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Hydrothermal Liquefaction of Agricultural and Biorefinery Residues

Final Report – CRADA #PNNL/277

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Summary

This project was performed as a Cooperative Research and Development Agreement (CRADA) with the participants: Archer-Daniels-Midland Company (ADM), ConocoPhillips (COP), and Pacific Northwest National Laboratory (PNNL). Funding from the federal government was provided by the Office of the Biomass Program within the Energy Efficiency and Renewable Energy assistant secretariat as part of the Thermochemical Conversion Platform. The three-year project was initiated in August 2007 with formal signing of the CRADA (#PNNL/277) in March 3, 2008 with subsequent amendments approved in November of 2008 and August of 2009.

This report describes the results of the work performed by PNNL and the CRADA partners ADM and ConocoPhillips. It is considered and is not available for public disclosure.

The work conducted during this project involved developing process technology at PNNL for hydrothermal liquefaction (HTL) of agricultural and biorefinery residues and catalytic hydrothermal gasification (CHG) of the aqueous byproduct from the liquefaction step. Related work performed by the partners included assessment of aqueous phase byproducts, hydroprocessing of the bio-oil product and process analysis and economic modeling of the technology.

As a part of this project three Battelle conceived three Subject Inventions and filed invention reports describing the new technology developed within the project:

| | | |
|--|------------|---------|
| Improvements to hydrothermal liquefaction | 9/25/2009 | 16490-E |
| Mineral separation in hydrothermal liquefaction | 10/12/2009 | 16525-E |
| Hydrothermal gasification with hydrothermal liquefaction | 12/21/2009 | 16611-E |

Task 1: Feedstock Effects

Initial tests in hydrothermal liquefaction (HTL) were performed in a micro-scale continuous-flow reactor at PNNL. The system required a clear or nearly clear feedstock because of the small orifices involved in the design. Appropriate model compounds and feedstocks were provided by ADM to PNNL for liquefaction tests.

- Model compounds were selected for the initial liquefaction tests.
- Pretreatment and fractionation of the feedstocks were evaluated by the ADM team.
- Micro-scale tests were undertaken in the existing continuous-flow reactor systems at PNNL.
- Feedstocks included agricultural and biorefinery residues and pretreated and fractionated versions thereof and were provided to PNNL by ADM.
- The incorporation of catalysts was tested at PNNL.

In parallel with the studies on feedstock effects, ADM and ConocoPhillips requested PNNL to make large samples of corn fiber and stover liquefaction oil using PNNL's current liquefaction technology. ConocoPhillips evaluated upgrading of this oil utilizing various standard refinery processes through lab scale tests.

Task 2: Process Optimization

Based on the experimental results in Task 1 and guided by the assessments in Task 5, bench-scale process optimization was undertaken at PNNL. The bench-scale tests also provided product oil in sufficient quantity for subsequent analysis and upgrading tests in Task 4.

- The bench-scale Continuous-flow Reactor System was used to optimize processing conditions for hydrothermal liquefaction, such as temperature, pressure, and residence time.
- The incorporation of alkali catalyst (sodium carbonate) was tested at PNNL

Task 3: Aqueous Product Assessment

- Detailed analysis of the aqueous phase determined composition and quantity of dissolved organic material as led by ADM with contribution from PNNL.
- Treatment or recovery of these potentially useful or valuable byproduct materials was a focus of the ADM team.
- Since the composition of the aqueous stream included only low levels of organic material, ConocoPhillips concluded that upgrading it to fuels was not reasonable.
- Following characterization of the stream, ConocoPhillips and ADM determined the best use of the stream would be as recycle to the hydrothermal liquefaction process, or hydrothermal gasification to methane which could then be used for combustion to generate high temperature steam for the process, or conversion to hydrogen to supply the hydrotreating process.
- PNNL performed bench-scale catalytic hydrothermal gasification tests to evaluate fuel gas production from the aqueous stream as a means of energy value recovery using this technology.

Task 4: Upgrading with Heteroatom Removal

Catalytic hydroprocessing of the hydrothermal liquefaction product oil was performed by Conoco-Phillips at the bench-scale. Mass balances around the process were determined and products recovered for detailed analysis for fuel applications.

- Hydroprocessing was utilized for upgrading the crude oil product.

- Targets for this processing included not only the oxygen heteroatoms, but also nitrogen, as well as sulfur.

ConocoPhillips sought to determine:

- Is the material suitable for directly blending into fuel?
- If not, what further treatments, including co-processing to make a material suitable for fuel in laboratory fixed bed test reactors are needed?

In addition, ConocoPhillips

- Evaluated catalytic cracking in laboratory screening reactors as an alternative upgrading option.
- Screened the fuel properties of the upgraded products.

Task 5: Techno-Economic Assessment

- Developed a baseline process model to allow techno-economic assessments.
- Utilized the model to identify the potential technical improvements that have the most significant impact on process economics.
- Permutations to the baseline model, such as the evaluation of the eliminations of catalyst and reducing gas in the liquefaction step were addressed.
- Using ADM's market information, an assessment of the scale of operation based on availability of feedstock was also undertaken. As experimental data was obtained, the model was updated throughout the life of the project.
- ConocoPhillips contributed the modeling of the upgrading portion to produce fuels.

Conclusions

Hydrothermal liquefaction can be applied to corn fiber, corn starch, or corn stover in water slurry to produce a bio-oil with 10-15% oxygen on a dry basis. Overall carbon basis yields for the several feedstocks ranged from 20% for starch, 50-55% for fiber and 30-35% for stover. The undesirable oxygen content of these HTL bio-oils is much lower than that achieved through fast pyrolysis of biomass, but at the expense of a lower bio-oil yield. The bio-oil can usually be gravity separated from the aqueous byproduct but the formation of a stable emulsion was seen during the processing of corn stover. It was thought that the mineral (ash) content of the feedstock caused this phenomenon therefore a mineral separation step prior to phase separation was developed. Only a small fraction of the biomass is converted to a gas byproduct (5-10% of the carbon) consisting mainly of carbon dioxide. The balance of the carbon is found in dissolved organics in the aqueous byproduct stream. Recycle of this aqueous stream as the solvent in the preparation of the feed slurry appears to facilitate the conversion of water soluble organics to bio-oil. Additionally, the aqueous byproduct stream can be processed via catalytic hydrothermal

gasification technology to produce fuel gas and a low biological oxygen demand (BOD) aqueous stream. The methane produced through gasification could be reformed into hydrogen and is sufficient to provide all the hydrogen required for upgrading the bio-oil to fuel.

The bio-oil product can be hydroprocessed in two stages to form hydrocarbons. Fractionation of the resulting product showed 14 wt% gasoline range, 58 wt% diesel range, and 28 wt% gas oil. The gasoline fraction had an octane value of 79 and could be used as a sub octane blending component. The diesel boiling range fraction had high aromatics content and would be suitable for distillate blending, solvent applications, or further processed via catalytic cracking. The gas oil fraction could be blended into fuel oil or further processed via catalytic cracking.

Based on the techno-economic analysis of the process the overall capital expense for a unit capable of processing 525,000 mt/year of biomass is approximately \$125 million. The annual operational expense is approximately \$72 million (including feedstock cost). Based on a yield of 42.5 gallons upgraded bio-oil per metric ton of corn stover, the minimum selling price of the bio-oil is \$4.11 per gallon (172.62/bbl). Because further refinery processing is required to incorporate the bio-oil into a final finished fuel, the bio-oil would have a refinery break-even value similar to light to medium gravity low sulfur crude oil or condensate. The current premiums for these grades of crude oil range from \$3-7 over NYMEX WTI. Thus the current price structure of the crude market does not support commercialization of this process at its current stage of development.

Currently both ADM and ConocoPhillips do not plan to conduct further research and development with this process. Significant barriers to commercialization of this technology are identified as follows:

- Low primary oil yield
- Energy consumption for grinding biomass into a slurry
- Process and product sensitivity to feedstock impurities.
- Hydrogen requirements for upgrading

Significant technical improvements addressing these barriers are needed before warranting additional evaluation of this technology.

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Introduction

The purpose of this project was to develop an understanding of hydrothermal liquefaction as applied to agricultural residue and co products produced in Archer-Daniels-Midland Company's (ADM) biorefinery operations. ADM, Pacific Northwest National Laboratory (PNNL), and ConocoPhillips Company (COP) coordinated bench-scale research efforts to generate process information to optimize the application and to allow scale-up of the technology.

Hydrothermal liquefaction of biomass with subsequent upgrading of the crude oil product-provides an efficient pathway to liquid transportation fuels to displace imported petroleum. Hydrothermal processing utilizes water and/or organic solvent at medium temperatures (300–350°C) and sufficient pressure (15.9–20.7 MPa) to maintain the water in the liquid phase. The processing option is particularly applicable to wet biomass feedstocks, such as biorefinery residues.

Hydrothermal processing of biomass to liquid fuels requires expanded process development to take the technology to an industrial demonstration scale. Technical challenges associated with the technology include mixing, pressurization, transport, and pressure let down of high solid slurries, but also understanding the relationship between crude oil product properties and feedstock composition. Other challenges include optimization of the liquefaction process variables; demonstration of separation techniques; and demonstration of bio-oil upgrading processes in order to produce a product with marketable commercial value.

Feedstock Effects

Initial tests in hydrothermal liquefaction were performed in a micro-scale continuous-flow reactor at PNNL. The system required a clear or nearly clear feedstock because of the small orifices involved in the design. Appropriate model feedstocks were provided by ADM to PNNL for liquefaction tests. The results of these tests were provided in a separate report (PNNL-18644)¹.

Pretreatment and fractionation of the feedstocks was evaluated by the ADM team. A single sample of a hydrolyzed feedstock was provided to PNNL. Micro-scale processing was attempted in the continuous-flow reactor systems, which was modified for liquid product collection. The majority of the tests all ended with plugging of the feed lines in the preheating stage of unit. The high level of sulfuric acid in the product required neutralization prior to liquefaction which was accomplished by the addition of alkali. The plugging was due to alkali precipitation at the operating temperatures needed for hydrothermal liquefaction.

In parallel with the studies on feedstock effects, ADM and ConocoPhillips initially requested PNNL to make large samples of corn fiber and stover liquefaction oil using PNNL's bench-scale liquefaction reactor system. ConocoPhillips evaluated the upgrading of this oil through lab scale tests on hydrotreating and fluidized-bed catalytic cracking.

Micro-scale Process Results with Model Compounds

The process diagram for the micro-scale hydrothermal liquefaction test system is shown in **Figure 1**. A picture of the system is given in **Figure 2**. The processing system is constructed of 316 stainless steel and is designed to operate at the process conditions of up to 400°C and up to 20.7 MPa.

The high-pressure metering syringe pumps from Isco were used to pump the feed solutions into the preheater/reactor component, pictured in **Figure 3**. The 1/4" preheater tube operated in a downflow configuration and fed into the bottom of the 1/2" tubular reactor, which had an internal volume of 50 cc. Temperature was monitored by thermocouples in the feed line; shortly after the preheater, about 2 inches into the reactor; and toward the end of the reactor, about 2 inches from the outlet. The product effluent left the reactor, passed through a chiller and then drained into two liquid sample collection vessels (samplers). The samplers were operated at system pressure and were valved in and out of the process flow to alternately fill and empty them. Product gases (along with nitrogen used to prepressurize the liquid sample collection vessels) were vented from the top of the samplers through the back-pressure regulator (BPR) and measured by a wet test

¹ Elliott, DC; Rotness, LJ; Hart, TR; Neuenschwander, GG; Hydrothermal Liquefaction of Agricultural and Biorefinery Residues – Interim Report Micro-scale Tests with Model Feed stocks, PNNL, Richland Washington, August 2009, PNNL-18644.

meter (WTM). Gas samples were manually collected from the vent line and analyzed by gas chromatography.

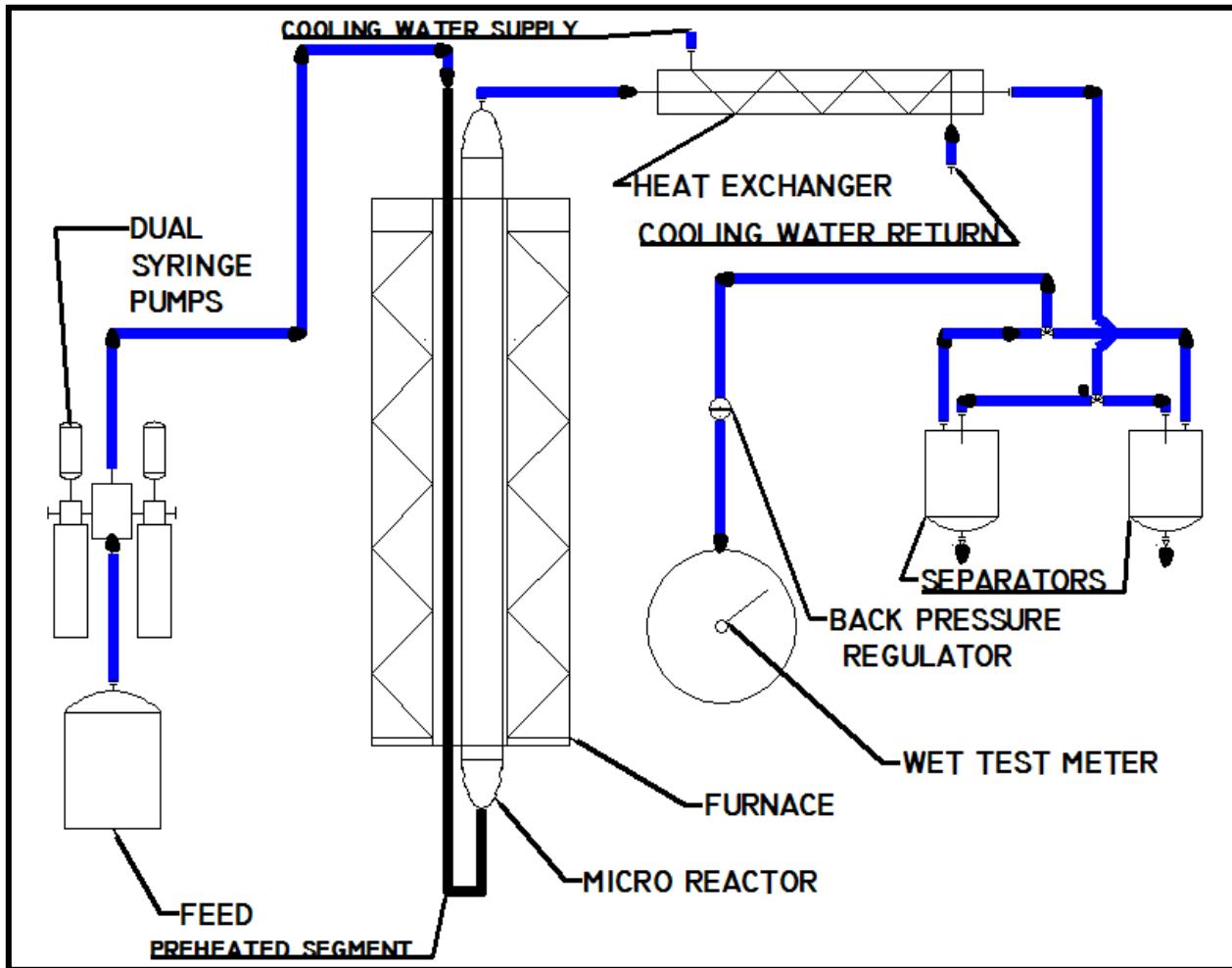


Figure 1. Process Flow Diagram of the Micro-Scale Hydrothermal Liquefaction System

In the process discussed in this report, the model feedstocks were reacted as water solutions to determine the yield of bio-oil product, which could be phase-separated from the aqueous byproduct. The bio-oil phase and the aqueous phase byproduct were recovered and separated by pouring and/or pipetting the low viscosity aqueous phase from the viscous bio-oil phase.

This microscale reactor had no internal agitation, such as a static mixer, but temperature management was viewed as adequate for the tests completed. Coking in the reactor was not found to be a major factor with these feedstocks. Other unsuccessful tests were attempted with a hydrolyzed corn fiber feedstock provided by ADM. This highly acidic material coked up immediately in the reactor and subsequent tests with neutralized (with sodium carbonate)

feedstock resulted in precipitation of solids in the preheating lines that plugged and stopped the flow.



Figure 2. Micro-scale Hydrothermal Liquefaction System

Table 1 summarizes the results of the micro-scale hydrothermal liquefaction process tests with model feedstocks. These results were all generated at 343-350°C and 20.4-21.2 MPa, with a 2.0 liter of solution per liter of reactor volume per hour liquid hourly space velocity (LHSV). All the feedstock solutions contained an added catalyst of 2 wt% Na₂CO₃ (on the total solution basis) to moderate the pH of the reactor system and maintain a near neutral processing environment. As a result, the feedstocks each had a pH of around 10, but the product aqueous phases had pH levels near 5 (7 in the isosorbide case).



Figure 3. Preheater/Reactor

Table 1. Process Results with Model Feedstocks

| | dextrose | HFC syrup | sorbitol | isosorbide |
|---|----------|-----------|----------|------------|
| Carbon conversion to oil, % | 44.5* | 50.9* | 1.8 | 1.5 |
| Carbon remaining in aqueous, % | 31.6* | 39.9* | 59.7 | 70.8 |
| Carbon conversion to gas, % | 3.6* | 0.5* | 0.05 | 0.3 |
| Carbon balance, % | 89 | 69 | 62 | 73 |
| Mass of recovered bio-oil, % of dry feed | 20.2 | 15.5 | 0.8 | 0.9 |
| Oxygen content in raw oil product, % | 19.6 | 23.5 | 15.5 | 15.9 |
| Oxygen content calculated to dry basis, % | 13.7 | 15.6 | 7.7 | 10.5 |
| pH of aqueous | 4.84 | 4.80 | 5.16 | 6.79 |
| Carbon content of aqueous, % | 3.63 | 2.79 | 12.44 | 16.62 |
| Carbon content of feed, % | 12.40 | 8.94 | 14.44 | 17.32 |

* for the glucose and HFC tests, the carbon balance was normalized to 100%, while including estimated char deposit in reactor (not shown in table)

These results showed a substantial bio-oil yield for the sugar feedstocks with very little bio-oil produced from the hydrogenated sugar (sorbitol) or the dehydrated sorbitol product, isosorbide. Gas yields were low in all cases. The bio-oil product was highly oxygenated (15-24 wt% oxygen, as recovered) in all cases. However, the raw bio-oil has a significant water content which contributes to the measured oxygen. On a dry basis, the bio-oil oxygen was much lower (8-16 wt% oxygen, dry basis). In the two cases where little bio-oil was produced; most of the carbon remained dissolved in the aqueous phase.

Liquid Chromatography analysis was used to further clarify the extent of reaction of the model feedstocks. **Figure 4, Figure 5, and Figure 6** compare the feedstock with the products of the several feedstocks. The dextrose was converted in the hydrothermal processing and the remaining dissolved organics were a collection of oxygenates with acetic and glycolic acid being the most prominent (phenolics were not determined in this analysis). Essentially the same product slate was seen with the high-fructose corn (HFC) syrup. The sorbitol was converted to a significant degree into what are believed to be dehydrated sugar alcohols, sorbitans, idatan and mannitan, (misidentified as C5 sugar alcohols and lactic acid in Figure 5) with a notable yield of isosorbide also forming. Isosorbide was essentially unchanged by processing at these conditions with a small yield by hydration to sorbitol.

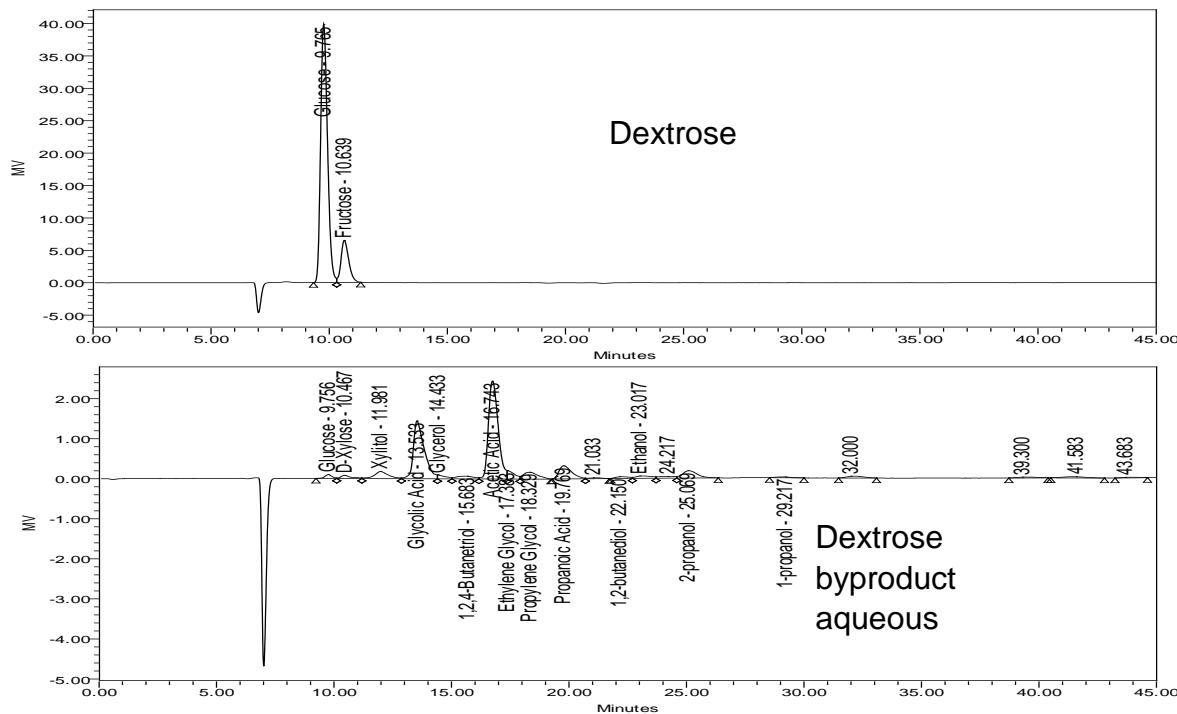


Figure 4. Liquid Chromatography of Dextrose Feedstock and Products

Subsequent tests were attempted with a biomass hydrolysate feedstock produced by ADM. The feedstock, as received, was an ultra-filtered (to remove protein), cation exchanged (to remove residual amino acids) hydrolysate produced from corn fiber. The material was determined to have a pH of 0.01. Sufficient sodium hydroxide was added to neutralize the feedstock (to a pH of 7.7) before it was processed. The sulfate content was measured by ion chromatography and found to be far outside the instrument's calibration range. of 2000 ppm, and perhaps as high as several weight percent.

These process tests were all ended prematurely with plugging in the preheater tube. Precipitation of sodium sulfate was suspected to be the cause. The small amount of bio-oil collected appeared to have a similar composition to the other bio-oils produced in this project, but no material balance or yield calculation could be obtained from the shortened runs. The aqueous byproduct carried 3.3% dissolved carbon and had a pH of 4.4. In one test, a small batch anion exchange attempt was made which raised the feedstock pH to 3.9. NaOH was then used to raise the starting pH to 7.3; however processing this feed gave the same result of a plugged preheater.

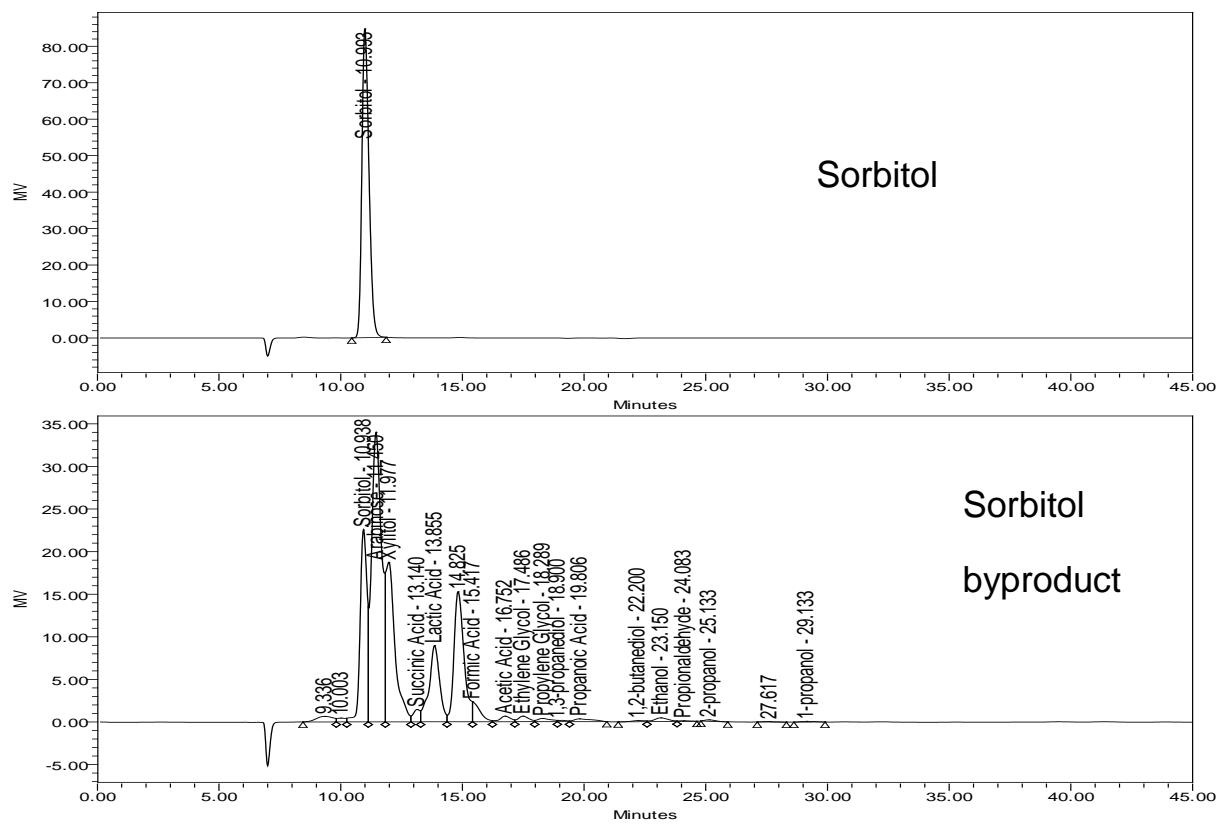


Figure 5. Liquid Chromatography of Sorbitol Feedstock and Products

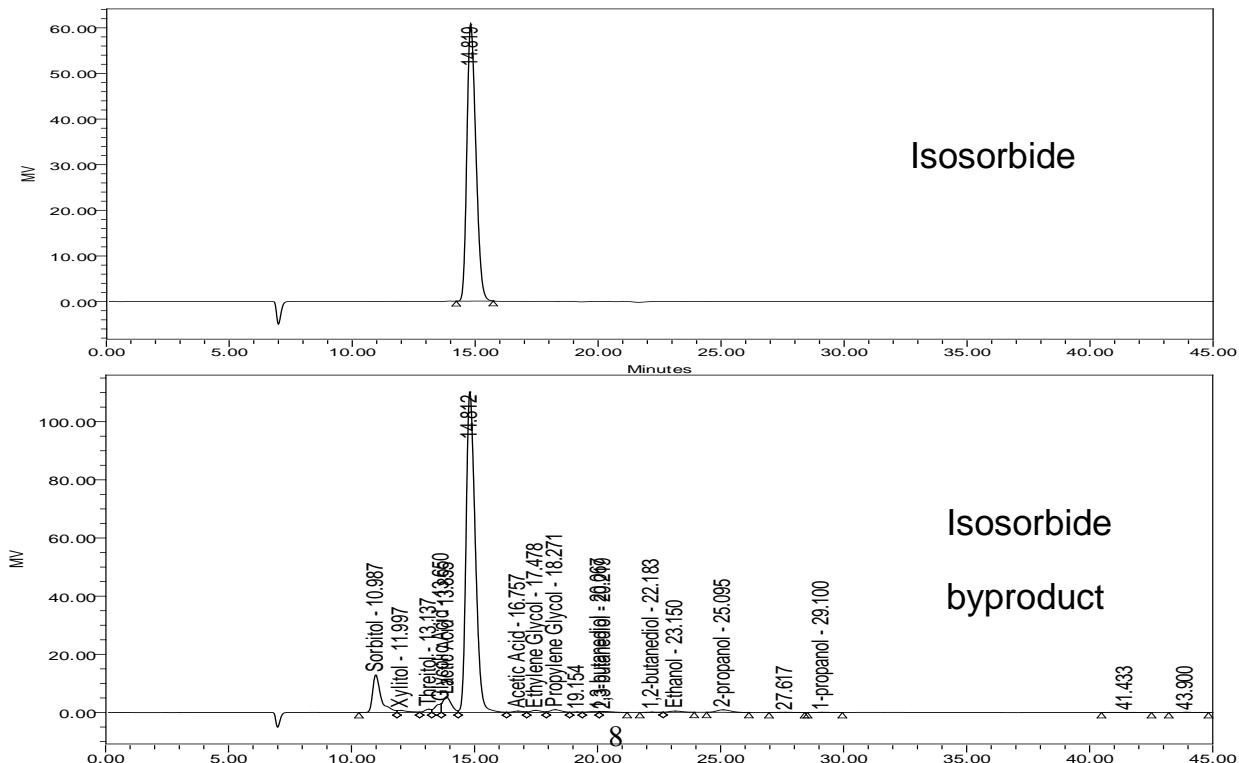


Figure 6. Liquid Chromatography of Isosorbide Feedstock and Products

These micro-scale hydrothermal liquefaction tests with model feedstocks demonstrated the principle of bio-oil formation from sugars in a pH moderated reaction system. At a temperature of 350°C and 21 MPa (pressure sufficient to maintain liquid water in the reactor system), solutions of dextrose and high-fructose corn syrup were reacted to produce a water immiscible bio-oil phase in a carbon yield of 45-50%. Minimal gas formation accompanied the reaction. A significant portion of the sugar feedstock remained dissolved in the byproduct aqueous phase. In these tests, conducted at around 30% dry solids solution, the residual yield of carbon to the aqueous phase was about 30-40%. Some coke development on the reactor wall was also seen. The processing rate was a LHSV of 2.0, which equates to a residence time of about 19 minutes based on the water density of 0.63 g/mL at the reaction conditions.

Non-sugar feedstocks did not form, to any significant degree (<2%), a separable bio-oil product. Sorbitol (a C6 sugar alcohol) was dehydrated to C6 sugar alcohol anhydrides and, isosorbide (the dianhydride). Isosorbide was almost unreactive, producing only a small yield of sorbitol, as evidenced by the HPLC result.

Process Optimization

Bench-scale process optimization was undertaken at PNNL. The bench-scale tests also provided product oil in sufficient quantity for subsequent analysis and upgrading tests in Task 4.

The bench-scale Continuous-flow Reactor System (CRS) was used to optimize processing conditions for hydrothermal liquefaction, such as temperature, pressure, and residence time.

The incorporation of alkali catalyst (sodium carbonate) was tested at PNNL

Reactor System Design

The CRS was composed of five major functional subsystems: feed pretreatment and preparation, pumping, preheater/reactor, reaction products separation, and instrumentation and control. The system was based on a throughput of 0.5-10 lb of slurry or solution per hour and was typically operated over a range of 1 to 3 liter/hour. The process flow diagram (without the pretreatment section) as initially configured is shown in **Figure 7**.

The CRS is designed for obtaining engineering data for continuous flow hydrothermal liquefaction process. The system consists of the high-pressure pump feeding system, product recovery system, data acquisition and control system, furnaces, and other equipment required to utilize the 1-liter Carberry stirred tank reactor (MAWP 6500 psi @ 800°F) and the 1-liter tubular reactor. The tubular reactor (MAWP 10,000 psi @ 72°F or approximately 7500 psi @ 400°C) can be run as a stand-alone unit or may utilize the Carberry as a stirred tank pre-heater. The CRS can be run with the removal of either of the two primary pressure vessels. The feed line, operated at ambient temperature, is ½" 316 SS tubing with 0.049" wall. All process lines at temperature of 200°C or above are ¼" 316 SS tubing with 0.065" wall). The product collection is done via two 1-liter Parr vessels.

The CRS feed system is a dual-barrel continuous-flow Isco syringe pump. After the pumps, the feed can be heated in the 1-liter stirred Carberry reactor. The feed continues to the 1-liter tubular reactor for the final process step, and then it is alternately sent to one of two PARR vessels to collect the liquids at pressure. The liquids collect in the temperature-controlled PARR vessels and gases are vented via a dome-loaded back-pressure regulator (BPR). The off gas is cooled by another chilled heat exchanger to further remove any entrained water, the liquid is accumulated in a weighed tank, and the off-gas is measured by a wet test meter and analyzed manually by a gas chromatograph. Pressure transducers on each vessel record pressures and note pressure drops due to restriction and plugging. Each vessel and most transfer lines are also monitored for temperature. Three rupture discs protect the system. A data acquisition/control system heats the furnaces and records the process parameters and offers off-normal warnings and auto-shut down.

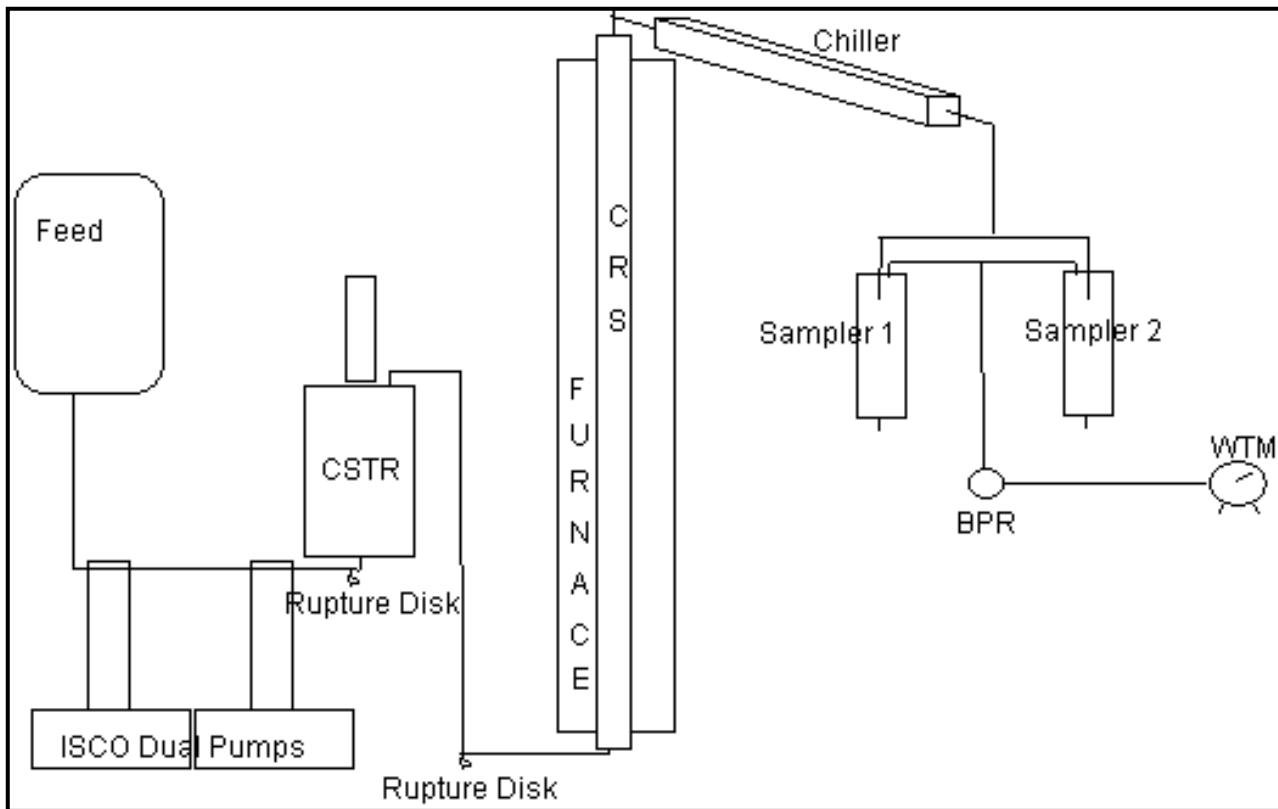


Figure 7. Initial Process Flow Schematic of the Bench-Scale Continuous-Flow Reactor System (CRS)

Feed Pretreatment and Preparation

The feedstock pretreatment and preparation method was designed to ensure a relatively homogeneous feed for the reactor. The feedstocks typically required a wet milling step in a Union Process Attrition Mill.

Pumping

The pumping subsystem was designed to operate at feed pressures up to 6000 psig and flow rates between 0.2 and 4.0 liter/hour. This system consists of a modified Isco 500D pump. The modification was the addition of a larger bore valve package in the unit that controls the feeding from one cylinder or the other. The valve package purchased consisted of four 3/8-inch air-actuated (6000 psi rated) ball valves with 3/8-inch stainless steel (SS) tubing connections installed on the Isco dual pump package. The valves and tubing were configured to fill and empty the pumps based on controller commands. We also installed oversize caps on the barrels that accommodate 3/8-inch NPT fittings. The large bore head, valve, and tubing allowed us to suction and pump the viscous slurries while still allowing the pump to operate at 3500 psi max. System piping included 0.5-inch (0.065-wall) 304 SS tubing on the outlet of the pump. Pump

inlet piping was 0.5-inch (0.035 wall) 304 SS tubing. All valves and valve trim (except the pressure-control valve) were also made of SS. Using the Isco pump, the feeding rates were measured directly by the screw drive of the positive displacement syringe pump.

Pre-Heater/Reactor

As initially tested, the preheater was a 1-liter 316 SS vessel equipped with a Carberry-type rotating basket. The preheater functioned as a continuous-flow, stirred-tank reactor (CSTR) in which the feedstock was brought to the reaction temperature.

The original reactor was a 1-inch ID X 72-inch-long 304 SS tube. The vessel had a maximum allowable working pressure of 10,000 psig at 22°C, which was derated to 5200 psig at 450°C. The vessel had bolted-closure endcaps with metal o-rings on each end. The reactor furnace was a 6-kWe resistance heater split into three separately controllable zones. The pressure was controlled with a dome-loaded diaphragm back-pressure regulator.

From our work in hydrothermal gasification we had previously learned to use an in-line system to separate precipitated solids, primarily mineral content². In the process of heat up, the organics in the biomass were pyrolyzed and liquefied while certain inorganic components, such as calcium phosphates, formed and precipitated as solids. We placed a vessel in the process line following the preheater to capture and remove the solids following heat-up to reaction temperature. The design of the separator was a simple dip leg vessel wherein the solids fell to the bottom of a vessel and the liquids passed overhead through a filter to the reactor. The solids could be removed by batch from the bottom of the vessel as they built up over time.

In later tests of hydrothermal liquefaction, we bypassed the tubular reactor and used only the CSTR as the combined preheater and reactor in line with the solids separation vessel. We included the solids removal vessel in order that the oil/water separation was more easily attained as prior experience had demonstrated. This configuration of the reactor system is depicted in **Figure 8**.

Reaction Products Separation

The liquid product collection was done via two 1-liter Parr vessels. The process effluent from the reactor was alternately sent to one of the two PARR vessels to collect the liquids at system pressure. The liquids collected in the temperature-controlled PARR vessels and gases were vented via a dome-loaded back-pressure regulator (BPR). At predetermined times the flow was

² Elliott, D.C.; Hart, T.R.; Neuenschwander, G.G. 2008. "Catalytic Hydrothermal Gasification of Biomass for the Production of Hydrogen-Containing Feedstock (Methane)" 2nd Symposium on Hydrogen from Renewable Sources and Refinery Applications, **Prep. Pap.-Am. Chem. Soc., Div. Pet. Chem.** 53 (1), 73-74.

redirected to the second Parr vessel by process line valving so that the first vessel could be emptied while the second was filling.

