

Research Performance Final Report

Project Title: Technology for GHG Emission Reduction and Cost-Competitive Mil-Spec Jet Fuel Production using CTL

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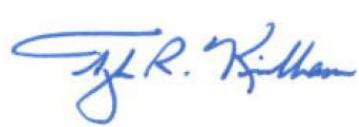
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ACCOMPLISHMENTS

Ceramatec Inc, in collaboration with IntraMicron (IM), the Energy Environmental Research Center (EERC) and Sustainable Energy Solutions, LLC (SES), have completed the thirteenth and final quarter of research towards the integration of proprietary technologies in key areas to demonstrate production of a jet fuel from coal and biomass sources.

Final Quarter efforts at Ceramatec and IM focused on the installation and operation of the nominal 2 bbl/day FT reactor and sulfur removal system skids at EERC. EERC hosted the Ceramatec and IM teams installing and operating their respective skids, operated the gasifier providing syngas to the sulfur and FT skids, and provided analytical chemistry support to the operations. SES activities were effectively complete at the submission of the previous quarterly report.

Table 1 Milestone Status Report

Milestone Title/Description	Planned Completion Date	Actual Completion Date	Verification Method	Comments (progress towards achieving milestone, explanation of deviation from plan, etc.)
Revise PMP	10/14	10/14	Approval by DOE	PMP Completed
Initial run of 4" reactor with MFEC containing catalyst on natural gas	3/15	3/15	Results contained in Q2 Report	The 4" reactor with MFEC containing catalyst was delivered by IntraMicron. Ceramatec performed the initial run of this reactor.
Design criteria for multi-tube reactor completed	3/15	3/15	Results contained in Q2 Report	Ceramatec performed a down select of reactor designs and produced a preliminary drawing
Initial run of gasifier on CWS at pressure of ~500 psi	6/15	Pending	Update in Q8 Report	EERC steady state operating conditions during Rosebud PRB and Biomass Blends Operation contained within this report
All component models underway and interim results reported	6/15	6/15	Results contained in Q3 Report	
Component modeling completed and integrated into overall system model	9/15	9/15	Results contained in Q4 Report	
Design drawings completed for FT reactor, sulfur clean up system, and gasifier	12/15	12/15	Results contained in Q5 Report; Updated in Q8 Report	Schematic of EERC TRDU gasifier contained in Q8 Report
P&ID, PFD, and fabrication drawings completed for all systems	3/16	3/16	Ceramatec drawings contained in	Q8 Report contains a Process Flow Diagram of EERC's gasifier unit

			Q6 report, PFD for gasifier contained in Q8 Report	
Multi-tube FT fabricated and installed at Ceramatec	6/16	4/3/17		Reactor fabrication complete, at IntraMicron for MFEC loading. Reactor mounted in skid during March and delivered 4/3
Sulfur removal system fabricated and shipped to EERC	6/16	Nov 2017		The vessels for the sulfur removal system have been fabricated and catalyst is being loaded. The vessels will be shipped directly to EERC when ready.
Component modeling completed and integrated into overall system model	9/16	Completed	Results contained in Q9 Report	The Ceramatec and IM models are completed. EERC has been tasked to supply a gasifier system model suitable for full system model integration.
Integrated testing of sulfur removal system and FT at Ceramatec	9/16	Dec 2017		The sulfur removal system will be integrated with the gasifier at EERC
All components shipped to gasifier location and assembly of integrated system started	12/16	Nov 2017		Shipments arrived early November 2017
System assembly completed at gasifier location with cold and hot flow tests completed	3/17	Nov 2017		FT and Sulfur skid integration with EERC gasifier complete Nov 2017
Testing of integrated system initiated. Initial production of FT liquids in Jet A range, with testing with CWS and bio-based feedstock	6/17	Dec 04, 2017		FT products were analyzed by GCMS and fractionated using the EERC laboratory scale distillation facility.
Continuous run of >= 100 hours completed. 10 gallons of FT liquids in Jet A range delivered for evaluation. As built drawings prepared and issued. Cost benefit analysis completed and issued.	9/17	Dec 04-13, 2017		The integrated system was run through the planned period; however, catalyst deactivation limited the quantity of product obtained.

Major Activities

Programmatic

Ceramatec announced the sale of a portion of the company August 22, 2017 and its plans to cease operations of the remaining portion of the company by the end of 2017. This required

compression of the work planned for the last three months of the schedule extension. All teams went to great efforts to complete the project before the end of 2017. The final integrated demonstration of the gasifier, sulfur processing unit and Fischer-Tropsch synthesis system was conducted December 3-14, 2017 at the EERC in Grand Forks, North Dakota.

