GENIFUEL CORPORATION
CLOSEOUT REPORT FOR NAABB PROGRAM, DE-FOA-0000123
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Background

The National Alliance for Advanced Biofuels and Bioproducts (NAABB) was formed in 2009 to prepare a proposal for the Bioenergy Technologies Office of the U.S. Department of Energy. Its purpose was to explore the potential for algae to contribute substantially to the supply of US transportation fuels. The NAABB was successful in its proposal, with funding and work beginning in 2010 and continuing for 3 1/2 years. Extensive reports have been published on the work of the consortium.

Genifuel Corporation was one of the original participants in NAABB. Prior to the proposed work under this project, Genifuel Corporation had been working closely with the Pacific Northwest National Laboratory (PNNL) to test algae provided by Genifuel in a PNNL-developed process known as Catalytic Hydrothermal Gasification (CHG). That work was highly successful, and Genifuel became a licensee of PNNL technology. In the NAABB program Genifuel and PNNL continued to work as a team to apply CHG to algae supplied by other members of the NAABB. Genifuel activities related to algae and other aspects of NAABB have already been included in the previously mentioned reports.

In addition, one of Genifuel's original NAABB goals was to fabricate and demonstrate a pilot-scale system to convert algae into fuels. The purpose of this pilot system was to show that processes developed in the laboratory at bench-scale during the program could be successfully scaled up to a pre-commercial level, and thereby provide visibility into the ultimate viability and cost of algae biofuels. The definition of the pilot system and the participants in its design, fabrication, and testing evolved over time during the NAABB program, causing the successful completion of this goal to extend past the original target date. A decision was made to complete the system and then prepare a report as an addendum to the rest of the reports previously submitted. The pilot system has now been completed and tested, and this report documents what has been achieved.

Evolution of the Pilot System

As originally proposed in NAABB, the pilot system was to be a CHG unit to gasify residual algae biomass left from other biofuel processes envisioned at the time. The algae was to be provided by one of the other founding members of the consortium, processed to extract algae oil for fuel production, with the residual designated for the pilot tests. Genifuel would work with the algae supplier and PNNL to define and build the pilot system. Laboratory-scale tests integrating all of these steps were performed and showed that the CHG process worked as expected and the scaled-up system should work as planned.

Approximately halfway through the three-year schedule, a parallel development at PNNL led to the observation that an alternate fuel-production pathway for algae might be possible. The parallel effort was called Hydrothermal Liquefaction (HTL), and enabled the conversion of biomass directly into biocrude oil. A small amount of funding was provided from NAABB reserves to perform an initial test of HTL with whole algae, and the test was successful.

This successful test of HTL with algae was destined to be exceptionally significant and led to one of the most important findings of the NAABB. What it meant was that algae could be converted into a biocrude which was substantially the same as fossil crude, and could be refined into finished fuels in the same way as fossil crude, using existing refineries. The yield of fuels was substantially higher than other algae biofuel processes, and provided significant advantages throughout the algae production process.

Because of the apparent importance of HTL, NAABB management decided to add HTL as a major research component in the second half of the program. With this decision the definition of the pilot system changed from a CHG system to a combined system with both HTL and CHG capability. The CHG capability was retained because the HTL process, while very efficient, leaves some residual organic matter which can be further processed by CHG both to yield additional fuel value and also to eliminate the remaining organic content, leaving only clean sterile water.

In addition to these technical developments, other events were occurring among the planned participants in the pilot system. The original consortium member who was to provide the algae changed its business model to a new focus of selling bioreactors to grow algae, but without growing algae itself. Fortunately, at the same time another company had applied for membership in NAABB. That company was Reliance Industries Ltd., the largest private company in India and the operator of the world's largest oil refinery.

Reliance had recently started an algae program to provide an alternative feedstock for its refinery. NAABB and Reliance both felt that membership could be mutually beneficial and Reliance joined NAABB. One of Reliance's primary interests was in CHG, and they volunteered to be the new sponsor of the pilot system. When the pilot system changed from CHG to a combined HTL-CHG system, Reliance was also interested in that and expanded their planned funding to include the combined system.

Once Reliance became the new sponsor for the pilot system, Genifuel, Reliance, and PNNL all began working closely together to design and build the pilot system. The project became known as the Hydrothermal Processing Pilot System, or HPPS.