Instrumentation and Control

The data acquisition and control system used in the CRS was a hybrid computer-based system employing discrete data acquisition devices and single-loop process controllers communicating to a central computer. The computer was used during experiments to monitor the process,

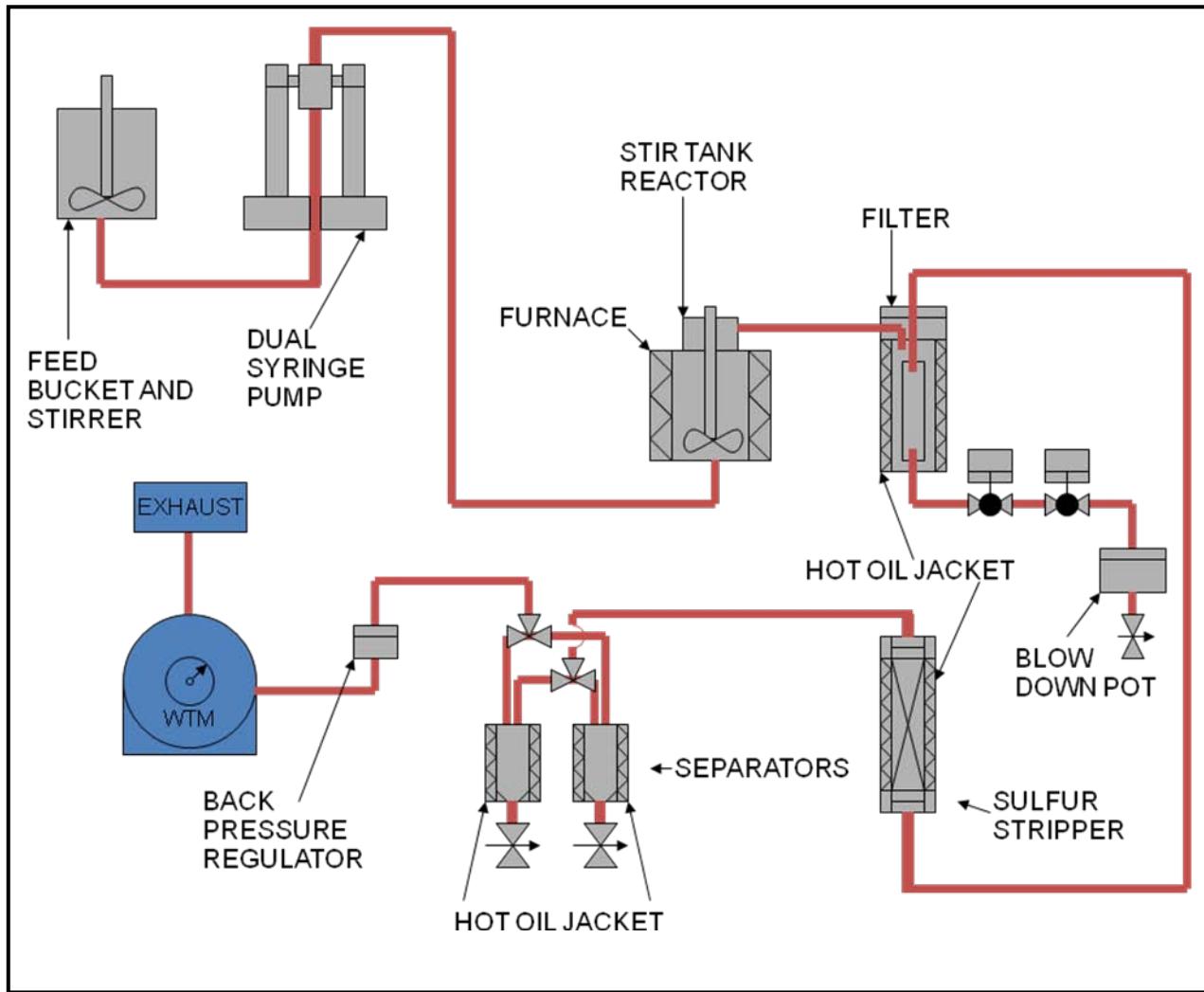


Figure 8. Hydrothermal Liquefaction System using a CSTR with a High-Pressure Solids Separator

calibrate instruments, and record data for later analysis. Labview is used to coordinate these activities. Non-control sensors such as thermocouples and pressure transducers were monitored via the data acquisition unit.

Bench-Scale Tests

A total of 23 separate process tests were performed within this project. These tests are summarized in **Table 2** and include processing corn fiber, starch and corn stover at various temperatures, flow rates and catalyst concentrations, with and without recycle of aqueous product as the solvent for the feed slurry

Table 2. Overview of Tests

| Test # | feedstock | Reactor Setup | Feed rate | Temp, °C | Length of test | Catalyst |
|--------|------------------------------|----------------|---------------|----------|--|-----------|
| 1 | 12.7% corn fiber | tubular | 2 L/h | 357 | 4 h – tube plug | none |
| 2 | 12.7% corn fiber | CSTR & tubular | 2 L/h | 356 | 3 h – tube plug | none |
| 3 | 14.7% corn fiber | CSTR & tubular | 2 L/h | 350 | 8.3 h - no plug | 2% NaCarb |
| 4 | 13.7% corn fiber | CSTR & tubular | 2 L/h | 350 | 7.5 h – transfer line plug | 1% NaCarb |
| 5 | 15.1% corn fiber | CSTR & tubular | 2 L/h | 350 | 37.8 h – transfer line plug | 2% NaCarb |
| 6 | 14.5% corn fiber | tubular | 2 L/h | 352 | 8.5 h – pump failed | 2% NaCarb |
| 7 | 8.2% corn fiber | tubular | 2 & 1.5 L/h | 190-340 | 10 h – temp out of control | 2% NaCarb |
| 8 | 13% corn fiber | tubular | 2 & 1.5 L/h | 341 | 9.9 h – no plug | 2% NaCarb |
| 9 | 13% corn fiber | tubular | 2 & 1.5 L/h | 330 | 10.2 h - no plug | 2% NaCarb |
| 10 | 5.3% corn stover | tubular | 1.5 & 1.3 L/h | 343 | 7 h – plug | none |
| 11 | 9.6% corn stover | tubular | 1.3 & 1.2 L/h | 350 | 8.9 h – no plug | 2% NaCarb |
| 12 | 40% starch | tubular | 1.3 L/h | 351 | 0.9 h – feed plug | 2% NaCarb |
| 13 | 9% starch | tubular | 1.3 L/h | 352 | 7 h – no plug | 2% NaCarb |
| 14 | 8.2% starch | tubular | 1.5 L/h | 344 | 7.4 h – no plug | 2% NaCarb |
| 15 | 10.6% corn stover | tubular | 1.5 L/h | 352 | 22.8 h – no plug | 1% NaCarb |
| 16 | 10.6% corn stover | CSTR | 1.5 L/h | 351 | 10.7 h – valve fail | 1% NaCarb |
| 17 | 13.3% corn stover | CSTR | 1.5 L/h | 350 | 75 h – valve fail | 1% NaCarb |
| 18 | 10.3% corn stover | CSTR | 1.5 L/h | 347 | 14 h – filter fail | 1% NaCarb |
| 19 | 13.3% corn stover | CSTR | 1.5 L/h | 346 | 11.2 h – valve fail | 1% NaCarb |
| 20 | 10.4% corn stover | CSTR | 1.2-1.8 L/h | 349-327 | 47.1 h – no plug | 1% NaCarb |
| 21 | 10% corn stover with recycle | CSTR | 1.5 L/h | | 4.8 h filter plug (residue from other) | 1% NaCarb |
| 22 | 10% corn stover with recycle | CSTR | 1.5 L/h | | 30.2 h pump fail | 1% NaCarb |
| 23 | 12% corn stover | CSTR | 1.5 L/h | | 88 h no plug | 1% NaCarb |

These tests suggest the following:

- Corn fiber can be processed effectively from 8 to 15 wt% in water; while corn stover can be processed from 5 to 13 wt%; limited testing with starch suggests processing at <10 wt% with heating to prepare pumpable slurry.
- Addition of base is required to maintain liquefaction conditions; 2 wt% of sodium carbonate facilitates corn fiber or starch; while only 1 wt% is needed for corn stover.
- A simple tubular reactor will work for hydrothermal liquefaction of corn fiber or starch, but liquefaction of corn stover requires a mineral separation step to facilitate oil/water separation in the collection system.

Corn Fiber Liquefaction

A total of nine process tests were performed using corn fiber slurries. The product yields and compositions are given in **Table 3**. The tests demonstrated bio-oil production with a significant formation of water soluble byproducts. Limited gas product was formed. Processing corn fiber slurry in a tubular reactor at nominally 350°C in the absence of pH adjustment resulted in char formation on the wall (test #1). Addition of the CSTR as a preheater before the tubular reactor did not eliminate the problem of wall coking (test #2). However, the addition of sodium carbonate to moderate the pH of the processing environment did result in no wall charring (test #3). Oxygen and moisture analysis in the product bio-oil appears faulty for this test. A subsequent test with a reduced level of sodium carbonate showed that it was not sufficient. Validation of the 2% level of sodium carbonate addition was seen in the longer term tests undertaken to produce bio-oil for subsequent hydroprocessing studies at CRADA partner, ConocoPhillips (test#5).

Table 3. Corn Fiber Liquefaction (average of data windows within test run)

| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|-----------------------------|-------|-------|-------|------|-------|-------|------|-------|-------|
| Oil yield, mass basis | 39.67 | 37.8* | 28.07 | 30.5 | 27.8 | 33.4 | 11.4 | 19.5* | 20.0 |
| Oil yield, carbon basis | 68.02 | 61.8* | 48.85 | 54.2 | 47.3 | 56.6 | 23.6 | 39.7* | 43.6 |
| Oil composition | | | | | | | | | |
| Carbon, wt% | 69.31 | 71.51 | 77.0 | 75.9 | 75.2 | 74.3 | 72.7 | 73.2 | 71.4 |
| Hydrogen, wt% | 7.40 | 7.52 | 8.0 | 8.4 | 8.6 | 7.5 | 8.4 | 8.2 | 8.1 |
| Oxygen, wt% | 19.85 | 19.39 | 9.9 | 12.2 | 11.3 | 13.3 | 16.0 | 14.5 | 16.0 |
| Oxygen, wt% dry basis | 9.93 | 12.99 | 1.7 | 5.0 | 7.9 | 8.4 | 9.9 | 11.5 | 13.0 |
| Nitrogen, wt% | 2.04 | 1.90 | 2.3 | 3.2 | 3.3 | 3.1 | 2.6 | 2.5 | 2.4 |
| Sulfur, wt% | 0.33 | 0.21 | 0.27 | 0.30 | 0.30 | 0.30 | 0.30 | 0.24 | 0.24 |
| Moisture, wt% | 12.56 | 8.43 | 9.4 | 8.7 | 4.2 | 6.0 | 7.8 | 3.9 | 4.0 |
| TAN, mg KOH/g | NA | NA | NA | NA | NA | 51 | 58 | 52 | 50 |
| density, g/mL | 1.1 | NA | NA | NA | 1.079 | 1.067 | NA | 1.071 | 1.072 |
| Aqueous yield, carbon basis | 25.74 | 31.2* | 30.6 | 30.0 | 33.1 | 34.7 | 72.5 | 55.3 | 46.3 |
| pH | 3.9 | 3.9 | 7.7 | 6.0 | 7.1 | 7.1 | NA | 7.7 | 7.7 |

| | | | | | | | | | |
|---------------------------------|------|-------|-------|-------|-------|-------|-----|-------|-------|
| COD | NA | 30700 | 72000 | 65000 | 64000 | 70000 | NA | 59000 | 60000 |
| Gas product yield, carbon basis | 3.76 | 7.1 | 8.0 | 7.6 | 6.0 | 6.0 | 2.6 | 4.6* | 9.2 |

* normalized carbon yield results

Red font indicates questionable result, likely faulty analysis

Subsequent tests used only the tubular reactor without the CSTR for preheating. Test #6 was halted because of pumping problems. Test #7 was the first test in the rebuilt system after moving to a new building. In the move process, the temperature control system lost its calibration and setup and was not functional until after a subsequent tuning session was completed. Following the tuning, lower temperature tests (tests #8 and #9) showed reduced bio-oil yields of lower quality.

Corn Starch Liquefaction

A total of three process tests were performed using corn starch slurries. The product yields and compositions are given in **Table 4** for the two successful tests. The first test with corn starch slurry (test #12) was attempted using a 40% dry starch in water slurry. Such slurry would not pump in the syringe pump and actually scoured the pump cylinder walls and seal rings and ruined the pump. The later two tests were performed with preheated starch slurry wherein the starch was “liquefied”. The tests demonstrated low levels of bio-oil production with a large amount of water soluble byproducts. Limited gas product was formed. Comparing processing corn starch slurry in a tubular reactor at 340°C and 1.3 L/h (test #13) with the lower severity test (test #14) with lower temperature, 330°C, and higher flow rate, 1.5 L/h, shows that the less severe conditions resulted in reduced bio-oil yield and the product was somewhat less deoxygenated. However, the difficulty in measuring the oxygen and moisture analysis in the product bio-oil appeared to produce inconsistent results for this test. An unrepresentative sample

Table 4. Corn Starch Liquefaction (average of data windows within test run)

| | 13 | 14 |
|-------------------------|------|-------|
| Oil yield, mass basis | 10.3 | 5.5* |
| Oil yield, carbon basis | 23.8 | 13.0* |
| Oil composition | | |
| Carbon, wt% | 72.2 | 63.9 |
| Hydrogen, wt% | 7.66 | 8.4 |
| Oxygen, wt% | 18.2 | 24.3 |
| Oxygen, wt% dry basis | 15.1 | 9.1 |
| Nitrogen, wt% | 0.06 | 1.26 |
| Sulfur, wt% | 0.02 | 0.01 |
| Moisture, wt% | 4.2 | 19.0 |
| TAN, mg KOH/g | 26 | 19 |

| | | |
|---------------------------------|-------|-------|
| density, g/mL | 1.08 | 1.04 |
| Aqueous yield, carbon basis | 66.9 | 81.5* |
| pH | 7.3 | 7.3 |
| COD | 56000 | 61000 |
| Gas product yield, carbon basis | 5.0 | 4.4* |

* normalized carbon yield results

Red font indicates questionable result, likely faulty analysis

may have also been used in the density measurement and the nitrogen analysis. The commercial starch product used had very little nitrogen (0.05-0.10 wt %). The lower density of the bio-oil does not reflect the higher oxygen content and is unlike any of the other HTL bio-oils in this project. Both bio-oil products had a low enough viscosity that they flowed at room temperature.

Corn Stover Liquefaction

A total of eleven process tests were performed using corn stover slurries. The product yields and compositions are given in **Table 5** for the successful tests. The first test with corn stover slurry (test #10) was attempted using low concentration slurry (5%) without alkali catalyst added. The test proceeded well but eventually plugged in the reactor. The next test was performed with higher concentration slurry (9.6%) with a 2% alkali catalyst added. This test ran smoothly and did not plug. The tests demonstrated significant levels of bio-oil production but with a significant amount of water soluble byproducts. Limited gas product was formed. A subsequent test with higher concentration corn stover and a 1% alkali catalyst added (test #15) performed well but there were problems with bio-oil separation from the aqueous byproduct and the significant amount of mineral precipitate formed in the process. Both the heavy bio-oil product and the mineral precipitate settle to the bottom of the condensate product and oil recovery is confounded.

Table 5. Corn Stover Liquefaction (average of data windows within test run)

| | 10 | 11 | 16 | 17 | 19 | 20a | 20b | 20c |
|-------------------------|-------|------|-------|-------|------|-------|------|-------|
| Oil yield, mass basis | 24.5* | 17* | 26.0* | 15.9* | 15.2 | 21.6* | 19.1 | 14.6* |
| Oil yield, carbon basis | 52.0* | 38* | 44.4* | 30.0* | 32.8 | 30.0* | 39.9 | 33.4* |
| Oil composition | | | | | | | | |
| Carbon, wt% | 60.5 | 72.5 | 72.05 | 63.6 | 68.9 | 55.1* | 68.1 | 69.5 |
| Hydrogen, wt% | 6.0 | 7.9 | 7.88 | 8.5 | 7.9 | 8.6 | 7.8 | 7.4 |
| Oxygen, wt% | 27.9 | 16.0 | 19.92 | 25.9 | 19.9 | 26.2 | 21.5 | 22.3 |
| Oxygen, wt% dry basis | 13.5 | 8.2 | 17.34 | 17.8 | 14.1 | 17.1 | 16.8 | 13.8 |
| Nitrogen, wt% | 1.11 | 1.2 | 1.35 | 1.06 | 1.15 | 0.92* | 1.23 | 1.2 |
| Sulfur, wt% | 0.08 | 0.07 | 0.07 | 0.06 | 0.08 | 0.06 | 0.07 | 0.08 |

| | | | | | | | | |
|---------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| Moisture, wt% | 19.05 | 9.6 | 3.61 | 11.3 | 7.74 | 12.6 | 6.62 | 11.2 |
| TAN, mg KOH/g | 35 | 32 | NA | 31 | 29 | 26 | 48 | 42 |
| density, g/mL | 1.09 | 1.10 | 1.04 | 1.08 | 1.094 | 1.073 | 1.066 | 1.109 |
| Aqueous yield, carbon basis | 37.6* | 55.3* | 43.9* | 56.1* | 56.8 | 53.4* | 42.1 | 52.6 |
| pH | 3.4 | 7.3 | 4.8 | 5.5 | 4.5 | 5 | 5 | 5 |
| COD | 28000 | 56000 | 53000 | 85000 | 71000 | 58000 | 54000 | 58000 |
| Gas product yield, carbon basis | 10.6* | 6.8* | 5.3* | 5.0* | 7.8 | 8.0* | 7.1 | 6.7* |

* normalized carbon yield results

Red font indicates questionable result, likely faulty analysis

Subsequent tests utilized the modified process flow shown in **Figure 8** which incorporated a solids separator and high-pressure filter prior to the condensate separation and collection. By this design the mineral components in the corn stover could be separated from the products while the bio-oil water separation could be accomplished more cleanly. The CSTR served as the only reactor in this flow configuration. In test #16 the new configuration was demonstrated for a period, but then a valve failure terminated the test. The next test shown in **Table 4** (test #17) was undertaken to produce bio-oil for subsequent hydroprocessing studies at CRADA partner, ConocoPhillips. Tests 19 and 20 included a series of process parameter tests starting at 346°C and 1.5 L/h (test #19) with variations in severity with lower flowrate 1.3 L/h @ 349°C (test #20a), lower temperature 340°C and higher flow rate 1.8 L/h (test #20b), and lower temperature, 327°C @ 1.5 L/h (test #20c). It is difficult to see any consistent trends in product yield or bio-oil product quality within this range of parameter variation.

Results from tests involving recycle of the aqueous byproduct are shown in **Table 6**. Test #21 was an initial test of recycle using aqueous product from test #19; it was terminated prematurely when a pressure drop could not be cleared. On disassembly the filter was found to be plugged. It was suggested that feedstock with a high lignin content used in a prior hydrothermal gasification test was the cause. A redesigned filter assembly was put in place for the final tests. In test 22 an attempt was made to recycle the aqueous stream within the limits of the test itself by preparing subsequent feed with the aqueous stream from earlier in the run. The recycle was accomplished but was limited by the extensive time required to grind the corn stover with

Table 6. Corn Stover Liquefaction with Aqueous Recycle (average of data windows within test run)

| | 20b | 22a | 22b | 22c | 23a | 23b | 23c |
|-------------------------|-------|-------|------|-------|-------|-------|-------|
| Oil yield, mass basis | 19.1 | 16.9* | 18.2 | 18.7* | 16.3* | 16.0* | 16.5 |
| Oil yield, carbon basis | 39.9 | 31.6* | 37.6 | 42.3* | 34.6* | 32.7* | 35.9 |
| Oil composition | | | | | | | |
| Carbon, wt% | 68.09 | 71.2* | 68.7 | 73.32 | 67.61 | 67.68 | 71.17 |

| | | | | | | | |
|----------------------------------|-------|-------|---------|-------|-------|-------|-------|
| Hydrogen, wt% | 7.84 | 7.99 | 7.70 | 7.94 | 8.58 | 8.62 | 8.26 |
| Oxygen, wt% | 21.53 | 18.0 | 20.0 | 17.64 | 22.55 | 20.18 | 19.93 |
| Oxygen, wt% dry basis | 16.75 | 12.4 | 13.0 | 8.71 | 12.81 | 13.09 | 11.75 |
| Nitrogen, wt% | 1.23 | 1.35 | 1.39 | 1.46 | 1.19 | 1.05 | 1.30 |
| Sulfur, wt% | 0.07 | 0.08 | 0.06 | 0.06 | 0.09 | 0.07 | 0.08 |
| moisture, wt% | 6.62 | 7.03 | 9.16 | 9.40 | 12.80 | 9.35 | 10.60 |
| ash, wt% | NA | NA | NA | NA | 0.18 | 0.10 | 0.20 |
| TAN, mg KOH/g | 48 | NA | NA | NA | 34.8 | 24.4 | 29.7 |
| density, g/mL | 1.066 | NA | NA | NA | NA | NA | NA |
| Aqueous yield, carbon basis | 42.1 | 57.3* | 53.6 | 49.8* | 50.2* | 53.0* | 54.18 |
| pH | 5 | 5.05 | 4.7-5.4 | 5.04 | 5 | 5.5 | 5.3 |
| COD | 53700 | 68600 | 77700 | 82500 | 54000 | 72500 | 77600 |
| Gas product yield, carbon basis | 7.07 | 10.9* | 9.3 | 7.3* | 12.3* | 8.3* | 8.62 |
| Carbon lost with solids, C basis | 6.23 | 0.22* | 0.94 | 0.52 | 2.9* | 6.0* | 2.63 |

* normalized carbon yield results

the recycled aqueous to produce pumpable slurry. Test 22a involves feedstock made with recycled aqueous from test #20 (given again in [Table 5](#) for reference). Test #22b involved recycled aqueous from test #22a, and #22c had recycle from #22b. It appears from these results that there is a trend an increasing carbon yield in the bio-oil at the expense of carbon yield in the aqueous phase, but there was also an increase in the organic loading in the aqueous byproduct. Test 23 was another extended run to produce bio-oil for subsequent hydroprocessing studies at CRADA partner, ConocoPhillips. This was an extended run to demonstrate operability of the process with some recycle. Deionized water was used for slurry preparation in the first reported phase (test #23a). Aqueous from test #17 was recycled in the second phase of the test (test #23b) and test #23a and b aqueous phase was recycled during the 23c portion of the test. The three data windows show only a slight but inconsistent trend in product yield and quality as a function of recycle but underline the inherent variability in the data generation (primarily inhomogeneous bio-oil sampling inconsistency). Additionally, carbon lost with solids is reported for each sampling window representing the amount of carbon that was carried out with the on-line blow down of the filtered solids from the system filter. While this represents an efficiency cost, it is possible that a change in design or operation of the solids filtering could result in restoring some of this lost carbon.

Data Correlation

The data for runs 17a through 20c, all CSTR runs without recycle, were correlated in terms of the empirical expression:

$$(PROPERTY\#) = a \cdot (LHSV)^b \cdot (BMF)^c \cdot \left(\frac{T}{300} \right)^d$$

where:

PROPERTY # is Moisture Free Oil Yield, Water Solubles Yield, H/C Atomic Ratio, O/C Atomic Ratio;

LHSV is the liquid hourly space velocity in h-1;

BMF is the biomass to water inlet mass fraction; and

T is temperature in °C

These correlations for Corn Stover HTL (parameter values and parity plots reported in Appendix B) were used within the experimental window of operation to show the various trends that dependent variables present in terms of the independent variables, namely Liquid Hourly Space Velocity (LHSV), Temperature (T) and Biomass Inlet Mass Fraction (BMF). All correlations are reported on a moisture free basis for the feed and the products.

Moisture Free Oil (MFO) Yield

Figure 9 shows the effect of the independent variables upon MFO yield.

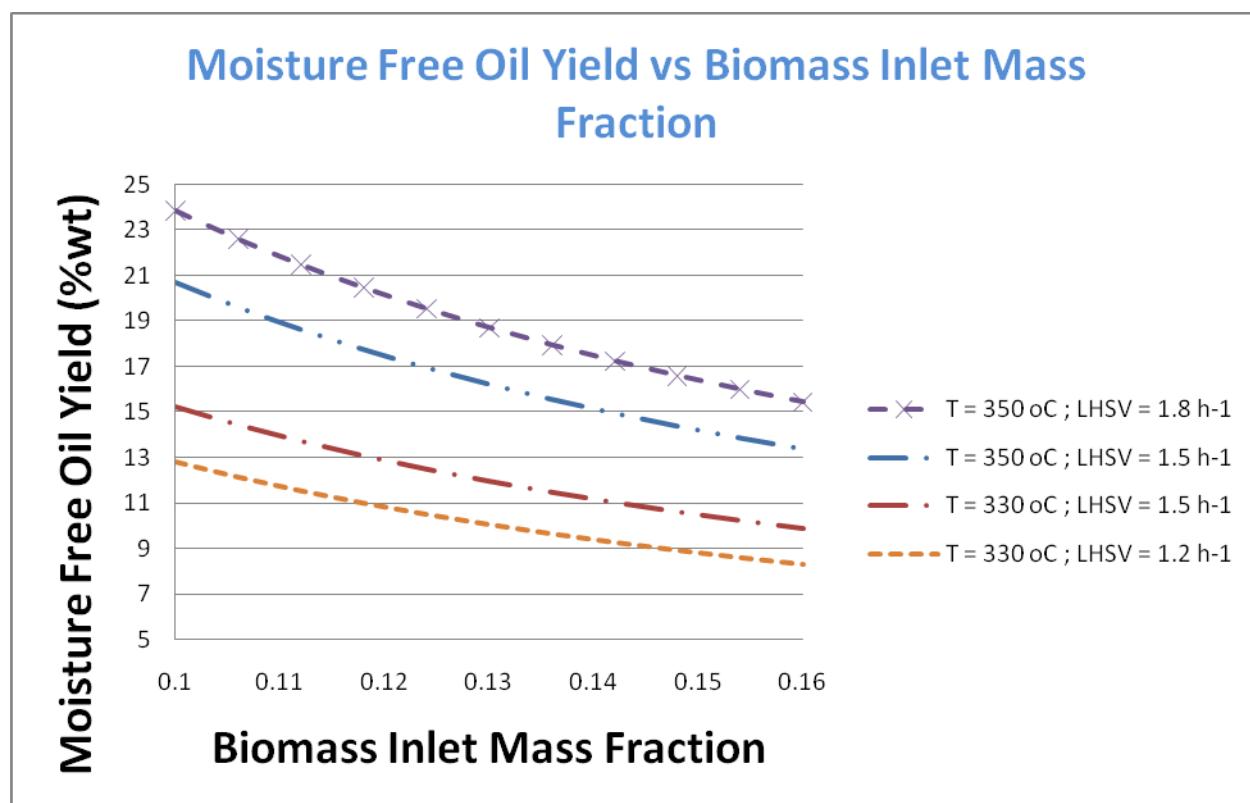


Figure 9. Moisture Free Oil Yields in terms of BMF, T and LHSV.

The MFO yield increases with temperature and LHSV, while it decreases with BMF. In this case, experimental data suggest that the maximum MFO production will benefit from processing dilute feeds, at the highest temperature and shortest residence time.

MFO H/C Atomic ratio

Figure 10 shows the effect of the independent variables upon MFO H/C atomic ratio. This demonstrates that -

The MFO H/C atomic ratio increases with temperature, decreasing BMF and LHSV. In this case, experimental data is suggesting that the maximum MFO H/C Atomic Ratio will benefit from processing dilute feeds, at the highest temperature and longest residence time.

MFO yield and its H/C atomic ratio are equally favored by processing dilute feeds and using high temperatures, but there is an opposite effect from residence time: longer times improve the MFO H/C ratio, but shorter times improve MFO yield. So, LHSV should be determined by operational and economic factors.

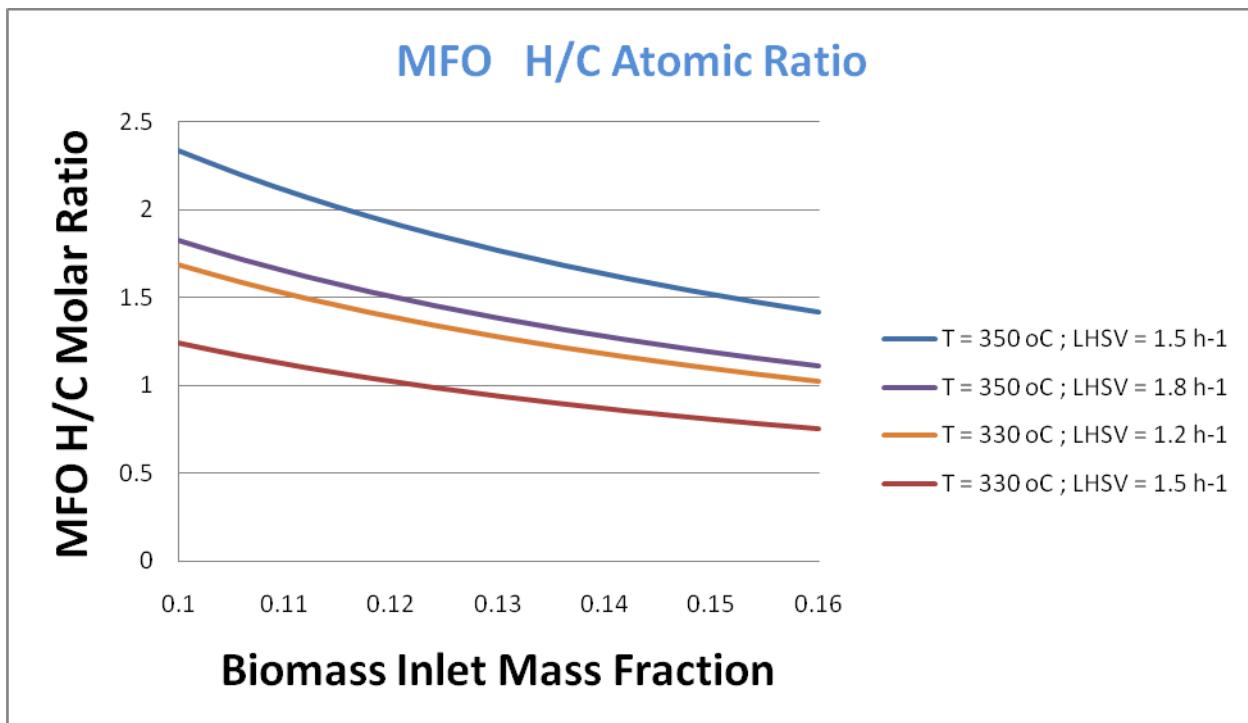


Figure 10. Moisture Free Oil H/C Atomic Ratio in terms of BMF, T and LHSV.

MFO O/C Atomic ratio

Figure 11 shows the effect of the independent variables upon MFO O/C atomic ratio.

The MFO H/C atomic ratio decreases with BMF and LHSV, while it increases with temperature. In this case, experimental data suggest that the minimum MFO O/C Atomic Ratio will benefit from processing concentrated feeds, at the lowest temperature and shortest residence time.

The MFO O/C atomic ratio optimum operating conditions are partially opposite to those that maximize MFO yield and MFO H/C atomic ratio. Because of the large impact the MFO O/C Atomic Ratio has upon the MFO HHV, it is anticipated that this parameter will override the MFO H/C Atomic Ratio maximization. There are also opposite trends for Biomass Inlet Mass Fraction and Temperature effects upon MFO yield and MFO O/C Atomic Ratio. However, short residence times favor both properties mentioned before.

There will be a trade off between MFO yield and its corresponding HHV that will dictate the final choice. This decision will be dependent also upon the downstream processing of the aqueous phase and the bio-oil upgrading.

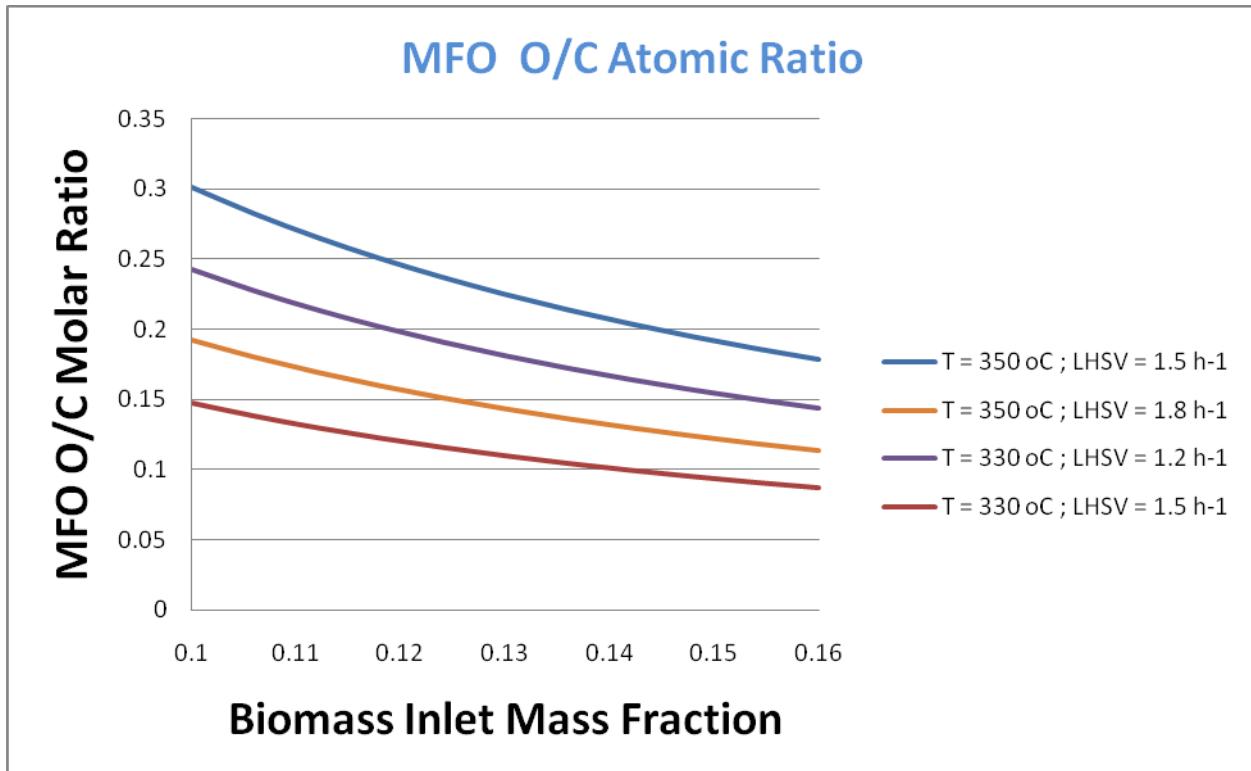


Figure 11. Moisture Free Oil O/C Atomic Ratio in terms of BMF, T and LHSV.

Water Solubles (WS) Yield

Figure 12 shows the effect of the independent variables upon WS yield. It

The WS yield decreases with temperature and BMF, but is independent of LHSV. In this case, experimental data suggest that the maximum WS production will benefit from processing dilute feeds, at the lowest temperature.

Trends for WS H/C and O/C atomic ratios are not presented due to the low number of valid data points.

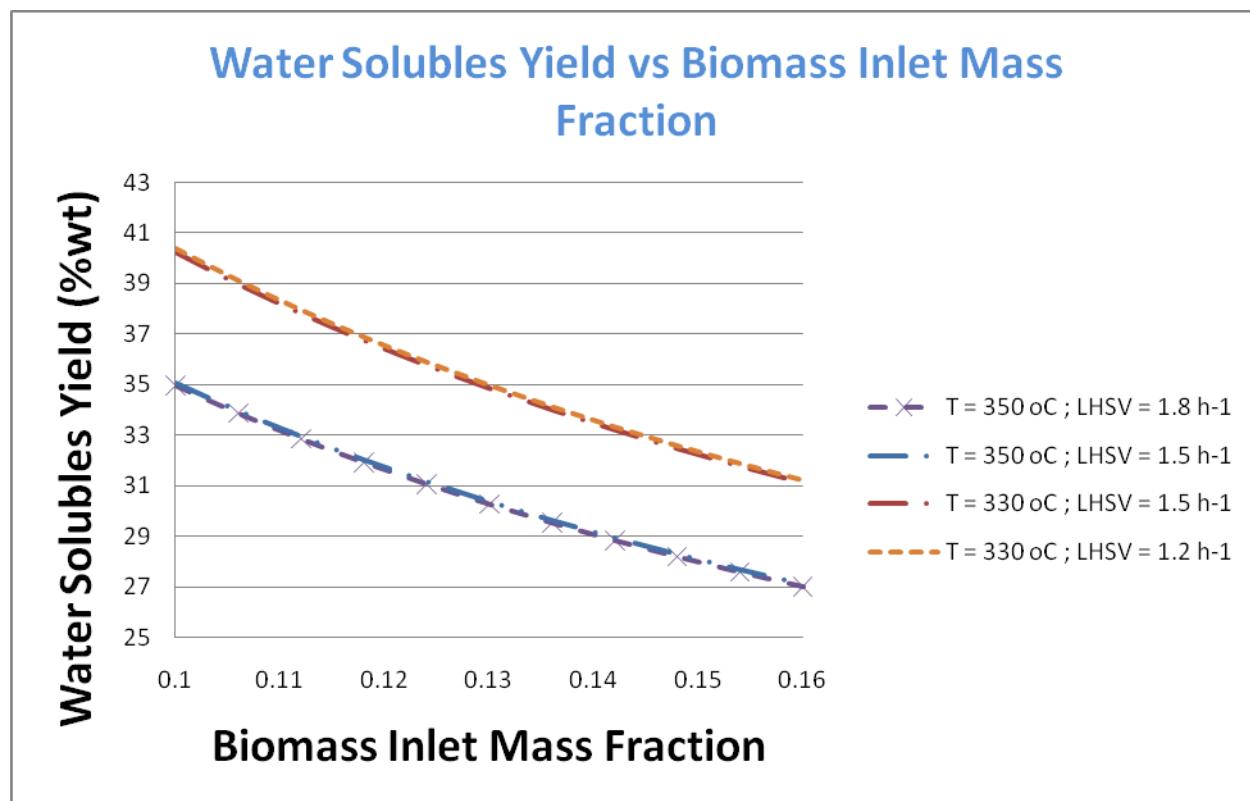


Figure 12. Water Solubles Yields in terms of BMF, T and LHSV.

Aqueous Product Assessment

Detailed analysis of the aqueous phase was undertaken to determine the composition and quantity of dissolved organic material as led by ADM with contribution from PNNL.

Treatment or recovery of these potentially useful or valuable byproduct materials was a focus of the ADM team. ConocoPhillips was involved in considering upgrading it to fuels.

Following characterization of the stream ConocoPhillips and ADM evaluated the best use of the aqueous stream. The options included stream, recycle to the hydrothermal liquefaction process, combustion to generate high temperature steam for the process, conversion to hydrogen to supply the hydrotreating process, conversion to mixed alcohols for fuels, or chemical applications. The aqueous stream analysis showed that it consisted primarily of acetic and glycolic acids (up to 2-4 wt% in some samples), with lesser amounts of acetone, other C1-C6 oxygenates, and phenols. This stream was not of interest for conversion to transportation fuels due to the low total organics concentration and high proportion of carboxylic acids. Thus, the most attractive options for monetizing this stream were determined to be: a) recycle to the liquefaction process, with the goal of further converting the dissolved organics to oil, or b) hydrothermal gasification to produce methane, which then could be converted to hydrogen through conventional steam methane reforming or burned for energy.