[7-Tube FT Reactor and Supporting System Completion and Commissioning](#)

During this final quarter; checkout of the mechanical, process units, piping, and electrical equipment was completed as shown Figure 1. The catalyst charge was reduced and a brief run on natural gas derived syngas was conducted at Ceramatec during the month of October.

Electrical control panels (main power distribution, process heaters, fan and pump variable speed drives, PID loop controllers, National Instruments PXI data system, and Unitronics PLC) were connected to process instruments and equipment and were nearing operational capability at the beginning of the quarter. Air compressor, chiller, fans and pumps as shown in Figure 2 were powered up and tested. With the exception of the two systems noted below, virtually all process equipment, piping, and electrical systems were operated successfully in the run at Ceramatec October 25-26, 2017.

It was determined that the initial runs would rely on the reactor's integral syngas preheat section rather than filling and draining the heat transfer oil for the short runs at Ceramatec before breaking down for the move to EERC. The internal syngas preheat system performed as designed in the run at Ceramatec.

The product separation system was designed around a Rosemount radar interface detection system capable of simultaneously measuring the water-oil and oil-syngas levels in the separation vessel. This instrument transmits interface level signals which are used to independently control flow of produced water and oil as required to maintain stationary interface levels in the separation vessel. The generic HART interface modem Ceramatec used to configure other control elements (control valves, Coriolis mass flow meters, etc.) was lacking specialized configuration data required to configure the Rosemount radar interface instrument. The Rosemount specific modem was ordered but was not received until just before the start of operations at EERC. Prior to reaching full operational status with this system at EERC on Dec 6, product oil and water were periodically drained and separated manually using a separatory flask.

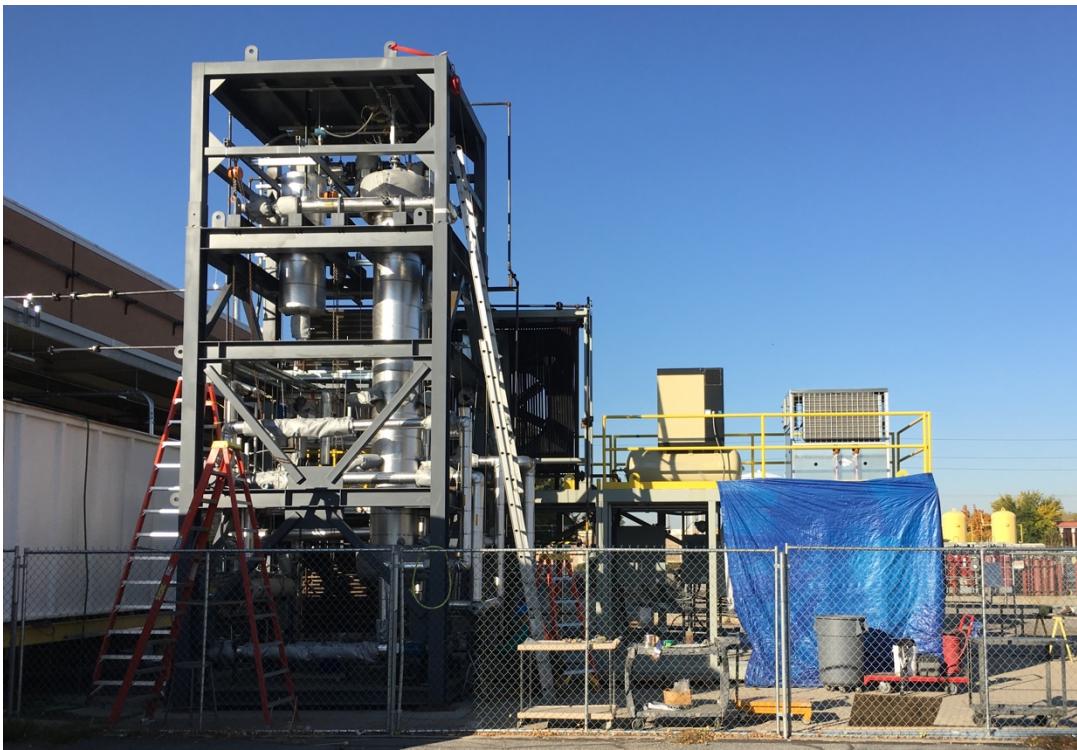


Figure 1. FT process skid assembly completion at Ceramatec September 2017.



