AGRICULTURAL RESEARCH FOUNDATION
FINAL REPORT
FUNDING CYCLE 2015 – 2017

TITLE: Evaluation of hydrothermal liquefaction for biofuels production from algal biomass and landfill leachate sludge.

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COOPERATORS: None

SUMMARY: Overall ***objective*** of this study is to investigate the potential of using algae and land fill leachate produced in Coffin Butte landfill in continuous hydrothermal liquefaction technology to produce biofuels. This project if successful will allow us to application of the HTL process for treatment of algae slurries and landfill leachate sludge to develop a practical and sustainable solution to the landfill leachate handling.

OBJECTIVES: *Our* **vision** *is to develop process technologies for production of environmentally sustainable and commercially viable algal biofuels that leverage existing infrastructure.* Overall ***objective*** of this study is to investigate the potential of using algae in continuous hydrothermal liquefaction technology to produce biofuels from land fill leachate produced in Coffin Butte landfill. While we have shown the ammonia separation and algal growth on landfill leachate in our laboratory, we have not demonstrated the biocrude production from algal biomass and sludge from the landfill leachate treatment. This project is aimed towards investigating and demonstrating the Hydrothermal liquefaction (HTL) treatment of algal biomass and landfill leachate sludge to produce biocrude.

PROCEDURES:

Task 1 Construction of a continuous algae HTL system

The HTL process which involves heating high moisture content feedstocks to near critical temperatures and pressure of water (373.95°C, 22.06 MPa), results in a biocrude which is similar to petroleum-based crudes and can be upgraded for use in existing petroleum refineries for fuels and chemicals production. The HTL process is similar to pyrolysis, but with the difference that the process can be used to process high moisture feedstocks (>50% w/w). During the HTL process, all fractions of biomass including starch, cellulose, hemicellulose, lignin and lipids present in the biomass are converted to biocrude. Thus the biocrude yields exceed the lipid content of the incoming feedstock. This is a critical advantage when processing algal biomass. Another advantage of the HTL process is that it has been shown to degrade pharmaceutical residues, pesticides and other toxic organic molecules. Hence the HTL process is a promising conversion process for both wet algal biomass and landfill leachate.

The HTL system constructed in this project (Fig. 1) consists of a diaphragm pump (LEWA-Nikkiso America, Inc.) with 300 bar working pressure capacity, 316 stainless still tube reactors (6.3 mm ID, 12.7 mm OD x 6.4 m length), heating elements with automatic high temperature shut off, fiberglass insulation, a cooling section consisting of a waterbath, a backpressure back pressure regulator which can adjust the reactor pressure to the desired value (up to 300 bar) and a product collection vessel. Two sump pumps were located inside the tanks in order to circulate the water and improve the efficiency of the cooling section. Three thermocouples were installed in three different sections of the system in order to monitor and control the temperature. An open-source electronics platform (Arduino®) was connected to relays to control electrical heaters, monitor reactor temperatures at three locations and display the reactor conditions.