PNNL performed bench-scale catalytic hydrothermal gasification tests to evaluate fuel gas production from the aqueous stream as a means of energy value recovery using this

Analysis of Aqueous Product

A gas chromatography with a mass spectrometer was used to identify and quantify compounds present in the aqueous fractions of biomass samples from hydrothermal liquefaction. Samples were injected neat onto a sixty meter wax column with 0.25 mm inner diameter and 0.25 micron film thickness using a split injector ratio of 10:1. The injector was maintained at 260°C. The carrier gas was helium at a linear column flow rate of 49 cm/s. The initial oven temperature was 35°C and held for ten minutes. The oven temperature was then ramped 10°C per minute to 260°C and held for 7.5 minutes. The interface temperature was set at 260°C and the ion source was set at 230°C. The detector voltage was set to tune plus 0.7 kilovolts. Mass spectral data was collected at a scan speed of 1250 over a mass to charge range of 15-600.

The compounds present were identified using library matching of the mass spectra using both the NIST and Wiley libraries. Compounds were then quantified using the areas the total ion chromatograms and the response factors of acetic acid and butanol. Results of this analysis are presented in **Table 7**.

Table 7. Identified Components in Aqueous Byproducts from Hydrothermal Liquefaction

| Feedstock | | HFCS | Starch | Dextrose | Stover | Stover | Stover | Stover | Stover | Stover | Stover |
|-------------------|-----------------|--------|--------|----------|--------|--------|--------|--------|---------|---------|---------|
| Run Number | | MHLT-2 | LF-13 | MHLT-1 | LF-10 | LF-11 | HTL-17 | HTL-19 | HTL 20A | HTL 20B | HTL-20C |
| Molecular Formula | Family Compound | % wt | % wt | % wt | % wt | % wt | % wt | % wt | % wt | % wt | % wt |
| CH4O | Alcohol | 0.5% | | 6.6% | 5.7% | 8.1% | 20.0% | 3.6% | 3.5% | 38.7% | 1.2% |
| C2H4O | Aldehyde | 2.8% | 5.0% | 2.8% | 0.3% | 1.8% | | | | | |
| C2H4O2 | Acid | 11.7% | | 1.5% | 1.1% | 1.2% | 0.9% | 9.1% | 10.2% | 0.1% | 0.3% |
| C2H6O | Alcohol | 2.2% | | 11.4% | 0.1% | 0.6% | 11.8% | 0.1% | 1.3% | 14.2% | 0.6% |
| C2H6O2 | Alcohol | 11.2% | 17.2% | 1.3% | | | 15.1% | 18.4% | 17.4% | 2.5% | 7.1% |
| C3H4O2 | Acid | | | | 1.9% | | | | | | |
| C3H6O | Ketone | 21.9% | 52.1% | 1.2% | 27.3% | 3.6% | 0.3% | 7.9% | 0.1% | 8.2% | 0.3% |
| C3H6O2 | Alcohol | | | | | | 0.6% | 0.2% | 0.6% | 1.7% | 0.9% |
| C3H6O2 | Ketone | | | | 2.1% | | | | | | |
| C3H6O2 | Acid | 4.7% | | 1.3% | 2.3% | | | | | | |
| C3H8O | Aldehyde | 1.6% | 1.3% | 0.2% | 1.3% | 0.1% | | | | | |
| C3H8O | Alcohol | 1.8% | 0.3% | 4.7% | 0.7% | 2.8% | 8.9% | 8.1% | 12.9% | 2.4% | 73.2% |
| C3H8O2 | Alcohol | | | | | | 5.0% | 0.9% | 6.4% | 0.1% | 3.1% |
| C3H8O3 | Alcohol | | | | | | 3.2% | 1.3% | 3.4% | 1.3% | 1.3% |
| C4H10O | Alcohol | | 4.6% | 0.9% | | 1.6% | 2.2% | 2.0% | 3.0% | 1.9% | 0.0% |
| C4H10O2 | Alcohol | | 0.5% | | | | 0.3% | 0.2% | 0.3% | 0.4% | 0.2% |
| C4H6O2 | Ketone | 3.4% | 16.0% | 2.3% | 1.4% | | 2.0% | 0.2% | 1.5% | 1.2% | 0.5% |
| C4H7NO | Ketone | | | | | | 0.1% | 0.9% | 0.9% | 0.4% | 0.2% |
| C4H8O2 | Acid | | | | 0.0% | 0.7% | | | | | |
| C4H8O2 | Ketone | | | | 3.1% | | | | | | |
| C5H10O | Ketone | 2.3% | | 3.6% | 0.4% | 1.1% | 0.3% | 0.9% | 0.6% | 1.3% | 0.1% |
| C5H10O | Alcohol | | | | 2.0% | | 0.4% | 9.3% | 7.7% | 1.8% | 0.3% |
| C5H4O2 | Aldehyde | | | | 1.4% | 0.2% | | | | | |
| C5H8O | Ketone | 14.4% | | 16.9% | 6.3% | 11.4% | 0.6% | 0.3% | 0.9% | 0.6% | |
| C6H10O | Ketone | 9.8% | | 1.3% | 11.4% | 1.2% | 3.0% | 0.7% | 5.0% | 6.5% | 2.2% |
| C6H10O3 | Acid | 1.6% | | 0.6% | 3.0% | | | | | | |
| C6H10O4 | Alcohol | | | | | | 4.1% | 1.1% | 0.5% | 0.3% | 0.0% |
| C6H6O | Alcohol | | | | 2.9% | 5.5% | 4.4% | 7.0% | 0.7% | 0.9% | 2.9% |
| C6H6O2 | Alcohol | 3.3% | | 20.7% | 13.7% | 1.7% | 0.1% | 1.9% | 1.2% | 1.9% | 0.8% |
| C7H10O | Ketone | 0.1% | 3.1% | 8.1% | 0.5% | 1.2% | 0.1% | 0.6% | 0.2% | 0.7% | 1.1% |
| C7H12O | Aldehyde | | | | | | 13.0% | 17.2% | 16.3% | 1.4% | 0.5% |
| C7H8O | Alcohol | | | | | | 0.9% | 1.1% | 1.0% | 0.2% | 0.1% |
| C7H8O2 | Alcohol | | | 1.1% | 0.3% | 0.4% | 0.8% | 1.8% | 0.8% | 3.3% | 1.5% |
| C7H8O2 | Alcohol | 5.4% | | 11.9% | | | | | | | |
| C8H10O | Alcohol | 1.4% | | 1.6% | 0.0% | 0.8% | 1.5% | 3.8% | 3.0% | 4.4% | 0.2% |
| C8H10O3 | Alcohol | | | | 6.2% | 3.5% | | 0.7% | 0.4% | 1.6% | 0.6% |
| C8H18O | Alcohol | | | | | | 0.2% | 0.7% | 0.2% | 0.9% | 0.3% |
| C9H12O2 | Alcohol | | | | 1.6% | | 0.3% | 0.1% | 0.0% | 1.1% | 0.4% |
| C10H12O4 | Ketone | 0.2% | | | 3.1% | 52.5% | | | | | |
| Total | | 100.0% | 100.0% | 100.0% | 100.0% | 100.0% | 100.0% | 100.0% | 100.0% | 100.0% | 100.0% |

Table 7 presents the chemical analysis of the aqueous phase on a moisture free basis. Important to note is that the carbon compounds contained in this process stream only represent approximately 3-6 wt% of its mass. From the table, it can be seen that the compounds in the aqueous phase are a heterogeneous mixture of alcohols, ketones, aldehydes, and acids. The majority of water soluble compounds are alcohols (54.7%), followed by ketones (33.4%), aldehydes (6.7%), and acids (5.2%). When considering carbon number, approximately 60% of the compounds are C4 minus and the mixture has an estimated 50% boiling point of 250°F on a dry basis. Because of the dilute nature of this stream, economic recovery of the carbon

compounds will be problematic. One method, catalytic gasification, was examined and the results are presented in the next section.

Catalytic Hydrothermal Gasification

The Continuous-flow Reactor System (CRS) used in hydrothermal liquefaction was used with slight modifications in for the hydrothermal gasification. Of the five major functional subsystems: feed pretreatment and preparation, pumping, preheater/reactor, reaction products separation, and instrumentation and control, changes were required in part of the reactor system and in the product separation system while feed pretreatment was not needed. The system throughput was similar at 1 to 3 liter/hour of aqueous product. The process flow diagram is shown in **Figure 13**. The modifications implemented for handling minerals and sulfur is indicated in the diagram labeled “NEW.”

Bench-scale Test Results

A preliminary batch reactor test was performed to evaluate the aqueous byproduct from hydrothermal liquefaction as a feedstock for catalytic hydrothermal gasification (CHG). The

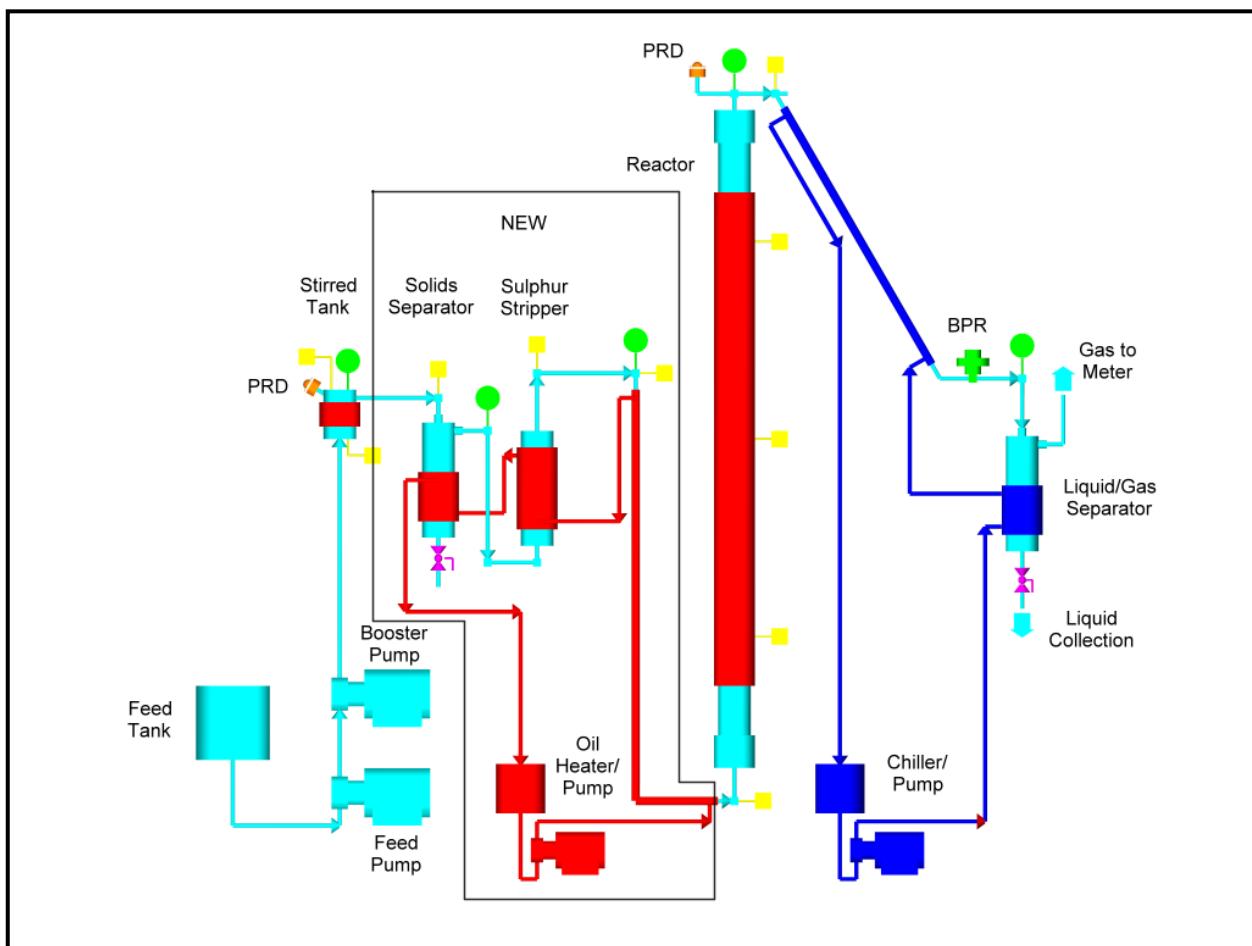


Figure 13. Schematic of the Bench-Scale Continuous-Flow Reactor System for Gasification.

aqueous stream came from test #6 which used a corn fiber feedstock. In a stirred batch reactor 261.5 g of aqueous byproduct were stirred with 35.4 g of a Ru on carbon catalyst, which had been developed and tested in other CHG applications (the catalyst had been reduced under hydrogen at 250°C overnight prior to the batch test). The test extended for 4 hours at nominally 350°C. The aqueous byproduct chemical oxygen demand (COD) was reduced from 60,300 ppm to 60 ppm. The pH of the solution increased from 7.51 to 7.98. The vented gas volume was 14.8 liters of which about 5.9 liters were the nitrogen cover gas initially left in the reactor following purge of oxygen from the system. The gas composition included 74% methane, with 12% each of carbon dioxide and hydrogen, and 1% ethane (on a nitrogen-free basis). This batch test verified that the organic contaminants left in the aqueous byproduct from hydrothermal liquefaction could be effectively gasified by CHG.

Two continuous-flow experiments were performed at bench-scale with the aqueous phase byproducts from corn stover hydrothermal liquefaction. In the tests the process was operated for 4 to 10 hours. In the first test there were plugging problems in the offgas system attributed to fine precipitate in the condensate water. In the second test, the 10-hour run was completed without stoppage although the same white precipitate (assumed to be sodium bicarbonate based on sodium analysis) was noticed in all condensate samples.

These tests, whose results are shown in **Table 8**, showed the high level of gasification achieved by this technology using the aqueous phase byproduct as the feedstock. In these tests almost all the organic material was converted to a medium-Btu gas. The gas could be used directly for process heating or further processed to generate pipeline quality natural gas substitute. Because of the low COD of the product water after gasification its reuse seems like a reasonable option and should be considered further. The recycle of the aqueous phase back to feedstock preparation for the liquefaction step would also be reasonable and has potential to reduce the cost of added alkali. The high-temperature and high-pressure separation of the bio-oil and aqueous needs to be further investigated. The solids separation prior to bio-oil separation also serves to

Table 8. CHG Process Results with Aqueous Phase Feedstock

| Process Parameters | 1 | 2 | Process Results | 1 | 2 |
|------------------------|-------------|-------------|-----------------------------|-------|-------|
| Preheater temp, °C | 324 | 345 | gas yield, L/g aqueous feed | 0.02 | 0.04 |
| Tubular temp, °C | 331-355-349 | 324-353-350 | Gas Composition, vol % | | |
| System Pressure, psig | 3041 | 2875 | methane | 59.7 | 65.8 |
| LHSV, L/L cat @ temp/h | 2.0 | 1.4 | carbon dioxide | 39.2 | 31.9 |
| Feed Composition, wt% | | | hydrogen | 0.5 | 1.5 |
| Carbon | 1.8 | 2.8 | ethane/propane | 0.06 | 0.1 |
| Hydrogen | 10.1 | 11.0 | carbon monoxide | 0.00 | 0.00 |
| Nitrogen | 0.00 | 0.04 | C conversion to gas, % | 65 | 71 |
| Oxygen, by dif | 84.2 | 84.2 | C conv adjust for carbonate | 120 | 88 |
| Sulfur | 0 | 0.0 | carbon lost with solids | 0 | 0 |
| ash | 3.9 | 1.9 | carbon balance | 120.4 | 95.8 |
| Feed COD, ppm | 54000 | 72867 | mass balance | 102.6 | 101.6 |
| Product COD, ppm | 606 | 236 | COD reduction | 98.9 | 99.7 |

pretreat the aqueous for use in the gasification by removing insoluble minerals. The tests gave no evidence of catalyst deactivation in this application in that the COD of the effluent remained constant and the gas product composition was essentially unchanged. However, the relatively short tests only provide an initial indication and longer term catalyst lifetime tests would be required.

The direct connection of the gasification technology following liquefaction technology with an intermediate separation of the bio-oil product should be straightforward since the technologies operate at the same conditions of temperature and pressure. There would be minimal requirement for reheating or repressurizing the aqueous phase to gasification reaction conditions. **This advantage would appear to override the issue of cost for processing a dilute phase feedstock as the gas product would be essentially net production without requirement for energy use in pressuring and heating.** A detailed engineering design and economic assessment would be required to confirm this preliminary assessment.

Upgrading with Heteroatom Removal

Catalytic hydroprocessing of the hydrothermal liquefaction product oil was performed by Conoco-Phillips at the bench-scale. Mass balances around the process were determined and products recovered for detailed analysis for fuel applications.

- Hydroprocessing was utilized for upgrading the crude oil product.
- Targets for this processing included not only the oxygen heteroatoms, but also nitrogen, as well as sulfur.

ConocoPhillips sought to determine:

- Is the material suitable for directly blending into fuel?

- If not, what further treatments, including co-processing to make a material suitable for fuel in laboratory fixed bed test reactors are needed?

In addition, ConocoPhillips

- Evaluated catalytic cracking in laboratory screening reactors as an alternative upgrading option.
- Screened the fuel properties of the upgraded products.

Catalytic Hydroprocessing for Heteroatom Removal

The hydrothermal liquefaction oil was subjected with catalytic hydroprocessing with catalysts supplied by Albemarle. The crude liquefaction oil was not miscible with typical petroleum fractions (naphtha, distillate, etc.), therefore is not suitable for direct blending. A lab-scale, fixed-bed hydroprocessing unit was employed along with a suitable catalyst to improve the quality of the crude oil. A diagram of the unit is shown in **Figure 14**. Three catalysts were screened: a commercially available hydrotreating catalyst, KF-757, and two developmental hydrodeoxygenation catalysts designated HDO-1 and HDO-2.

Experimental Details

An aliquot of desired catalyst was charged into the reactor and converted into its active forms following a presulfiding procedure provided by the vendor. In brief, a mixture of petroleum distillate and dimethyl disulfide was combined with hydrogen gas and flowed over the catalyst while increasing the catalyst temperature. The DMDS in contact with hydrogen at elevated temperature is converted into hydrogen sulfide, H_2S , which in turn converts the catalyst from the oxide form into the active sulfide form. Following this activation procedure, ultralow sulfur diesel fuel (ULSD) was flowed through the catalyst bed to remove residual sulfur to prevent contamination during the reaction stage. Crude liquefaction oil was loaded into one of the ISCO syringe pumps and introduced into the activated catalyst bed. The pump, transfer lines leading to the reactor, and transfer lines carrying the product were heated to reduce the liquid viscosity and allow smooth flow of the reactants and products. Products were collected in a heated trap along with simultaneous analyses of the product off-gas.

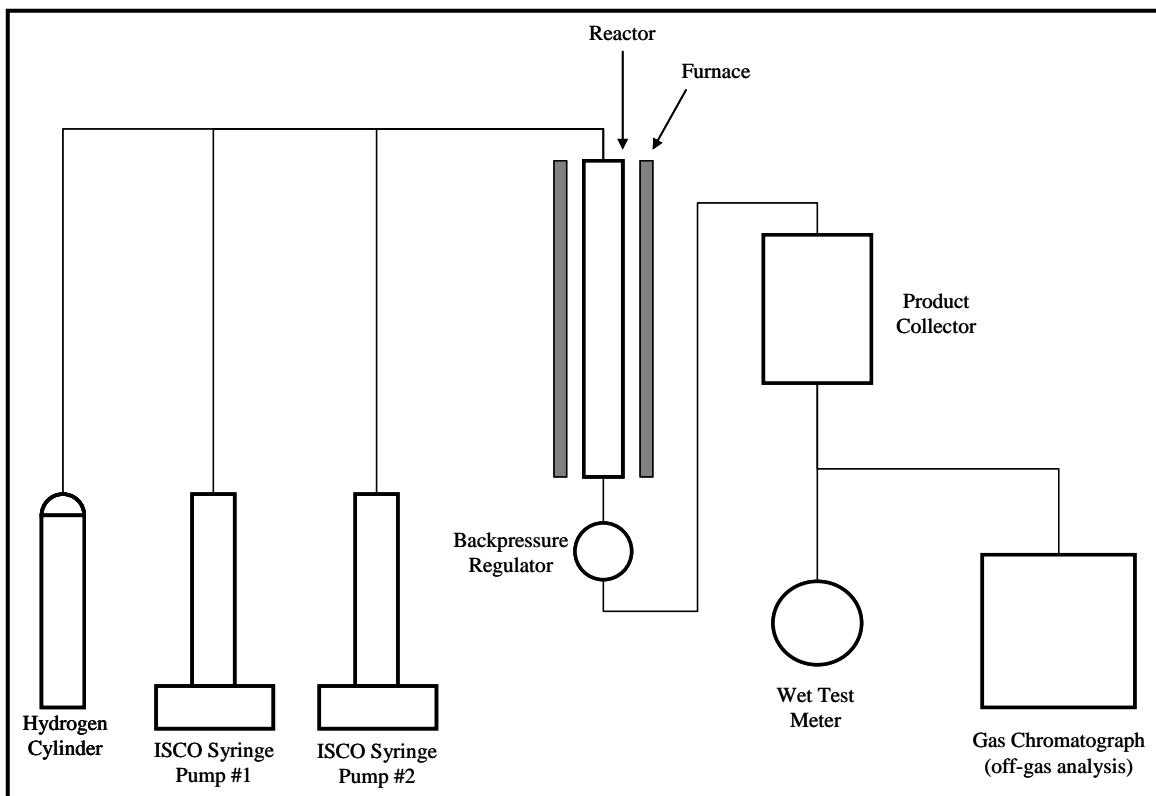


Figure 14. Diagram of Hydroprocessing Unit.

Liquefaction oil and hydroprocessed products were subjected to a standard combustion method to determine the elemental composition (CHNS, O by difference), Karl Fischer analysis for water, and total acidity number (TAN) titration with potassium hydroxide. Product improvements are indicated are a decrease in the oxygen and nitrogen content, decrease in the water content, and decrease in the TAN value. If these preliminary analyses indicated significant improvement in product quality, the product was subjected to additional analyses to determine its suitability as a fuel or fuel blending component.

Process Operations

Short-duration scoping experiments were initially performed to evaluate the upgradability of the liquefaction oil. Values for reaction temperature, reaction pressure, and space velocity were determined with the scoping tests.

Following reactor parameter scoping temperatures for the hydroprocessing were selected to be 250 – 320 °C. The thermal stability of the liquefaction oil was the primary concern for determining the temperature operating envelope. Thermal degradation leads to conversion of the liquid oil into solid carbon (coke) which would plug fill the processing system and require early termination of the experimental trial. This situation must be avoided. Since the hydroprocessed

product is predicted to be more thermally stable, operation at high conversion is desirable. Conversion was maximized by operating at high pressure and low space velocity with typical values of 1200 psig and 0.13 h⁻¹.

When preliminary analysis indicated successful heteroatom removal in the 250 – 320 °C temperature window, a second stage of hydrotreating was performed on the first stage product. The increase in thermal stability induced by the first stage hydrotreating was required before the second stage hydrotreating could occur.

Summary of operation results

The sample of crude liquefaction oil used for the scoping experiments possessed the qualities given in **Table 9**.

Table 9. Properties of crude liquefaction oil

| Property | Value |
|-----------------------|-------|
| C, wt% | 71.5 |
| H, wt% | 8.2 |
| N, wt% | 1.4 |
| O, wt% | 18.9 |
| Total Acidity | 17.3 |
| H ₂ O, wt% | 9.8 |

Attempts at hydrotreating this sample of liquefaction oil at 320 °C with Albemarle KF-757 resulted in formation coking of the oil inside the hydrotreating reactor. This phenomenon resulted in total plugging of the fixed bed reactor and required a shutdown of the system. No upgraded products were produced during this trial.

Attempts to avoid coke formation at 320 °C by dissolving the crude liquefaction oil in methyl-tetrahydrofuran were not successful. While the feedstock was soluble, no upgraded products were produced. All of the liquefaction oil resulted in coke formation of the catalyst surface. The addition of a high boiling solvent, diesel fuel, likewise had no effect.

A substantially lower temperature was required to avoid coke production. As the heteroatom removal would be less effective at this temperature, the space velocity was decreased to allow maximum reaction time. Properties of the crude liquefaction oil appear in the first row. The results appear in **Table 10**.

Table 10. Properties of crude and hydrotreated liquefaction oil

| Hydrotreating Temperature °C | %C | %H | %N | %O | TAN mg KOH / g |
|------------------------------|-------|------|------|-------|----------------|
| --- | 70.44 | 8.32 | 1.45 | 19.79 | 24.61 |
| 270 | 81.08 | 9.64 | 1.68 | 7.60 | 15.07 |
| 280 | 81.35 | 9.25 | 1.79 | 7.61 | 14.95 |
| 290 | 82.53 | 9.57 | 1.76 | 6.14 | 18.80 |
| 300 | 81.79 | 9.58 | 1.71 | 6.92 | 4.36 |

Little oxygen removal was observed. The drop in oxygen content from the crude oil value of 19.79% to 6-7% is the result of removing the entrained water. Some improvement in total acid number was observed dropping from 24.6 mg KOH/g to 18.8 mg KOH /g at 290 °C and 4.36 mg KOH / g.

A sample of crude liquefaction oil produced with water recycle was subjected to hydrotreating with Albemarle KF-757 with similar results as shown in **Table 11**.

Table 11. Properties of crude and hydrotreated liquefaction oil

| Hydrotreating Temperature °C | %C | %H | %N | %O | TAN mg KOH / g |
|------------------------------|-------|-------|------|------|----------------|
| --- | 71.28 | 8.43 | 1.58 | 18.7 | 30.27 |
| 270 | 81.11 | 9.70 | 1.80 | 7.39 | 12.02 |
| 280 | 81.57 | 9.63 | 1.88 | 6.92 | 7.52 |
| 290 | 82.78 | 9.97 | 1.87 | 5.38 | 4.49 |
| 300 | 84.18 | 10.28 | 1.77 | 3.77 | 3.19 |

Some improvement was observed, but the product is still unacceptably high in total acid number.

The final sample processed was produced during a production run. Some initial bench-top measurements indicated higher thermally stability with the crude liquefaction oil and heteroatom removal was attempted with both Albemarle HDO-1 and HDO-2 catalysts. **Table 12** shows the results from hydrotreating at 290 – 320 °C.

Table 12. Properties of crude and hydrotreated liquefaction oil using developmental catalysts

| Catalyst | Hydrotreating Temperature °C | %C | %H | %N | %O | TAN mg KOH / g |
|----------|------------------------------|------|------|------|-----|----------------|
| | --- | 84.2 | 7.9 | 1.71 | 6.1 | 13.0 |
| HDO-2 | 290 | 84.0 | 11.0 | 1.38 | 3.6 | 0.32 |
| HDO-2 | 300 | 84.8 | 10.6 | 1.73 | 2.8 | <0.05 |
| HDO-2 | 310 | 85.4 | 10.8 | 1.62 | 2.2 | <0.05 |
| HDO-2 | 320 | 84.2 | 10.3 | 1.77 | 1.6 | 0.09 |
| HDO-1 | 290 | 83.8 | 10.8 | 1.54 | 3.9 | <0.05 |
| HDO-1 | 300 | 84.9 | 10.9 | 1.49 | 2.8 | <0.05 |
| HDO-1 | 310 | 82.3 | 11.0 | 1.32 | 5.4 | <0.05 |
| HDO-1 | 320 | 85.9 | 11.4 | 1.16 | 1.5 | <0.05 |

The total acid number in the upgraded product is substantially lower at 290 °C than previous trials. Acid number less than 1 remained after hydrotreating at 290 °C compared with 4.49 and 18.8 mg KOH / g using the KF-757 catalyst. Using the HDO-1 catalyst at 320 °C also appeared to remove a portion of the nitrogen heteroatom, a phenomenon not observed previously. These results, coupled with the higher apparent thermal stability, led to attempts at a second, higher temperature hydrotreating stage. The results using the products collected in the first stage hydrotreated at 350 – 380 °C appear in **Table 13**. The listed values indicated with a hydrotreating temperature range are the mean values of the properties after the first hydrotreating

Table 13. Properties of crude and hydrotreated oil using developmental catalysts after 2nd stage

| Catalyst | Hydrotreating Temperature °C | %C | %H | %N | %O | TAN mg KOH / g |
|--------------|------------------------------|-------------|-------------|------------|------------|-----------------|
| HDO-2 | 290-320 | 84.6 | 10.7 | 1.6 | 2.6 | 0.21 |
| HDO-2 | 350 | 87.0 | 12.5 | 0.47 | 0.0 | <0.05 |
| HDO-2 | 365 | 87.5 | 12.1 | 0.29 | 0.1 | <0.05 |
| HDO-1 | 290-320 | 84.2 | 11.0 | 1.4 | 3.4 | <0.05 |
| HDO-1 | 350 | 87.4 | 12.6 | 0.00 | 0.1 | <0.05 |
| HDO-1 | 365 | 87.1 | 12.1 | 0.00 | 0.8 | <0.05 |
| HDO-1 | 380 | 87.7 | 12.2 | 0.00 | 0.0 | <0.05 |

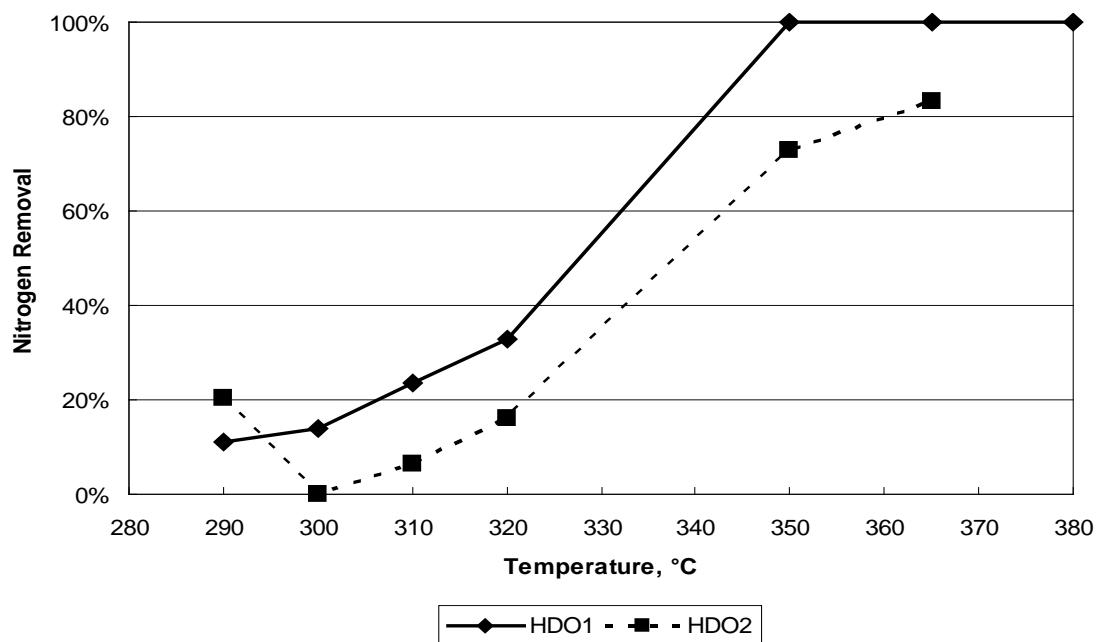


Figure 15. Apparent nitrogen removal activity as a function of hydrotreating temperature.

stage conducted within the 290-320 °C range. The products collected at 290, 300, 310, and 320 °C were combined to generate the feed for the second, higher temperature, hydrotreating stage. The preliminary analysis indicates nearly complete removal of the oxygen content and nitrogen content. HDO-1 showed higher apparent nitrogen removal activity as depicted in **Figure 15**.

The hydrotreated products from the HDO-1 trial were combined into one sample and fractionated into transportation fuel boiling ranges with the results appearing in **Table 14**.

Table 14. Boiling range composition of upgraded liquefaction oil

| Fraction | Mass Percent | Silicon ppm | Nitrogen ppm |
|-----------------------|--------------|-------------|--------------|
| Gasoline 0-380 °F | 13.7 | 70.7 | 24.4 |
| Distillate 380-650 °F | 58.3 | 36.5 | 288.9 |
| Heavy 650-1000 °F | 26.1 | 63.5 | |
| Ultraheavy >1000 °F | 1.8 | n/a | |

Each fraction was subjected to a battery of analytical assays to aid in determining the value of each fraction. Metals analyses showed only silicon as levels of all other metals were below the limit of detection, typically ~1 ppm. Trace nitrogen was about 10-fold higher in the distillate fraction than in the gasoline fraction. It can be assumed that the nitrogen content in the heavy and ultraheavy fractions was higher than the distillate, but available assays were not suitable for these samples.

Fuels and compositional properties of each fraction appear in the following **Tables 15 - 18**.

Table 15. Distillation properties of each fraction

| %OFF | Gasoline | Distillate | Heavy |
|------|----------|------------|-------|
| ibp | 189.5 | 374.1 | 560.4 |
| 10% | 278.0 | 430.7 | 659.8 |
| 50% | 358.0 | 543.7 | 743.1 |
| 90% | 411.1 | 654.3 | 854.5 |
| fbp | 474.7 | 782.0 | 999.9 |

Table 16. Fuel properties of liquefaction oil fractions

| Property | Gasoline | Distillate | Heavy |
|------------------|----------|------------|-------|
| Specific Gravity | 0.827 | 0.973 | 1.157 |
| API Gravity | 39.6 | 14 | -9.2 |
| RVP, psi | 6.173 | | |
| Cetane Index | n/a | 22.48 | n/a |

Table 17. Results from detailed hydrocarbon analysis of gasoline fraction produced from hydrotreating liquefaction oil

| Gasoline | | |
|------------------------------|-------------|-------------|
| Group | %Wgt | %Vol |
| Aromatics | 26.2 | 24.1 |
| Paraffin | 3.6 | 4.0 |
| I-Paraffins | 18.8 | 20.7 |
| Naphthenes | 22.4 | 23.1 |
| Unidentified | 28.9 | 28.1 |
| Plus | 0.1 | 0.1 |
| Olefins | 0.0 | 0.0 |
| Oxygenates | 0.0 | 0.0 |
| | | |
| Carbon Number | %Wgt | %Vol |
| C4 | 0.0 | 0.0 |
| C5 | 0.0 | 0.0 |
| C6 | 0.4 | 0.4 |
| C7 | 1.3 | 1.5 |
| C8 | 4.5 | 4.7 |
| C9 | 17.0 | 17.5 |
| C10 | 29.4 | 29.3 |
| C11 | 13.4 | 13.6 |
| C12 | 4.7 | 4.6 |
| C13 | 0.2 | 0.2 |
| C14 | 0.1 | 0.1 |
| | | |
| Group | RON | MON |
| Aromatics | 24.4 | 25.3 |
| Paraffin | -1.8 | -1.8 |
| I-Paraffins | 15.1 | 15.5 |
| Naphthenes | 19.2 | 16.4 |
| Unidentified | 20.8 | 21.7 |
| Plus | 0.1 | 0.1 |
| Olefins | 0.0 | 0.0 |
| Oxygenates | 0.0 | 0.0 |
| Total Linear RON = 77.81 | | |
| Total Linear MON = 77.08 | | |
| Total Calculated RON = 79.30 | | |
| Total Calculated MON = 78.76 | | |

Table 18. Results from NOISE analysis of distillate and heavy fractions produced from hydrotreating liquefaction oil

| | Distillate | Heavy |
|--------------------------|-------------------|--------------|
| Group | %Wgt | %Wgt |
| Paraffin | 6.97 | 9.72 |
| I-Paraffins | 2.85 | 1.85 |
| Cycloalkanes | 61.11 | 15.34 |
| 1-ring Aromatics | 23.74 | 23.53 |
| 2-ring Aromatics | 5.10 | 34.75 |
| 3-ring Aromatics | 0.23 | 12.30 |
| 4-ring Aromatics | 0.00 | 2.43 |
| 5-ring Aromatics | 0.00 | 0.07 |
| | | |
| Carbon Number | %Wgt | %Wgt |
| 5 | 0.00 | 0.00 |
| 6 | 0.00 | 0.03 |
| 7 | 0.00 | 0.00 |
| 8 | 0.00 | 0.00 |
| 9 | 0.01 | 0.00 |
| 10 | 0.32 | 0.00 |
| 11 | 3.70 | 0.02 |
| 12 | 10.28 | 0.07 |
| 13 | 12.74 | 0.09 |
| 14 | 13.17 | 0.09 |
| 15 | 12.63 | 0.10 |
| 16 | 12.52 | 0.44 |
| 17 | 10.37 | 1.64 |
| 18 | 10.24 | 4.09 |
| 19 | 5.59 | 7.40 |
| 20 | 3.69 | 10.48 |
| 21 | 2.23 | 12.13 |
| 22 | 1.34 | 12.55 |
| 23 | 0.70 | 11.51 |
| 24 | 0.33 | 9.65 |
| 25 | 0.11 | 7.24 |
| 26 | 0.04 | 5.99 |
| 27 | 0.00 | 4.29 |
| 28 | 0.00 | 3.81 |
| 29 | 0.00 | 3.03 |
| 30 | 0.00 | 1.78 |
| 31 | 0.00 | 1.15 |
| 32 | 0.00 | 0.96 |
| 33 | 0.00 | 0.57 |
| 34 | 0.00 | 0.40 |
| 35 | 0.00 | 0.25 |
| 36 | 0.00 | 0.16 |
| 37 | 0.00 | 0.04 |
| 38 | 0.00 | 0.00 |
| 39 | 0.00 | 0.00 |
| 40 | 0.00 | 0.00 |
| average # of carbons | 15.5 | 23.1 |
| average # of hydrogens | 27.2 | 36.4 |
| average # of oxygens | 0.00 | 0.00 |
| average # of sulfurs | 0.00 | 0.00 |
| average # of nitrogens | 0.00 | 0.00 |
| average molecular weight | 213.4 | 313.2 |
| phenolic oxygen, wt% | 0.00 | 0.00 |
| thiophenic sulfur, wt% | 0.00 | 0.00 |
| carbazolic nitrogen, wt% | 0.00 | 0.00 |

Evaluation of Bio-Oil for Catalytic Cracking

The bio-oil from the 1 gallon sample of corn fiber was assessed for its potential for catalytic cracking co-processing with refinery gas oil feedstock. Attempts were made to solubilize or suspend the bio-oil in gas oil for feeding into an ACE™ FCC laboratory testing reactor. Two different gas oils were used, one with a high aromatic content and one with a high aliphatic content. The samples were warmed to 40 °C in an ultrasonic bath to facilitate mixing. The bio-oil was immiscible with both gas oils at concentrations ranging from 5 wt% - 20 wt% bio-oil. In a second set of experiments, the emulsifiers, Atlox 4919 and Atlox 4912, were added to the blends at 1 wt% and 4 wt% in conjunction with warming and ultrasonication. In all cases, within a few minutes the bio-oil separated and formed a streaky film on the walls of the vessel and collected at the bottom. Because of the concern that a phase separation in the feed line to the ACE™ unit would lead to variable (and unverifiable) feed rate of bio-oil into the reactor, efforts were instead focused on hydroprocessing upgrading.

Other properties of the bio-oil that need to be considered for the FCC processing are the high nitrogen content and thermal instability. The bio-oils from corn fiber and corn stover had nitrogen contents of 4 wt% and 1.27 wt%, respectively. Higher catalyst/oil ratios would be needed to compensate for the deactivation by nitrogen. Typical inlet temperatures to the FCC reactor are in the range of 290 – 340°C. A distillation experiment with corn stover bio-oil showed that it converted to coke at 320°C, therefore one would quickly foul (coke up) the process feed preheaters prior to entering the FCCU.

Techno-Economic Assessment

- Developed a baseline process model to allow techno-economic assessments.
- Utilized the model to identify the potential technical improvements that have the most significant impact on process economics.
- Permutations to the baseline model, such as the evaluation of the eliminations of catalyst and reducing gas in the liquefaction step were addressed.
- Using ADM's market information, an assessment of the scale of operation based on availability of feedstock was also undertaken. As experimental data was obtained, the model was updated throughout the life of the project.
- ConocoPhillips contributed the modeling of the upgrading portion to produce fuels.