Figure 2. Electrical completion of skids at Ceramatec

Catalyst Reduction

The 50kg catalyst charge produced at a toll manufacturing company for Chevron had been reduced and passivated at Ceramatec prior to loading in MFEC by Intramicron in 2017. The total mass of reduced and passivated catalyst loaded in MFEC by Intramicron was 35.38kg as reported in Q08 of the project. The reactor was sealed off with blind flanges after MFEC loading to protect the catalyst from contamination during transportation and installation. After completion of the process piping installation, the reactor and syngas piping was purged with nitrogen. and slowly heated using the coolant pump and circulation heater. The coolant loop uses water as the coolant. At the target re-reduction cycle maximum temperature of 260°C, the coolant pressure is just under 700 psig, safely below the vessel code stamp maximum allowable working pressure (MAWP) of 738 psig. However the steam control valve pressure rating was based on the normal operating temperature limit of 240°C, which results in a nominal steam pressure about 500 psig. During the reduction run the steam control valve was replaced with a blind flange to enable heating the reactor to 260°C. This required completely cooling the reactor after reduction in order to reinstall the steam valve. Two pressure relief valves were still in place to prevent exceeding the vessel MAWP.

The reactor reduction run was started at Ceramatec on October 20, 2017 with the reactor temperature under 100°C. Nitrogen supplied from a 3,000-gallon liquid nitrogen tank and hydrogen from two 16-tank clusters (300 SCF per tank) were fed to the reactor at rates targeting a nominal 10% hydrogen concentration. The H₂/N₂ reducing gas pressure was just a few psi, enough to allow monitoring of the tailgas using a Vaisala HTM330 dew point instrument. FT catalyst can lose surface area and resulting activity if exposed to high steam concentrations during reduction. We typically target limiting water vapor to 1% during reduction. The strategy to prevent catalyst coarsening required monitoring exit dew point and pausing the reactor temperature ramp when the dew point exceeded 4°C, and resuming the ramp once the dew point had fallen below 1°C. An initial evolution of water vapor desorbing from the zeolite catalyst support early in the run (T ~ 100°C) reached a dew point of 10°C, and for a brief period at about 11.5 hours into the run, during the actual reduction process, the dew point spiked to about 5.5°C. Neither of these events were a concern with respect to exposing the catalyst to excess steam during reduction. The reduction run dew point history is shown in Figure 3. Reduction was considered complete after reaching the target temperature of 260°C and holding until a stable dry dew point (T_{dp} < -35°C) for several hours. The reactor was then cooled below 80°C so that the steam control valve could be reinstalled.