<u>System Description</u>
One of the first decisions for HPPS was what size system should be built considering algae availability, cost, risk, schedule, and ability to demonstrate pilot-scale feasibility. Sizes between 1 and 10 t/d (metric tonnes per day) were considered. The size finally selected was nominally 1 t/d, though as will be described later the actual system is capable of running at higher rates. This size measurement is based on processing algae slurry containing 20% dry algae solids mixed in water. In hydrothermal processing the feedstock is never actually dried, so a wet weight is easy to measure. However, an alternative metric would be to measure the dry weight of the algae as though it were dried, and in this case the system size would be 200 kg/d dry algae corresponding to 1 t/d wet weight, or 400 kg/d dry algae corresponding to 2 t/d wet weight.

Within this range of tested throughput, the system was actually tested for the Factory Acceptance Test (FAT) using a throughput of 1.5 t/d wet weight, or 300 kg/d dry weight. This setting was agreed with Reliance as a conservative choice without pushing the system to a higher limit. The system size will hereafter be referred to as 1.5 t/d wet weight or 300 kg/d dry weight. Using this size, the HPPS was larger than the largest previously built combined HTL-CHG system by a factor of 30.

As described previously, the system was planned to be a combined HTL-CHG system, though the two processes would be run separately and not in a combined single pass. In operation, the algae slurry would first be introduced to the system in HTL mode, producing oil and effluent water containing the remaining organic matter not already in the oil. This effluent water would be reserved in a separate tank and then later the system would be configured to CHG mode and the remaining organic matter converted to methane gas, which is renewable natural gas (RNG).

Since the user of the HPPS was Reliance, the system would ultimately be installed in India near their refinery. This site had several advantages, including the fact that the algae growing facility would be co-located with the hydrothermal processing facility. The choice of location meant the system would be shipped to India by ocean freight, and would therefore need to be designed to fit into ocean-going shipping containers. The design was a skid-mount system built on two skids which could go into standard containers. Top-level specifications were developed by Genifuel Corporation in consultation with PNNL, detailed design and engineering was performed by Merrick & Company, and fabrication was performed by Springs Fabrication Inc. (SFI). Genifuel is located in Utah, while Merrick and SFI are located in Colorado.

After the overall size and operating mode were agreed, and the necessary contracts prepared and signed, front-end engineering work began in April 2013. The system was built and ready to begin commissioning tests by August 2014. The engineering and fabrication were somewhat more complex than originally anticipated because Reliance needed the system to be designed in the same way as their refinery systems, with similar HAZOP review, instrumentation, and control system. This would allow the HPPS to be integrated seamlessly into a control environment familiar to their refinery operators.

Commissioning and Acceptance Testing

Commissioning of the HPPS began in late August 2014 after completion of fabrication. The commissioning process took longer than originally scheduled, with a total duration of slightly over one year. However, of this year only six months was actual work time because a contractual hiatus took place while additional commissioning funds were requested and approved within Reliance.

The difficulties in commissioning were not due to design or process deficiencies, but rather were the result of a series of overlapping equipment failures which proved to be hard to diagnose. Failures occurred in the high-pressure pump, flow-measurement meter, continuous stirred-tank reactors (CSTRs), several digital instruments, and parts of the control software. All of the failures were relatively minor but because they overlapped it was hard to diagnose the exact source of the problems encountered. Diagnosing and repairing the problems required the support of the equipment vendors, specifically with onsite support from their headquarters, which was often slow to arrive. Ultimately the problems were resolved with minimal expense, but significant schedule delay.

An example of the type of problem encountered was with the high-pressure pump. This pump is ideally suited for the service required, but the model installed had recently been redesigned to incorporate a new hydraulic relief valve to protect the pump from overpressure. The new overpressure regulator inside the pump proved to have a high failure rate, and the manufacturer had to redesign the valve and retrofit all pumps already in the field. After repeated failures of the HPPS pump the new valve was installed and the problem was solved—but with a delay of more than three months.

Once the startup bugs were resolved the system demonstrated stable, reliable operation. After the initial repairs no further failures occurred for the rest of the commissioning and testing. The system operates at 350ºC (662ºF) and 207 bar (3,000 psi), and it was discovered that after the first few hours of operation all fittings and bolts needed to be re-torqued to prevent leaks. The same process was needed a second time after about a week of operation. After that no further adjustments were needed and no new leaks developed. In operation the HPPS was almost silent—in fact within the often noisy factory environment in was hard to tell at times if it was actually operating.