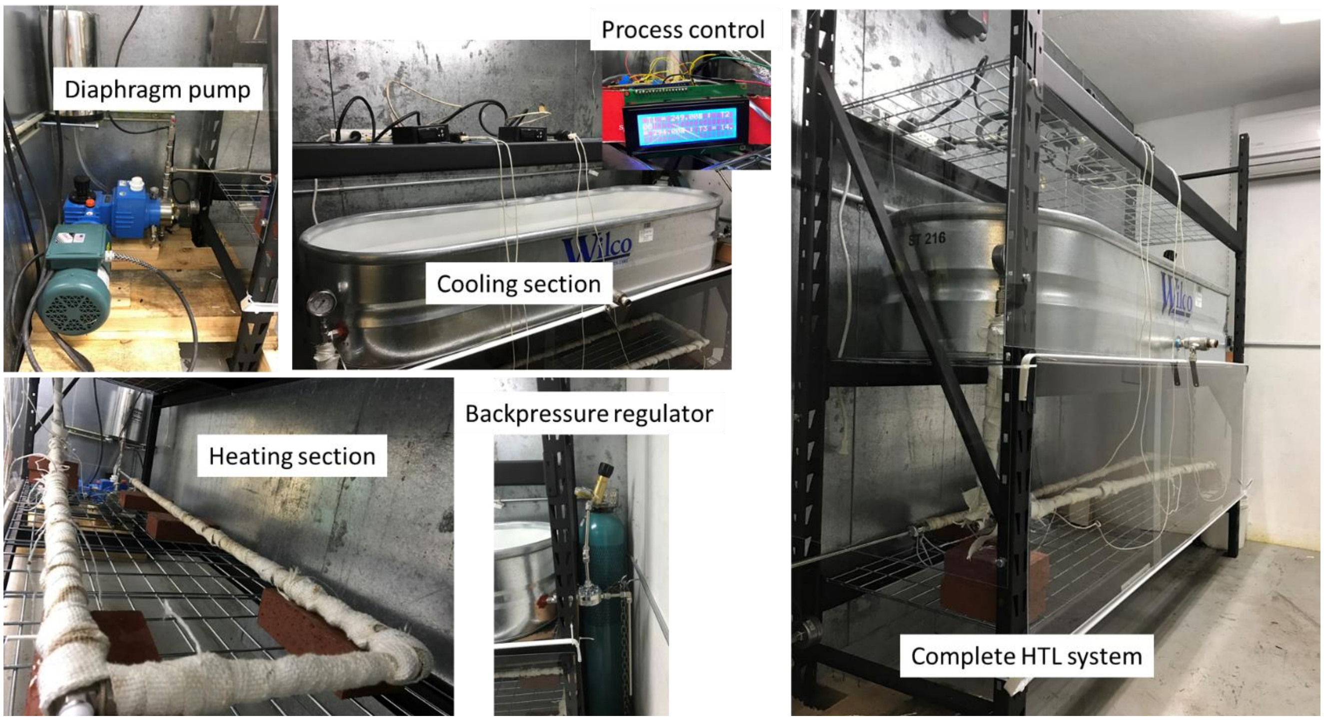

Figure 1. A continuous hydrothermal liquefaction system

<u>*Task 2 Experimental studies using algal biomass in a continuous HTL system*</u>

For a typical experiment, the HTL system was started-up by pumping water to achieve the desired processing conditions including flow rate, pressure and temperature. The flow rate was set to 8 mL/min and pressure was set to 300 bar in order to keep all the materials inside the reactor in liquid phase. Once the system reached steady state, the reactor feed was switched to the premixed algal slurry. In order to avoid any clogging, the solids content of the slurry was maintained at 10 % (w/w). To ensure steady-state operation and sampling, the reactor was operated for a minimum of 45 min (equivalent to purging the reactor with three reactor volumes of the feed solution) before any sampling was undertaken. At each operating temperature, product samples (oil–water–solid mixture) was collected in 1000 ml Duran flasks. The oil–water–solid sample mixture was extracted for 60 min in a shaker flask with dichloromethane (DCM) and then the mixture was separated and filtered to isolate the bio-crude (dissolved in DCM), solid and aqueous phase fractions. The DCM solvent was allowed to evaporate over 4 days in a fume hood at room temperature. Any remaining traces of the solvent were removed by overnight drying at 40°C in a hot air oven. The samples were weighed to determine the yields of the biocrude.

Two sets of experiments were conducted to assess the system performance using algal biomass and de-ammonified landfill leachate. In the first set of experiments, an algae slurry (10 % w/w, 7% algae ash content on dry basis) was processed in HTL reactor at three temperatures. Three products (biocrude, aqueous phase and hydrochar) were obtained (Fig. 2 and 3). The algae biocrude yields at 250, 300 and 325 °C were 35, 45 and 50% (g biocrude /g algae) respectively. Similarly landfill leachate was also processed in the HTL system.