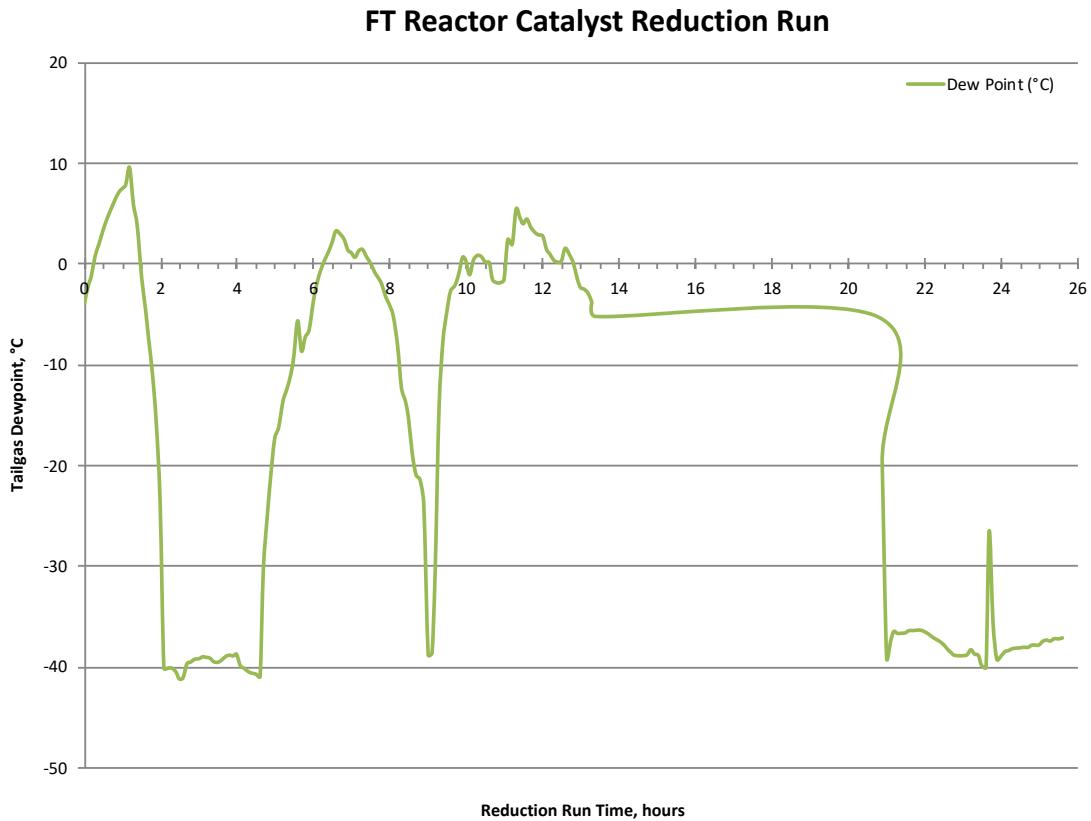


Figure 3. Dew point history during catalyst reduction at Ceramatec, Oct 20-21, 2017

Syngas generation at Ceramatec

The FT system was run at Ceramatec October 25-26, 2017 using natural gas derived syngas. A plasma catalyzed autothermal reformer and a syngas compression and storage system of sufficient capacity to supply the 2 bbl/day FT system was in place from earlier commercial FT system development projects. The reformate was delivered to a two-stage liquid cooled Blackmer HDL613 oil free gas compressor at 10-12 psig. The compressor employed a pre-cooler, interstage cooler and after cooler, each with condensate removal as shown in Figure 4. This compressor was sized to deliver reformate to a 200 psig, 240-gallon buffer tank on the suction side of a Blackmer HDL322 single stage two-cylinder liquid cooled compressor used to boost the syngas pressure to 760 psig in two 500 gallon syngas storage tanks as shown in Figure 5. During the syngas system startup, the reformer was operating at full capacity, but it was determined that the HDL613 compressor 2nd stage check valve was leaking such that it wasn't able to reach the needed 200 psig delivery pressure that triggered operation of the HDL322 final compression stage.

There was no time in the schedule to repair the leaking check valve HDL322 so rather than storing syngas at high pressure the outside syngas system was reconfigured as a direct flow through to the FT reactor. Pressure switches controlling the HDL322 were set to turn on at a suction pressure less than 150 psig (rather than 200 psig) and stay on with suction pressures above 80 psig (rather switching off when the buffer tank pressure dropped to 150 psig). The syngas exit pressure regulator was bypassed, and the FT syngas control valve was commanded to full open. The FT reactor pressure control valve still functioned normally, but the FT system took all the syngas that could be delivered at the reactor pressure.