Hydrothermal Liquefaction: CapEx and OpEx

CapEx

The liquefaction section costs have been estimated after the paper by Goudriaan et al. (2000)³. It is assumed that the unit that has been designed in the referred document can be scaled to a 525 kton/yr biomass feed on a dry basis, with a service factor of 0.959 (350 days per year). The reference case contemplates the treatment of the waste water, so a 20% reduction in capital, electricity and external fuel was implemented for discounting this part of the process that is not employed in the present setup.

The reference has a 30 million dollar (M\$) CapEx (15% contingency) for a feedstock of 130 kton/yr (db), on 2000 US\$. This represents a 73.76M\$ investment for a 525 kton/yr feedstock today, using CEPCI Ann.Index 2000 at 394.1, and the December Preliminary Index 2009 at 524.2, along with the 0.6 scale up rule and the 20% discount because of waste water treatment not used here.

The net biocrude production has been taken from test #17a, at a 17.82%wt yield on a dry basis.

The OSBL has been calculated at 25% of the Equipment Installed Cost and is 18.44M\$. The Total Installed Cost (TIC) is 92.20M\$ for a plant processing 525 kton/yr of dry feedstock.

³ Goudriaan, F., van de Beld, B., Boerefijn, F.R., Bos, G.M., Naber, J.E., van der Wal, S. and Zeevalkink, J.A., Thermal efficiency of the HTU® Process for Biomass Liquefaction, in "Progress in Thermochemical Biomass Conversion", Tyrol, Austria, Sept.2000.

OpEx

Feedstock has been priced at \$60/ton (db), capital is charged at 10% (linear), and maintenance + overhead+ insurance+ taxes + royalties were taken at 6% as in the reference paper, for the sake of similarity. Labor was charged for the same number of workers per shift, at the same level as in reference, but the salaries were adjusted by inflation using inflationdata.com and the feedstock flow rate. It was taken the inflation estimated since June 2000 to January 2010 that it turned to be 25.69%.

Electricity was priced at 50.00 \$/MWh and natural gas was priced 5.00 \$/MBTU. The OpEx shown by Goudriaan et al. was used on a feedstock flow rate basis and was scaled up using the updated prices referred above and feedstock flow rate. Electricity and natural gas contributed 16.84 \$/ton and 6.21\$/ton oil product respectively. **Table 19** summarizes all the results above.

Table 19. Hydrothermal liquefaction Economics for a 525 kton/yr Dry Biomass Feed. [1 ton = 1,000 kg]

| | | | |
|--------------------------------|--------|-------------------|--------|
| Capacity intake (kton/yr) (db) | 525 | | |
| Net biocrude prod.(kton/yr) | 93.56 | | |
| Installed capital(M\$) | 73.76 | (15% contingency) | |
| | M\$/yr | \$/ton prod | % |
| Capital charge (10%) | 7.38 | 78.84 | 15.62 |
| Feedstock (\$/dt) | 31.50 | 336.70 | 66.71 |
| Labor | 1.76 | 18.81 | 3.73 |
| Maintenance,Ovh,etc 6% | 4.43 | 47.31 | 9.37 |
| Electricity | 1.58 | 16.84 | 3.34 |
| External Fuel | 0.58 | 6.21 | 1.23 |
| <hr/> | | | |
| TOTAL | 47.22 | 504.71 | 100.00 |
| <hr/> | | | |
| OSBL(25% IC) | 18.44 | | |
| TIC(M\$) | 92.20 | | |
| \$/GJ | 15.086 | | |
| \$/gal | 2.121 | | |

The HHV estimated for the Biocrude composition from test # 17a was used for calculating the OpEx on a product energy basis, as in the referred paper.

The correlation by Dermirbas⁴ was used for a dry basis composition of Biocrude of: 75.05%wt C, 7.42%wt H, 13.23%wt O and 1.41%wt N.

The OpEx can also be reported on a gallon basis, with an oil density of 1,110 kg/cm as reported in test #17a. In this case the resulting cost was 2.12 \$/gal.

Two major differences can be noticed when compared with Goudriaan et al. work: 1) feedstock has been incorporated in this work, when in the reference this input was ignored; 2) the biocrude yield in the present work (17.82%) is noticeable lower than the 37% yield reported by Goudriaan et al.

For this reason sensitivities were estimated around these two parameters that are shown in **Tables 20 and 21.**

Table 20. Operational Cost Sensitivity to Biocrude Yield at 60\$/ton Biomass

| Biocrude Yield (%wt) | OpEx (k\$/ton product) | OpEx (\$/gal) |
|----------------------|------------------------|---------------|
| 10 | 0.899 | 3.779 |
| 20 | 0.450 | 1.890 |
| 30 | 0.300 | 1.260 |
| 40 | 0.225 | 0.945 |

Notes: Costs on a dry basis (db). All other OpEx costs fixed as base case on a 525 kton/yr (db) biomass feed.

Table 21. Operational Costs Sensitivity to Feedstock price (\$/ton,) at 17.82%wt Biocrude Yield. All other OpEx costs fixed as base case on a 525 kton/yr (db) biomass feed.

| Feedstock price (\$/ton) (db) | OpEx (k\$/ton product) | OpEx (\$/gal) |
|-------------------------------|------------------------|---------------|
| 20 | 0.280 | 1.178 |
| 60 | 0.505 | 2.121 |
| 100 | 0.729 | 3.064 |
| 140 | 0.954 | 4.007 |

Notes: Costs on a dry basis (db). All other OpEx costs fixed as base case on a 525 kton/yr (db) biomass feed.

⁴ Demirbas, A., Calculation of higher heating values of biomass fuels, Fuel, 76, 431-434(1997).

The cost per unit mass of oil product improves with increased biocrude yield (see Table 19) at a fixed feedstock price. A four-fold bio-oil yield increase translates into a 75% OpEx reduction on a product oil basis. The feedstock price also has a significant impact upon OpEx value on a product oil basis as seen from **Table 21**.

As a reference market value, NYMEX Heating Oil Future price closed at 2.25 \$/gal on 04/23/2010. Biocrude yield improvements have the potential of greatly enhancing the HTL economics, along with the usage of the most economic feedstock.

Hydrotreating: CapEx and OpEx

The CapEx and OpEx for the dual-stage hydrotreating process have been estimated at \$58.6 M and \$31.8 M, respectively. The estimate for the CapEx (**Table 22**) is based on January 2008 costs of equipment inflation escalated to present day. The magnitude of the process was based on 1500 dry tons per day corn stover fed to the hydrothermal liquefaction stage producing 267.3 tons per day raw oil (dry basis) with a composition of 77.2%C, 7.6%H, 13.6%O, 1.5%N, and 0.1% S. With the final product after two stages of hydrotreating having a composition of 87.8%C and 12.3%H, the total hydrogen consumption is 15.3 MM SCFD. The breakdown of the OpEx is shown in **Table 23**.

Table 22. Major Equipment for Capital Expense Estimate

| Component Name | Equipment Cost | No. of items | Total Equipment Cost | Cost Factor | Total Direct Costs | % of Cost |
|----------------------------------|----------------|--------------|----------------------|----------------------|--|--------------|
| Charge Tank | \$ 547,300 | 1 | \$ 547,300 | 2.00 | \$ 1,094,600 | 4.1 |
| Feed Pump | \$ 90,400 | 2 | \$ 180,800 | 4.00 | \$ 723,200 | 2.7 |
| Alcohol Rxn Preheater | \$ 30,600 | 1 | \$ 30,600 | 3.50 | \$ 107,100 | 0.4 |
| Alcohol Rxr Rxfeed/RX exchanger | \$ 111,900 | 1 | \$ 111,900 | 3.50 | \$ 391,650 | 1.5 |
| Low temp reactor | \$ 1,920,000 | 1 | \$ 1,920,000 | 4.00 | \$ 7,680,000 | 28.9 |
| RXR Feed/Effluent Heat Exchanger | \$ 98,000 | 1 | \$ 98,000 | 3.50 | \$ 343,000 | 1.3 |
| Trim Heater | \$ 256,700 | 1 | \$ 256,700 | 3.50 | \$ 898,450 | 3.4 |
| High temp reactor | \$ 1,920,000 | 1 | \$ 1,920,000 | 4.00 | \$ 7,680,000 | 28.9 |
| Reactor HP Flash Column | \$ 106,900 | 1 | \$ 106,900 | 4.00 | \$ 427,600 | 1.6 |
| Recycle Compressor | \$ 1,323,900 | 1 | \$ 1,323,900 | 2.20 | \$ 2,912,580 | 11.0 |
| Reactor Cooler | \$ 924,600 | 1 | \$ 924,600 | 3.50 | \$ 3,236,100 | 12.2 |
| UPGRADE.DMDSPUMP | \$ 38,900 | 2 | \$ 77,800 | 4.00 | \$ 311,200 | 1.2 |
| Feed 2/RXR Effluent HX | \$ 32,200 | 1 | \$ 32,200 | 3.50 | \$ 112,700 | 0.4 |
| 150# steam HX | \$ 50,300 | 1 | \$ 50,300 | 3.50 | \$ 176,050 | 0.7 |
| Product Storage | \$ 200,000 | 1 | \$ 200,000 | 2.00 | \$ 400,000 | 1.5 |
| DMDS Storage Tank | \$ 9,300 | 1 | \$ 9,300 | 4.00 | \$ 37,200 | 0.1 |
| Intermediate Storage Tank | \$ 241,900 | 1 | \$ 241,900 | 2.00 | \$ 483,800 | 1.8 |
| | | | \$ 8,032,200 | | | |
| | | | | | TOTAL Equipment Cost | Jan-08 |
| | | | | \$ 26,531,430 | Total Direct Field Cost | |
| | | | | \$ 13,265,715 | Indirect Costs (% of DFC) | 50 |
| | | | | \$ 39,797,145 | Total Direct and Indirect Costs | |
| | | | | \$ 11,939,144 | Contingency and Cost Growth Allowance | 30 |
| | | | | | | CE Index |
| | | | | \$ 51,736,289 | Current Costs (1st Qtr 2008) | 539.9 |
| | | | | \$ 51,065,509 | Escalation to current costs | 0.9870 532.9 |
| | | | | \$ 51,065,509 | Total Capital Cost (Future) | |
| | | | | \$ 51,065,509 | Total Capital Cost (Location Adjusted) | |
| | | | | \$ 51,000,000 | Rounded Capital Cost | 1 Midwest |
| | | | | 4.95 | "Lang Factor" before contingency | |
| | | | | 6.35 | "Lang Factor" after contingency | |

Table 23. Estimate of Operating Expenses

| | Factor | | |
|--|----------------------|----------|--------------|
| Fixed Capital Investment | | | \$51,000,000 |
| Working Capital | 15% | | \$7,650,000 |
| Total Plant Investment | | | \$58,650,000 |
| Operating Expenses | | Units/hr | |
| <i>Raw Materials:</i> | | | |
| Feed to upgrader, \$/lb | 0 | 24503 | \$0 |
| <i>Labor:</i> | | | |
| Operating Labor, \$/yr/(operator/shift) | 279,552 | 3 | \$838,656 |
| Maintenance Labor | 1.5 % FCI | | \$765,000 |
| Laboratory | 20 % operating labor | | \$167,731 |
| Supervision | 15 % operating labor | | \$125,798 |
| <i>Utilities:</i> | | | |
| 1000# Steam, \$/Klbs | 8.00 | 62.27 | \$3,945,427 |
| Electricity, \$/kwh | 0.050 | 1957 | \$774,972 |
| Cooling Water, \$/Mgal circulating (20 F dT) | 0.060 | 621.4 | \$295,289 |
| Waste water treatment, \$/Kgal | 3.00 | 7.19 | \$170,834 |
| 150# Steam credit, \$/Klbs | 5 | -18.365 | -\$727,254 |
| <i>Catalysts and Chemicals:</i> | | | |
| Maintenance materials | 1.5 % FCI | | \$765,000 |
| General | 0.5 % FCI | | \$255,000 |
| Hydrogen, 435# (from NG), \$/KSCF | 2.48 | 637.5 | \$12,521,520 |
| Hydrogen, 435# (from offgas stream), \$/KSCF | 2.67 | 0 | \$0 |
| HT Catalyst 1 Costs, \$/lb | 20.00 | 128963 | \$2,579,260 |
| HT Catalyst Replacements per yr | 1 | | |
| HT Catalyst 2 Costs, \$/lb | 20.00 | 128963 | \$2,579,260 |
| Alcohol HT Catalyst Replacements per yr | 1 | | |
| DMDS usage, \$/KT | 2300 | 0.00424 | \$77,236 |
| Plant Overhead | 60 % Total Labor | | \$1,138,311 |
| Insurance and Taxes | 2 % FCI | | \$1,020,000 |
| Corporate | 80% Total Labor | | \$1,517,748 |
| Operating Costs (Reformer for offgas) | | | \$3,000,000 |
| TOTAL OPERATING COSTS | | | \$31,809,790 |
| Hours on stream | 7920 | | |

Catalytic Hydrothermal Gasification: Technoeconomic Assessment

The CapEx of the catalytic hydrothermal gasification of the aqueous phase byproduct was assessed. Using the information in the PNNL study of Catalytic Hydrothermal Gasification of Lignin-Rich Biorefinery Residues and Algae⁵ a rough order of magnitude estimate was generated for gasification of the aqueous phase. The yield of aqueous phase was used from the same experiment, test #17a, as was used for the HTL CapEx estimates and the scale was adjusted from that in the report to that used in this study for HTL. The gas yields are based on test #2 in [Table 8](#). Based on a Total Equipment Cost from the report of \$14M for a plant processing 3.64 Mtonne/y, a plant of 4.93 Mtonne/yr for the HTL case would cost about \$16.8M (using a power of 0.6 to scale). In the report the Total Project Investment is given as \$52M including all the direct (installations, instrumentation and control, piping, electrical, building and services, yard improvements) costs and the indirect (engineering, construction, legal & contract fees, contingency) costs.

An important question also addressed in this assessment was the potential for hydrogen production using the gas product. Again, using the data in [Table 8](#) for gas yield and composition, and scaling to the plant size used in the HTL assessment, the yield of methane gas could be used to generate well in excess of the hydrogen requirement for the upgrading of the bio-oil to hydrocarbon fuels. The stoichiometric yield of hydrogen from the 10.5 MSCF/d methane when processed by steam reforming could generate as much as 42 MSCF/d of hydrogen compared to the requirement for hydrotreating of 15 MSCF/d. With other assumptions of reduced yield due to process efficiency, clearly there is sufficient methane produced with excess available for other process heating requirements.

Conclusions

Hydrothermal liquefaction can be applied to corn fiber, corn starch, or corn stover in water slurry to produce a bio-oil with 10-15% oxygen on a dry basis. Overall carbon basis yields for the several feedstocks ranged from 20% for starch, 50-55% for fiber and 30-35% for stover. The undesirable oxygen content of these HTL bio-oils is much lower than that achieved through fast pyrolysis of biomass, but at the expense of a lower bio-oil yield. The bio-oil can usually be gravity separated from the aqueous byproduct but the formation of a stable emulsion was seen during the processing of corn stover. It was thought that the mineral (ash) content of the feedstock caused this phenomenon therefore a mineral separation step prior to phase separation was developed. Only a small fraction of the biomass is converted to a gas byproduct (5-10% of

⁵ Elliott, DC, et al. *Catalytic Hydrothermal Gasification of Lignin-Rich Biorefinery Residues and Algae: Final Report*, PNNL-18944, Pacific Northwest National Laboratory, Richland, WA, **October 2009**.

the carbon) consisting mainly of carbon dioxide. The balance of the carbon is found in dissolved organics in the aqueous byproduct stream. Recycle of this aqueous stream as the solvent in the preparation of the feed slurry appears to facilitate the conversion of water soluble organics to bio-oil. Additionally, the aqueous byproduct stream can be processed via catalytic hydrothermal gasification technology to produce fuel gas and a low BOD aqueous stream. The methane produced through gasification could be reformed into hydrogen and is sufficient to provide all the hydrogen required for upgrading the bio-oil to fuel.

The bio-oil product can be hydroprocessed in two stages to form hydrocarbons. Fractionation of the resulting product showed 14 wt% gasoline range, 58 wt% diesel range, and 28 wt% gas oil. The gasoline fraction had an octane value of 79 and could be used as a sub octane blending component. The diesel boiling range fraction had high aromatics content and would be suitable for distillate blending, solvent applications, or further processing via catalytic cracking. The gas oil fraction could be blended into fuel oil or further processed via catalytic cracking.

Based on the techno-economic analysis of the process, the overall capital expense for a unit capable of processing 525,000 mt/year is approximately \$125 million. The annual operational expense including feedstock costs is approximately \$72 million. Based on a yield of 42.5 gallons upgraded bio-oil per metric ton of corn stover, the minimum selling price of the bio-oil is \$4.11 per gallon (\$172.62/bbl). Because further refinery processing is required to incorporate the bio-oil into a final finished fuel, the bio-oil would have a break-even value to a refiner similar to light to medium gravity low sulfur crude oil or condensate. The current premiums for these grades of crude oil range from \$3-7 over NYMEX WTI. Thus the current price structure of the crude market does not support commercialization of this process at its current stage of development.

Currently both ADM and ConocoPhillips do not plan to conduct further research and development with this process. Significant barriers to commercialization of this technology are identified as follows:

- Low primary oil yield
- Energy consumption for grinding biomass into a slurry
- Process and product sensitivity to feedstock impurities.
- Hydrogen requirements for upgrading

Significant technical improvements addressing these barriers are needed before warranting additional evaluation of this technology.