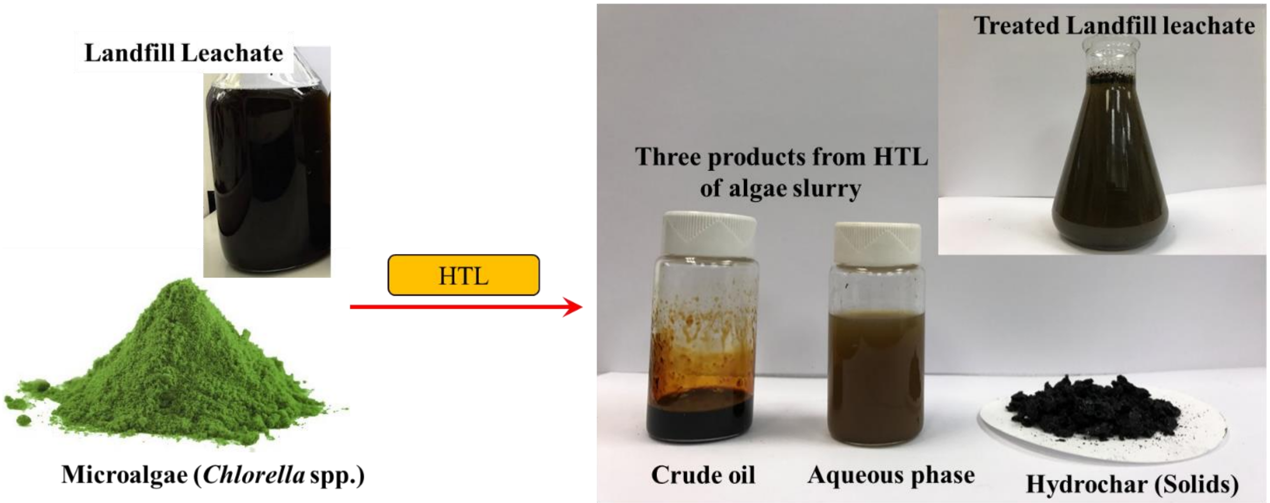

Figure 2. Products from HTL of algal biomass slurry

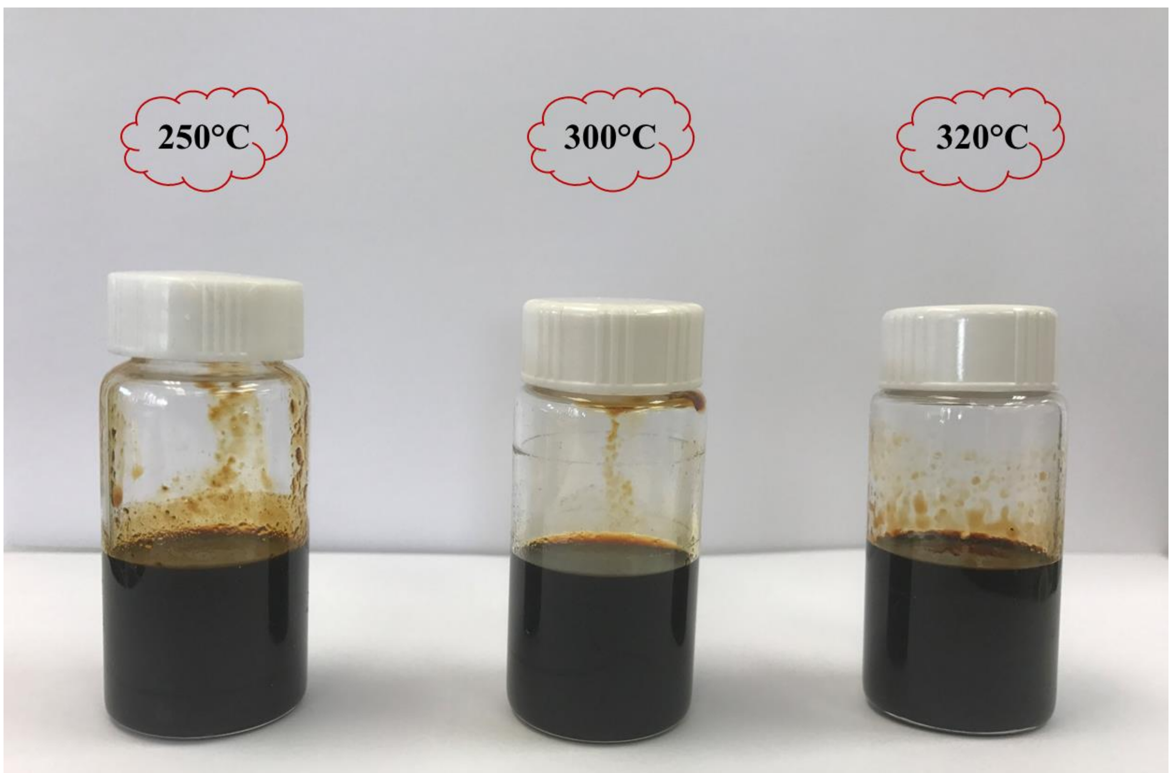

Figure 3. Biocrude from algal biomass slurry at different temperatures.

SIGNIFICANT ACCOMPLISHMENTS:

The project has been successfully completed. Based on our interactions with other researchers at other Universities and published literature, if successfully tested, this design will represent the lowest cost (~$16,000) continuous HTL system in the world. Due to this extensive redesign and long lead times for the slurry pump, the construction of the system setup was delayed and that impacted our experimental schedules. We are glad to report that the setup has been completed now (Fig. 1) and successfully tested using both algae biomass and landfill leachate as promised in the proposal.

BENEFITS & IMPACT:

This project has been very successful in developing new capabilities at PI's Laboratory and OSU. Benefits and impacts of this research are summarized below:

1. Development of a continuous HTL system that enables many research projects.
2. Successfully produced biocrude using the continuous system from both algal biomass and landfill leachate sludge.
3. Resulted in development of new proposals to NSF and USDA.
4. The HTL setup will enable continued development of strategies for treating landfill leachate sludge and algal biomass.

ADDITIONAL FUNDING RECEIVED DURING PROJECT TERM:

PI used some of his funds (salary savings and retention funds) to develop the system further and has completed the system and experiments.

FUTURE FUNDING POSSIBILITIES:

One proposal was submitted to NSF in 2015 and was not funded but received high ratings. The proposal has been resubmitted in 2016 and we are awaiting results of the panel review. The completion of this system adds unique capabilities not only to PI's laboratory but also to OSU. There are perhaps three such systems in US and OSU is one of them and will enable a large range of research to be undertaken in future. PI is already pursuing many research ideas which will generate additional proposals and collaborations.