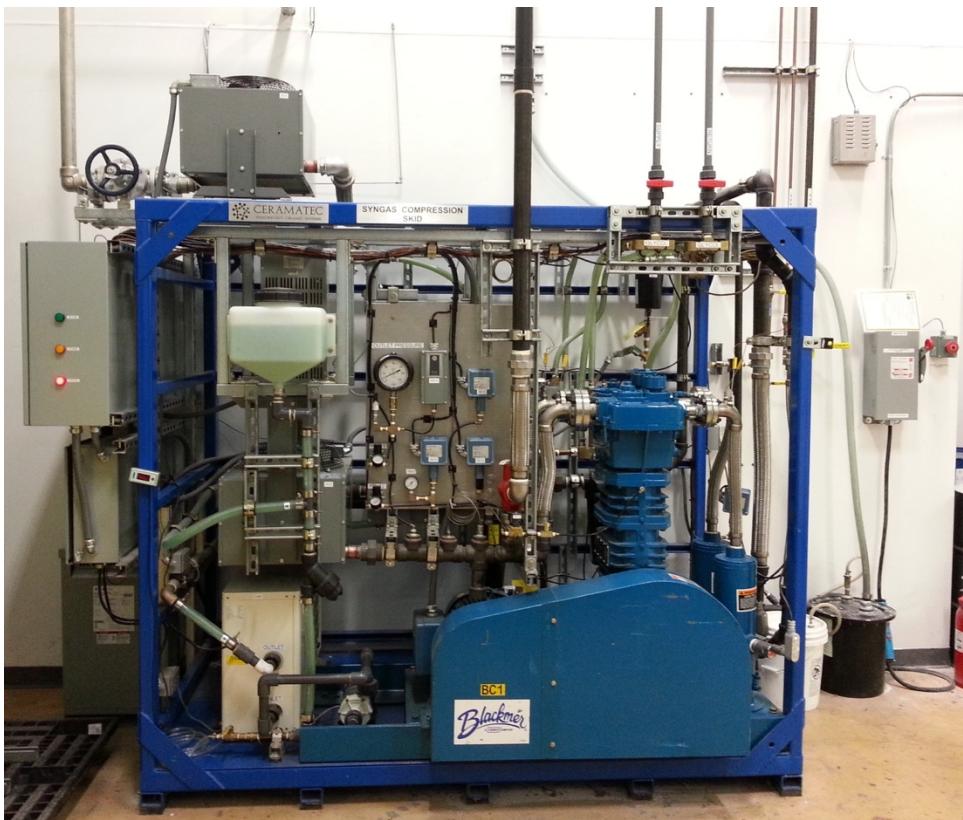


Figure 4. Ceramatec syngas compression system.



Figure 5. Ceramatec syngas compression system.

There were two issues interfering with obtaining a reliable quantitative measurement of syngas flow to the FT reactor during the Ceramatec run that were corrected for the run at EERC. The syngas fresh feed control valve had been inadvertently installed upstream of the Coriolis mass flow meter which interferes with readings due to the vibration imparting turbulence created by the valve. The other complication was conversion of the native kg/h reading to SCFM which assumed a syngas composition from Q03 testing (30 kg/h = 22 SCFM). Runs with the single tube reactor in April of 2015 producing 5-6 gal/day from a 2kg charge of the same catalyst formulation required 5-6 SCFM of fresh feed and 10 SCFM of recycle, suggest a flow rate of 70-100 SCFM depending on composition and catalyst activity. However the syngas fresh feed rate readings from the Coriolis mass flow meter were typically in the range of 1-2 SCFM, while our syngas production estimates based on reformer operating point was at least 20-30 times higher, though the flow bypassing the compressor directly to the flare was not measured.

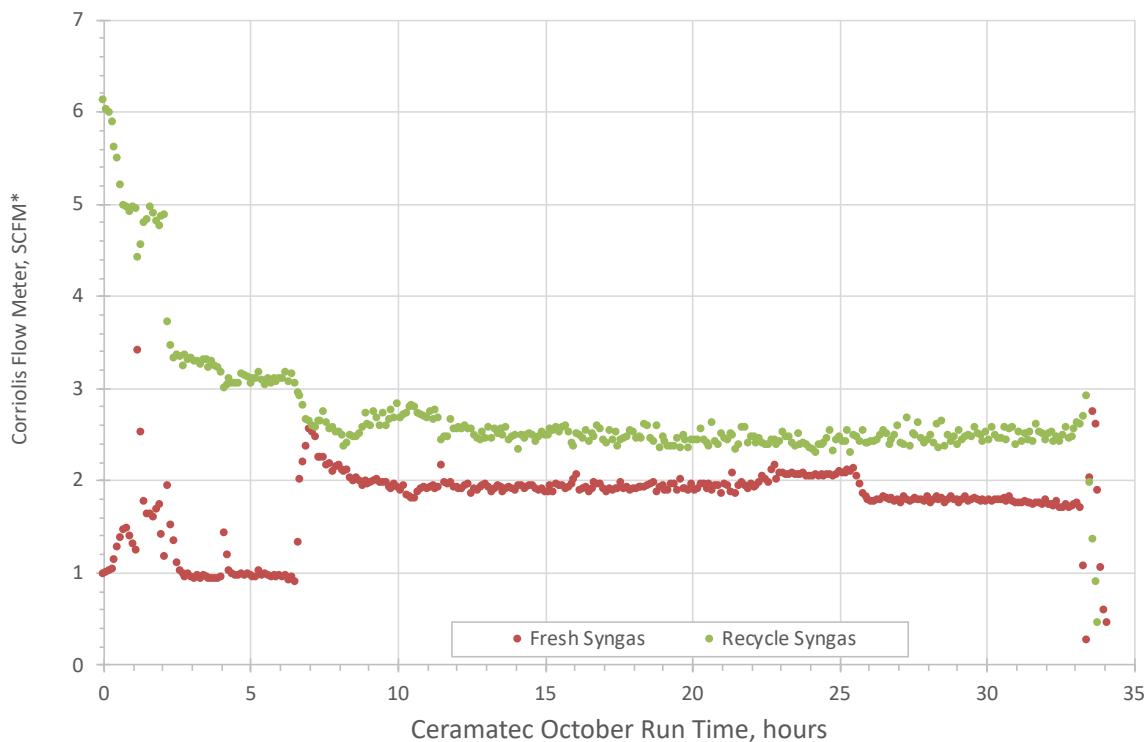


Figure 6. Ceramatec syngas feed rate readings, October 25-26, 2017.

