Figure 5 (b) shows the yield increased with time in relation to increase in temperature, clearly showing the impact of temperature. The results also showed that acetic acid was the dominant VFA produced under the conditions.

- NH₄-N production was 33 kg /t dry solids. NH₄-N concentration increased with time as the reactions progressed.
- Soluble phosphates showed a tendency to reduce as a result of precipitation during the process.
- Maximum yields were achieved after two hours of operation.

Further pilot plant test work is currently underway to evaluate continuous and semi batch operation at higher solids concentration, and development of a mass and energy balance model that will form a basis for scale up to commercial scale plant. Other process engineering aspects like hydrodynamic performance of the reactor, reaction kinetics and mass transfer limitations will be investigated.

5 MAJOR CHALLENGES

The following are challenges and lessons learnt in the process development of the pilot plant.

- Equipment sourcing and purchasing: Significant delays in construction resulted due to long lead times for material supply, especially for the large number of items that were ordered from overseas (control valves, high pressure pumps).
- Communications: Often complex (designer, owner, contractor etc). These must be managed extremely well, as it is easy to get breakdown in communication between all parties concerned resulting in further delays.
- Commissioning and startup: This takes time, and should not be underestimated.
- Design modifications: For new project type work, this must be considered likely, and will cause delays unless considered as a normal part of pilot plant development.
- Equipment failure: Should be considered normal, particularly for bespoke builds where new processes being trialled.

6 CONCLUSION

In conclusion, this paper has revealed that with the right partnerships, technology development can be achieved. Scion in conjunction with Rotorua District council developed a technology platform for the destruction of biosolids. Proof of concept bench work tests showed the effectiveness of the technology. Development of a pilot plant for development of the case to establish a commercial sized plant was successfully completed through involvement of external engineering experts in the relevant field. Pilot scale tests which are currently underway have shown encouraging results. Significant challenges which can be encountered when embarking on pilot plant operations have been described. Despite these challenges, and acknowledging that such problems are likely in a major capital build containing a significant bespoke aspect to its construction, a fully functional thermal oxidation pilot plant was successfully achieved.

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