APPENDIX A
Data Sheets for Hydrothermal Liquefaction

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | | | |
|---|-----------------------|-------------------------------------|----------------------------|--|---------------------------------|----------------|-------------------|-----------|---------|-----------------|--|
| Run No. | HTL-1 | | | | | reactor vloume | 1000 | total mL | | | |
| Pressure | 20.2 MPa 2918 psig | Reactor Temperature degrees C | 158 346 357 | Time | 10:30-11:20 | 600 | mL at temperature | | | | |
| Total Feed | 1657 cc | Feed rate | 2000 cc/hr 2020.00 g/hr | | | | | | | | |
| ground corn fiber | | | | | | | | | | | |
| Total Product | 1697.4 g | Product oil | 116.4 g/hr 1920.5 g/hr | sum of two phases | 2036.9 g/hr | | | | | | |
| Elemental Analyses | | C | H | O | density | moisture | N | S | ash | | |
| feed | 46.24% | 6.58% | 46.55% | 1.01 | 87.30% | | 2.04% | 0.33% | 0.89% | 102.61% | |
| product oil | 69.31% | 7.40% | 19.85% | 1.14 | | (2.56%) | 2.70% | 0.29% | 99.55% | actual measured | |
| oxygen by difference | 1.59% | 11.20% | 87.16% | 1.0 est | 97.00% | | 0.05% | | 100.00% | 84.64% | |
| Material Balance | | g C/hr | g H/hr | g O/hr | | Total | TAN | Viscosity | | | |
| feed | 118.61 | 214.55 | 1685.19 | | | 2018.35 | | | | | |
| product oil | 80.68 | 8.61 | 23.11 | | | | | | | | |
| aqueous | 30.54 | 215.10 | 1673.91 | | | | | | | | |
| gas | 4.46 | 0.00 | 11.87 | | | | | | | | |
| Total Products | 115.67 | 223.70 | 1708.88 | | | 2048.26 | | | | | |
| Elemental Balance | 98% | 104% | 101% | | | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | >>>>>> | | 101% | | | | | |
| GAS CALCULATIONS | | | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 | g H/hr | | | | | | |
| Gas Out | 304.8 L/hr | 12.70 | moles/hr | | | | | | | | |
| Gas Composition | | moles/hr | | | | | | | | | |
| | volume% | C | H | O | | | | | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 | gH/hr | | | | | |
| CarbDioxide | 2.92% | 0.37 | | 0.74 | | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | | | | | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | | | | | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | | | |
| | 2.92% | | | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | | | |
| Total Gas, C1-C4 | 4.46 | 0.00 | 11.87 | 16.33 | Total gas | | | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | | | |
| | 4.46 | 0.00 | 11.87 | 16.33 | Total gas mass out | | | | | | |
| Yields | | | | | 68.02% carbon conversion to oil | | | | | | |
| Oil Product Yield | 101.78 g/hr | 89.28 ml/hr | | 0.04 L/L feed | | | | | | | |
| | | density | 1.14 g/ml | 39.67% mass conversion to oil | | | | | | | |
| Oil Loss in Aqueous | 57.62 g/hr | 57.62 ml/hr | | 0.03 L/L feed | | | | | | | |
| | | density, est | 1 g/ml | 25.74% carbon conversion to water solubles | | | | | | | |
| Gasification of Carbon | 3.76% | | | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | | | | |
| | | | | 0.00 L/hr consumption | | | | | | | |
| | | | | 0.00 L/L feed | | | | | | | |
| | | | | 0 g/g feed | | | | | | | |
| Space velocity | 3.33 L/L/hr | LHSV | | | | | | | | | |
| Chemical Hydrogen Consumption | 0.00 g/hr | 0 L/L | | 0 nM3/tonne | | | | | | | |
| Calculation of Deoxygenation | | | | | | | | | | | |
| O content of dry product | 9.93% | | | | | | | | | | |
| O in Dry Product | 10.11 g/hr | | | | | | | | | | |
| O in Organics(H2O) | 18.01 g/hr-----> | | | | | | | | | | |
| O in Dry Feed | 119.41 g/hr | | | | | | | | | | |
| Deoxygenation | 76.45% | | | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|----------------------------|--------------------------|----------------------------|-------------------|--|--------------|-----------|-------|-------------------------|
| Run No. | HTL-2 | CSTR Reactor | 266 | CSTR volume | 1000 mL | | | | |
| Pressure | 20.9 MPa 3019 psig | Tube temp., degrees C | 313 bottom 356 top | reactor volume | 800 mL, at temp | 1000 mL | | | |
| Total Feed | 4002 cc 193467 COD, ppm | Feed rate | 2001 cc/hr 2021.01 g/hr | Time | 9:30-11:30 | Date | 12-Jun-08 | | |
| Total Product | 4076.1 g | Product oil | 52 g/hr | sum of two phases | 2038 | | | | |
| Elemental Analyses | C | H | O | density | moisture | N | S | ash | |
| feed | 47.81% | 5.69% | 47.20% | 1.01 | 87.28% | 1.90% | 0.21% | 0.89% | 103.68% |
| product oil | 71.51% | 7.52% | 19.39% | 1.14 | 8.43% | 2.80% | 0.29% | | 101.51% actual measured |
| oxygen by difference | 0.95% | 9.16% | 89.84% | 1.0 est | 97.00% | 0.05% | | | 100.00% measured |
| Material Balance | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | | | |
| feed | 122.89 | 212.35 | 1687.53 | 2022.77 | | | | | |
| product oil | 37.19 | 3.91 | 10.08 | | | | | | |
| aqueous | 18.77 | 181.92 | 1784.22 | | | | | | |
| gas | 4.27 | 0.00 | 11.38 | | | | | | |
| Total Products | 60.22 | 185.83 | 1805.68 | 2051.73 | | | | | |
| Elemental Balance | 49% | 88% | 107% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>> | 101% | | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 | g H/hr | | | | |
| Gas Out | 292 L/hr | 12.17 | moles/hr | | | | | | |
| Gas Composition | volume% | C | H | O | | | | | |
| Hydrogen | 0.00% | 0.00 | | | 0.00 gH/hr | | | | |
| CarbDioxide | 2.92% | 0.36 | | 0.71 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | | | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | | | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | |
| | 2.92% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | Total gas | | | | | |
| Total Gas, C1-C4 | 4.27 | 0.00 | 11.38 | 15.65 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 4.27 | 0.00 | 11.38 | 15.65 | Total gas mass out | | | | |
| Yields | | | | | 30.26% carbon conversion to oil | | | | |
| Oil Product Yield | 47.62 g/hr | | 41.77 ml/hr | | 0.02 L/L feed | | | | |
| | | density, | 1.14 g/ml | | 18.52% mass conversion to oil | | | | |
| Oil Loss in Aqueous | 59.58 g/hr | | 59.58 ml/hr | | 0.03 L/L feed | | | | |
| | | density, est | 1 g/ml | | 15.27% carbon conversion to water solubles | | | | |
| Gasification of Carbon | 3.47% | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | |
| | | | | | 0.00 L/hr consumption | | | | |
| | | | | | 0.00 L/L feed | | | | |
| | | | | | 0 g/g feed | | | | |
| Space velocity | 2.50 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | 0.00 g/hr | | 0 L/L | | 0 nM3/tonne | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 12.99% | | | | | | | | |
| O in Dry Product | 6.19 g/hr | | | | | | | | |
| O in Organics(H2O) | 71.85 g/hr-----> | | | | | | | | |
| O in Dry Feed | 121.33 g/hr | | | | | | | | |
| Deoxygenation | 35.68% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|---|--|---|---|---|--|--|------------------|------------------|
| Run No. | HTL-3 | CSTR Reactor | 347 | CSTR volume | 1000 mL | | | | |
| Pressure | 20.5 MPa 2963 psig | Tube temp degrees C | 338 bottom 350 top | reactor volume | 900 mL, at temp | | | | 1000 mL, total |
| Total Feed ground corn fiber | 5837 cc 10.22 pH 199.867 COD, ppm | Feed rate | 2001 cc/hr 2021.01 g/hr | | | | | | |
| Total Product | 5787.2 g | 6.60 wt% Na | Product oil | 86.2 g/hr | sum of two phases | 1984.2 g/hr | | | |
| Elemental Analyses | | 47.81% C 47.20% H 5.69% O 1.01% N 0.21% S 0.89% ash | Product aqueous | 1898 g/hr | 7.71 pH | | | | 103.68% measured |
| normalized | feed | 46.00% C 77.65% product oil 2.21% aqueous | 45.40% H 8.06% O 9.34% N 88.60% S 1.0 est | 85.26% density 7.79% moisture | 1.90% N 2.01% S 0.05% ash | 0.21% O 0.27% S 100.00% ash | 0.89% O 97.32% measured 99.96% ash | 100.00% measured | |
| Material Balance | q C/hr feed | 137.03 | q H/hr product oil | 209.84 6.95 | q O/hr aqueous | 1665.20 8.05 | Total gas | TAN | Viscosity |
| | product oil | 66.93 | aqueous | 41.99 | gas | 172.62 1681.63 | | | |
| | aqueous | 9.20 | gas | 9.20 | | 0.00 24.51 | | | |
| | Total Products | 118.13 | | 179.57 | | 1714.19 | | | 2011.89 |
| | Elemental Balance | 86% | | 86% | | 103% | | | |
| Total Material Balance | >>>>> | >>>>>>>>> | >>>>>> | >>>>> | | 100% | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | | 0.00 q H/hr | | | | |
| Gas Out | 301.4 L/hr | | | 12.56 moles/hr | | | | | |
| Gas Composition | volume% | C Hydrogen CarbDioxide CarbMonoxide Methane Ethane Propane Butanes Pentanes Higher HC (C7H14) | moles/hr | H 0.00 0.77 0.00 0.00 0.00 0.00 0.00 0.00 0.15 | O 0.00 1.53 0.00 0.00 0.00 0.00 0.00 0.00 0.30 | 0.00 qH/hr | | | |
| | | 6.10% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.17% | | | | | | | |
| | | 6.27% | | | | | | | |
| | | C g/hr Total Gas, C1-C4 Total Gas Oil, C5-C7 | H g/hr 9.20 1.79 | O g/hr 0.00 24.51 0.30 | 33.71 2.10 35.81 | Total gas Total gas oil Total gas mass out | | | |
| Yields | | | | | 48.85% carbon conversion to oil | | | | |
| Oil Product Yield | 79.49 g/hr | | 72.26 ml/hr | | 0.04 L/L feed | | | | |
| Oil Loss in Aqueous | 136.09 g/hr | | 1.1 g/ml 136.09 ml/hr | | 26.68% mass conversion to oil 0.07 L/L feed | | | | |
| Gasification of Carbon | 6.71% | | density, est | 1 g/ml | 30.64% carbon conversion to water solubles | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | 0.00 g/hr consumption 0.00 L/hr consumption 0.00 L/L feed 0 g/g feed | | | | |
| Space velocity | 2.22 L/L/hr | | LHSV | | | | | | |
| Chemical Hydrogen Consumption | | | 0.00 g/hr | | 0 L/L | | 0 nm3/tonne | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 2.61% | | | | | | | | |
| O in Dry Product | 2.08 g/hr | | | | | | | | |
| O in Organics(H2O) | 115.48 g/hr.....> | | | | | | | | |
| O in Dry Feed | 135.25 g/hr | | | | | | | | |
| Deoxygenation | 13.08% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | | | | |
|---|-------------------|--------------|----------------------|--|---------------------------------|-------------------|-------------|-----------|-------|---------|-----------------|--|
| Run No. | HTL-3 | | CSTR Reactor | 347 | CSTR volume | 1000 mL | | | | | | |
| Pressure | 20.5 MPa | | Tube temp, degrees C | 338 bottom 350 top | reactor volume | 900 mL, at temp | | | | | | |
| | 2967 psig | | | | Time | 12:55:15:35 | | | | | | |
| | | | | | Date | 18-Jun-08 | | | | | | |
| Total Feed | 5323 cc | | Feed rate | 1996.1 cc/hr | | | | | | | | |
| ground corn fiber | 10.22 pH | | | 2016.06 g/hr | | | | | | | | |
| | 199.867 COD, ppm | | | 15.2 wt% Na ₂ CO ₃ on dry corn fiber | | | | | | | | |
| Total Product | 5321.8 g | | 6.60 wt% Na | Product oil 94.9 g/hr | | sum of two phases | 1995.7 g/hr | | | | | |
| | | | | Product aqueous 1900.8 g/hr | | | | | | | | |
| | actual feed | 47.81% | 5.69% | 47.20% | | 1.90% | 0.21% | | | | | |
| Elemental Analyses | | C | H | O | density | moisture | N | S | ash | | | |
| normalized | feed | 46.00% | 5.60% | 45.40% | 1.01 | 85.26% | 1.90% | 0.21% | 0.89% | 100.00% | | |
| | product oil | 76.44% | 8.01% | 10.53% | 1.1 | 11.10% | 2.53% | 0.27% | | 97.78% | actual measured | |
| oxygen by difference | aqueous | 2.20% | 9.08% | 88.70% | 1.0 est | 95.00% | 0.05% | | | 100.03% | | |
| Material Balance | | g C/hr | g H/hr | g O/hr | | Total | TAN | Viscosity | | | | |
| | feed | 136.70 | 209.33 | 1661.12 | | 2007.15 | | | | | | |
| | product oil | 72.54 | 7.60 | 9.99 | | | | | | | | |
| | aqueous | 41.77 | 172.59 | 1686.01 | | | | | | | | |
| | gas | 12.94 | 0.00 | 34.47 | | | | | | | | |
| | Total Products | 127.24 | 180.20 | 1730.48 | | 2037.92 | | | | | | |
| | Elemental Balance | 93% | 86% | 104% | | | | | | | | |
| Total Material Balance | | >>>>> | >>>>>>> | >>>>> | >>>>> | 102% | | | | | | |
| GAS CALCULATIONS | | | | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | 0.00 g H/hr | | | | | | | | |
| Gas Out | 311.5 L/hr | | 12.98 moles/hr | | | | | | | | | |
| Gas Composition | | moles/hr | | | | | | | | | | |
| | volume% | C | H | O | | | | | | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 g H/hr | | | | | | | |
| CarbDioxide | 8.30% | 1.08 | | 2.15 | | | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | | | | | | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | | | | | | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | | | | |
| Higher HC (C7H14) | 0.40% | 0.36 | 0.73 | 0.00 | | 2.94 | | | | | | |
| | 8.70% | | | | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | | | | |
| Total Gas, C1-C4 | 12.94 | 0.00 | 34.47 | 47.41 | Total gas | | | | | | | |
| Total Gas Oil, C5-C7 | 4.36 | 0.73 | 0.00 | 5.10 | Total gas oil | | | | | | | |
| | 17.30 | 0.73 | 34.47 | 52.51 | Total gas mass out | | | | | | | |
| Yields | | | | | 53.06% carbon conversion to oil | | | | | | | |
| Oil Product Yield | 84.37 g/hr | | 76.70 ml/hr | 0.04 L/L feed | | | | | | | | |
| | | density, | 1.1 g/ml | 28.39% mass conversion to oil | | | | | | | | |
| Oil Loss in Aqueous | 95.04 g/hr | | 95.04 ml/hr | 0.05 L/L feed | | | | | | | | |
| | | density, est | 1 g/ml | 30.56% carbon conversion to water solubles | | | | | | | | |
| Gasification of Carbon | 9.46% | | | | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | 0.00 g/hr consumption | | | | | | | | |
| | | | | 0.00 L/hr consumption | | | | | | | | |
| | | | | 0.00 L/L feed | | | | | | | | |
| | | | | 0 g/g feed | | | | | | | | |
| Space velocity | 2.22 L/L/hr | LHSV | | | | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | 0 L/L | 0 nM3/tonne | | | | | | | | |
| Calculation of Deoxygenation | | | | | | | | | | | | |
| O content of dry product | 0.75% | | | | | | | | | | | |
| O in Dry Product | 0.63 g/hr | | | | | | | | | | | |
| O in Organics(H ₂ O) | 80.89 g/hr-----> | | | | | | | | | | | |
| O in Dry Feed | 134.91 g/hr | | | | | | | | | | | |
| Deoxygenation | 39.58% | | | | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|-------------------|--------------|--|-------------|--|-----------------------|-----------------|----------------|-------------------------|
| Run No. | HTL-4 | CSTR Reactor | 349 | CSTR volume | 1000 mL | reactor volume | 900 mL, at temp | 1000 mL, total | |
| Pressure | 20.1 MPa | Tube temp, | 339 bottom | Time | 9:35:13-25 | | | | |
| | 2911 psig | degrees C | 350 top | Date | 19-Jun-08 | | | | |
| Total Feed | 7667 cc | Feed rate | 2000 cc/hr | | | | | | |
| ground corn fiber | 7.96 pH | | 2020.10 g/hr | | | | | | |
| | 194,933 COD, ppm | | 7.28 wt% Na ₂ CO ₃ on dry corn fiber | | | | | | |
| Total Product | 7692.7 g | 3.16 wt% Na | Product oil | 89.2 g/hr | sum of two phases | 2006.8 g/hr | | | |
| | | | Product aqueous | 1917.6 g/hr | 5.89 pH | | | | |
| actual feed | 47.81% | 5.69% | 47.20% | | 1.90% | 0.21% | 0.89% | 103.68% | |
| Elemental Analyses | | C | H | O | density | moisture | N | S | ash |
| normalized | feed | 46.00% | 5.60% | 45.40% | 1.0 | 86.27% | 1.90% | 0.21% | 0.89% 100.00% |
| | product oil | 76.20% | 8.49% | 12.31% | 1.1 | 8.70% | 3.16% | 0.30% | 100.46% actual measured |
| oxygen by difference | aqueous | 1.99% | 0.08% | 88.85% | 1.0 est | 97.00% | 0.08% | | 100.00% |
| Material Balance | | q C/hr | q H/hr | q O/hr | | product moisture est. | | | |
| | feed | 127.59 | 210.89 | 1673.31 | | Total | | | |
| | product oil | 67.97 | 7.58 | 10.98 | | | | | |
| | aqueous | 38.21 | 174.12 | 1703.79 | | | | | |
| | gas | 10.49 | 0.00 | 27.95 | | | | | |
| | Total Products | 116.67 | 181.69 | 1742.73 | | 2041.09 | | | |
| | Elemental Balance | 91% | 86% | 104% | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | >>>>> | 101% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 | moles/hr | | 0.00 g H/hr | | | |
| Gas Out | 308.1 L/hr | | 12.84 | moles/hr | | | | | |
| Gas Composition | | moles/hr | | | | | | | |
| | volume% | C | H | O | | | | | |
| Hydrogen | 0.00% | | 0.00 | | | 0.00 g H/hr | | | |
| CarbDioxide | 6.81% | 0.87 | | 1.75 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | | | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | | | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.14% | 0.13 | 0.25 | 0.00 | | 2.94 | | | |
| | 6.95% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 10.49 | 0.00 | 27.95 | 38.45 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 1.51 | 0.25 | 0.00 | 1.77 | Total gas oil | | | | |
| | 12.00 | 0.25 | 27.95 | 40.21 | Total gas mass out | | | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 81.44 g/hr | | 74.04 ml/hr | | 53.28% carbon conversion to oil | | | | |
| | | density, | 1.1 g/ml | | 0.04 L/L feed | | | | |
| Oil Loss in Aqueous | 57.53 g/hr | | 57.53 ml/hr | | 29.36% mass conversion to oil | | | | |
| | | density, est | 1 g/ml | | 0.03 L/L feed | | | | |
| Gasification of Carbon | 8.22% | | | | 29.95% carbon conversion to water solubles | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | |
| | | | | | 0.00 L/hr consumption | | | | |
| | | | | | 0.00 L/L feed | | | | |
| | | | | | 0 g/q feed | | | | |
| Space velocity | 2.22 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | | 0 L/L | | 0 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 5.02% | | | | | | | | |
| O in Dry Product | 4.09 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 50.39 g/hr-----> | | | | | | | | |
| O in Dry Feed | 125.92 g/hr | | | | | | | | |
| Deoxygenation | 56.74% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|-----------------------|----------------------|-----------------------------------|----------------|--|-----------------------|-------|-----------|-------------------------|
| Run No. | HTL-4 | CSTR Reactor | 349 | CSTR volume | 1000 | mL | | | |
| Pressure | 20.1 MPa 2911 psig | Tube temp, degrees C | 339 bottom 350 top | reactor volume | 900 | mL, at temp | 1000 | mL, total | |
| Total Feed | 7667 cc | Feed rate | 2000.1 cc/hr | | | | | | |
| ground corn fiber | 7.96 pH | | 2020.10 g/hr | | | | | | |
| | 194.933 COD, ppm | | 7.28 wt% Na2CO3 on dry corn fiber | | | | | | |
| Total Product | 7692.7 g | 3.16 wt% Na | Product oil | 89.2 g/hr | sum of two phases | 2006.8 g/hr | | | |
| | | | Product aqueous | 1917.6 g/hr | 5.89 pH | 0.21% g/hr | 0.89% | 103.68% | |
| actual feed | 47.81% | 5.69% | 47.20% | | | | | | |
| Elemental Analyses | | C | H | O | density | moisture | N | S | ash |
| normalized | feed | 46.00% | 5.60% | 45.40% | 3.01 | 86.27% | 1.90% | 0.21% | 0.89% |
| | product oil | 76.20% | 8.49% | 12.31% | 1.1 | 8.70% | 3.16% | 0.30% | 100.00% |
| oxygen by difference | aqueous | 1.99% | 9.06% | 88.85% | 1.0 est | 97.00% | 0.08% | | 100.46% actual measured |
| Material Balance | | g C/hr | g H/hr | g O/hr | | product moisture est. | | | |
| | feed | 127.59 | 210.89 | 1673.31 | | | Total | TAN | Viscosity |
| | product oil | 67.97 | 7.58 | 10.98 | | | | | |
| | aqueous | 38.21 | 174.12 | 1703.79 | | | | | |
| | gas | 10.49 | 0.00 | 27.95 | | | | | |
| | Total Products | 116.67 | 181.69 | 1742.73 | | 2041.09 | | | |
| | Elemental Balance | 91% | 86% | 104% | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | >>>>>> | | 101% | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | | 0.00 g H/hr | | | | |
| Gas Out | 308.1 L/hr | | | 12.84 moles/hr | | | | | |
| Gas Composition | | volume% | moles/hr | | | | | | |
| | | C | H | O | | | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 gH/hr | | | | |
| CarbDioxide | 6.81% | 0.87 | | 1.75 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | | | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | | | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.14% | 0.13 | 0.25 | 0.00 | | 2.94 | | | |
| | 6.95% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 10.49 | 0.00 | 27.95 | 38.45 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 1.51 | 0.25 | 0.00 | 1.77 | Total gas oil | | | | |
| | 12.00 | 0.25 | 27.95 | 40.21 | Total gas mass out | | | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 81.44 g/hr | | 74.04 ml/hr | | 53.28% carbon conversion to oil | | | | |
| | | density, | 1.1 g/ml | | 0.04 L/L feed | | | | |
| Oil Loss in Aqueous | 57.53 g/hr | | 57.53 ml/hr | | 29.36% mass conversion to oil | | | | |
| | | density, est | 1 g/ml | | 0.03 L/L feed | | | | |
| Gasification of Carbon | 8.22% | | | | 29.95% carbon conversion to water solubles | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | |
| | | | | | 0.00 L/hr consumption | | | | |
| | | | | | 0.00 U/L feed | | | | |
| | | | | | 0 g/g feed | | | | |
| Space velocity | 2.22 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | | 0 L/L | | 0 nm3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 5.02% | | | | | | | | |
| O in Dry Product | 4.09 g/hr | | | | | | | | |
| O in Organics(H2O) | 50.39 g/hr-----> | | | | | | | | |
| O in Dry Feed | 125.92 g/hr | | | | | | | | |
| Deoxygenation | 56.74% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|--|--|----------|-----------------------|-----------------------|--|-----------------------|-----------|------------------------|
| Run No. | HTL-4 | CSTR Reactor | | | CSTR volume | 1000 mL | | | |
| Pressure | 20.1 MPa 2908 psig | Tube temp, degrees C | | | reactor volume | 900 mL, at temp | | | 1000 mL, total |
| Total Feed | 1987 cc 7.96 pH 194.933 COD, ppm | Feed rate | | | Time | 13:55-14:55 | | | |
| ground corn fiber | | 2020.10 g/hr | | | Date | 19-Jun-08 | | | |
| Total Product | 986 g | 7.28 wt% Na ₂ CO ₃ on dry corn fiber | | | Product oil | 93.1 g/hr | sum of two phases | 986 g/hr | |
| | | 3.16 wt% Na | | | Product aqueous | 1878.9 g/hr | 5.66 pH | | |
| Elemental Analyses | actual feed | 47.81% | 5.69% | 47.20% | | | 1.90% | 0.21% | 0.89% 103.68% |
| normalized | feed | 46.00% | 5.60% | 45.40% | 1.0 | 86.27% | 1.90% | 0.21% | 0.89% 100.00% |
| oxygen by difference | product oil | 75.69% | 8.30% | 12.04% | 1.1 | 18.98% | 3.19% | 0.30% | 99.51% actual measured |
| aqueous | 2.03% | 10.40% | 87.50% | 1.0 est | 97.00% | 0.09% | | | 100.02% |
| Material Balance | actual | 8.67% | 89.22% | | product moisture est. | | product moisture est. | | |
| | g C/hr | g H/hr | g O/hr | | Total | | TAN | Viscosity | |
| feed | 127.59 | 210.89 | 1673.31 | | 2011.78 | | | | |
| product oil | 70.47 | 7.73 | 11.20 | | | | | | |
| aqueous | 38.14 | 195.41 | 1644.04 | | | | | | |
| gas | 8.85 | 0.00 | 23.59 | | | | | | |
| Total Products | 117.46 | 203.13 | 1678.83 | | 1999.43 | | | | |
| Elemental Balance | 92% | 96% | 100% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>>>> | | 99% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 moles/hr | | 0.00 g H/hr | | | | | |
| Gas Out | 310 L/hr | 12.92 moles/hr | | | | | | | |
| Gas Composition | volume% | C | moles/hr | H | O | | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 | 0.00 gH/hr | | | |
| CarbDioxide | 5.71% | 0.74 | | 1.47 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | | 0.00 | 0.00 | | | | |
| Methane | 0.00% | 0.00 | | 0.00 | 0.00 | | | | |
| Ethane | 0.00% | 0.00 | | 0.00 | 0.00 | | | | |
| Propane | 0.00% | 0.00 | | 0.00 | 0.00 | | | | |
| Butanes | 0.00% | 0.00 | | 0.00 | 0.00 | | | | |
| Pentanes | 0.00% | 0.00 | | 0.00 | 0.00 | | | | |
| Higher HC (C7H14) | 0.11% | 0.10 | | 0.20 | 0.00 | | 2.94 | | |
| | 5.82% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 8.85 | 0.00 | 23.59 | | 32.44 | Total gas | | | |
| Total Gas Oil, C5-C7 | 1.18 | 0.20 | 0.00 | | 1.38 | Total gas oil | | | |
| | 10.04 | 0.20 | 23.59 | | 33.82 | Total gas mass out | | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 75.43 g/hr | 68.57 ml/hr | | 0.03 L/L feed | | 55.23% carbon conversion to oil | | | |
| | density, 1.1 g/ml | 27.20% mass conversion to oil | | | | | | | |
| Oil Loss in Aqueous | 56.37 g/hr | 56.37 ml/hr | | 0.03 L/L feed | | 29.89% carbon conversion to water solubles | | | |
| Gasification of Carbon | 6.94% | 1 g/ml | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | | |
| | | 0.00 L/hr consumption | | 0.00 L/L feed | | | | | |
| | | 0.00 g/g feed | | | | | | | |
| Space velocity | 2.22 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | | 0 L/L | | 0 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | -5.97% | | | | | | | | |
| O in Dry Product | -4.50 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 24.01 g/hr-----> | | | | | | | | |
| O in Dry Feed | 125.92 g/hr | | | | | | | | |
| Deoxygenation | 84.51% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|------------------------------|------------------------------------|-----------------------|----------------|--|-----------------------|-------------------|-----------|------------------------|
| Run No. | HTL-5 | CSTR Reactor | 330 | CSTR volume | 1000 | mL | | | |
| Pressure | 20.1 MPa 2902 psig | Tube temp, degrees C | 326 bottom 350 top | reactor volume | 900 | mL, at temp | | | 1000 mL, total |
| Total Feed | 5759 cc | Feed rate | 1974.5 cc/hr | | | | | | |
| ground corn fiber | 9.64 pH | | 1994.25 g/hr | | | | | | |
| Total Product | 185,867 COD, ppm 5967.8 g | 13.02 wt% Na2CO3 on dry corn fiber | | Product oil | 85.54 | g/hr | sum of two phases | 2046.1 | |
| | | 5.65 wt% Na | Product aqueous | 1960.56 | g/hr | 6.79 | pH | g/hr | |
| actual feed | 47.81% | 5.69% | 47.20% | | | 1.90% | 0.21% | 0.89% | 103.68% |
| Elemental Analyses | | C | H | O | density | moisture | N | S | ash |
| normalized | feed | 46.00% | 5.60% | 45.40% | 1.01 | 84.94% | 1.90% | 0.21% | 0.89% |
| oxygen by difference | product oil | 75.79% | 8.61% | 11.22% | 1.147 | 3.82% | 3.28% | 0.33% | 99.22% actual measured |
| | aqueous | 2.35% | 9.45% | 88.90% | 1.0 est | 96.00% | 0.07% | 0.05% | 100.02% |
| Material Balance | | g C/hr | g H/hr | g O/hr | | product moisture est. | | | |
| | feed | 138.15 | 206.70 | 1640.38 | | Total | TAN | Viscosity | |
| | product oil | 64.83 | 7.36 | 9.60 | | | | | |
| | aqueous | 45.98 | 169.59 | 1742.94 | | | | | |
| | gas | 4.53 | 0.08 | 11.37 | | | | | |
| | Total Products | 115.33 | 177.03 | 1763.91 | | 2056.27 | | | |
| | Elemental Balance | 83% | 86% | 108% | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>: | >>>>> | | 104% | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 | moles/hr | | 0.00 | g H/hr | | |
| Gas Out | 324.3 L/hr | | 13.51 | moles/hr | | | | | |
| Gas Composition | | moles/hr | | | | | | | |
| | volume% | C | H | O | | | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 | g H/hr | | | |
| CarbDioxide | 2.63% | 0.36 | | 0.71 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.10% | 0.01 | 0.05 | | | | | | |
| Ethane | 0.03% | 0.01 | 0.02 | | | | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.08% | 0.08 | 0.16 | 0.00 | | 2.94 | | | |
| | 2.84% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 4.53 | 0.08 | 11.37 | 15.98 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.94 | 0.16 | 0.00 | 1.10 | Total gas oil | | | | |
| | 5.47 | 0.24 | 11.37 | 17.08 | Total gas mass out | | | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 82.27 g/hr | | 71.73 ml/hr | | 46.93% carbon conversion to oil | | | | |
| | | density, g/ml | 1.147 | | 0.04 L/L feed | | | | |
| Oil Loss in Aqueous | 78.42 g/hr | | 78.42 ml/hr | | 27.39% mass conversion to oil | | | | |
| | | density, est | 1 g/ml | | 0.04 L/L feed | | | | |
| Gasification of Carbon | 3.28% | | | | 33.28% carbon conversion to water solubles | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | |
| | | | | | 0.00 L/hr consumption | | | | |
| | | | | | 0.00 L/L feed | | | | |
| | | | | | 0 g/q feed | | | | |
| Space velocity | 2.19 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | | 0 L/L | | 0 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 8.14% | | | | | | | | |
| O in Dry Product | 6.69 g/hr | | | | | | | | |
| O in Organics(H2O) | 69.93 g/hr-----> | | | | | | | | |
| O in Dry Feed | 136.35 g/hr | | | | | | | | |
| Deoxygenation | 43.81% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | | | | |
|---|---------------------------------------|--------------|-----------------|----------|----------------------|-------------------------------------|--------------------|---------------------------------|-----------------|-----------|-----------------|----------------|
| Run No. | HTL-5 | | | | CSTR Reactor | 334 | | CSTR volume | 1000 mL | | | |
| Pressure | 20.7 MPa 2986 psig | | | | Tube temp, degrees C | 305 bottom 350 top | | reactor volume | 700 mL, at temp | | | 1000 mL, total |
| Total Feed | 5796 cc 9.52 pH 191.13 COD, ppm | | | | Feed rate | 1987.2 cc/hr 2007.07 g/hr | | Time | 02:00-04:55 | | | |
| | | | | | | | | Date | 24-Jun-08 | | | |
| Total Product | 5916.1 g | | | | | | | | | | | |
| | | | | | | | | | | | | |
| Elemental Analyses | | | | | | | | | | | | |
| normalized | feed | C | H | O | density | moisture | | N | S | ash | | |
| | product oil | 46.00% | 5.60% | 45.40% | 1.01 | 84.81% | | 1.90% | 0.21% | 0.89% | 100.00% | |
| | oxygen by difference | 75.17% | 8.68% | 11.45% | 1.023 | 4.77% | | 3.40% | 0.33% | 99.02% | actual measured | |
| | aqueous | 2.38% | 9.45% | 88.80% | 1.0 est | 96.00% | | 0.10% | 0.05% | 99.98% | | |
| Material Balance | | g C/hr | g H/hr | g O/hr | | | | | | | | |
| | feed | 140.24 | 207.89 | 1649.80 | | | | Total | TAN | Viscosity | | |
| | product oil | 68.30 | 7.89 | 10.40 | | | | | | | | |
| | aqueous | 46.11 | 167.59 | 1720.50 | | | | | | | | |
| | gas | 10.69 | 0.12 | 27.43 | | | | | | | | |
| | Total Products | 125.10 | 175.60 | 1758.33 | | | | 2059.03 | | | | |
| Elemental Balance | | 89% | 84% | 107% | | | | | | | | |
| Total Material Balance | | >>>>> | >>>>>>> | >>>>> | >>>>> | | | 103% | | | | |
| GAS CALCULATIONS | | | | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 | moles/hr | | 0.00 | g H/hr | | | | | |
| Gas Out | 314.4 L/hr | | | | | 13.10 | moles/hr | | | | | |
| Gas Composition | | volume% | C | moles/hr | | | | | | | | |
| | Hydrogen | 0.00% | | 0.00 | | | 0.00 gH/hr | | | | | |
| | CarbDioxide | 6.54% | 0.86 | | 1.71 | | | | | | | |
| | CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | | | |
| | Methane | 0.15% | 0.02 | 0.08 | | | | | | | | |
| | Ethane | 0.05% | 0.01 | 0.04 | | | | | | | | |
| | Propane | 0.00% | 0.00 | 0.00 | | | | | | | | |
| | Butanes | 0.00% | 0.00 | 0.00 | | | | | | | | |
| | Pentanes | 0.00% | 0.00 | 0.00 | | | | | | | | |
| | Higher HC (C7H14) | 0.16% | 0.15 | 0.29 | 0.00 | | | 2.94 | | | | |
| | | 6.90% | | | | | | | | | | |
| | | C g/hr | H g/hr | O g/hr | | | | | | | | |
| Total Gas, C1-C4 | 10.69 | 0.12 | 27.43 | | 38.24 | Total gas | | | | | | |
| Total Gas Oil, C5-C7 | 1.77 | 0.30 | 0.00 | | 2.07 | Total gas oil | | | | | | |
| | | 12.46 | 0.42 | 27.43 | | 40.30 | Total gas mass out | | | | | |
| Yields | | | | | | | | | | | | |
| Oil Product Yield | 86.54 g/hr | | 84.59 ml/hr | | 0.04 | L/L feed | | 48.70% carbon conversion to oil | | | | |
| | | density, | 1.023 g/ml | | 28.38% | mass conversion to oil | | | | | | |
| Oil Loss in Aqueous | 77.50 g/hr | | 77.50 ml/hr | | 0.04 | L/L feed | | | | | | |
| | | density, est | 1 g/ml | | 32.88% | carbon conversion to water solubles | | | | | | |
| Gasification of Carbon | 7.62% | | | | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | 0.00 | g/hr consumption | | | | | | |
| | | | | | 0.00 | L/hr consumption | | | | | | |
| | | | | | 0.00 | L/L feed | | | | | | |
| | | | | | 0 | g/g feed | | | | | | |
| Space velocity | 2.84 L/L/hr | LHSV | | | | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | | 0 L/L | | 0 | nm3/tonne | | | | | |
| Calculation of Deoxygenation | | | | | | | | | | | | |
| O content of dry product | 7.57% | | | | | | | | | | | |
| O in Dry Product | 6.55 g/hr | | | | | | | | | | | |
| O in Organics(H2O) | 67.17 g/hr-----> | | | | | | | | | | | |
| O in Dry Feed | 138.41 g/hr | | | | | | | | | | | |
| Deoxygenation | 46.74% | | | | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|----------------------|--------------|--|-------------|-------------------|--|--------------------|----------------|------------------------|
| Run No. | HTL-5 | CSTR Reactor | 335 | CSTR volume | 1000 mL | reactor volume | 750 mL, at temp | 1000 mL, total | |
| Pressure | 20.7 MPa | Tube temp, | 309 bottom | Time | 10:00-12:55 | | | | |
| | 2985 psig | degrees C | 350 top | Date | 24-Jun-08 | | | | |
| Total Feed | 5865 cc | Feed rate | 2010.86 cc/hr | | | | | | |
| ground corn fiber | 9.48 pH | | 2030.97 g/hr | | | | | | |
| | COD, ppm | | 12.3 wt% Na ₂ CO ₃ on dry corn fiber | | | | | | |
| Total Product | 5961 g | 5.34 wt% Na | Product oil | 92.63 g/hr | sum of two phases | 2042.8 g/hr | | | |
| | | | Product aqueous | 1951.2 g/hr | 7.064 pH | | | | |
| Elemental Analyses | actual feed | 47.81% | 5.69% | 47.20% | 1.90% | 0.21% | 0.89% | 103.68% | |
| | normalized | C | H | O | density | moisture | N | S | ash |
| | feed | 46.00% | 5.60% | 45.40% | 1.01 | 83.96% | 1.90% | 0.21% | 0.89% |
| | product oil | 74.59% | 8.54% | 11.33% | 1.067 | 4.07% | 3.28% | 0.32% | 98.06% actual measured |
| | oxygen by difference | aqueous | 2.54% | 0.65% | 88.70% | 1.0 est | 96.00% | 0.06% | 0.05% |
| Material Balance | | q C/hr | q H/hr | q O/hr | | Total | TAN | Viscosity | |
| | feed | 149.85 | 209.39 | 1661.95 | | 2021.20 | | | |
| | product oil | 69.09 | 7.91 | 10.49 | | | | | |
| | aqueous | 49.56 | 168.78 | 1730.71 | | | | | |
| | gas | 9.96 | 0.15 | 25.18 | | | | | |
| | Total Products | 128.61 | 176.84 | 1766.39 | | 2071.84 | | | |
| | Elemental Balance | 86% | 84% | 106% | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | >>>>> | | 103% | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 | moles/hr | | 0.00 g H/hr | | | |
| Gas Out | 315.8 L/hr | | 13.16 | moles/hr | | | | | |
| Gas Composition | | volume% | C | moles/hr | | | | | |
| | Hydrogen | 0.00% | | 0.00 | | 0.00 g H/hr | | | |
| | CarbDioxide | 5.98% | 0.79 | | 1.57 | | | | |
| | CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | |
| | Methane | 0.20% | 0.03 | 0.11 | | | | | |
| | Ethane | 0.06% | 0.02 | 0.05 | | | | | |
| | Propane | 0.00% | 0.00 | 0.00 | | | | | |
| | Butanes | 0.00% | 0.00 | 0.00 | | | | | |
| | Pentanes | 0.00% | 0.00 | 0.00 | | | | | |
| | Higher HC (C7H14) | 0.12% | 0.11 | 0.23 | 0.00 | | 2.94 | | |
| | | 6.36% | | | | | | | |
| | | C g/hr | H g/hr | O g/hr | | | | | |
| Total Gas, C1-C4 | 9.96 | 0.15 | 25.18 | | 35.29 | Total gas | | | |
| Total Gas Oil, C5-C7 | 1.37 | 0.23 | 0.00 | | 1.60 | Total gas oil | | | |
| | | 11.33 | 0.38 | 25.18 | | 36.89 | Total gas mass out | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 88.86 g/hr | | 83.28 ml/hr | | | 46.11% carbon conversion to oil | | | |
| | | density, | 1.067 g/ml | | | 0.04 L/L feed | | | |
| Oil Loss in Aqueous | 78.05 g/hr | | 78.05 ml/hr | | | 27.28% mass conversion to oil | | | |
| | | density, est | 1 g/ml | | | 0.04 L/L feed | | | |
| Gasification of Carbon | 6.64% | | | | | 33.07% carbon conversion to water solubles | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | | 0.00 g/hr consumption | | | |
| | | | | | | 0.00 L/hr consumption | | | |
| | | | | | | 0.00 L/L feed | | | |
| | | | | | | 0 g/q feed | | | |
| Space velocity | 2.68 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | | 0 L/L | | 0 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 8.04% | | | | | | | | |
| O in Dry Product | 7.14 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 65.69 g/hr-----> | | | | | | | | |
| O in Dry Feed | 147.90 g/hr | | | | | | | | |
| Deoxygenation | 50.75% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|-----------------------|------------------------|----------------------------|--|--------------------|-------------------|-------------|-------|------------------------|
| Run No. | HTL-6 | Reactor | 231 | reactor volume | 1000 | total mL | | | |
| Pressure | 20.3 MPa 2936 psig | Temperature, degrees C | 323 351 | | 400 | mL at temperature | | | |
| Total Feed ground corn fiber | 6942 cc | Feed rate | 1983 cc/hr 2002.83 g/hr | | | | | | |
| Total Product | 7106.1 g | | | Product oil | 95.6 g/hr | sum of two phases | 2030.3 g/hr | | |
| | | | | Product aqueous | 1934.7 g/hr | | | | |
| Elemental Analyses | | C | H | O | density | moisture | N | S | ash |
| normalized | feed | 46.00% | 5.60% | 45.40% | 1.01 | 81.72% | 1.90% | 0.21% | 0.89% |
| | product oil | 74.73% | 7.30% | 13.44% | 1.067 | 6.60% | 3.25% | 0.32% | 99.03% actual measured |
| oxygen by difference | aqueous | 2.48% | 10.40% | 86.85% | 1.0 est | 95.00% | 0.28% | | 100.00% |
| Material Balance | | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | | |
| | feed | 168.41 | 203.97 | 1619.46 | 1991.85 | | | | |
| | product oil | 71.44 | 6.97 | 12.85 | | | | | |
| | aqueous | 47.88 | 201.21 | 1680.29 | | | | | |
| | gas | 7.73 | 0.17 | 17.94 | | | | | |
| | Total Products | 127.06 | 208.35 | 1711.07 | 2046.48 | | | | |
| | Elemental Balance | 75% | 102% | 106% | | | | | |
| Total Material Balance | | >>>>> | >>>>>>> | >>>>>> | >>>>>> | 103% | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | 0.00 g H/hr | | | | | |
| Gas Out | 321.1 L/hr | | 13.38 moles/hr | | | | | | |
| Gas Composition | | moles/hr | | | | | | | |
| | volume% | C | H | O | 0.00 qH/hr | trace | | | |
| Hydrogen | 0.00% | | 0.00 | | | | | | |
| CarbDioxide | 4.19% | 0.56 | | 1.12 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.09% | 0.08 | 0.17 | 0.00 | | 2.94 | | | |
| | 4.28% | | | | | | | | |
| | C q/hr | H q/hr | O q/hr | | | | | | |
| Total Gas, C1-C4 | 6.73 | 0.00 | 17.94 | 24.67 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 1.00 | 0.17 | 0.00 | 1.17 | Total gas oil | | | | |
| | 7.73 | 0.17 | 17.94 | 25.84 | Total gas mass out | | | | |
| Yields | | | | 42.42% carbon conversion to oil | | | | | |
| Oil Product Yield | 89.29 g/hr | | 83.68 ml/hr | 0.04 L/L feed | | | | | |
| | | density, | 1.067 g/ml | 24.39% mass conversion to oil | | | | | |
| Oil Loss in Aqueous | 96.74 g/hr | | 96.74 ml/hr | 0.05 L/L feed | | | | | |
| | | density, est | 1 g/ml | 28.43% carbon conversion to water solubles | | | | | |
| Gasification of Carbon | 4.00% | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | | |
| | | | | 0.00 L/hr consumption | | | | | |
| | | | | 0.00 U/L feed | | | | | |
| | | | | 0 g/g feed | | | | | |
| Space velocity | 4.96 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | 0 L/L | 0 nm3/tonne | | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 8.11% | | | | | | | | |
| O in Dry Product | 7.24 g/hr | | | | | | | | |
| O in Organics(H2O) | 46.54 g/hr-----> | | | | | | | | |
| O in Dry Feed | 166.22 g/hr | | | | | | | | |
| Deoxygenation | 67.64% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|------------------------------|-----------------|------------------------------|-----------------------------|--|--------------------|-----------|-------|---------|
| Run No. | HTL-6 | Reactor | 225 | reactor volume | 1000 | total mL | | | |
| Pressure | 20.4 MPa 2941 psig | Temperature | 344 352 | | 450 | mL at temperature | | | |
| | | | | Time | 14:00-17:00 | | | | |
| | | | | Date | 24-Jul-08 | | | | |
| Total Feed | 5809 cc ground corn fiber | Feed rate | 1936.3 cc/hr 1955.66 g/hr | | | | | | |
| Total Product | 5889 g | Product oil | 100.1 g/hr | sum of two phases | 1963 | g/hr | | | |
| | | Product aqueous | 1862.9 g/hr | | | | | | |
| | | | | measured 81.7% based on COD | | | | | |
| Elemental Analyses | | C | H | O | density | N | S | ash | |
| normalized | feed | 46.00% | 5.60% | 45.40% | 1.01 | 85.50% | 1.90% | 0.21% | 0.89% |
| | product oil | 73.81% | 7.70% | 13.20% | 1.067 | 5.53% | 3.05% | 0.30% | 98.06% |
| oxygen by difference | aqueous | 2.43% | 11.41% | 86.10% | 1.0 est | 97.00% | 0.10% | 0.03% | 100.03% |
| Material Balance | | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | | |
| | feed | 130.44 | 203.32 | 1613.40 | 1947.16 | | | | |
| | product oil | 73.89 | 7.71 | 13.21 | | | | | |
| | aqueous | 45.22 | 212.46 | 1603.96 | | | | | |
| | gas | 8.73 | 0.15 | 20.87 | | | | | |
| | Total Products | 127.84 | 220.33 | 1638.03 | 1986.20 | | | | |
| | Elemental Balance | 98% | 108% | 102% | | | | | |
| Total Material Balance | >>>>> | >>>>>>>> | >>>>>>>>> | >>>>>> | 102% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 g H/hr | | | | | |
| Gas Out | 300.7 L/hr | 12.53 | moles/hr | | | | | | |
| Gas Composition | | moles/hr | | | | | | | |
| | volume% | C | H | O | | | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 gH/hr | trace | | | |
| CarbDioxide | 5.20% | 0.65 | | 1.30 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | trace | trace | trace | | |
| Ethane | 0.00% | 0.00 | 0.00 | | trace | trace | trace | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.09% | 0.07 | 0.15 | 0.00 | 2.94 | | | | |
| | 5.29% | | | | | | | | |
| | | C g/hr | H g/hr | O g/hr | | | | | |
| Total Gas, C1-C4 | | 7.83 | 0.00 | 20.87 | 28.70 | Total gas | | | |
| Total Gas Oil, C5-C7 | | 0.90 | 0.15 | 0.00 | 1.05 | Total gas oil | | | |
| | | 8.73 | 0.15 | 20.87 | 29.75 | Total gas mass out | | | |
| Yields | | | | | 56.64% carbon conversion to oil | | | | |
| Oil Product Yield | | 94.57 g/hr | density, ▼ | 88.63 ml/hr | 0.05 L/L feed | | | | |
| | | | | 1.067 g/ml | 33.35% mass conversion to oil | | | | |
| Oil Loss in Aqueous | | 55.89 g/hr | | 55.89 ml/hr | 0.03 L/L feed | | | | |
| | | | density, est | 1 g/ml | 34.67% carbon conversion to water solubles | | | | |
| Gasification of Carbon | 6.00% | 0.00 g/hr in - | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | |
| Hydrogen Consumption | | | | | 0.00 L/hr consumption | | | | |
| | | | | | 0.00 L/L feed | | | | |
| | | | | | 0 g/g feed | | | | |
| Space velocity | 4.30 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | 0 L/L | | 0 nM3/tonne | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 8.77% | | | | | | | | |
| O in Dry Product | 8.29 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | -2.28 g/hr-----> | | | | | | | | |
| O in Dry Feed | 128.74 g/hr | | | | | | | | |
| Deoxygenation | 95.33% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|-----------------------|------------------------|----------------------------|---------------|--|-------------|-------------------|-------------|---------------|
| Run No. | HTL-7 | Reactor | 151 | +/-30 | reactor volume | 1000 | total mL | | |
| Pressure | 20.2 MPa 2912 psig | Temperature, degrees C | 324 339 | +/-20 +/-5 | | 600 | mL at temperature | | |
| Total Feed | 3740 cc | Feed rate | 1496 cc/hr 1510.96 g/hr | | Time | 21:40:00:10 | | | |
| Total Product | 3918.9 g | Product oil | 15.3 g/hr 1552.3 g/hr | | Date | 16-Sep-08 | sum of two phases | 1567.6 g/hr | |
| Elemental Analyses | C | H | O | density | moisture | N | S | ash | |
| adjusted to C balance | feed | 38.00% | 5.60% | 53.40% | 1.01 | 91.80% | 1.90% | 0.21% | 0.89% 100.00% |
| normalized | product oil | 72.70% | 8.40% | 16.00% | 1.072 | 7.75% | 2.55% | 0.28% | 99.93% |
| normalized | aqueous | 2.20% | 11.70% | 85.70% | 1.0 est | 98.00% | 0.43% | 0.005% | 100.03% |
| Material Balance | q C/hr | q H/hr | q O/hr | | Total | TAN | Viscosity | | |
| feed | 47.08 | 162.42 | 1297.74 | | 1507.24 | | | | |
| product oil | 11.12 | 1.29 | 2.45 | | | | | | |
| aqueous | 34.15 | 181.62 | 1330.32 | | | | | | |
| gas | 1.40 | 0.03 | 3.31 | | | | | | |
| Total Products | 46.68 | 182.93 | 1336.08 | | 1565.69 | | | | |
| Elemental Balance | 99% | 113% | 103% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>>>>>>>>>>> | | | 104% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | | 0.00 | q H/hr | | | |
| Gas Out | 230 L/hr | 9.58 | moles/hr | | | | | | |
| Gas Composition | volume% | C | H | O | | | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 | qH/hr | trace | | |
| CarbDioxide, est. | 1.08% | 0.10 | | 0.21 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | trace | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | trace | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.02% | 0.01 | 0.03 | 0.00 | | 2.94 | | | |
| | 1.10% | | | | | | | | |
| | C q/hr | H q/hr | O q/hr | | | | | | |
| Total Gas, C1-C4 | 1.24 | 0.00 | 3.31 | 4.56 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.16 | 0.03 | 0.00 | 0.19 | Total gas oil | | | | |
| | 1.40 | 0.03 | 3.31 | 4.74 | Total gas mass out | | | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 14.11 g/hr | 13.17 ml/hr | | | 23.63% carbon conversion to oil | | | | |
| | | density, 1.072 g/ml | | | 0.01 L/L feed | | | | |
| Oil Loss in Aqueous | 31.05 g/hr | 31.05 ml/hr | | | 11.39% mass conversion to oil | | | | |
| | | density, est 1 g/ml | | | 0.02 L/L feed | | | | |
| Gasification of Carbon | 2.64% | | | | 72.54% carbon conversion to water solubles | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | 0.00 g/hr out = | | | 0.00 g/hr consumption | | | | |
| | | | | | 0.00 L/hr consumption | | | | |
| | | | | | 0.00 L/L feed | | | | |
| | | | | | 0 g/g feed | | | | |
| Space velocity | 2.49 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | 0.00 g/hr | | 0 L/L | | 0 m3/tonne | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 9.88% | | | | | | | | |
| O in Dry Product | 1.39 g/hr | | | | | | | | |
| O in Organics(H2O) | -21.90 g/hr----- | | | | | | | | |
| O in Dry Feed | 66.16 g/hr | | | | | | | | |
| Deoxygenation | 131.00% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|-----------------------|------------------------|----------------------------|-------------------|--|-------------------|-----------|--------|------------------------|
| Run No. | HTL-8 | Reactor | 147 | reactor volume | 1000 | total mL | | | |
| Pressure | 19.8 MPa 2867 psig | Temperature, degrees C | 340 341 | | 600 | mL at temperature | | | |
| Total Feed | 3280 cc | Feed rate | 2008 cc/hr 2028.08 g/hr | | | | | | |
| ground corn fiber with 2% Na2CO3 | | | | | | | | | |
| Total Product | 3385 g | Product oil | 37.3 g/hr | sum of two phases | 2072 | | | | |
| | | Product aqueous | 2035 g/hr | | | | | | |
| Elemental Analyses | | C | H | O | 152.4g/h organics based on COD | | | | |
| normalized | feed | 37.49% | 6.30% | 53.97% | 1.01 | 87.98% | 1.18% | 0.10% | 0.89% |
| | product oil | 73.11% | 7.98% | 14.52% | 1.070 | 3.85% | 2.52% | 0.24% | 98.36% actual measured |
| oxygen by difference | aqueous | 2.39% | 10.94% | 84.50% | 1.0 est | 93.25% | 0.05% | 0.005% | 97.89% |
| Material Balance | | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | | |
| | feed | 91.39 | 215.37 | 1715.85 | | 2022.62 | | | |
| | product oil | 27.27 | 2.98 | 5.41 | | | 51.95 | | |
| | aqueous | 48.59 | 222.68 | 1719.58 | | | | | |
| | gas | 3.79 | 0.00 | 10.11 | | | | | |
| | Total Products | 79.65 | 225.66 | 1735.10 | | 2040.40 | | | |
| | Elemental Balance | 87% | 105% | 101% | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>> | >>>>> | | 101% | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 | g H/hr | | | | |
| Gas Out | 307.3 L/hr | 12.80 | moles/hr | | | | | | |
| Gas Composition | | moles/hr | | | | | | | |
| | volume% | C | H | O | 0.00 gH/hr | trace | | | |
| Hydrogen | 0.00% | | 0.00 | | | | | | |
| CarbDioxide | 2.47% | 0.32 | | 0.63 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | trace | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | trace | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | |
| | 2.47% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 3.79 | 0.00 | 10.11 | 13.90 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 3.79 | 0.00 | 10.11 | 13.90 | Total gas mass out | | | | |
| Yields | | | | | 29.84% carbon conversion to oil | | | | |
| Oil Product Yield | 35.86 g/hr | 33.52 ml/hr | | | 0.02 L/L feed | | | | |
| | | density, 1.07 g/ml | | | 14.71% mass conversion to oil | | | | |
| Oil Loss in Aqueous | 137.36 g/hr | 137.36 ml/hr | | | 0.07 L/L feed | | | | |
| | | density, est 1 g/ml | | | 53.16% carbon conversion to water solubles | | | | |
| Gasification of Carbon | 4.15% | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | 0.00 g/hr out = | | | 0.00 g/hr consumption | | | | |
| | | | | | 0.00 L/hr consumption | | | | |
| | | | | | 0.00 L/L feed | | | | |
| | | | | | 0 g/g feed | | | | |
| Space velocity | 3.35 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | | 0 L/L | | 0 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 11.54% | | | | | | | | |
| O in Dry Product | 4.14 g/hr | | | | | | | | |
| O in Organics(H2O) | 32.79 g/hr-----> | | | | | | | | |
| O in Dry Feed | 131.57 g/hr | | | | | | | | |
| Deoxygenation | 71.94% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | |
|--|-----------------------|--------------------------|----------------------------|-------------------|--|-------------------|-------------------------------------|
| Run No. | HTL-8 | Reactor | 166 | reactor volume | 1000 | total mL | |
| Pressure | 19.7 MPa 2852 psig | Temperature degrees C | 340 341 | | 600 | mL at temperature | |
| Total Feed ground corn fiber with 2% Na ₂ CO ₃ | 3750 cc | Feed rate | 1500 cc/hr 1515.00 q/hr | Time | 16:50-19:20 | | |
| Date | 14-Oct-08 | | | | | | |
| Total Product | 3797.8 g | Product oil | 39.4 g/hr | sum of two phases | 1519 | | |
| | | Product aqueous | 1479.6 g/hr | | | | |
| Elemental Analyses | | C | H | O | density | moisture | |
| normalized | feed | 37.49% | 6.30% | 53.97% | 1.01 | 87.06% | |
| | product oil | 73.20% | 8.43% | 14.53% | 1.072 | 3.95% | 1.18% 0.10% 0.89% 99.93% moisture |
| oxygen by difference | aqueous | 2.14% | 11.18% | 86.63% | 1.0 est | 92.25% | 2.57% 0.244% 98.97% actual measured |
| 0.05% 0.005% 100.00% | | | | | | | |
| Material Balance | | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity |
| feed | 73.50 | 160.20 | 1276.91 | | 1510.61 | | |
| product oil | 28.84 | 3.32 | 5.72 | | | 52.55 | |
| aqueous | 31.66 | 165.38 | 1281.78 | | | | |
| gas | 2.84 | 0.00 | 7.58 | | | | |
| Total Products | 63.35 | 168.70 | 1295.08 | | 1527.13 | | |
| Elemental Balance | 86% | 105% | 101% | | | | |
| Total Material Balance | >>>>> | >>>>>>>> | >>>>>> | >>>>> | 101% | | |
| GAS CALCULATIONS | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | | 0.00 q H/hr | | |
| Gas Out | 230.4 L/hr | | 9.60 moles/hr | | | | |
| Gas Composition | | moles/hr | | | | | |
| volume% | C | H | O | | | | |
| Hydrogen | 0.00% | 0.00 | | 0.00 qH/hr | trace | | |
| CarbDioxide, est. | 2.47% | 0.24 | 0.47 | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | trace | | |
| Ethane | 0.00% | 0.00 | 0.00 | trace | trace | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | | 2.94 | | |
| no good GC | 2.47% | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | |
| Total Gas, C1-C4 | 2.84 | 0.00 | 7.58 | 10.42 | Total gas | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | |
| | 2.84 | 0.00 | 7.58 | 10.42 | Total gas mass out | | |
| Yields | | | | | 39.24% carbon conversion to oil | | |
| Oil Product Yield | 37.84 g/hr | | 35.30 ml/hr | | 0.02 L/L feed | | |
| | | density, est. | 1.072 g/ml | | 19.30% mass conversion to oil | | |
| Oil Loss in Aqueous | 114.67 g/hr | | 114.67 ml/hr | | 0.08 L/L feed | | |
| | | density, est | 1 g/ml | | 43.08% carbon conversion to water solubles | | |
| Gasification of Carbon | 3.87% | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out - | | 0.00 g/hr consumption | | |
| | | | | | 0.00 L/hr consumption | | |
| | | | | | 0.00 L/L feed | | |
| | | | | | 0 g/g feed | | |
| Space velocity | 2.50 L/L/hr | LHSV | | | | | |
| Chemical Hydrogen Consumption | 0.00 g/hr | | 0 L/L | | 0 nm3/tonne | | |
| Calculation of Deoxygenation | | | | | | | |
| O content of dry product | 11.47% | | | | | | |
| O in Dry Product | 4.34 g/hr | | | | | | |
| O in Organics(H ₂ O) | 68.51 g/hr-----> | | | | | | |
| O in Dry Feed | 105.80 g/hr | | | | | | |
| Deoxygenation | 31.15% | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|-------------------------------|-------------------------------------|--|-------------------|--|----------|-----------|--------|-------------------------|
| Run No. | HTL-9 | reactor v'lume | 1000 total mL 600 mL at temperature | | | | | | |
| Pressure | 20.2 MPa 2917 psig | Reactor Temperature degrees C | 146 333 325 | Time | 13:00-15:30 Date 16-Oct-08 | | | | |
| Total Feed | 5000 cc | Feed rate | 2000 cc/hr 2020.00 g/hr | | | | | | |
| ground corn fiber with 2% Na ₂ CO ₃ | | | | | | | | | |
| Total Product | 5061.5 g | Product oil | 49.8 g/hr | sum of two phases | 2024.6 g/hr | | | | |
| | | Product aqueous | 1974.8 g/hr | | | | | | |
| Elemental Analyses | | C | H | O | density@25C | moisture | N | S | ash |
| normalized | feed | 46.60% | 5.60% | 45.40% | 1.01 | 87.06% | 1.90% | 0.21% | 0.89% |
| | product oil | 71.29% | 8.11% | 15.35% | 1.072 | 3.80% | 2.28% | 0.24% | 97.27% |
| oxygen by difference | aqueous | 2.58% | 10.91% | 86.45% | 1.0 est | 93.25% | 0.05% | 0.005% | 100.00% actual measured |
| Material Balance | | q C/hr | q H/hr | q O/hr | Total | TAN | Viscosity | | |
| | feed | 120.24 | 211.77 | 1680.15 | 2012.16 | | | | |
| | product oil | 35.50 | 4.04 | 7.65 | | 51.55 | | | |
| | aqueous | 50.95 | 215.45 | 1707.21 | | | | | |
| | gas | 6.84 | 0.03 | 18.23 | | | | | |
| | Total Products | 93.30 | 219.51 | 1733.09 | 2045.90 | | | | |
| | Elemental Balance | 78% | 104% | 103% | | | | | |
| | Total Material Balance | >>>>> | >>>>>>> | >>>>>> | 102% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 moles/hr | 0.00 q H/hr | | | | | | |
| Gas Out | 303.3 L/hr | 12.63 moles/hr | | | | | | | |
| Gas Composition | | moles/hr | | | | | | | |
| | volume% | C | H | O | 0.03 qH/hr | trace | | | |
| Hydrogen | 0.10% | 0.03 | | | | | | | |
| CarbDioxide | 4.51% | 0.57 | | 1.14 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | trace | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | trace | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | |
| | 4.61% | | | | | | | | |
| | C q/hr | H q/hr | O q/hr | | | | | | |
| Total Gas, C1-C4 | 6.84 | 0.00 | 18.23 | 25.08 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 6.84 | 0.00 | 18.23 | 25.08 | Total gas mass out | | | | |
| Yields | | | | | 29.53% carbon conversion to oil | | | | |
| Oil Product Yield | 47.91 q/hr | density, est | 44.69 ml/hr 1.072 g/ml | 0.02 L/L feed | | | | | |
| Oil Loss in Aqueous | 133.30 q/hr | | 133.30 ml/hr | 0.07 L/L feed | 18.33% mass conversion to oil | | | | |
| Gasification of Carbon | 5.69% | | 1 g/ml | | 42.37% carbon conversion to water solubles | | | | |
| Hydrogen Consumption | 0.00 q/hr in - | | 0.03 q/hr out = | | -0.03 q/hr consumption | | | | |
| | | | | | -0.30 L/hr consumption | | | | |
| | | | | | -0.15 L/L feed | | | | |
| | | | | | -1.26333E-05 q/g feed | | | | |
| Space velocity | 3.33 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | -0.02 q/hr | 0 L/L | 0 nM3/tonne | | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 12.45% | | | | | | | | |
| O in Dry Product | 5.96 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 70.32 g/hr-----> | | | | | | | | |
| O in Dry Feed | 118.67 g/hr | | | | | | | | |
| Deoxygenation | 35.71% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | | |
|--|-----------------------|-------------------------------------|----------------------------|-------------------|--|-------------|-----------|--------|-------|--------|
| Run No. | HTL-9 | reactor volume | 1000 | total mL | | | | | | |
| Pressure | 20.2 MPa 2912 psig | Reactor Temperature degrees C | 165 337 325 | 600 | mL at temperature | | | | | |
| Total Feed ground corn fiber with 2% Na ₂ CO ₃ | 3770 cc | Feed rate | 1508 cc/hr 1523.08 g/hr | Time Date | 17:00-19:30 16-Oct-08 | | | | | |
| Total Product | 3832 g | Product oil | 44.6 g/hr | sum of two phases | 1532.8 g/hr | | | | | |
| Product aqueous | | Product aqueous | 1488.2 g/hr | | | | | | | |
| Elemental Analyses | | C | H | O | density@25C | moisture | N | S | ash | |
| normalized | feed | 34.43% | 5.79% | 49.57% | 1.01 | 85.96% | 1.08% | 0.09% | 0.89% | 91.84% |
| | product oil | 71.41% | 8.04% | 16.70% | 1.072 | 4.10% | 2.51% | 0.23% | | 98.89% |
| oxygen by difference | aqueous | 2.29% | 11.34% | 86.30% | 1.0 est | 92.75% | 0.05% | 0.005% | | 99.98% |
| Material Balance | | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | | | |
| | feed | 73.63 | 159.13 | 1268.47 | 1501.23 | | | | | |
| | product oil | 31.85 | 3.59 | 7.45 | | 48.35 | | | | |
| | aqueous | 34.12 | 168.69 | 1284.32 | | | | | | |
| | gas | 6.81 | 0.06 | 18.15 | | | | | | |
| | Total Products | 72.78 | 172.33 | 1309.92 | 1555.02 | | | | | |
| | Elemental Balance | 99% | 108% | 103% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | >>>>> | 104% | | | | | |
| GAS CALCULATIONS | | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 | moles/hr | 0.00 | g H/hr | | | | |
| Gas Out | 228.8 L/hr | | 9.53 | moles/hr | | | | | | |
| Gas Composition | | moles/hr | | | | | | | | |
| | volume% | C | H | O | 0.06 | gH/hr | trace | | | |
| Hydrogen | 0.29% | | 0.06 | | | | | | | |
| CarbDioxide | 5.95% | 0.57 | | 1.13 | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | trace | | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | trace | | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | | |
| | 6.24% | | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | | |
| Total Gas, C1-C4 | 6.81 | 0.00 | 18.15 | 24.96 | Total gas | | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | | |
| | 6.81 | 0.00 | 18.15 | 24.96 | Total gas mass out | | | | | |
| Yields | | | | | 43.26% carbon conversion to oil | | | | | |
| Oil Product Yield | 42.77 g/hr | | 39.92 ml/hr | | 0.03 L/L feed | | | | | |
| | | density, | 1.0715 g/ml | | 20.00% mass conversion to oil | | | | | |
| Oil Loss in Aqueous | 107.89 g/hr | | 107.89 ml/hr | | 0.07 L/L feed | | | | | |
| | | density, est | 1 g/ml | | 46.34% carbon conversion to water solubles | | | | | |
| Gasification of Carbon | 9.25% | | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.06 g/hr out = | | -0.06 g/hr consumption | | | | | |
| | | | | | -0.66 L/hr consumption | | | | | |
| | | | | | -0.44 L/L feed | | | | | |
| | | | | | -3.66667E-05 g/g feed | | | | | |
| Space velocity | 2.51 L/L/hr | LHSV | | | | | | | | |
| | | | | | | | | | | |
| Chemical Hydrogen Consumption | | -0.05 g/hr | | 0 L/L | | 0 nM3/tonne | | | | |
| | | | | | | | | | | |
| Calculation of Deoxygenation | | | | | | | | | | |
| O content of dry product | 13.61% | | | | | | | | | |
| O in Dry Product | 5.82 g/hr | | | | | | | | | |
| O in Organics(H ₂ O) | 57.38 g/hr----- | | | | | | | | | |
| O in Dry Feed | 105.99 g/hr | | | | | | | | | |
| Deoxygenation | 40.37% | | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|-------------------------------|------------------------|----------------------------|-----------------|--|--------------------------|-------------------|---------|------------------------|
| Run No. | HTL-10 | Reactor | 162 | reactor volume | 1000 | total mL | | | |
| Pressure | 19.9 MPa 2875 psig | Temperature, degrees C | 329 345 | | 500 | mL at temperature | | | |
| | | | | Time | 12:15-14:55 | | | | |
| | | | | Date | 31-Dec-08 | | | | |
| Total Feed | 3470 cc ground corn stover | Feed rate | 1301 cc/hr 1314.01 g/hr | | | | | | |
| Total Product | 3438 g | | | Product oil | 27 g/hr | | sum of two phases | 1289.3 | |
| | | | | Product aqueous | 1262.3 g/hr | | | | q/hr |
| Elemental Analyses | C | H | O | density@25C | moisture | N | S | ash | |
| feed | 42.25% | 5.28% | 41.40% | 1.01 | 94.74% | 0.66% | 0.06% | 11.94% | 101.58% moisture |
| product oil | 66.32% | 6.40% | 25.51% | 1.100 | 26.05% | 1.28% | 0.08% | 0.0% | 99.58% actual measured |
| oxygen by difference | aqueous | 1.03% | 9.73% | 89.20% | 1.0 est | 99.00% | <0.05% | <0.005% | 99.95% |
| Material Balance | g C/hr | g H/hr | g O/hr | | Total | TAN | Viscosity | | |
| feed | 29.20 | 143.20 | 1133.95 | | 1306.35 | | | | |
| product oil | 17.91 | 1.73 | 6.89 | | | 36.3 | solid | | |
| aqueous | 12.94 | 122.76 | 1125.97 | | | | | | |
| gas | 3.65 | 0.03 | 9.44 | | | | | | |
| Total Products | 34.49 | 124.52 | 1142.30 | | 1301.31 | | | | |
| Elemental Balance | 118% | 87% | 101% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>>>>>>>>>>> | | | 100% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 | moles/hr | 0.00 | g H/hr | | | |
| Gas Out | 210.8 L/hr | | 8.78 | moles/hr | | | | | |
| Gas Composition | volume% | C | moles/hr | | | | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 | gH/hr | trace | | |
| CarbDioxide | 3.36% | 0.30 | | 0.59 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.06% | 0.01 | 0.02 | | trace | | trace | | |
| Ethane | 0.01% | 0.00 | 0.01 | | trace | | trace | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | | 2.94 | | | | |
| | 3.44% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 3.64 | 0.03 | 9.44 | 13.12 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 3.65 | 0.03 | 9.44 | 13.12 | Total gas mass out | | | | |
| Yields | | | | | 61.32% carbon conversion to oil | | | | |
| Oil Product Yield | 19.97 g/hr | | 18.15 ml/hr | | 0.01 L/L feed | | | | |
| | | density, | 1.1 g/ml | | 28.89% mass conversion to oil | 13% mass yield of solids | | | |
| Oil Loss in Aqueous | 12.62 g/hr | | 12.62 ml/hr | | 0.01 L/L feed | plugged reactor tube | | | |
| | | density, est | 1 g/ml | | 44.31% carbon conversion to water solubles | | | | |
| Gasification of Carbon | 12.48% | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | 0.00 g/hr consumption | | | | |
| | | | | | 0.00 L/hr consumption | | | | |
| | | | | | 0.00 L/L feed | | | | |
| | | | | | 0 g/g feed | | | | |
| Space velocity | 2.60 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | | 0.00 g/hr | | 0 L/L | | 0 nm3/tonne | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 3.18% ??? | | | | | | | | |
| O in Dry Product | 0.63 g/hr | | | | | | | | |
| O in Organics(H2O) | 15.15 g/hr-----> | | | | | | | | |
| O in Dry Feed | 28.61 g/hr | | | | | | | | |
| Deoxygenation | 44.84% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | |
|---|-----------------------|------------------------|----------------------------|-------------------|--|--------------------------|----------------|
| Run No. | HTL-10 | Reactor | 152 | reactor volume | 1000 | total mL | |
| Pressure | 19.9 MPa 2878 psig | Temperature, degrees C | 326 341 | Time | 16:10-16:40 | 500 mL at temperature | Date 31-Dec-08 |
| Total Feed | 750 cc | Feed rate | 1500 cc/hr 1515.00 g/hr | | | | |
| Total Product | 743 g | Product oil | 63 g/hr | sum of two phases | 1494 | | |
| | | Product aqueous | 1431 g/hr | | | | g/hr |
| Elemental Analyses | | C | H | O | density@25C | moisture | |
| feed | 42.25% | 5.28% | 41.40% | 1.01 | 94.74% | 0.66% | 0.06% |
| product oil | 60.46% | 5.95% | 27.89% | 1.090 | 19.05% | 1.11% | 0.08% |
| oxygen by difference | 0.84% | 10.07% | 89.10% | 1.0 est | 99.00% | <0.05% | -0.005% |
| Material Balance | | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity |
| feed | 33.67 | 165.10 | 1307.40 | | 1506.17 | | |
| product oil | 38.09 | 3.75 | 17.57 | | | 35.1 | solid |
| aqueous | 12.02 | 144.10 | 1275.02 | | | | |
| gas | 3.91 | 0.02 | 10.20 | | | | |
| Total Products | 54.02 | 147.87 | 1302.78 | | 1504.67 | | |
| Elemental Balance | 160% | 90% | 100% | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | >>>>> | 100% | | |
| GAS CALCULATIONS | | | | | | | |