Reformate (fresh feed) and FT tailgas composition were monitored with an Agilent 490 (aka Inficon 3000) micro GC at approximately 5-minute intervals. The GC was plumbed online with solenoid valves to select either reformer output, FT fresh feed or FT tailgas sources to the GC. The source of the readings is inferred by the composition. FT fresh feed composition is shown in Figure 7 to have approximately 30% H₂ and 16% CO. Methane at ~4% was higher than the 0.4% typical for this reformer suggesting it was due for some maintenance. Nitrogen and carbon dioxide at ~ 50% and 4%-5% respectively were within the expected range. Compared to the syngas composition provided by the gasifier at EERC, hydrogen and CO were nearly double in the Ceramatec syngas while the EERC syngas had similar N₂ concentration and an order of magnitude higher CO₂ concentration.

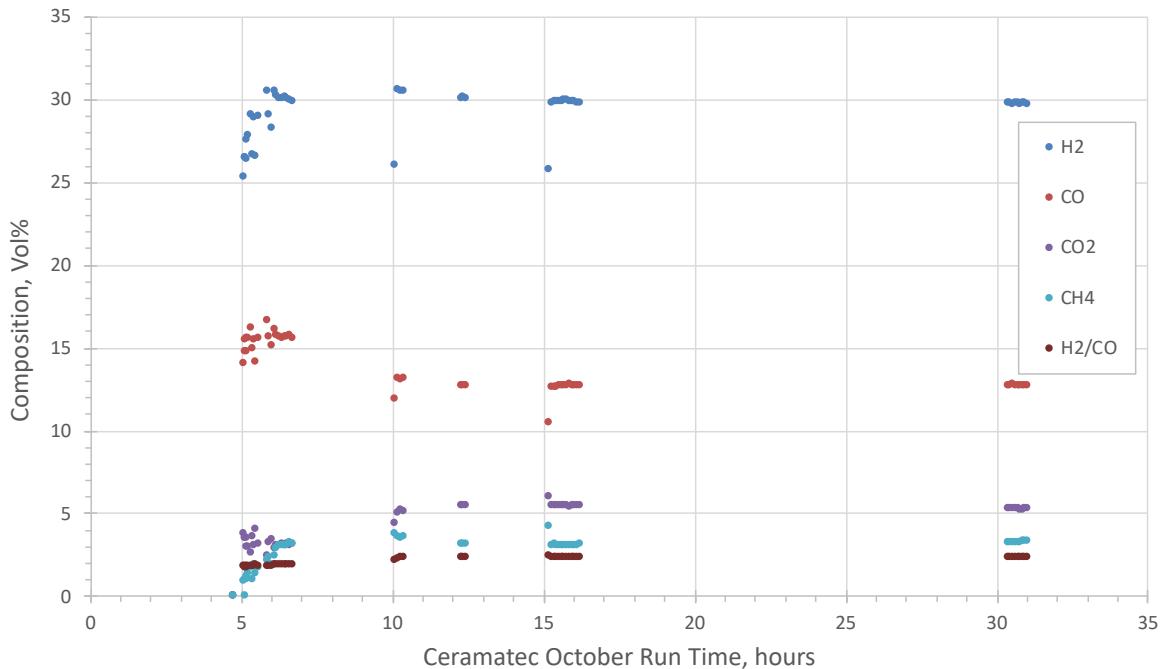


Figure 7. Ceramatec syngas feed gas composition, October 25-26, 2017.