System Specifications

The table below gives several key operating specifications of the HPPS.

Table 1: HPPS System Specifications

ITEM	MEASURE
Operating pressure	207 bar (3000 psi)
Operating temperature	350ºC (662ºF)
Flow rate range (wet slurry)	1,000 to 2,500 L/d (264 to 660 GPD)
Flow rate as tested for FAT	1,500 L/d (396 GPD)
Liters per hour space velocity range	1.5 to 3.75
Liters per hour space velocity as tested for FAT	2.25
Power requirement at full rate	App. 50 kW
Size (total of both skids)	2m x 10m x 3.5m high (6.6’ x 33’ x 11.5’)*
Weight (total of both skids)	13,000 kg (28,660 lbs.)

*Several items extended above the envelope of the shipping container and were removed for shipping.

Factory Acceptance Test (FAT)

The FAT included a predefined series of steps with gradually increasing feedstock slurry concentrations, temperatures, pressures, and flow rates. The system was operated continuously 24 hours per day for the week of the FAT. Feedstock slurry was prepared throughout the testing period using dried algae purchased and stored in preparation for the testing. The testing started in HTL mode, saving both the oil and water produced during liquefaction. The plan included HTL testing as a first step, followed by CHG testing with the saved effluent water from the HTL tests.

As the testing in HTL mode proceeded, it was clear that the system was performing reliably with expected outputs. At this point a meeting was held with Reliance to assess progress and review the remaining schedule for running CHG. In order to run CHG, the system would have to be at least partially shut down to allow for the safe loading of catalyst into the CHG reactors. Reliance made a decision at this point to forego further testing in the CHG mode for the following reasons:

- CHG had been tested very extensively at PNNL and witnessed by Reliance during NAABB
- Much of the process pathway through the system is the same between HTL and CHG
- Once the catalyst is loaded into the system and run (even for a short time) it is quite difficult to safely repackage and ship to India, with the possibility of significant loss

Accordingly, the FAT was concluded after the HTL mode was complete and the acceptance certificate was signed by Reliance.

<u>Shipping</u>

After completion of the FAT, the HPPS was thoroughly cleaned inside and out with both water and a mild solution of acetic acid (vinegar). When cleaning was complete, the system was shut down and disconnected from power, then the two skids were disconnected electrically and mechanically. Items which extended above the skid outside the envelope of the shipping container were removed for separate packing. All spares and supplies were collected.

All parts were then crated, wrapped, and inventoried. The containers, which had been previously obtained and situated, were cleaned and prepared for shipment. All items were loaded and stabilized and all shipping and customs documents completed. At the time of this report the system is on a container ship bound for India.

<u>Next Steps</u>

When the system arrives in India it will be transferred by truck to its final installation site, where a pad, roof, utilities, storage tanks, and control room have already been prepared. The system will be installed, the two skids reconnected, all removed parts re-installed, and all electrical wiring reconnected. The system will then begin a series of tests similar to the commissioning and FAT tests.

Once startup and testing are complete, Reliance will begin using its own algae to produce biocrude oil and biogas.

<u>Testing and Results</u>

During testing at PNNL, the standard procedure is to schedule each run with a specific objective, run conditions and duration, usually approximately 15 hours including heatup and cooldown. The steady-state operation is usually six to eight hours. Samples are taken during the test and analyzed afterward. The results are then compiled into a complete description and documentation of the test. The entire process takes approximately six weeks.

In the case of the HPPS FAT, the testing period was much longer than the PNNL tests and was continuously varied to show operation under different conditions. For this reason it was impossible to collect steady-state data at a single previously defined temperature, pressure, flow rate, and feedstock makeup. Nevertheless, the testing team did obtain data in the final stages of the test at the last two sets of conditions.

The table below shows the variables in these final two test ranges.

Table 2: Range of variables for in final stages of HPPS testing.

VARIABLE	MEASURE
Temperature	325ºC to 355ºC
Pressure	175 bar to 205 bar
Feedstock solids concentration	15% to 20%
Flow rate	1,500 to 1,750 L/d
Algae used	Spirulina (Arthrospira platensis)

The output of the system was directed to an empty recovery tank at approximately the time when the HPPS was running with the foregoing set of conditions. The runtime with this set of conditions was approximately 14 hours. Measurements of oil and water levels in the recovery tank were taken, and samples of oil were drawn for delivery to a testing lab in Wyoming. Samples were delivered to the lab immediately after the completion of FAT.