| Gas In | 0 L/hr | | 0.00 | moles/hr | 0.00 | g H/hr | |
| Gas Out | 242 L/hr | | 10.08 | moles/hr | | | |
| Gas Composition | | moles/hr | | | | | |
| | volume% | C | H | O | | | |
| Hydrogen | 0.00% | | 0.00 | | 0.00 | gH/hr | trace |
| CarbDioxide | 3.16% | 0.32 | | 0.64 | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | |
| Methane | 0.03% | 0.00 | 0.01 | | trace | trace | |
| Ethane | 0.01% | 0.00 | 0.01 | | trace | trace | |
| Propane | 0.00% | 0.00 | 0.00 | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | |
| | 3.20% | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | |
| Total Gas, C1-C4 | 3.89 | 0.02 | 10.20 | 14.11 | Total gas | | |
| Total Gas Oil, C5-C7 | 0.02 | 0.00 | 0.00 | 0.02 | Total gas oil | | |
| | 3.91 | 0.02 | 10.20 | 14.13 | Total gas mass out | | |
| Yields | | | | | 113.13% carbon conversion to oil | | |
| Oil Product Yield | 51.00 g/hr | | 46.79 ml/hr | | 0.03 L/L feed | | |
| | | density, g/ml | 1.09 g/ml | | 64.00% mass conversion to oil | 13% mass yield of solids | |
| Oil Loss in Aqueous | 14.31 g/hr | | 14.31 ml/hr | | 0.01 L/L feed | plugged reactor tube | |
| | | density, est | 1 g/ml | | 35.70% carbon conversion to water solubles | | |
| Gasification of Carbon | 11.56% | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.00 g/hr out = | | 0.00 g/hr consumption | | |
| | | | | | 0.00 L/hr consumption | | |
| | | | | | 0.00 L/L feed | | |
| | | | | | 0 g/g feed | | |
| Space velocity | 3.00 L/L/hr | LHSV | | | | | |
| Chemical Hydrogen Consumption | | 0.00 g/hr | | 0 L/L | 0 nM3/tonne | | |
| Calculation of Deoxygenation | | | | | | | |
| O content of dry product | 13.53% | | | | | | |
| O in Dry Product | 6.90 g/hr | | | | | | |
| O in Organics(H ₂ O) | 15.74 g/hr-----> | | | | | | |
| O in Dry Feed | 32.99 g/hr | | | | | | |
| Deoxygenation | 31.37% | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|-----------------------|--------------------------|----------------------------|--|------------------------------------|-------------------|---------|--------|------------------------|
| Run No. | HTL-11 | Reactor | 162 | reactor volume | 1000 | total mL | | | |
| Pressure | 20.0 MPa 2884 psig | Temperature degrees C | 346 349 | | 500 | mL at temperature | | | |
| Total Feed | 2350 cc | Feed rate | 1175 cc/hr 1186.75 q/hr | | Time | 12:35-14:35 | | | |
| ground corn stover slurry w/2% sodium carbonate | | | | | Date | 7-Jan-09 | | | |
| Total Product | 2227.5 q | Product oil | 18 q/hr | sum of two phases | 1113.75 | | | | |
| | | Product aqueous | 1095.75 q/hr | | | | | | |
| Elemental Analyses | C | H | O | density@25C | moisture | N | S | ash | |
| feed | 34.02% | 3.85% | 42.59% | 1.01 | 90.40% | 0.72% | 0.03% | 18.97% | 100.16% moisture |
| product oil | 65.92% | 6.83% | 16.12% | 1.135 | 7.39% | 1.04% | 0.06% | 0.5% | 90.46% actual measured |
| oxygen by difference | 1.74% | 10.12% | 88.10% | 1.0 est | 98.00% | <0.05% | <0.005% | | 99.96% |
| Material Balance | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | | | |
| feed | 38.75 | 124.64 | 1001.08 | 1164.48 | | | | | |
| product oil | 11.86 | 1.23 | 2.90 | | 29.7 | tar | | | |
| aqueous | 19.07 | 110.84 | 965.36 | | | | | | |
| gas | 3.27 | 0.07 | 8.68 | | | | | | |
| Total Products | 34.20 | 112.13 | 976.94 | 1123.27 | | | | | |
| Elemental Balance | 88% | 90% | 98% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | >>>>> | 96% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | 0.00 q H/hr | | | | | |
| Gas Out | 249.5 L/hr | | 10.40 moles/hr | | | | | | |
| Gas Composition | | | moles/hr | | | | | | |
| volume% | C | H | O | | | | | | |
| Hydrogen | 0.31% | 0.07 | | 0.07 qH/hr | trace | | | | |
| CarbDioxide | 2.61% | 0.27 | 0.54 | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | | 2.94 | | | | |
| | 2.93% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 3.27 | 0.00 | 8.68 | 11.95 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 3.27 | 0.00 | 8.68 | 11.96 | Total gas mass out | | | | |
| Yields | | | | 30.62% carbon conversion to oil | | | | | |
| Oil Product Yield | 16.67 g/hr | | 14.69 ml/hr | 0.01 L/L feed | | | | | |
| | | density, | 1.135 q/ml | 14.63% mass conversion to oil | 5% mass yield of solids recovered | | | | |
| Oil Loss in Aqueous | 21.92 g/hr | | 21.92 ml/hr | 0.02 L/L feed | wall deposits but no pressure drop | | | | |
| | | density, est | 1 q/ml | 49.20% carbon conversion to water solubles | | | | | |
| Gasification of Carbon | 8.44% | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.07 g/hr out - | -0.07 g/hr consumption | | | | | |
| | | | | -0.78 L/hr consumption | | | | | |
| | | | | -0.66 L/L feed | | | | | |
| | | | | -5.53855E-05 g/q feed | | | | | |
| Space velocity | 2.35 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | -0.07 g/hr | | -1 L/L | | -1 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 10.31% | | | | | | | | |
| O in Dry Product | 1.72 g/hr | | | | | | | | |
| O in Organics(H2O) | 10.84 g/hr-----> | | | | | | | | |
| O in Dry Feed | 48.52 g/hr | | | | | | | | |
| Deoxygenation | 74.13% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|-----------------------|-----------------|----------------------------|--|------------------------------------|-------------------|-----------|---------|--------|
| Run No. | HTL-11 | Reactor | 161 | reactor volume | 1000 | total mL | | | |
| Pressure | 20.0 MPa 2890 psiq | Temperature | 344 degrees C | | 600 | mL at temperature | | | |
| | | | | Time | 14:35:15:50 | | | | |
| | | | | Date | 7-Jan-09 | | | | |
| Total Feed | 1640 cc | Feed rate | 1312 cc/hr 1325.12 g/hr | | | | | | |
| ground corn stover slurry w/2% sodium carbonate | | | | | | | | | |
| Total Product | 2227.5 g | Product oil | 23.2 g/hr | sum of two phases | 1220.4 | | | | |
| | | Product aqueous | 1197.2 g/hr | | g/hr | | | | |
| Elemental Analyses | | C | H | O | density@25C | moisture | N | S | ash |
| feed | 34.02% | 3.85% | 42.59% | 1.01 | 90.40% | | 0.72% | 0.03% | 18.97% |
| product oil | 72.47% | 7.89% | 15.98% | 1.00 | 9.59% | | 1.18% | 0.07% | 97.58% |
| oxygen by difference | 1.86% | 10.01% | 88.10% | 1.0 est | 98.00% | | <0.05% | <0.005% | 99.97% |
| Material Balance | g C/hr | g H/hr | g O/hr | | Total | TAN | Viscosity | | |
| feed | 43.27 | 139.17 | 1117.81 | | 1300.25 | | | | |
| product oil | 16.81 | 1.83 | 3.71 | | | 31.6 | tar | | |
| aqueous | 22.27 | 119.84 | 1054.73 | | | | | | |
| gas | 1.67 | 0.03 | 4.42 | | | | | | |
| Total Products | 40.75 | 121.70 | 1062.86 | | 1225.30 | | | | |
| Elemental Balance | 94% | 87% | 95% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>> | >>>>> | 94% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 | g H/hr | | | | |
| Gas Out | 189 L/hr | | | 7.50 | moles/hr | | | | |
| Gas Composition | volume% | C | H | O | | | | | |
| Hydrogen | 0.15% | 0.02 | | 0.02 | gH/hr | trace | | | |
| CarbDioxide | 1.84% | 0.14 | | 0.28 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | |
| | 2.00% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 1.67 | 0.00 | 4.42 | 6.09 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 1.67 | 0.00 | 4.42 | 6.09 | Total gas mass out | | | | |
| Yields | | | | | 38.85% carbon conversion to oil | | | | |
| Oil Product Yield | 20.98 g/hr | density, | 19.07 ml/hr | 0.01 L/L feed | | | | | |
| | | | 1.1 g/ml | 16.49% mass conversion to oil | 5% mass yield of solids recovered | | | | |
| Oil Loss in Aqueous | 23.94 g/hr | | 23.94 ml/hr | 0.02 L/L feed | wall deposits but no pressure drop | | | | |
| | | density, est | 1 g/ml | 51.46% carbon conversion to water solubles | | | | | |
| Gasification of Carbon | 3.85% | | | | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.02 g/hr out = | -0.02 g/hr consumption | | | | | |
| | | | | -0.28 L/hr consumption | | | | | |
| | | | | -0.21 L/L feed | | | | | |
| | | | | -1.76067E-05 g/g feed | | | | | |
| Space velocity | 2.19 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | -0.03 g/hr | | 0 L/L | 0 nM3/tonne | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 8.25% | | | | | | | | |
| O in Dry Product | 1.73 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 11.84 g/hr-----> | | | | | | | | |
| O in Dry Feed | 54.18 g/hr | | | | | | | | |
| Deoxygenation | 74.95% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|-----------------------|--------------|----------------------------|-----------------|--|-------------------|--------------|---------|------------------------|
| Run No. | HTL-13 | Reactor | 232 | reactor volume | 1000 | total mL | | | |
| Pressure | 20.7 MPa 2988 psig | Temperature | 353 degrees C 348 | | 600 | mL at temperature | | | |
| Total Feed | 1740 cc | Feed rate | 1305 cc/hr 1318.05 g/hr | | | | | | |
| corn starch slurry feed 1 w/2% sodium carbonate | | | | | | | | | |
| Total Product | 1761.5 g | | | Product oil | 13.9 g/hr | sum of two phases | 1321.16 g/hr | | |
| | | | | Product aqueous | 1307.3 g/hr | | | | |
| Elemental Analyses | C | H | O | density@25C | moisture | N | S | ash | |
| feed | 33.15% | 4.37% | 50.33% | 1.01 | 91.00% | 0.08% | <0.005 | 21.31% | 109.23% moisture |
| product oil | 69.35% | 7.74% | 16.25% | 1.084 | 4.45% | 0.07% | 0.02% | 1.0% | 94.43% actual measured |
| oxygen by difference | 1.98% | 9.83% | 88.20% | 1.0 est | 96.00% | <0.05% | <0.005% | 100.01% | |
| Material Balance | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | COD | | |
| feed | 39.32 | 139.63 | 1124.67 | 1303.63 | | 27.6 | 5070 ??? | | |
| product oil | 9.64 | 1.08 | 2.26 | | | NA | | | |
| aqueous | 25.82 | 128.51 | 1153.04 | | pH 7.29-7.41 | | 56670 | | |
| gas | 2.14 | 0.13 | 5.69 | | | | | | |
| Total Products | 37.60 | 129.71 | 1160.98 | 1328.30 | | | | | |
| Elemental Balance | 96% | 93% | 103% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | 102% | | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | 0.00 g H/hr | | | | | |
| Gas Out | 204 L/hr | | 8.58 moles/hr | | | | | | |
| Gas Composition | volume% | moles/hr | | | | | | | |
| Hydrogen | 0.74% | C | H | O | 0.13 gH/hr | trace | | | |
| CarbDioxide | 2.07% | | 0.18 | | 0.36 | | | | |
| CarbMonoxide | 0.00% | | 0.00 | | 0.00 | | | | |
| Methane | 0.00% | | 0.00 | | trace | trace | | | |
| Ethane | 0.00% | | 0.00 | | trace | trace | | | |
| Propane | 0.00% | | 0.00 | | | | | | |
| Butanes | 0.00% | | 0.00 | | | | | | |
| Pentanes | 0.00% | | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | | 0.00 | | 0.00 | 2.94 | | | |
| | 2.81% | | | | | | | | |
| Total Gas, C1-C4 | | C g/hr | H g/hr | O g/hr | 7.83 Total gas | | | | |
| Total Gas Oil, C5-C7 | | 2.14 | 0.00 | 5.69 | 0.00 Total gas oil | | | | |
| | | | | | 7.83 Total gas mass out | | | | |
| Yields | | | | | 24.51% carbon conversion to oil | | | | |
| Oil Product Yield | 13.28 g/hr | | 12.25 ml/hr | | 0.01 L/L feed | | | | |
| | | density, | 1.084 g/ml | | 11.20% mass conversion to oil | | | | |
| Oil Loss in Aqueous | 52.29 g/hr | | 52.29 ml/hr | | 0.04 L/L feed | | | | |
| | | density, est | 1 g/ml | | 65.66% carbon conversion to water solubles | | | | |
| Gasification of Carbon | 5.45% | | | | 5.45% carbon conversion to gas | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.13 g/hr out = | | -0.13 g/hr consumption | | | | |
| | | | | | -1.52 L/hr consumption | | | | |
| | | | | | -1.17 L/L feed | | | | |
| | | | | | -9.73436E-05 g/g feed | | | | |
| Space velocity | 2.18 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | -0.14 g/hr | | -1 L/L | | -1 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 12.87% | | | | | | | | |
| O in Dry Product | 1.71 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 37.48 g/hr----- | | | | | | | | |
| O in Dry Feed | 59.70 g/hr | | | | | | | | |
| Deoxygenation | 34.36% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | |
|---|-----------------------|------------------------|----------------------------|----------------------|---|
| Run No. | HTL-14 | Reactor | 217 | reactor volume | 1000 total mL 500 mL at temperature |
| Pressure | 20.0 MPa 2887 psig | Temperature, degrees C | 345 344 | Time | 13:20-16:50 |
| | | | | Date | 26-Jan-09 |
| Total Feed | 5280 cc | Feed rate | 1509 cc/hr 1524.09 g/hr | | |
| corn starch slurry w/2% sodium carbonate | | | | | |
| Total Product | 1761.5 g | Product oil | 6.9 g/hr | sum of two phases | 1480.3 g/hr |
| | | Product aqueous | 1473.4 g/hr | | |
| Elemental Analyses | C | H | O | density@25C moisture | |
| feed | 33.59% | 4.34% | 46.72% | 1.01 | 91.80% 0.05% < 0.005 |
| product oil | 63.92% | 8.41% | 24.28% | 1.040 | 1.26% 0.01% 1.9% 99.74% |
| oxygen by difference | 1.88% | 10.51% | 87.60% | 1.0 est | 96.00% 0.68% < 0.005% 99.99% |
| Material Balance | g C/hr | g H/hr | g O/hr | Total | TAN Viscosity COD |
| feed | 41.98 | 162.26 | 1300.66 | 1504.90 | 77070 |
| product oil | 4.41 | 0.58 | 1.68 | | NA |
| aqueous | 27.70 | 154.85 | 1290.70 | pH 7.20-7.36 | 61470 |
| gas | 1.86 | 0.07 | 4.92 | | |
| Total Products | 33.97 | 155.50 | 1297.30 | 1486.77 | |
| Elemental Balance | 81% | 96% | 100% | | |
| Total Material Balance | >>>>> | >>>>>>>> | >>>>>> | 99% | |
| GAS CALCULATIONS | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 g H/hr | |
| Gas Out | 217.7 L/hr | 9.07 | moles/hr | | |
| Gas Composition | volume% | moles/hr | | | |
| Hydrogen | 0.37% | C | H | O | |
| CarbDioxide | 1.70% | 0.15 | 0.07 | 0.31 | 0.07 gH/hr trace |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | |
| Methane | 0.00% | 0.00 | 0.00 | trace | trace |
| Ethane | 0.00% | 0.00 | 0.00 | trace | trace |
| Propane | 0.00% | 0.00 | 0.00 | | |
| Butanes | 0.00% | 0.00 | 0.00 | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 2.94 | |
| | 2.06% | | | | |
| Total Gas, C1-C4 | 1.86 | C g/hr | H g/hr | O g/hr | 6.78 Total gas |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 Total gas oil |
| | 1.86 | 0.00 | 4.92 | | 6.79 Total gas mass out |
| Yields | | | | | 10.51% carbon conversion to oil |
| Oil Product Yield | 5.59 g/hr | density | 5.37 ml/hr | 1.04 g/ml | 0.00 L/L feed 4.47% mass conversion to oil |
| Oil Loss in Aqueous | 58.94 g/hr | density, est | 58.94 ml/hr | 1 g/ml | 0.04 L/L feed 65.98% carbon conversion to water solubles |
| Gasification of Carbon | 4.42% | | | | 4.43% carbon conversion to gas |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.07 g/hr out = | | -0.07 g/hr consumption -0.80 L/hr consumption -0.53 L/L feed -4.40017E-05 g/g feed |
| Space velocity | 3.02 L/L/hr | LHSV | | | |
| Chemical Hydrogen Consumption | | -0.07 g/hr | -1 L/L | -1 | nM3/tonne |
| Calculation of Deoxygenation | | | | | |
| O content of dry product | 9.11% | | | | |
| O in Dry Product | 0.51 g/hr | | | | |
| O in Organics(H ₂ O) | 33.40 g/hr-----> | | | | |
| O in Dry Feed | 58.38 g/hr | | | | |
| Deoxygenation | 41.92% | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|------------------|-----------------|-----------------|-------------------------|--|-------------------|---------|--------|---------|
| Run No. | HTL-15 | Reactor | 157-173 | reactor volume | 1000 | total mL | | | |
| Pressure | 20.1 MPa | Temperature | 343-353 | | 700 | mL at temperature | | | |
| 40-140psig pressure drop | 2900 psig | degrees C | 350-355 | | | | | | |
| Total Feed | 32259 cc | Feed rate | 1500 cc/hr | | | | | | |
| corn stover slurry #1 | | | 1515.00 q/hr | | | | | | |
| wt% sodium carbonate | | | | estimates | | | | | |
| Total Product | 33050 g | Product oil | 40 g/hr | | sum of two phases | 1537 | | | |
| | | Product aqueous | 1497 g/hr | | | q/hr | | | |
| | | | | | 12.42 in CS | | | | |
| Elemental Analyses | C | H | O | density@25C | moisture | N | S | ash | |
| estimates from LF11 | 32.70% | 3.70% | 40.60% | 1.01 | 89.40% | 0.70% | 0.03% | 22.39% | 100.09% |
| feed | 72.82% | 7.63% | 19.99% | 1.040 | 3.65% | 1.24% | 0.05% | 0.0% | 101.73% |
| product oil | | | | | | <0.05 | <0.005% | | 99.97% |
| oxygen by difference | 1.39% | 10.28% | 88.30% | 1.0 est | 97.00% | | | | |
| aqueous | | | | | | | | | |
| Material Balance | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | COD | | |
| ASH-Free Basis | | | | | | | | | |
| feed | 52.51 | 157.77 | 1267.78 | 1478.06 | NA | 91583 | | | |
| product oil | 29.13 | 3.05 | 8.00 | | 25.6 | NA | | | |
| aqueous | 20.73 | 153.89 | 1321.85 | | pH 4.8-5.6 | 52000 | | | |
| gas | 2.61 | 0.02 | 6.92 | | | | | | |
| Total Products | 52.47 | 156.96 | 1336.76 | 1546.20 | | | | | |
| Elemental Balance | 100% | 99% | 105% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | 105% | | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | 0.00 q H/hr | | | | | |
| Gas Out | 247 L/hr | | 10.29 moles/hr | | | | | | |
| Gas Composition | volume% | C | moles/hr | | | | | | |
| Hydrogen | 0.10% | | 0.02 | 0.02 qH/hr | | | | | |
| CarbDioxide | 2.10% | 0.22 | 0.43 | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | | | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | | | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | | 2.94 | | | | |
| | 2.20% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 2.61 | 0.00 | 6.92 | 9.52 Total gas | | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 Total gas oil | | | | | |
| | 2.61 | 0.00 | 6.92 | 9.53 Total gas mass out | | | | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 38.54 g/hr | | 37.06 ml/hr | | 55.47% carbon conversion to oil | | | | |
| | | density, | 1.04 g/ml | | 0.02 L/L feed | | | | |
| Oil Loss in Aqueous | 44.91 g/hr | | 44.91 ml/hr | | 24.00% mass conversion to oil | | | | |
| | | density, est | 1 g/ml | | 0.03 L/L feed | | | | |
| Gasification of Carbon | 4.96% | | | | 39.48% carbon conversion to water solubles | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | | 0.02 g/hr out - | | 4.97% carbon conversion to gas | | | | |
| | | | | | -0.02 g/hr consumption | | | | |
| | | | | | -0.25 L/hr consumption | | | | |
| | | | | | -0.16 L/L feed | | | | |
| | | | | | -1.37222E-05 g/q feed | | | | |
| Space velocity | 2.14 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | -0.02 g/hr | 0 L/L | 0 m3/tonne | | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 17.38% | | | | | | | | |
| O in Dry Product | 6.70 g/hr | | | | | | | | |
| O in Organics(H2O) | 31.10 g/hr-----> | | | | | | | | |
| O in Dry Feed | 65.20 g/hr | | | | | | | | |
| Deoxygenation | 42.02% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|------------------|---------------------|--------------|--|--------------------|--------------|---------|------------|-----------------------|
| Run No. | HTL-16 | CSTR | 320 | reactor volume | 1000 | total mL | | | |
| Pressure | 19.8 MPa | CSTR | 351 | Time | 16:30-19:45 | | | | |
| 45 psig differential | 2860 psig | filter | 334 | Date | 3-Mar-09 | | | | |
| Total Feed | 3750 cc | Feed rate | 1154 cc/hr | | | | | | |
| corn stover slurry #3 w/1% sodium carbonate | | | 1165.54 g/hr | | | | | | |
| Total Product | 3257.5 g | Product oil | 27 g/hr | blowdown | 10.66% | % dry solids | | | |
| Blowdown | 408 g | Product aqueous | 975 g/hr | blowdown | 125.5 g/hr | | | | |
| | | dry blowdown | 13.38 g/hr | sum of two phases | 1002 g/hr | | | | |
| Elemental Analyses | C | H | O | density@25C moisture | N | S | | | |
| estimates from LF11 | 32.80% | 3.70% | 40.70% | 1.01 | 0.70% | 0.026% | 22.12% | 100.02% | |
| product oil | 72.05% | 7.88% | 19.92% | 1.040 | 3.36% | 1.35% | 0.068% | 0.0% | 101.27% |
| oxygen by difference | 1.96% | 10.02% | 88.00% | 1.0 est | 96.00% | <0.05 | <0.005% | | 99.98% |
| not blowdown solids! | 23.37% | 2.74% | 12.04% | | | 0.76% | 0.019% | 0.00% | 99.92% too much C and |
| Material Balance | q C/hr | q H/hr | q O/hr | Total | TAN | Viscosity | COD | | too little ash |
| feed | 36.85 | 122.22 | 980.85 | | | | | 915B3 est. | |
| product oil | 19.45 | 2.13 | 5.38 | | | | | NA | |
| aqueous | 19.11 | 97.70 | 858.00 | | | | | 53270 | |
| gas | 2.29 | 0.04 | 6.09 | | | | | | |
| blowdown | 3.13 | 0.37 | 1.61 | | | | | | |
| Total Products | 43.98 | 100.23 | 871.08 | | | | | | |
| Elemental Balance | 119% | 82% | 89% | | | | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>>> | | 89% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 | g H/hr | | | | |
| Gas Out | 147.4 L/hr | 6.14 | moles/hr | | | | | | |
| Gas Composition | volume% | moles/hr | | | | | | | |
| Hydrogen | 0.30% | C | H | O | 0.04 gH/hr | trace | | | |
| CarbDioxide | 3.10% | 0.19 | | 0.38 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | |
| | 3.40% | | | | | | | | |
| | C q/hr | H q/hr | O q/hr | | | | | | |
| Total Gas, C1-C4 | 2.29 | 0.00 | 6.09 | 8.39 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 2.29 | 0.00 | 6.09 | 8.39 | Total gas mass out | | | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 26.03 g/hr | 25.03 ml/hr | | 52.79% carbon conversion to oil | | | | | |
| | | density, 1.04 g/ml | | 0.02 L/L feed | | | | | |
| Oil Loss in Aqueous | 39.00 g/hr | 39.00 ml/hr | | 23.16% mass conversion to oil | | | | | |
| | | density, est 1 g/ml | | 0.03 L/L feed | | | | | |
| Gasification of Carbon | 6.22% | | | 51.85% carbon conversion to water solubles | | | | | |
| Hydrogen Consumption | 0.00 g/hr in - | 0.04 g/hr out = | | 6.22% carbon conversion to gas | | | | | |
| | | | | -0.04 g/hr consumption | | | | | |
| | | | | -0.44 L/hr consumption | | | | | |
| | | | | -0.38 L/L feed | | | | | |
| | | | | -3.19324E-05 g/q feed | | | | | |
| Space velocity | 1.65 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | -0.40 g/hr | | -5 L/L | | | | | |
| | | | | -5 nM3/tonne | | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 17.34% | | | | | | | | |
| O in Dry Product | 4.51 g/hr | | | | | | | | |
| O in Organics(H2O) | 26.00 g/hr-----> | | | | | | | | |
| O in Dry Feed | 45.73 g/hr | | | | | | | | |
| Deoxygenation | 33.27% | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | | |
|---|-------------|--------------|---------------|--------|------------------------------------|--------------------------|------------------------|-----------|--------------|---------|
| Run No. | HTL-17 | | | | reactor volume | 1000 | total mL | | | |
| Pressure | 20.3 MPa | CSTR | 319 | | | 1000 | mL at temperature | | | |
| 45 psig differential | 2936 psig | CSTR | 346 | | | | | | | |
| | | filter | 334 | | | | | | | |
| Time | 11:00-17:45 | | | | | | | | | |
| Date | 31-Mar-09 | | | | | | | | | |
| Total Feed | 10084 cc | Feed rate | 1494 cc/hr | | 215.8 tot oil | | | | | |
| corn stover slurry # 1 | | | 1508.94 g/hr | | 9368 tot aq | | | | | |
| w/1% sodium carbonate | | | | | Product oil | 32 | blowdown g/hr | 20.13% | % dry solids | |
| Total Product | 9583.8 g | | | | Product aqueous | 1387.9 | blowdown g/hr | 107.8 | g/hr | |
| Blowdown | 566 g | | | | dry blowdown | 21.70 | sum of two phases g/hr | 1419.9 | g/hr | |
| | | | | | | | | | | |
| Elemental Analyses | | C | H | O | density@25C | moisture | N | S | ash | |
| feed | 38.61% | 4.49% | 38.86% | 1.01 | | 90.10% | 0.66% | 0.041% | 19.99% | 102.60% |
| product oil | 69.46% | 7.75% | 19.87% | 1.110 | | 8.79% | 1.29% | 0.097% | 0.0% | 97.46% |
| oxygen by difference | 2.76% | 11.05% | 86.00% | | 1.0 est | 96.00% | <0.05 | <0.005% | | 99.80% |
| aqueous | | | | | | | 0.20% | 0.020% | 78.26% | 100.19% |
| dry rinsed soil | 11.27% | 1.12% | 9.33% | | | | | | | |
| oil, dry basis | 77.20% | 7.63% | 13.61% | | | | 1.45% | 0.11% | | |
| | | | | | | | | | | |
| Material Balance | | g C/hr | g H/hr | g O/hr | Total | | TAN | Viscosity | COD | |
| feed | 57.68 | 159.11 | 1265.20 | | 1481.98 | | | | 88867 | |
| product oil | 21.91 | 2.48 | 6.36 | | | | 30.4 | NA | | |
| aqueous | 38.24 | 153.29 | 1193.59 | | | | pH 4.6-4.8 | | 52868 | |
| gas | 5.79 | 0.08 | 15.39 | | | | | | | |
| blowdown | 2.44 | 0.24 | 2.02 | | | | | | | |
| Total Products | 68.37 | 156.09 | 1217.37 | | 1441.84 | | | | | |
| | | | | | | | | | | |
| Elemental Balance | 119% | 98% | 96% | | | | | | | |
| Ash Balance | >>>>> | >>>>>>> | >>>>>>> | >>>>>> | | 56.87% (without aqueous) | | | | |
| Total Material Balance | >>>>> | >>>>>> | >>>>> | >>>>> | | 97% | | | | |
| | | | | | | | | | | |
| GAS CALCULATIONS | | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | | 0.00 g H/hr | | | | | |
| Gas Out | 213.8 L/hr | | 8.91 moles/hr | | | | | | | |
| | | | | | | | | | | |
| Gas Composition | | moles/hr | | | | | | | | |
| volume% | C | H | O | | | | | | | |
| Hydrogen | 0.42% | | 0.07 | | 0.08 gH/hr | | trace | | | |
| CarbDioxide | 5.40% | 0.48 | 0.96 | | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | | | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | trace | | trace | | | |
| Ethane | 0.00% | 0.00 | 0.00 | | trace | | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | | |
| | 5.82% | | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | | |
| Total Gas, C1-C4 | 5.79 | 0.00 | 15.39 | | 21.18 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | | 0.00 | Total gas oil | | | | |
| | 5.79 | 0.00 | 15.39 | | 21.18 | Total gas mass out | | | | |
| | | | | | | | | | | |
| Yields | | | | | 37.98% carbon conversion to oil | | 32.04% C bal. adjusted | | | |
| Oil Product Yield | 29.19 g/hr | | 26.30 mL/hr | | 0.02 L/L feed | | | | | |
| | | density, | 1.11 g/ml | | 19.54% mass conversion to oil | | 16.49% C bal. adjusted | | | |
| Oil Loss in Aqueous | | | | | | | | | | |
| | | density, est | 1 g/ml | | 66.29% C conversion to water solub | | 55.92% C bal. adjusted | | | |
| Gasification of Carbon | 10.03% | | | | 10.03% carbon conversion to gas | | 8.46% C bal. adjusted | | | |
| Carbon loss in solids | 2.44 g/hr | | | | 4.24% % carbon loss in solids | | 3.58% C bal. adjusted | | | |
| | | | | | | | | | | |
| Space velocity | 1.49 L/L/hr | LHSV | | | | | | | | |
| | | | | | | | | | | |
| Calculation of Deoxygenation | | | | | | | | | | |
| O content of dry product | 13.22% | | | | | | | | | |
| O in Dry Product | 3.86 g/hr | | | | | | | | | |
| O in Organics(H ₂ O) | 9.25 g/hr | | | | | | | | | |
| O in Dry Feed | 58.04 g/hr | | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | | | |
|---|------------------|-----------------|---------------|--------------------------------------|--------------------------|------------------------|---------|-------------------|---------|--|--|
| Run No. | HTL-17 | reactor volume | 320 | 1000 | total mL | | | | | | |
| Pressure | 21.6 MPa | CSTR | 320 | 1000 | mL at temperature | | | | | | |
| 45 psig differential | 3120 psig | CSTR | 345 | Time | 00:15:07:00 | | | | | | |
| filter | 347 | filter | 347 | Date | 2-Apr-09 | | | | | | |
| Total Feed | 10130 cc | Feed rate | 1500.7 cc/hr | 225 tot oil | 9134.5 tot aq | blowdown | 20.80% | % dry solids | | | |
| corn stover slurry # 7 | | | 1515.71 g/hr | | | blowdown | 87.3 | g/hr | | | |
| w/1% sodium carbonate | | | | Product oil | 33.3 g/hr | sum of two phases | 1386.6 | g/hr | | | |
| Total Product | 9359.5 g | Product aqueous | 1363.3 g/hr | dry blowdown | 18.16 g/hr | | 12.42 | In CS | | | |
| Blowdown | 589 g | | | | | | | | | | |
| Elemental Analyses | C | H | O | density@25C moisture | N | S | ash | | | | |
| feed | 36.50% | 4.40% | 36.44% | 7.01 | 86.75% | 0.46% | 0.028% | 23.64% | 101.44% | | |
| product oil | 63.15% | 8.35% | 26.57% | 1.088 | 10.27% | 1.12% | 0.068% | 0.7% | 99.26% | | |
| oxygen by difference | 2.57% | 10.13% | 86.00% | 1.0 est | 96.00% | <0.05 | <0.005% | | 98.70% | | |
| aqueous | | | | | | 0.26% | 0.017% | 74.22% | 97.19% | | |
| dry rinsed so | 12.60% | 1.16% | 8.94% | | | | | | | | |
| oil, dry basis | 70.96% | 8.09% | 19.61% | | | 1.26% | 0.08% | | | | |
| Material Balance | g C/hr | g H/hr | g O/hr | Total | TAN | Viscosity | COD | | | | |
| feed | 73.30 | 156.22 | 1240.66 | 1470.19 | | | 145130 | | | | |
| product oil | 21.03 | 2.78 | 8.85 | | 31.31 | | NA | | | | |
| aqueous | 34.78 | 137.09 | 1163.84 | | pH 4.85-5.70 | | 86760 | | | | |
| gas | 6.18 | 0.12 | 16.27 | | | | | | | | |
| blowdown | 2.29 | 0.21 | 1.62 | | | | | | | | |
| Total Products | 64.27 | 140.20 | 1190.58 | 1395.05 | | | | | | | |
| Elemental Balance | 88% | 90% | 96% | | | | | | | | |
| Ash Balance | >>>>>>>>>>>>>>>> | | | | | | 28.39% | (without aqueous) | | | |
| Total Material Balance | >>>>>>>>>>>>>>> | | | | | | 95% | | | | |
| GAS CALCULATIONS | | | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | 0.00 | q H/hr | | | | | | |
| Gas Out | 217.9 L/hr | | 9.08 moles/hr | | | | | | | | |
| Gas Composition | moles/hr | | | | | | | | | | |
| volume% | C | H | O | | 0.10 qH/hr | trace | | | | | |
| Hydrogen | 0.55% | 0.10 | 1.02 | | | | | | | | |
| CarbDioxide | 5.60% | 0.51 | 1.02 | | | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | | trace | | | | | |
| Ethane | 0.03% | 0.01 | 0.02 | trace | | trace | | | | | |
| Propane | | 0.00 | 0.00 | | | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 2.94 | | | | | | | |
| | 6.18% | | | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | | | |
| Total Gas, C1-C4 | 6.18 | 0.02 | 16.27 | 22.46 | Total gas | | | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | | | |
| | 6.18 | 0.02 | 16.27 | 22.46 | Total gas mass out | | | | | | |
| Yields | | | | 28.69% | carbon conversion to oil | 32.72% C bal. adjusted | | | | | |
| Oil Product Yield | 29.88 q/hr | | 27.46 ml/hr | 0.02 L/L feed | | | | | | | |
| | | density, | 1.088 g/ml | 14.88% mass conversion to oil | | 16.97% C bal. adjusted | | | | | |
| Oil Loss in Aqueous | | | | 47.45% C conversion to water soluble | 54.11% C bal. adjusted | | | | | | |
| Gasification of Carbon | 8.43% | | | 8.43% carbon conversion to gas | 9.61% C bal. adjusted | | | | | | |
| Carbon loss in solids | | 2.29 g/hr | | 3.12% % carbon loss in solids | 3.56% C bal. adjusted | | | | | | |
| Space velocity | 1.50 L/L/hr | LHSV | | | | | | | | | |
| Chemical Hydrogen Consumption | -0.35 g/hr | | -3 L/L | | -3 nM3/tonne | | | | | | |
| Calculation of Deoxygenation | | | | | | | | | | | |
| O content of dry product | 19.44% | | | | | | | | | | |
| O in Dry Product | 5.81 g/hr | | | | | | | | | | |
| O in Organics(H ₂ O) | 9.02 g/hr | | | | | | | | | | |
| O in Dry Feed | 73.18 g/hr | | | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|----------------|-----------------|--------------|-------------------|------------------------------------|------------------------|-------|-----------|---------|
| Run No. | HTL-17 | | | reactor v'lume | 1000 | total mL | | | |
| Pressure | 20.3 MPa | CSTR | 319 | | 1000 | mL at temperature | | | |
| 45 psig differential | 2935 psig | CSTR | 345 | | | | | | |
| | | filter | 352 | | | | | | |
| | | | | | Time | 06:15:10:45 | | | |
| | | | | | Date | 3-Apr-09 | | | |
| Total Feed | 6760 cc | Feed rate | 1502 cc/hr | 131.2 tot oil | | | | | |
| corn stover slurry # 7 | | | 1517.02 g/hr | 6231 tot aq | | | | | |
| w/1% sodium carbonate | | | | | blowdown | 19.02% dry solids | | | |
| Total Product | 9583.8 g | Product oil | 29.2 g/hr | sum of two phases | 157.6 g/hr | | | | |
| Blowdown | 709 g | Product aqueous | 1384.7 g/hr | | 1413.9 g/hr | | | | |
| | | dry blowdown | 29.98 g/hr | | | 12.42 in CS | | | |
| Elemental Analyses | | C | H | O | density@25C | moisture | N | S | ash |
| | feed | 35.95% | 4.40% | 35.52% | 7.01 | 86.60% | 0.47% | 0.013% | 23.95% |
| | product oil | 64.19% | 8.71% | 25.22% | 1.081 | 12.33% | 0.99% | 0.048% | 0.78% |
| oxygen by difference | | 2.79% | 11.21% | 86.00% | 1.0 est | 96.00% | <0.05 | <0.005% | 100.00% |
| | aqueous | 13.96% | 1.25% | 9.31% | | | 0.21% | 0.014% | 74.22% |
| | dry rinsed soi | | | | | | | | 98.95% |
| | oil, dry basis | 73.93% | 8.44% | 16.44% | | | 1.14% | 0.06% | |
| Material Balance | | g C/hr | g H/hr | g O/hr | Total | | TAN | Viscosity | COD |
| | feed | 73.07 | 156.21 | 1238.67 | 1467.95 | | | 136400 | |
| | product oil | 18.74 | 2.54 | 7.36 | | 30.91 | NA | | |
| | aqueous | 38.63 | 155.16 | 1190.84 | | pH 6.15-6.24 | | 82600 | |
| | gas | 4.74 | 0.09 | 12.60 | | | | | |
| | blowdown | 4.18 | 0.37 | 2.79 | | | | | |
| | Total Products | 66.30 | 158.16 | 1213.59 | 1438.06 | | | | |
| Elemental Balance | | 91% | 101% | 98% | | | | | |
| Ash Balance | | >>>>> | >>>>> | >>>>> | 45.70% | (without aqueous) | | | |
| Total Material Balance | | >>>> | >>>>> | >>>>> | 98% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 | moles/hr | 0.00 | g H/hr | | | |
| Gas Out | 210 L/hr | | 8.75 | | | | | | |
| Gas Composition | | moles/hr | | | | | | | |
| | volume% | C | H | O | | | | | |
| Hydrogen | 0.50% | 0.09 | | | 0.09 | gH/hr | trace | | |
| CarbDioxide | 4.50% | 0.39 | | | 0.79 | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | | | trace | | |
| Ethane | 0.00% | 0.00 | 0.00 | trace | | | trace | | |
| Propane | 0.00 | 0.00 | | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | 2.94 | | | | |
| | 5.00% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 4.74 | 0.00 | 12.60 | 17.34 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 4.74 | 0.00 | 12.60 | 17.34 | Total gas mass out | | | | |
| Yields | | | | | 25.65% carbon conversion to oil | 28.27% C bal. adjusted | | | |
| Oil Product Yield | 25.60 g/hr | | 23.68 ml/hr | | 0.02 L/L feed | | | | |
| | | density, | 1.081 g/ml | | 12.59% mass conversion to oil | 13.88% C bal. adjusted | | | |
| Oil Loss in Aqueous | | | | | 52.87% C conversion to water solub | 58.27% C bal. adjusted | | | |
| Gasification of Carbon | 6.48% | | | | 6.49% carbon conversion to gas | 7.15% C bal. adjusted | | | |
| Carbon loss in solids | | 4.18 g/hr | | | 5.72% % carbon loss in solids | 6.31% C bal. adjusted | | | |
| Space velocity | 1.50 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | -0.48 g/hr | | -3 L/L | | -3 nM3/tonne | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 16.26% | | | | | | | | |
| O in Dry Product | 4.16 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 9.23 g/hr | | | | | | | | |
| O in Dry Feed | 72.20 g/hr | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|-----------------------|------------------------|-------------------|-------------------------------------|------------------------|---|-------------------|---------------------|---------|
| Run No. | HTL-19 | reactor vluome | 1000 | total mL | 1000 | mL at temperature | | | |
| Pressure | 20.4 MPa 2950 psig | CSTR CSTR filter | 322 346 340 | Time | 16:18:19:40 | Date | 28-May-09 | | |
| Total Feed | 5020 cc | Feed rate | 1491 cc/hr | 1505.91 g/hr | 105.5 tot oil | 4549 tot aq | blowdown | 14.23% % dry solids | |
| corn stover slurry #2 w/1% sodium carbonate | | | | | | | blowdown | 185.6 g/hr | |
| Total Product | 4654.5 g | Product oil | 31.3 g/hr | | | | sum of two phases | 1382.5 g/hr | |
| Blowdown | 625 g | Product aqueous | 1351.2 g/hr | | | | | | |
| | | dry blowdown | 26.42 g/hr | | | | | | |
| | | | | | | | | 12.42 in CS | |
| Elemental Analyses | C | H | O | density@25C | moisture | N | S | ash | |
| feed | 34.65% | 4.66% | 38.24% | 1.03 | 87.40% | 0.52% | 0.032% | 25.69% | 103.74% |
| product oil | 68.89% | 7.91% | 19.89% | 1.094 | 7.74% | 1.15% | 0.083% | 0.0% | 97.92% |
| oxygen by difference | 2.77% | 10.67% | 86.60% | 1.0 est | 96.00% | <0.05 | <0.005% | | 100.04% |
| aqueous | | | | | | | | 0.23% 0.019% | 74.53% |
| dry rinsed solids | 10.61% | 1.18% | 12.53% | | | | | | 99.08% |
| oil, dry basis | 76.39% | 7.81% | 14.43% | | | | | | |
| | | | | | | 1.28% | 0.09% | | |
| Material Balance | q C/hr | q H/hr | q O/hr | Total | TAN | Viscosity | COD | | |
| feed | 65.74 | 156.37 | 1241.18 | 1463.28 | | | | | |
| product oil | 21.56 | 2.48 | 6.22 | | 28.87 | >1500 cSt @ 40C | | | |
| aqueous | 37.36 | 144.17 | 1170.14 | | 4.5 | | 71250 | | |
| gas | 5.10 | 0.19 | 12.98 | | | | | | |
| blowdown | 2.80 | 0.31 | 3.31 | | | | | | |
| Total Products | 66.82 | 147.15 | 1192.65 | 1406.62 | | | | | |
| Elemental Balance | 102% | 94% | 96% | | | | | | |
| Ash Balance | >>>>> | >>>>>>> | >>>>>> | | 40.38% | (based on solids recovered and without aqueous) | | | |
| Total Material Balance | >>>>> | >>>>>>> | >>>>>> | | 96% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 | q H/hr | | | | |
| Gas Out | 217 L/hr | 9.04 | moles/hr | | | | | | |
| Gas Composition | volume% | C | H | O | 0.13 qH/hr | trace | | | |
| Hydrogen | 0.73% | | 0.13 | | | | | | |
| CarbDioxide | 4.49% | 0.41 | | 0.81 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | trace | trace | | | |
| Ethane | 0.10% | 0.02 | 0.05 | | trace | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | |
| | 5.32% | | | | | | | | |
| | C q/hr | H q/hr | O q/hr | | | | | | |
| Total Gas, C1-C4 | 5.10 | 0.06 | 12.98 | 18.13 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 5.10 | 0.06 | 12.98 | 18.13 | Total gas mass out | | | | |
| Yields | | | | 32.80% carbon conversion to oil | 32.27% C bal. adjusted | | | | |
| Oil Product Yield | 28.88 q/hr | 26.41 ml/hr | 1.0935 q/ml | 0.02 L/L feed | | | | | |
| | density, | | | 15.22% mass conversion to oil | 14.97% C bal. adjusted | | | | |
| Oil Loss in Aqueous | | | | 56.83% C conversion to water solubl | 55.91% C bal. adjusted | | | | |
| Gasification of Carbon | 7.75% | | | 7.75% carbon conversion to gas | 7.63% C bal. adjusted | | | | |
| Carbon loss in solids | | 2.80 g/hr | | 4.26% % carbon loss in solids | 4.19% C bal. adjusted | | | | |
| Space velocity | 1.49 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | -0.47 g/hr | -4 L/L | | -3 nM3/tonne | | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 14.10% | | | | | | | | |
| O in Dry Product | 4.07 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 17.12 g/hr | | | | | | | | |
| O in Dry Feed | 72.55 g/hr | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|-------------------------|-----------------------|---------------------------------|--------------------------------------|------------------------|-------------------------------|---------------|--------|-------------|
| Run No. | HTL-20 | reactor volume | 1000 | total mL | | | | | |
| Pressure | 21.1 MPa +/-100 psig | CSTR filter | 321 340 343 | 1000 | mL at temperature | | | | |
| Total Feed | 11688 cc | Feed rate | 1798 cc/hr | 254.5 tot oil | blowdown | 22.38% | % dry solids | | |
| corn stover slurry #3 w/1% sodium carbonate | | | 1815.98 g/hr | 9,728.5 tot aq | blowdown | 172.2 g/hr | | | |
| Total Product | 9983 g | Product oil | 40.4 g/hr | sum of two phases | | | | | 1661.8 g/hr |
| Blowdown | 861 g | #12 missing | Product aqueous dry blowdown | 1621.4 g/hr | | | 12.42 | In CS | |
| Elemental Analyses | | C | H | O | density@25C moisture | N | S | ash | |
| feed | 34.98% | 4.61% | 40.45% | 1.01 | 89.14% | 0.58% | 0.040% | 23.60% | 104.22% |
| product oil | 68.09% | 7.84% | 21.53% | 1.066 | 6.62% | 1.23% | 0.074% | | 98.75% |
| oxygen by difference | 1.79% | 10.98% | 87.20% | 1.0 est | 96.00% | <0.05 | <0.005% | | 99.97% |
| aqueous | 11.15% | 1.15% | 11.44% | | | 0.20% | 0.016% | 82.97% | 106.92% |
| dry rinsed soil | | | | | | 1.33% | 0.08% | | |
| oil, dry basis | 73.91% | 7.70% | 16.98% | | | | | | |
| Material Balance | | q C/hr | q H/hr | q O/hr | Total | TAN | Viscosity | COD | |
| feed | 68.98 | 190.55 | 1517.08 | | 1776.61 | | | 93467 | |
| product oil | 27.51 | 3.17 | 8.70 | | | 48.17 | >1500 cSt@40C | | |
| aqueous | 29.02 | 177.95 | 1413.86 | | | pH = 5 | | 53725 | |
| gas | 4.87 | 0.10 | 12.75 | | | | | | |
| blowdown | 4.29 | 0.44 | 4.41 | | | | | | |
| Total Products | 65.70 | 181.66 | 1439.72 | | 1687.07 | | | | |
| Elemental Balance | 95% | 95% | 95% | | | | | | |
| Ash Balance | >>>>> >>>>>> >>>>>> | | | | 68.69% | (solids only without liquids) | | | |
| Total Material Balance | >>>>> >>>>>> >>>>> | | | | 95% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 moles/hr | | 0.00 q H/hr | | | | | |
| Gas Out | 251.7 L/hr | 10.49 moles/hr | | | | | | | |
| Gas Composition | | moles/hr | | | | | | | |
| volume% | C | H | O | | | | | | |
| Hydrogen | 0.38% | | 0.08 | | 0.08 qH/hr | trace | | | |
| CarbDioxide | 3.80% | 0.40 | 0.80 | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Ethane | 0.03% | 0.01 | 0.02 | trace | | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | | 2.94 | | | | |
| | 4.21% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 4.87 | 0.02 | 12.75 | 17.65 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 4.87 | 0.02 | 12.75 | 17.65 | Total gas mass out | | | | |
| Yields | | | | 39.88% carbon conversion to oil | 41.87% C bal. adjusted | | | | |
| Oil Product Yield | 37.73 g/hr | 35.38 ml/hr | | 0.02 L/L feed | | | | | |
| | | density, 1.06624 g/ml | | 19.13% mass conversion to oil | 20.08% C bal. adjusted | | | | |
| Oil Loss in Aqueous | | | | 42.08% C conversion to water soluble | 44.18% C bal. adjusted | | | | |
| Gasification of Carbon | 7.06% | | | 7.07% carbon conversion to gas | 7.42% C bal. adjusted | | | | |
| Carbon loss in solids | | 4.29 g/hr | | 6.23% % carbon loss in solids | 6.54% C bal. adjusted | | | | |
| Space velocity | 1.80 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | -0.55 g/hr | | -3 L/L | | -3 nM3/tonne | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 16.75% | | | | | | | | |
| O in Dry Product | 6.32 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 30.27 g/hr | | | | | | | | |
| O in Dry Feed | 79.77 g/hr | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|--------------------------------------|-------------------|---|--|-------------------------------|------------------------|--------------------------------------|---------|---------|
| Run No. | HTL-20 | | | | reactor volume | 1000 total mL | | | |
| Pressure | 21.0 MPa +/-100 | CSTR 3040 psig | 306 inlet-bottom CSTR filter 330 | | | 1000 mL at temperature | | | |
| | | | | | Time | 22-10-06 10 | | | |
| | | | | | Date | 6/3/2009 | | | |
| Total Feed | 11890 cc | Feed rate | 1486.25 cc/hr | | | | | | |
| corn stover slurry #4 w/1% sodium carbonate | | | 1501.11 g/hr | 178 tot oil 11069 tot aq | | blowdown blowdown | 24.03% % dry solids 110.875 g/hr | | |
| Total Product | 11247 g | | | Product oil 1383.625 g/hr | sum of two phases | 1405.875 g/hr | | | |
| Blowdown | 887 g | | | Product aqueous dry blowdown density@25C | | 12.42 in CS | | | |
| Elemental Analyses | C | H | O | moisture | N | S | ash | | |
| feed | 34.31% | 4.60% | 34.59% | 1.01 | 90.02% | 0.55% | 0.035% | 23.48% | 97.53% |
| product oil | 69.47% | 7.35% | 22.29% | 1.109 | 11.25% | 1.25% | 0.077% | | 100.44% |
| oxygen by difference | aqueous | 1.76% | 10.44% | 87.80% | 1.0 est | 96.00% | <0.05 | <0.005% | 100.00% |
| | dry rinsed solids | 12.57% | 1.31% | 12.29% | | 0.25% | 0.014% | 63.45% | 109.88% |
| | oil, dry basis | 77.90% | 6.82% | 13.79% | | 1.40% | 0.09% | | |
| Material Balance | q C/hr | q H/hr | q O/hr | Total | TAN | Viscosity | COD | | |
| feed | 51.39 | 158.37 | 1251.64 | | 1461.41 | | | 142333 | |
| product oil | 15.46 | 1.63 | 4.96 | | | 42.26 >1500 cSt@40C | | | |
| aqueous | 24.35 | 144.38 | 1214.82 | | | pH = 5 | | 58100 | |
| gas | 3.11 | 0.06 | 8.26 | | | | | | |
| blowdown | 3.35 | 0.35 | 3.27 | | | | | | |
| Total Products | 46.27 | 146.42 | 1231.32 | | 1424.01 | | | | |
| Elemental Balance | 90% | 92% | 98% | | | | | | |
| Ash Balance | >>>>> >>>>>> >>>>>> >>>>>>> >>>>>>>> | | | | | | 63.19% (solids only without liquids) | | |
| Total Material Balance | >>>>> >>>>>> >>>>>> >>>>>>> | | | | | | 97% | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | | 0.00 | g H/hr | | | |
| Gas Out | 221.375 L/hr | 9.22 | moles/hr | | | | | | |
| Gas Composition | volume% | C | H | O | 0.06 | gH/hr | trace | | |
| Hydrogen | 0.30% | | 0.06 | | | | | | |
| CarbDioxide | 2.80% | 0.26 | | 0.52 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | | trace | | trace | | |
| Ethane | 0.00% | 0.00 | 0.00 | | trace | | trace | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 0.00 | | 2.94 | | | |
| | 3.10% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 3.11 | 0.00 | 8.26 | 11.38 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 3.11 | 0.00 | 8.26 | 11.38 | Total gas mass out | | | | |
| Yields | | | | 30.08% carbon conversion to oil | 33.41% C bal. adjusted | | | | |
| Oil Product Yield | 19.75 g/hr | 17.80 ml/hr | density, 1.10938 g/ml | 0.01 L/L feed | 13.18% mass conversion to oil | 14.64% C bal. adjusted | | | |
| | | | | | | | | | |
| Oil Loss in Aqueous | | | | 47.38% C conversion to water soluble | 52.63% C bal. adjusted | | | | |
| Gasification of Carbon | 6.05% | | | 6.06% carbon conversion to gas | 6.73% C bal. adjusted | | | | |
| Carbon loss in solids | | 3.35 g/hr | | 6.51% % carbon loss in solids | 7.23% C bal. adjusted | | | | |
| Space velocity | 1.49 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | -0.44 g/hr | | -3 L/L | | -3 nM3/tonne | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 13.84% | | | | | | | | |
| O in Dry Product | 2.73 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 34.13 g/hr | | | | | | | | |
| O in Dry Feed | 51.82 g/hr | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|-----------------------|-----------------------|---------------|----------------------------|--------------------------------------|------------------------|-------------------|---------------------|---------|
| Run No. | HTL-22 | | | | reactor volume | 1000 mL | | | |
| Pressure | 20.5 MPa 2963 psig | CSTR filter | 319 329 | inlet-bottom outlet-top | Time | 10:15:19:15 | total mL | | |
| | | | | | Date | 5-Aug-09 | mL at temperature | | |
| Total Feed | 13260 cc | Feed rate | 1473 cc/hr | | 296.5 g, total oil | | blowdown | 27.48% % dry solids | |
| corn stover slurry w/HTL20 recycle water w/1% sodium carbonate | | | 1487.73 g/hr | | 12438 g, total aqueous | | blowdown | 4.8 g/hr | |
| Total Product | 12734.5 g | Product oil | 32.94 g/hr | | | sum of two phases | 1414.94 g/hr | | |
| Blowdown | 43 g | Product aqueous | 1382 g/hr | | | | | | |
| | | dry blowdown | 1.33 g/hr | | | | | | |
| | | | | | | | | 12.42 in CS | |
| Elemental Analyses | | C | H | O | moisture | N | S | ash | |
| | feed | 34.61% | 4.05% | 42.49% | 1.01 | 86.15% | 0.43% | 0.009% | 22.70% |
| | product oil | 60.21% | 7.99% | 15.20% | 1.109 | 7.03% | 1.14% | 0.077% | 84.61% |
| oxygen by difference | | 2.60% | 10.11% | 87.30% | 1 | 96.00% | <0.05 | | 100.01% |
| oxygen by difference | | 10.48% | 0.87% | 8.70% | | | 0.16% | 79.81% | 100.01% |
| | oil, dry basis | 77.60% | 9.28% | 11.55% | | | 1.47% | 0.10% | |
| Material Balance | | g C/hr | g H/hr | g O/hr | | Total | TAN | Viscosity | COD |
| | feed | 71.29 | 152.01 | 1225.57 | | 1448.87 | | | 130.467 |
| | product oil | 19.83 | 2.63 | 5.01 | | | | | |
| | aqueous | 35.93 | 139.65 | 1206.49 | | | pH = 5.05 | | 68.610 |
| | gas | 6.83 | 0.21 | 17.35 | | | | | |
| | blowdown | 0.14 | 0.01 | 0.12 | | | | | |
| | Total Products | 62.73 | 142.50 | 1228.96 | | 1434.19 | | | |
| | Elemental Balance | 88% | 94% | 100% | | | | | |
| Ash Balance | >>>>>>>>>>>>>>>> | | | | 2.26% (solids only without liquids) | | | | |
| Total Material Balance | >>>>>>>>>>>>>>> | | | | 99% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | | 0.00 g H/hr | | | | |
| Gas Out | 209.9 L/hr | | 8.75 moles/hr | | | | | | |
| Gas Composition | | | moles/hr | | | | | | |
| | volume% | C | H | O | | | | | |
| Hydrogen | 0.67% | 0.12 | | | 0.12 gH/hr | trace | | | |
| CarbDioxide | 6.20% | 0.54 | | 1.08 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.13% | 0.01 | 0.05 | | trace | trace | | | |
| Ethane/ethylene | 0.08% | 0.01 | 0.04 | | trace | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | | 2.94 | | | | |
| | 7.08% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 6.83 | 0.09 | 17.35 | 24.27 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 6.83 | 0.09 | 17.35 | 24.27 | Total gas mass out | | | | |
| Yields | | | | | 27.82% carbon conversion to oil | 31.61% C bal. adjusted | | | |
| Oil Product Yield | 30.63 g/hr | | 27.61 ml/hr | | 0.02 L/L feed | | | | |
| | | density, 1.10938 g/ml | | | 14.87% mass conversion to oil | 16.89% C bal. adjusted | | | |
| Oil Loss in Aqueous | | | | | 50.41% C conversion to water soluble | 57.28% C bal. adjusted | | | |
| Gasification of Carbon | 9.57% | | | | 9.58% carbon conversion to gas | 10.88% C bal. adjusted | | | |
| Carbon loss in solids | | 0.14 g/hr | | | 0.20% % carbon loss in solids | 0.22% C bal. adjusted | | | |
| Space velocity | 1.47 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | -0.14 g/hr | | -1 L/L | | -1 nM3/tonne | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 9.63% | | | | | | | | |
| O in Dry Product | 2.95 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 27.18 g/hr | | | | | | | | |
| O in Dry Feed | 87.52 g/hr | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | | |
|---|---|---|---|----------------------------|--|--------------------------------------|---|---------------------------------------|---|---|
| Run No. | HTL-22 | | | | reactor volume | 1000 total mL | | | | |
| Pressure | 20.8 MPa 3001 psig | CSTR filter | 314 350 331 | inlet-bottom outlet-top | | 1000 mL at temperature | | | | |
| Total Feed | 10110 cc | Feed rate | 1473 cc/hr | | | | | | | |
| corn stover slurry w/HTL22 recycle water w/100g (1 wt%) sodium carbonate added | | | 1487.73 g/hr | | 288 g, total oil 9766.5 g, total aqueous | blowdown blowdown | 30.40% % dry solids 26.5 g/hr | | | |
| Total Product | 10054.5 g | | | Product oil | 42.67 g/hr | sum of two phases | 1489.57 g/hr | | | |
| Blowdown | 179 g | | | Product aqueous | 1446.9 g/hr | | | | | |
| | | | | dry blowdown | 8.06 g/hr | | 12.42 in CS | | | |
| Elemental Analyses | C feed product oil aqueous dry rinsed solids oil, dry basis | H 36.49% 7.70% 2.89% 0.13% | O 4.33% 7.70% 10.06% 0.82% 7.52% | | 1.01 1.109 1 | moisture density@25C | N 85.65% 9.16% 96.00% 0.16% | S 0.58% 1.39% <0.05 0.16% | ash 22.89% 0.028% 0.057% 83.61% | 101.65% 97.81% 100.04% 100.02% |
| | | | | | | | 1.56% | 0.06% | | |
| Material Balance | q C/hr feed product oil aqueous gas blowdown | q H/hr 77.90 29.31 41.74 7.22 0.74 | q O/hr 152.07 3.28 145.49 0.27 0.07 | | Total 1211.16 8.52 1260.25 18.20 0.51 | | TAN 1441.14 | Viscosity 166,800 | COD pH = 4.7->5.4 | 77.700 |
| | | | | | | | | | | |
| Elemental Balance | 101% | 98% | 106% | | | | | | | |
| Ash Balance | >>>>> >>>>>> >>>>>> | | | | | | 13.78% (solids only without liquids) | | | |
| Total Material Balance | >>>>> >>>>>> >>>>>> | | | | | | 105% | | | |
| GAS CALCULATIONS | | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | | 0.00 | q H/hr | | | | |
| Gas Out | 231.4 L/hr | | | | 9.64 moles/hr | | | | | |
| Gas Composition | moles/hr | | | | | | | | | |
| volume% | C Hydrogen CarbDioxide CarbMonoxide Methane Ethane/ethylene Propane Butanes Pentanes Higher HC (C7H14) | H 0.83% 5.90% 0.00% 0.15% 0.09% 0.00% 0.00% 0.00% 0.00% 6.97% | O 0.16 1.14 0.00 0.06 0.05 0.00 0.00 0.00 0.00 | | 0.16 gH/hr | trace | | | | |
| | | | | | | | | | | |
| Total Gas, C1-C4 | 7.22 | 0.11 | 18.20 | | 25.53 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | | 0.00 | Total gas oil | | | | |
| | 7.22 | 0.11 | 18.20 | | 25.54 | Total gas mass out | | | | |
| Yields | | | | | | | | | | |
| Oil Product Yield | 38.76 g/hr | | 34.94 ml/hr | | 0.02 L/L feed | 37.63% carbon conversion to oil | 37.10% C bal. adjusted | | | |
| | | | density, 1.10938 g/ml | | | 18.16% mass conversion to oil | 17.90% C bal. adjusted | | | |
| Oil Loss in Aqueous | | | | | | 53.58% C conversion to water soluble | 52.83% C bal. adjusted | | | |
| Gasification of Carbon | 9.27% | | | | | 9.27% carbon conversion to gas | 9.14% C bal. adjusted | | | |
| Carbon loss in solids | | 0.74 g/hr | | | | 0.94% % carbon loss in solids | 0.93% C bal. adjusted | | | |
| | | | | | | | | | | |
| Space velocity | 1.47 L/L/hr | LHSV | | | | | | | | |
| | | | | | | | | | | |
| Chemical Hydrogen Consumption | | -0.23 g/hr | | -2 L/L | | -2 nm3/tonne | | | | |
| | | | | | | | | | | |
| Calculation of Deoxygenation | | | | | | | | | | |
| O content of dry product | 13.02% | | | | | | | | | |
| O in Dry Product | 5.05 g/hr | | | | | | | | | |
| O in Organics(H ₂ O) | 25.56 g/hr | | | | | | | | | |
| O in Dry Feed | 79.76 g/hr | | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|---|-----------------------|----------------|-----------------------|-------------------------------------|-------------------------------------|------------------------|-------------------|---------------------|---------|
| Run No. | HTL-22 | | | reactor volume | 1000 | total mL | | | |
| Pressure | 21.2 MPa 3057 psig | CSTR filter | 315 352 330 | inlet-bottom outlet-top | 1000 | mL at temperature | | | |
| | | | | | Time | 11:00-14:00 | | | |
| | | | | | Date | 6-Aug-09 | | | |
| Total Feed | 4140 cc | Feed rate | 1380 cc/hr | | | | | | |
| corn stover slurry w/HTL22 recycle water | | | 1393.80 g/hr | | 114 g, total oil | | blowdown | 32.70% g dry solids | |
| no sodium carbonate added | | | | | 3364 g, total aqueous | | blowdown | 10.55% g/hr | |
| Total Product | 3478 g | | | Product oil | 38 g/hr | | sum of two phases | 1159 g/hr | |
| Blowdown | 31.666 g | | | Product aqueous | 1121 g/hr | | | | |
| | | | | dry blowdown | 3.45 g/hr | | | | |
| | | | | moisture | | | 12.42 in CS | | |
| Elemental Analyses | C | H | O | density@25C | N | S | ash | | |
| based on HTL22b feed | | | | | | | | | |
| feed | 56.89% | 4.33% | 37.36% | 1.01 | 55.65% | 0.59% | 0.028% | 22.69% | 101.65% |
| product oil | 73.32% | 7.94% | 17.64% | 1.109 | 9.40% | 1.46% | 0.063% | | 100.42% |
| oxygen by difference | 2.93% | 9.87% | 87.20% | 1.0 est | 96.00% | <0.05 | | | 100.00% |
| oxygen by difference | 9.99% | 0.88% | 4.40% | | 0.17% | 84.62% | | | 100.05% |
| oil, dry basis | 80.56% | 7.56% | 10.21% | | 1.60% | 0.07% | | | |
| Material Balance | q C/hr | q H/hr | q O/hr | Total | TAN | Viscosity | COD | | |
| feed | 72.98 | 142.47 | 1134.69 | 1350.15 | | | | | |
| product oil | 27.86 | 3.02 | 6.70 | | | | | | |
| aqueous | 32.85 | 110.64 | 977.51 | | pH = 5.04 | | 82.533 | | |
| gas | 4.83 | 0.26 | 11.96 | | | | | | |
| blowdown | 0.34 | 0.03 | 0.15 | | | | | | |
| Total Products | 65.88 | 113.95 | 996.32 | 1176.16 | | | | | |
| Elemental Balance | 90% | 80% | 88% | | | | | | |
| Ash Balance | >>>>> | >>>>>> | >>>>>> | >>>>>> | 6.38% (solids only without liquids) | | | | |
| Total Material Balance | >>>>> | >>>>> | >>>>> | >>>>> | 87% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | 0.00 | moles/hr | 0.00 | q H/hr | | | | |
| Gas Out | 179 L/hr | 7.46 | moles/hr | | | | | | |
| Gas Composition | moles/hr | | | | | | | | |
| volume% | C | H | O | | | | | | |
| Hydrogen | 1.05% | 0.16 | | 0.16 qH/hr | trace | | | | |
| CarbDioxide | 5.01% | 0.37 | 0.75 | | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.20% | 0.01 | 0.06 | trace | trace | | | | |
| Ethane/ethylene | 0.09% | 0.01 | 0.04 | trace | trace | | | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | 2.94 | | | | | |
| | 6.35% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 4.83 | 0.10 | 11.96 | 16.89 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 4.83 | 0.10 | 11.96 | 16.89 | Total gas mass out | | | | |
| Yields | | | | 38.18% carbon conversion to oil | 42.29% C bal. adjusted | | | | |
| Oil Product Yield | 34.43 g/hr | 31.03 ml/hr | density, 1.10938 g/ml | 0.02 L/L feed | 17.21% mass conversion to oil | 19.07% C bal. adjusted | | | |
| | | | | | | | | | |
| Oil Loss in Aqueous | | | | 45.00% C conversion to water solubl | 49.85% C bal. adjusted | | | | |
| Gasification of Carbon | 6.62% | | | 6.62% carbon conversion to gas | 7.34% C bal. adjusted | | | | |
| Carbon loss in solids | | 0.34 g/hr | | 0.47% carbon loss in solids | 0.52% C bal. adjusted | | | | |
| Space velocity | 1.38 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | -0.24 g/hr | -2 L/L | | -2 nM3/tonne | | | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 10.25% | | | | | | | | |
| O in Dry Product | 3.53 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 20.93 g/hr | | | | | | | | |
| O in Dry Feed | 74.72 g/hr | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|--|--|---|--|--|-------------------------------|---------------------------|--|--|
| Run No. | HTL-23 | | | reactor volume | 1000 total mL | | | | |
| Pressure | 19.9 MPa 2877 psig | CSTR filter | 313 inlet-bottom 351 outlet-top 329 | | 1000 mL at temperature | | | | |
| Total Feed | 16740 cc | Feed rate | 1488 cc/hr | | | | | | |
| corn stover slurry #3 w/1% sodium carbonate | | | 1502.88 g/hr | 258 g, total oil | blowdown | 22.41% % dry solids | | | |
| Total Product (less 1 sample) | 14718 g 735 g | | | 14460 g, total aqueous Product oil Product aqueous dry blowdown density@25C | 24.57 g/hr 1377 g/hr 14.64 g/hr moisture | blowdown sum of two phases | 65.33 g/hr 1401.7 g/hr | | |
| Elemental Analyses | C feed product oil aqueous dry rinsed solids oil, dry basis | H 36.49% 67.61% 1.75% 9.45% 77.37% | O 4.33% 8.58% 10.21% 1.14% 8.17% 12.80% 1.01 1.109 1 1.36% 0.10% | N 90.30% 12.80% 1.19% 96.00% 0.19% 0.021% 0.50% 0.086% 0.2% 12.42 in CS 101.65% 100.19% 99.96% 83.18% 107.64% | S 0.028% 0.086% <0.05 0.085% 0.19% 0.021% 24.89% 0.2% 10.24% 101.65% 100.19% 99.96% 83.18% 107.64% | ash | | | |
| Material Balance | q C/hr feed product oil aqueous gas blowdown | q H/hr 158.43 2.11 5.54 140.59 1211.76 0.17 2.00 | q O/hr 1259.44 1471.06 34.8 1425.21 | Total 1471.06 34.8 1425.21 | TAN 113.600 | Viscosity pH = 5.0 | COD 53.950 | | |
| Elemental Balance | 90% 90% 98% | | | | | | | | |
| Ash Balance | >>>>> | >>>>>> | >>>>>> | >>>>>> | 36.49% (solids only without liquids) | | | | |
| Total Material Balance | >>>>> | >>>>>> | >>>>>> | >>>>>> | 97% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | | 0.00 g H/hr | | | | |
| Gas Out | 227.3 L/hr | | 9.47 moles/hr | | | | | | |
| Gas Composition | | | moles/hr | | | | | | |
| | volume% | C 0.44% 4.92% 0.00% 0.13% 0.06% 0.00% 0.00% 0.00% 5.55% | H 0.08 0.47 0.00 0.05 0.01 0.03 0.00 0.00 0.00 | O 0.93 0.00 0.00 0.00 trace trace trace trace 2.94 | 0.08 qH/hr | trace | | | |
| | | C q/hr 5.88 0.00 | H q/hr 0.08 14.90 0.00 | O q/hr 14.90 0.00 | 20.87 Total gas 0.00 Total gas oil | | | | |
| | | 5.88 0.09 | 14.90 | 20.87 | Total gas mass out | | | | |
| Yields | | | | | | | | | |
| Oil Product Yield | 21.43 g/hr density, 1.10938 g/ml | | 19.31 ml/hr | | 31.23% carbon conversion to oil 0.01 L/L feed | 34.63% C bal. adjusted | | | |
| | | | | | 14.70% mass conversion to oil | 16.30% C bal. adjusted | | | |
| Oil Loss in Aqueous | | | | | 45.30% C conversion to water soluble | 50.23% C bal. adjusted | | | |
| Gasification of Carbon | 11.06% | | | | 11.06% carbon conversion to gas | 12.26% C bal. adjusted | | | |
| Carbon loss in solids | | 1.38 g/hr | | | 2.60% carbon loss in solids | 2.88% C bal. adjusted | | | |
| Space velocity | 1.49 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | -0.28 g/hr | | -2 L/L | | -2 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 12.81% | | | | | | | | |
| O in Dry Product | 2.75 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 36.72 g/hr | | | | | | | | |
| O in Dry Feed | 54.46 g/hr | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | | | | | | |
|--|---------------------------|------------------------|---|---|--|---------------------------------|------------------------|--------------------------|---|
| Run No. | HTL-23 | | | | reactor volume | 1000 total mL | | | |
| Pressure | 20.1 MPa 2900 psig | CSTR CSTR filter | 312 inlet-bottom 351 outlet-top 328 | | Time | 06:45:14:15 | 1000 mL at temperature | | |
| Total Feed | 11220 cc | Feed rate | 1496 cc/hr | | | | | | |
| corn stover slurry #5 w/1% sodium carbonate | | | 1510.96 g/hr | 218 g, total oil 9815 g, total aqueous | blowdown blowdown | 24.35% dry solids 107.6 g/hr | | | |
| Total Product | 10033 g 807 g | | | Product oil Product aqueous dry blowdown density@25C | 29.07 g/hr 1308.7 g/hr 26.20 g/hr | sum of two phases | 1337.77 g/hr | | |
| Blowdown | | | | moisture | | | 12.42 in CS | | |
| Elemental Analyses | C based on HTL22b feed | H | O | N oxygen by difference | S feed and product densities based on earlier measurements | ash | | | |
| feed | 36.49% | 4.33% | 37.36% | 1.01 | 87.90% 1.109 | 0.58% 9.35% 1 | 0.028% <0.05 | 21.89% 0.1% 77.31% | 101.65% 97.71% 100.65% 112.15% |
| product oil | 67.68% | 8.62% | 20.18% | | | | | | |
| aqueous | 2.44% | 10.21% | 88.00% | | | | | | |
| dry rinsed solids | 13.85% | 1.64% | 19.13% | | | | | | |
| oil, dry basis | 76.60% | 8.57% | 13.44% | | | | | | |
| | | | | estimated | | | | | |
| Material Balance | q C/hr | q H/hr | q O/hr | | Total | TAN | Viscosity | COD | |
| feed | 66.71 | 156.79 | 1247.56 | | 1471.06 | | | 165.230 | |
| product oil | 19.67 | 2.51 | 5.87 | | | 24.4 | | | |
| aqueous | 31.93 | 133.55 | 1151.66 | | | pH = 5.5 | | 72.500 | |
| gas | 4.98 | 0.11 | 13.07 | | | | | | |
| blowdown | 3.63 | 0.43 | 5.01 | | | | | | |
| Total Products | 60.21 | 136.60 | 1175.60 | | 1372.42 | | | | |
| Elemental Balance | 90% | 87% | 94% | | | | | | |
| Ash Balance | >>>>> | >>>>> | >>>>> | >>>>> | 48.39% (solids only without liquids) | | | | |
| Total Material Balance | >>>>> | >>>>> | >>>>> | >>>>> | 93% | | | | |
| GAS CALCULATIONS | | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | | 0.00 q H/hr | | | | |
| Gas Out | 218.8 L/hr | | 9.12 moles/hr | | | | | | |
| Gas Composition | | | moles/hr | | | | | | |
| | volume% | C | H | O | | | | | |
| Hydrogen | 0.52% | | 0.09 | | 0.10 qH/hr | trace | | | |
| CarbDioxide | 4.48% | 0.41 | | 0.82 | | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | | |
| Methane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Ethane/ethylene | 0.03% | 0.01 | 0.02 | trace | | trace | | | |
| Propane | 0.00% | 0.00 | 0.00 | trace | | trace | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | | 2.94 | | | | |
| | 5.03% | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | | |
| Total Gas, C1-C4 | 4.98 | 0.02 | 13.07 | 18.07 | Total gas | | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | | |
| | 4.98 | 0.02 | 13.07 | 18.07 | Total gas mass out | | | | |
| Yields | | | | | 29.49% carbon conversion to oil | 32.67% C bal. adjusted | | | |
| Oil Product Yield | 26.35 g/hr | | 23.75 ml/hr | | 0.02 L/L feed | | | | |
| | | density | 1.10938 g/ml | | 14.41% mass conversion to oil | 15.97% C bal. adjusted | | | |
| Oil Loss in Aqueous | | | | | 47.86% C conversion to water soluble | 53.03% C bal. adjusted | | | |
| Gasification of Carbon | 7.46% | | | | 7.47% carbon conversion to gas | 8.27% C bal. adjusted | | | |
| Carbon loss in solids | | 3.63 g/hr | | | 5.44% carbon loss in solids | 6.02% C bal. adjusted | | | |
| Space velocity | 1.50 L/L/hr | LHSV | | | | | | | |
| Chemical Hydrogen Consumption | | -0.60 g/hr | | -4 L/L | | -4 nM3/tonne | | | |
| Calculation of Deoxygenation | | | | | | | | | |
| O content of dry product | 13.09% | | | | | | | | |
| O in Dry Product | 3.45 g/hr | | | | | | | | |
| O in Organics(H ₂ O) | 34.90 g/hr | | | | | | | | |
| O in Dry Feed | 68.30 g/hr | | | | | | | | |