FT system initial run at Ceramatec

The reactor was at temperature and pressure with syngas flowing for a period of about 30 hours on October 25-26, 2017 before it had to be shut down and readied for shipment to EERC on November 1. With the exceptions of the problems with the syngas compressor and fresh syngas flow measurement the system balance of plant and reactor appeared to perform as expected. The target operating temperature for this hybrid cobalt catalyst is 240°C, about 20°C higher than for typical cobalt FT catalyst. Our usual startup protocol brings pressure and temperature up from 10 bar (145 psig) and 190°C to 20 bar (290 psig) and 240°C over a period of 1-2 days. This allows time for a cautious reaction thermal stability assessment and dampening of the dry finely divided catalyst with product oil before reaching the full reaction rate at 240°C and 20 bar (190 psig). This ramp up period was shortened to a few hours as shown in Figure 8, due to the hard deadline to end the run, in order to meet the shipment schedule. However no signs of thermal instability were observed. The syngas compressor problem led us to reduce the FT exit pressure from 20 bar (290 psig) to 16 bar (232 psig) in an attempt to increase the fresh syngas flow rate. Reactor temperatures were taken as high as 230°C, but reduced to 220°C since with the limited syngas flow rate the reactor was achieving full conversion of the syngas (exit CO concentration was near zero) as seen in Figure 9.

The first oil and produced water was yellowed with rust from the carbon steel reactor and process piping, but was observed to clear up with time as the rust settled out of samples and was flushed from the piping. A light wax precipitate was seen in the clear oil as the sample sat in a cool room. Samples of the first oil are shown in Figure 10. On disassembly in preparation for shipping some slugs of white FT wax were seen at low points in the recycle piping. This hybrid catalyst is wax free when operated at the nominal 240°C reactor temperature. Seeing some wax was not a surprise given the fact that the reactor was never allowed to reached full operating temperature in the initial run as a result of time and syngas supply limitations.

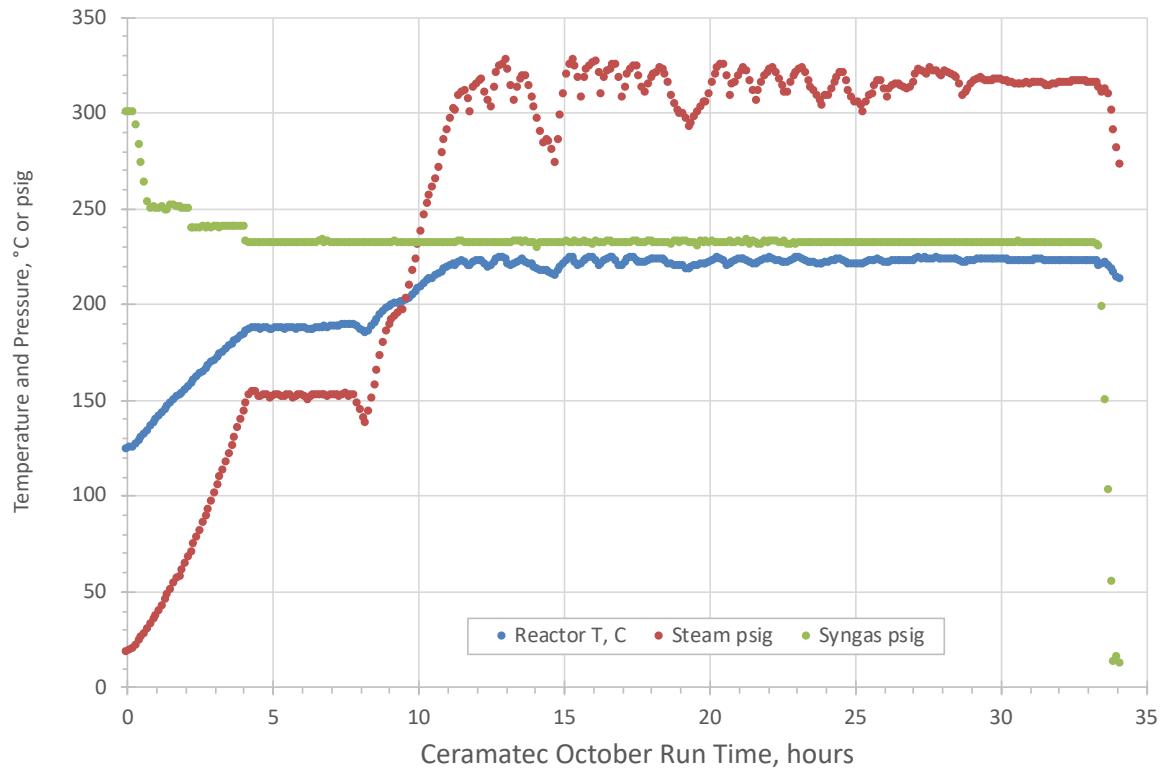


Figure 8. FT reactor temperatures and pressures at Ceramatec, October 25-26, 2017.