In addition, a number of samples of the oil and water were taken for delivery to Reliance and PNNL. The purpose of delivering the samples to PNNL was to allow PNNL to perform independent tests so that the same testing procedures could be used for this oil as for other HTL oil samples previously analyzed and documented by PNNL.

The results of the immediate testing in Wyoming and the later testing at PNNL gave very similar results, which serves as a check on the testing procedures. Both tests showed that HPPS performance and oil production were very similar to the performance and oil production reported by PNNL from many bench-scale tests with a variety of algae samples.

Table 3: Typical results for HTL oil

ITEM	WEIGHT %, DRY ASH-FREE BASIS
Oil Yield	38%
Carbon	78.6%
Hydrogen	10.4%
Oxygen	5.3%
Nitrogen	4.2%
Sulfur	0.5%
TOTAL	**99.0% ***

Density	0.953
TAN	59 mg KOH/g
Viscosity	114 cP @ 75ºF

*Remainder is small amounts of other elements including phosphorus.

Conclusion

The results of the HPPS project are quite positive for the commercialization of Hydrothermal Processing. The rule of thumb for scaling process systems is that a scale-up of about 25x is a reasonable target for good results. The HPPS scale-up factor was approximately 30x and was successful once several equipment problems were identified and solved. It is noteworthy that the particular equipment issues which caused problems during commissioning were not specifically related to scale-up.

The oil produced by the process was very similar to the oil produced by PNNL at smaller scale, indicating that scaling did not hurt oil volume or quality. If anything, the results of the larger system seemed somewhat better, though more precise testing would be needed to verify that.

Overall, once the commissioning was complete the FAT showed that the system was safe, reliable and controllable. During operation the HPPS was nearly silent and gave the impression of a stable and well-mannered system.

Finally, though it was agreed that the FAT would be conducted at 1,500 L/d capacity, it was clear from tests during the commissioning that the HPPS could perform at a higher capacity, possibly much higher. If a much higher throughput was to be used for long-term operation, several pieces of equipment would have to be changed, especially the trims on the pressure regulator valves, which were specified for the lower capacity in the original conservative design. Based on results achieved by PNNL, it is possible that the HPPS could be used at capacities as high as 4,000 L/d (corresponding to a space velocity of 6.0) with appropriate upgrades of several relatively small items of equipment. This would reduce the capital cost per unit of production by 75% compared to the original target throughput of 1,000 L/d. Such a result would have a dramatic effect on the cost of biofuels produced by Hydrothermal Processing.

Photos

The following pages show a series of photographs documenting the design, fabrication, testing, and shipment of the HPPS.


Engineering review of 3D Layout

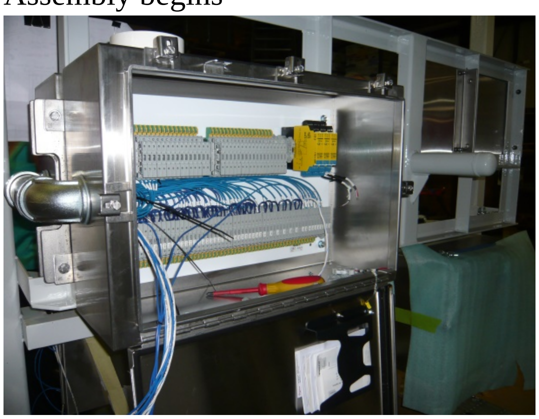

Skid fabrication begins


Skid in paint booth


Parts arrive


Assembly begins


Assembly continues

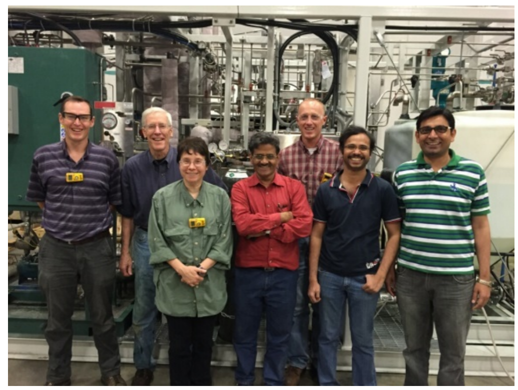

Electrical assembly


Reliance first review of system


Assembly complete


Mixing algae feedstock for FAT


HTL oil from algae


Team completes the FAT


Loading HPPS into container


Container ship departs for India


Merrick engineering team


Springs fabrication team