| HYDROTHERMAL LIQUEFACTION CALCULATION SHEET | | | | reactor volume | 1000 mL | total mL | | |
|--|-----------------------|-----------------------|---------------|------------------------------------|--------------------------------------|---------------------|----------------|---------|
| Run No. | HTL-23 | CSTR | 310 | inlet-bottom | 1000 | 1000 mL | at temperature | |
| Pressure | 20.0 MPa 2896 psig | CSTR filter | 351 327 | outlet-top | Time | 04:30:17:45 | | |
| | | | | | Date | 11 Sep 09 | | |
| Total Feed | 19790 cc | Feed rate | 1494 cc/hr | | | | | |
| corn stover slurry #7 w/1% sodium carbonate | | | 1508.94 g/hr | 430 g, total oil | blowdown | 33.59% % dry solids | | |
| Total Product | 19226.5 g | Product oil | 35.83 g/hr | 18796.5 g, total aqueous | blowdown | 65.36 g/hr | | |
| Blowdown | 915 g | Product aqueous | 1409.6 g/hr | sum of two phases | 1451.1 g/hr | | | |
| | | dry blowdown | 21.96 g/hr | | | | | |
| | | density@25°C | moisture | | | | | |
| Elemental Analyses | C | H | O | N | S | ash | | |
| based on HTL22b feed | | | | | | | | |
| feed | 36.49% | 4.33% | 37.36% | 1.01 | 87.10% | 0.58% | 0.628% | 22.8% |
| product oil | 71.17% | 8.26% | 19.93% | 1.109 | 10.60% | 1.30% | 0.075% | 0.2% |
| oxygen by difference | 2.73% | 10.19% | 87.10% | 1 | 96.00% | <0.05 | 0.006% | 100.03% |
| aqueous | | | | | | | | |
| dry rinsed solid | 8.52% | 1.00% | 10.40% | | | 0.17% | 0.011% | 84.11% |
| oil, dry basis | 78.79% | 7.82% | 11.64% | | | | | 104.20% |
| | | | | | | | | |
| | | | | | | | | |
| Material Balance | q C/hr | q H/hr | q O/hr | Total | TAN | Viscosity | COD | |
| | feed | 71.03 | 155.75 | 1239.68 | | | 174,200 | |
| | product oil | 25.50 | 2.96 | 7.14 | | 29.7 | | |
| | aqueous | 38.48 | 143.64 | 1227.76 | | | 77,560 | |
| | gas | 6.12 | 0.20 | 15.31 | | | | |
| | blowdown | 1.87 | 0.22 | 2.28 | | | | |
| | Total Products | 71.97 | 147.02 | 1252.50 | | 1471.49 | | |
| | | | | | | | | |
| | | | | | | | | |
| Elemental Balance | 101% | 94% | 101% | | | | | |
| Ash Balance | >>>>> | >>>>>> | >>>>> | >>>>>> | 41.45% (solids only without liquids) | | | |
| Total Material Balance | >>>>> | >>>>>> | >>>>> | >>>>>> | 100% | | | |
| | | | | | | | | |
| GAS CALCULATIONS | | | | | | | | |
| Gas In | 0 L/hr | | 0.00 moles/hr | 0.00 | q H/hr | | | |
| Gas Out | 214.4 L/hr | | 8.93 moles/hr | | | | | |
| | | | | | | | | |
| Gas Composition | moles/hr | | | | | | | |
| | volume% | C | H | O | | | | |
| Hydrogen | 0.50% | 0.09 | | 0.09 | qH/hr | trace | | |
| CarbDioxide | 5.36% | 0.48 | | 0.96 | | | | |
| CarbMonoxide | 0.00% | 0.00 | 0.00 | 0.00 | | | | |
| Methane | 0.18% | 0.02 | 0.06 | | trace | trace | | |
| Ethane/ethylene | 0.08% | 0.01 | 0.04 | | trace | trace | | |
| Propane | 0.00% | 0.00 | 0.00 | | | | | |
| Butanes | 0.00% | 0.00 | 0.00 | | | | | |
| Pentanes | 0.00% | 0.00 | 0.00 | | | | | |
| Higher HC (C7H14) | 0.00% | 0.00 | 0.00 | | | 2.94 | | |
| | 6.12% | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | C g/hr | H g/hr | O g/hr | | | | | |
| Total Gas, C1-C4 | 6.12 | 0.11 | 15.31 | 21.54 | Total gas | | | |
| Total Gas Oil, C5-C7 | 0.00 | 0.00 | 0.00 | 0.00 | Total gas oil | | | |
| | 6.12 | 0.11 | 15.31 | 21.55 | Total gas mass out | | | |
| | | | | | | | | |
| Yields | | | | 35.90% carbon conversion to oil | 35.43% C bal. adjusted | | | |
| Oil Product Yield | 32.03 g/hr | | 28.87 ml/hr | 0.02 L/L feed | | | | |
| | | density, 1.10938 g/ml | | 16.46% mass conversion to oil | 16.24% C bal. adjusted | | | |
| | | | | | | | | |
| Oil Loss in Aqueous | | | | 54.18% C conversion to water solub | 53.47% C bal. adjusted | | | |
| Gasification of Carbon | 8.62% | | | 8.62% carbon conversion to gas | 8.51% C bal. adjusted | | | |
| Carbon loss in solids | | 1.87 g/hr | | 2.63% C carbon loss in solids | 2.60% C bal. adjusted | | | |
| | | | | | | | | |
| Space velocity | 1.49 L/L/hr | LHSV | | | | | | |
| | | | | | | | | |
| Chemical Hydrogen Consumption | -0.3 g/hr | | -2 L/L | -2 m3/tonne | | | | |
| | | | | | | | | |
| Calculation of Deoxygenation | | | | | | | | |
| O content of dry product | 11.75% | | | | | | | |
| O in Dry Product | 3.76 g/hr | | | | | | | |
| O in Organics(H ₂ O) | 24.90 g/hr | | | | | | | |
| O in Dry Feed | 72.72 g/hr | | | | | | | |

APPENDIX B
Correlations for Corn Stover HTL Performance

Correlation Definitions

The data for runs 17a through 20c, this is for all CSTR runs without recycle, were correlated in terms of the empirical expression:

$$\boxed{PROPERTY\# = a \cdot (LHSV)^b \cdot (BMF)^c \cdot \left(\frac{T}{300} \right)^d}$$

where:

PROPERTY # is Moisture Free Oil Yield, Water Solubles Yield, H/C Atomic ratio, O/C Atomic Ratio

LHSV is the liquid hourly space velocity in h^{-1}

BMF is the biomass to water inlet mass fraction

T is temperature in $^{\circ}C$

- Correlations were developed based on experimental Hydrothermal Upgrading (HTU) data (series 17 through 20), on a Moisture Free Basis.
- Experimental Data fit to the equation $y = a(LHSV)^b(BMF)^c (Tout/300^{\circ}C)^d$ using solver application in Excel.
- Runs 17c, 19 and 20c were excluded of the Aqueous Phase correlations because of Hydrogen balance closure; however, Oxygen concentration seems to be also affected.

Correlations-Series 17 through 20 CSTR with solids separation, no recycle

Corn Stover

Oil phase

| | Oil yield | wt% C | wt% H | wt% O |
|---|-------------|-------------|-----------|-------------|
| a | 0.799720875 | 826.3700867 | 4.0477318 | 4.093247664 |

| | | | | |
|-------------------------|--------------|--------------|--------------|--------------|
| b | 0.782901291 | 1.110112671 | -0.251848595 | -0.151057362 |
| c | -0.927041409 | 0.96833327 | -0.142373758 | -0.320426642 |
| d | 5.191310295 | -7.518533298 | 3.565760281 | 6.024895087 |
| Average relative error | 8.22% | 5.33% | 0.81% | 6.90% |
| Correlation coefficient | 0.715626111 | 0.862477212 | 0.984463039 | 0.715287204 |

Corn Stover

Aqueous phase

| | Water sol. yield | wt% C | wt% H | wt% O |
|-------------------------|------------------|--------------|--------------|--------------|
| a | 14.31367888 | 4.23218E+11 | 1.22122E+16 | 2.21081E-06 |
| b | -0.018669219 | 0.6221798 | 2.221538427 | -1.110382955 |
| c | -0.548714742 | 7.841925287 | 12.64247126 | -5.762025988 |
| d | -2.331731615 | -50.96627331 | -74.52618053 | 35.29832976 |
| Average relative error | 0.01% | 0.05% | 0.07% | 26.26% |
| Correlation coefficient | 0.999999784 | 0.99999993 | 0.999999563 | 0.883712492 |

Range of Experimental Conditions

| | LHSV | BMR | Temp (degrees C) |
|---------------|-------|-------------|------------------|
| Minimum value | 1.26 | 0.109877913 | 330 |
| Maximum value | 1.798 | 0.154734411 | 352 |

Corn Stover

Oil phase

| | H/C Ratio | O/C Ratio |
|-------------------------|--------------|--------------|
| a | 0.067836903 | 0.005750578 |
| b | -1.358147169 | -1.186200106 |
| c | -1.060251972 | -1.115042791 |
| d | 10.69267687 | 12.14759115 |
| Average relative error | 6.32% | 0.111449813 |
| Correlation coefficient | 0.938299279 | 0.824830367 |

Corn Stover

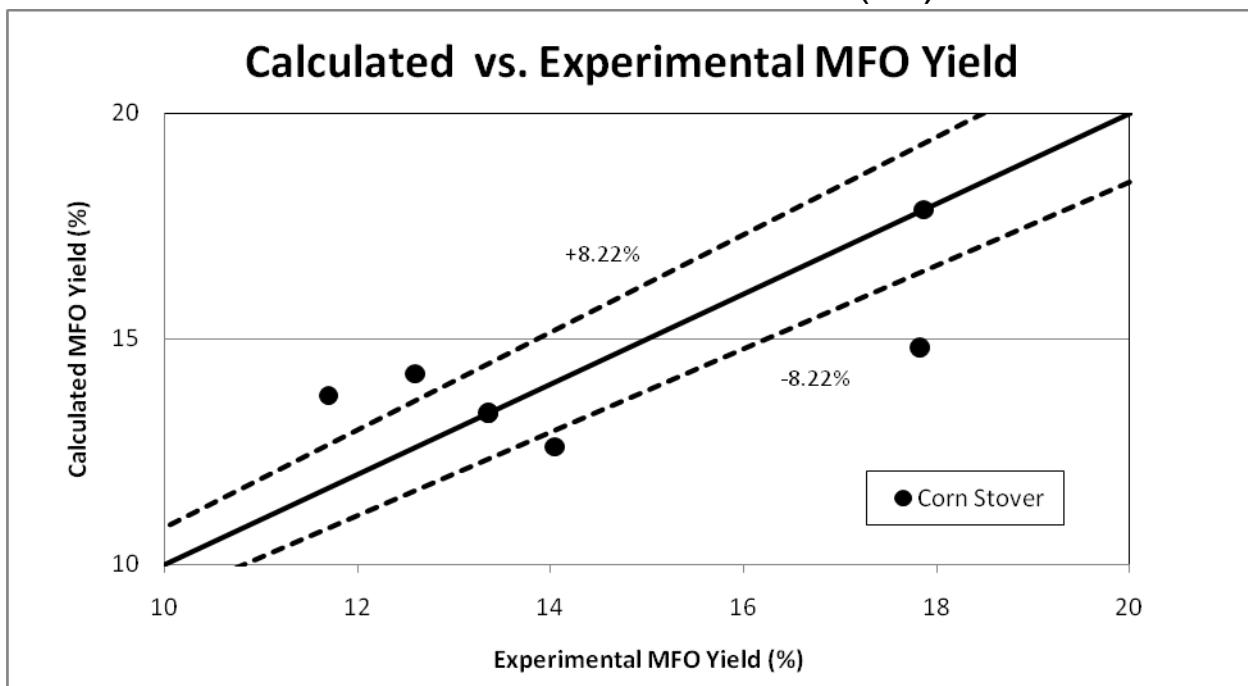
Aqueous phase

| | H/C Ratio | O/C Ratio |
|---|--------------|-------------|
| a | 342422.0312 | 1.3519E-05 |
| b | 1.599264989 | 5.802908514 |
| c | 4.799433216 | -2.61179426 |
| d | -23.55129316 | 14.81613352 |

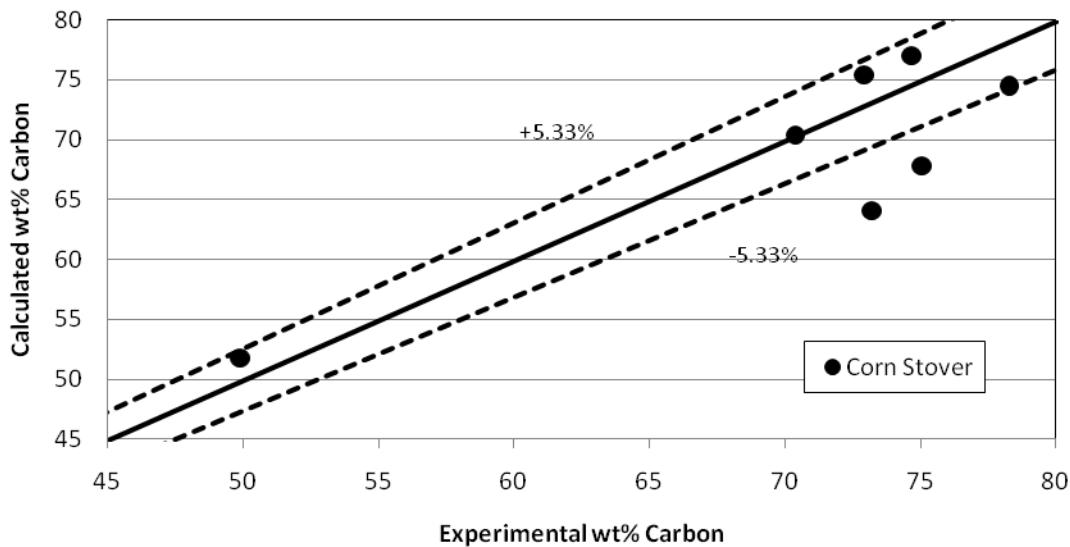
| | | |
|-------------------------|-------------|-------------|
| Average relative error | 0.000279036 | 0.275986289 |
| Correlation coefficient | 0.999999577 | 0.199329643 |

| Range of Experimental Conditions | | LHSV | BMR | Temp (degrees C) |
|----------------------------------|---------------|-------|-------------|------------------|
| | Minimum value | 1.26 | 0.109877913 | 330 |
| | Maximum value | 1.798 | 0.154734411 | 352 |

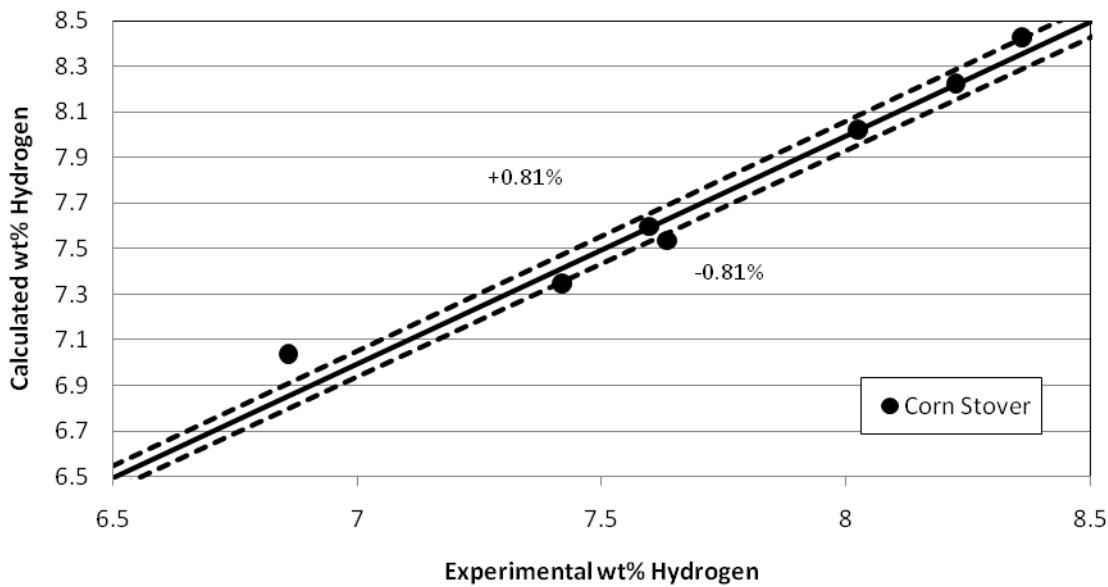
PARITY PLOTS FOR THE OIL PHASE (db)



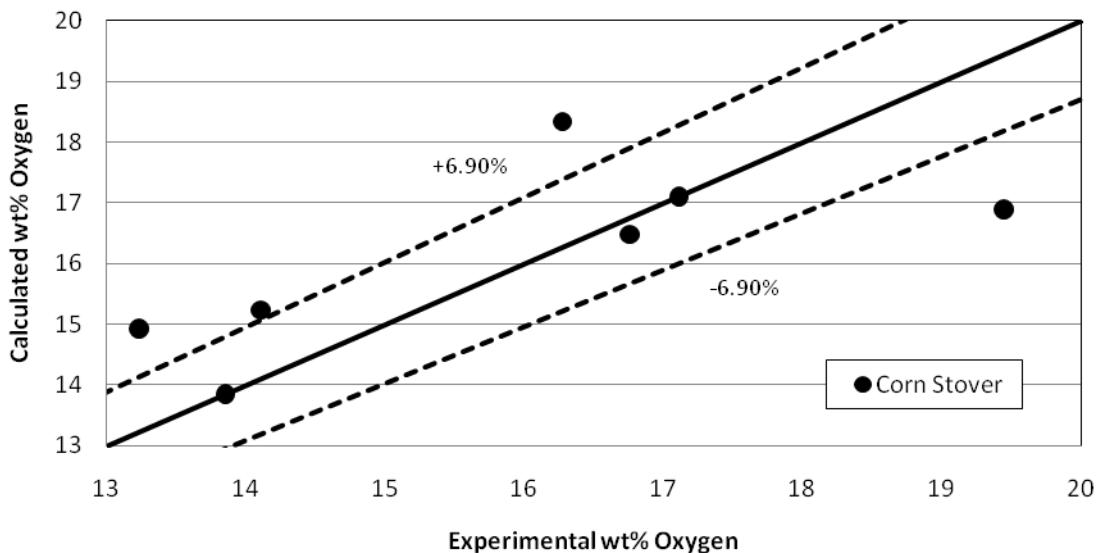
Calculated vs. Experimental wt% Carbon



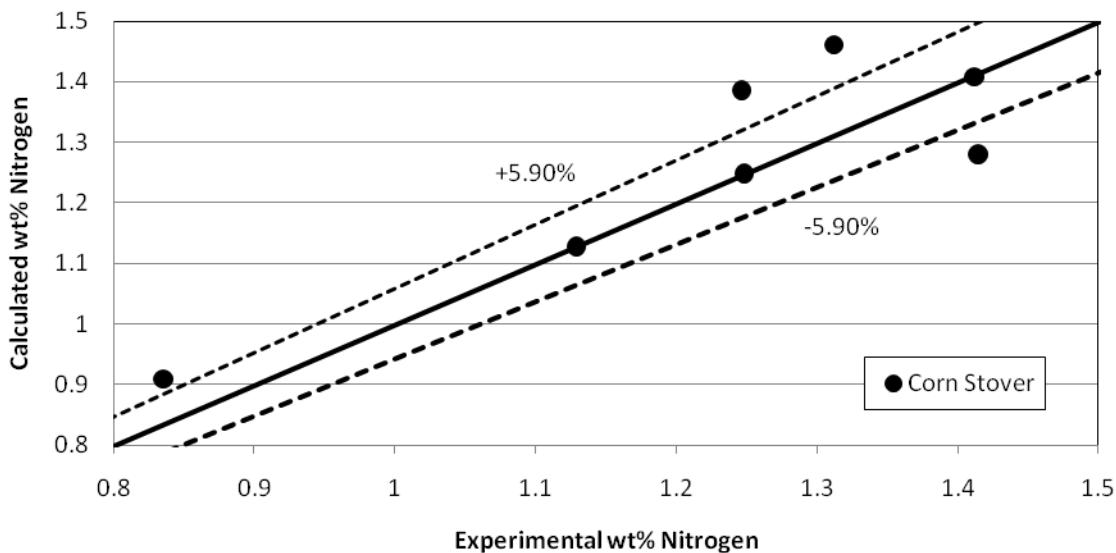
Calculated vs. Experimental wt% Hydrogen



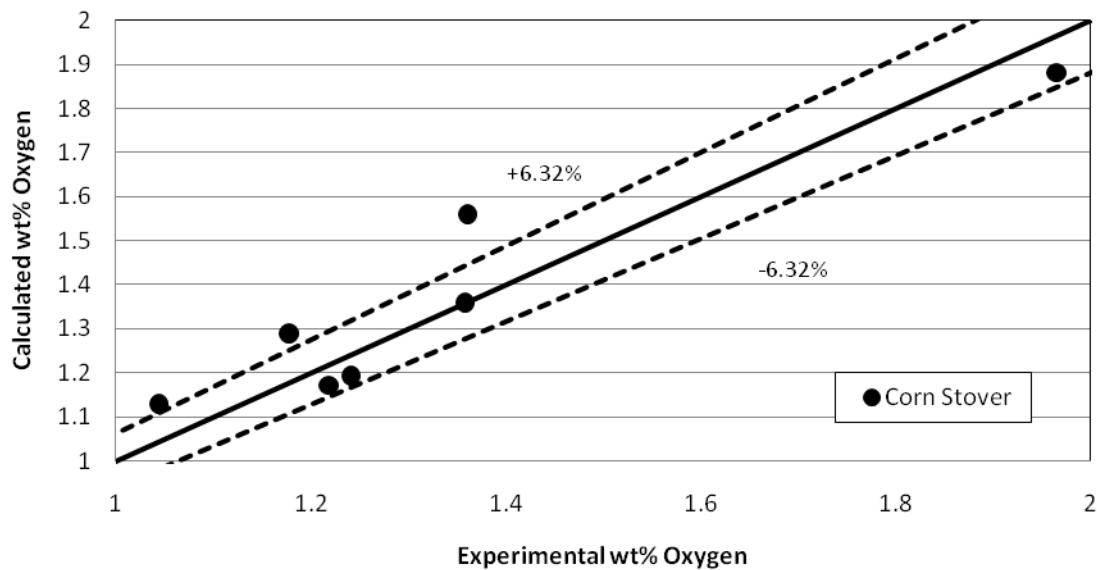
Calculated vs. Experimental wt% Oxygen



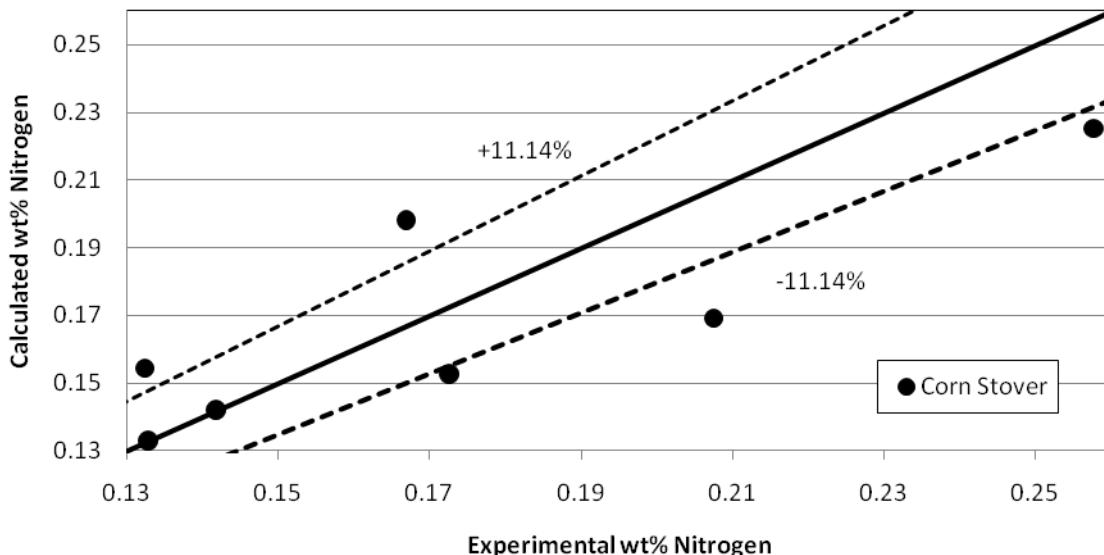
Calculated vs. Experimental wt% Nitrogen



Calculated vs. Experimental H/C Ratio



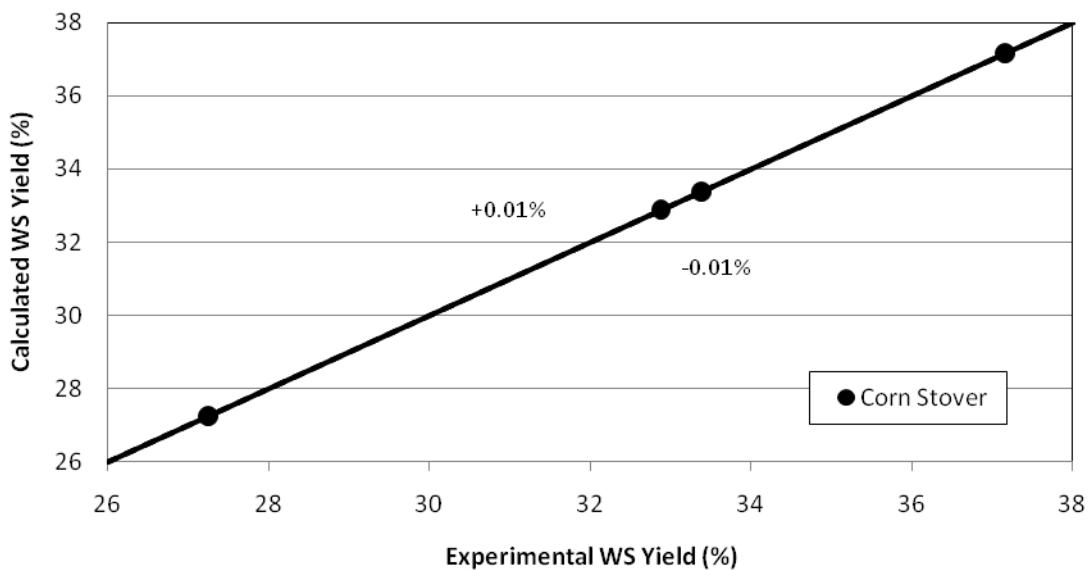
Calculated vs. Experimental O/C Ratio



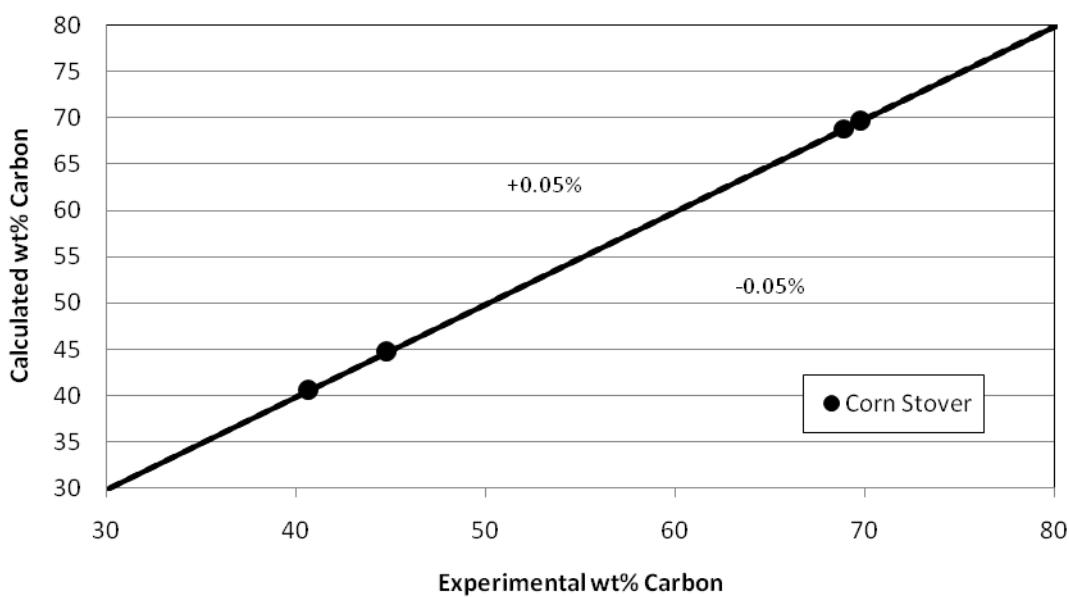
PARITY PLOTS FOR THE AQUEOUS PHASE (db)

Runs 17c, 19 and 20c were excluded of the Aqueous Phase correlations because of Hydrogen balance closure; however, Oxygen concentration seems to be also affected.

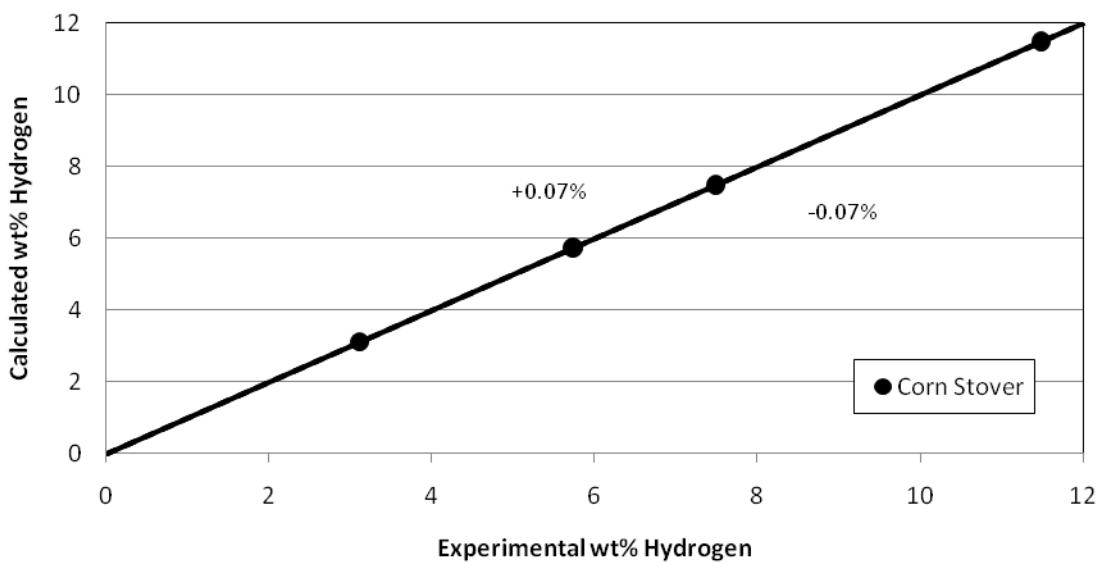
Calculated vs. Experimental WS Yield



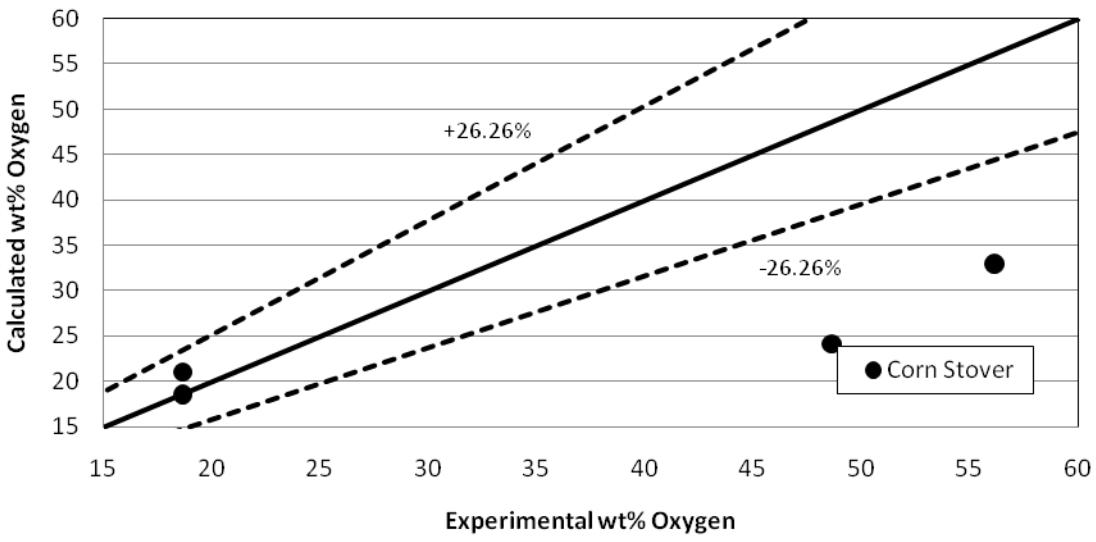
Calculated vs. Experimental wt% Carbon



Calculated vs. Experimental wt% Hydrogen



Calculated vs. Experimental wt% Oxygen



Calculated vs. Experimental H/C Ratio