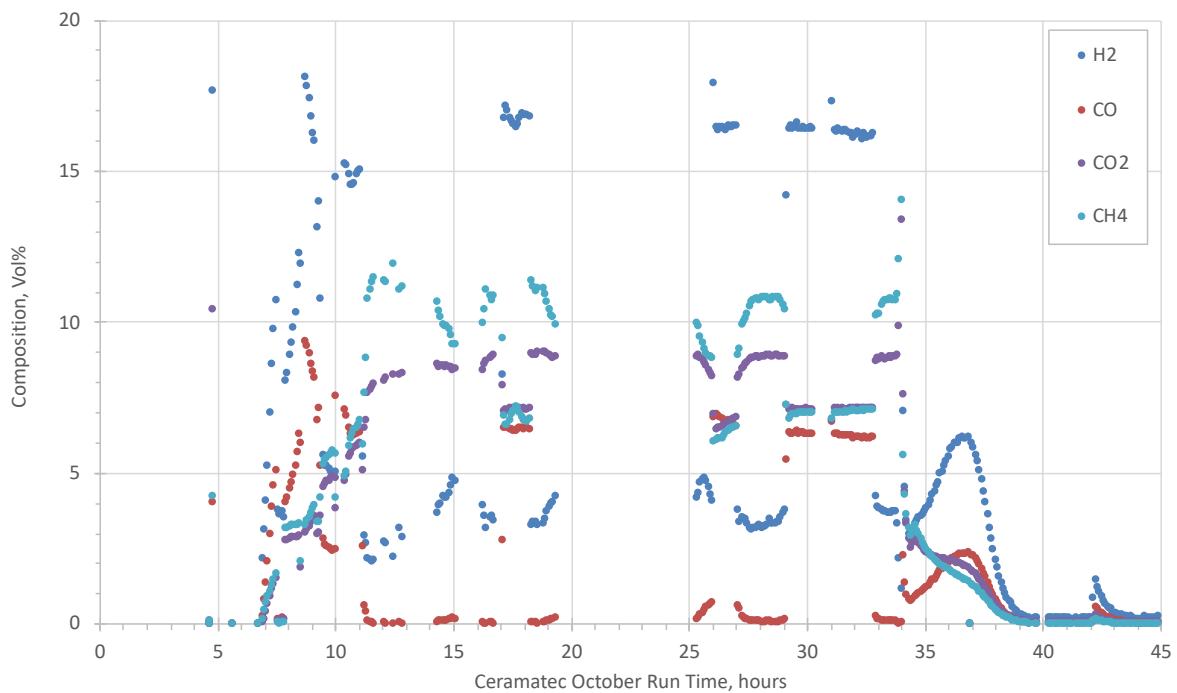


Figure 9. FT reactor tail gas composition at Ceramatec, October 25-26, 2017.



Figure 10. FT first oil and produced water at Ceramatec, October 26, 2017.

System move and integration at EERC

The FT system was broken down to move from Friday October 27, to Tuesday October 31 with trucks and cranes scheduled to load the skids starting the afternoon of Nov 1. We were notified of a delayed arrival of the first truck from Wednesday afternoon to Thursday morning, so both skids needed to be loaded Thursday morning. With the benefit of good preparation and skilled crane operators and riggers the two trucks were loaded as planned Thursday morning, Nov 2 as seen in Figure 11. A 3rd truck was loaded Friday morning with the remaining smaller items.

All three trucks arrived and were unloaded at the EERC on Monday Nov 6, 2017. Ceramatec engineers Don Claus and Piotr Czernichowski left for EERC on Tuesday Nov 7, and worked through two solid weeks with the EERC team, returning home for the Thanksgiving holiday on Tuesday Nov 21. They returned a week later to complete final preparations for the gasifier run that was scheduled to begin late evening on Sunday December 3, 2017. The Ceramatec PI, Joseph Hartvigsen joined Don and Piotr at EERC for the run Dec 03-14 to enable round the clock manning of the system.

Since the extensive electrical wiring on the horizontal skid made it impractical to separate the two halves as originally planned, EERC was able to devise an angled orientation of the skid in the Fuels of the Future high bay at the National Center for Hydrogen Technology (NCHT). The planned layout and installation photos are shown in Figure 12.



Figure 11. FT system move from Ceramatec, November 2, 2017.

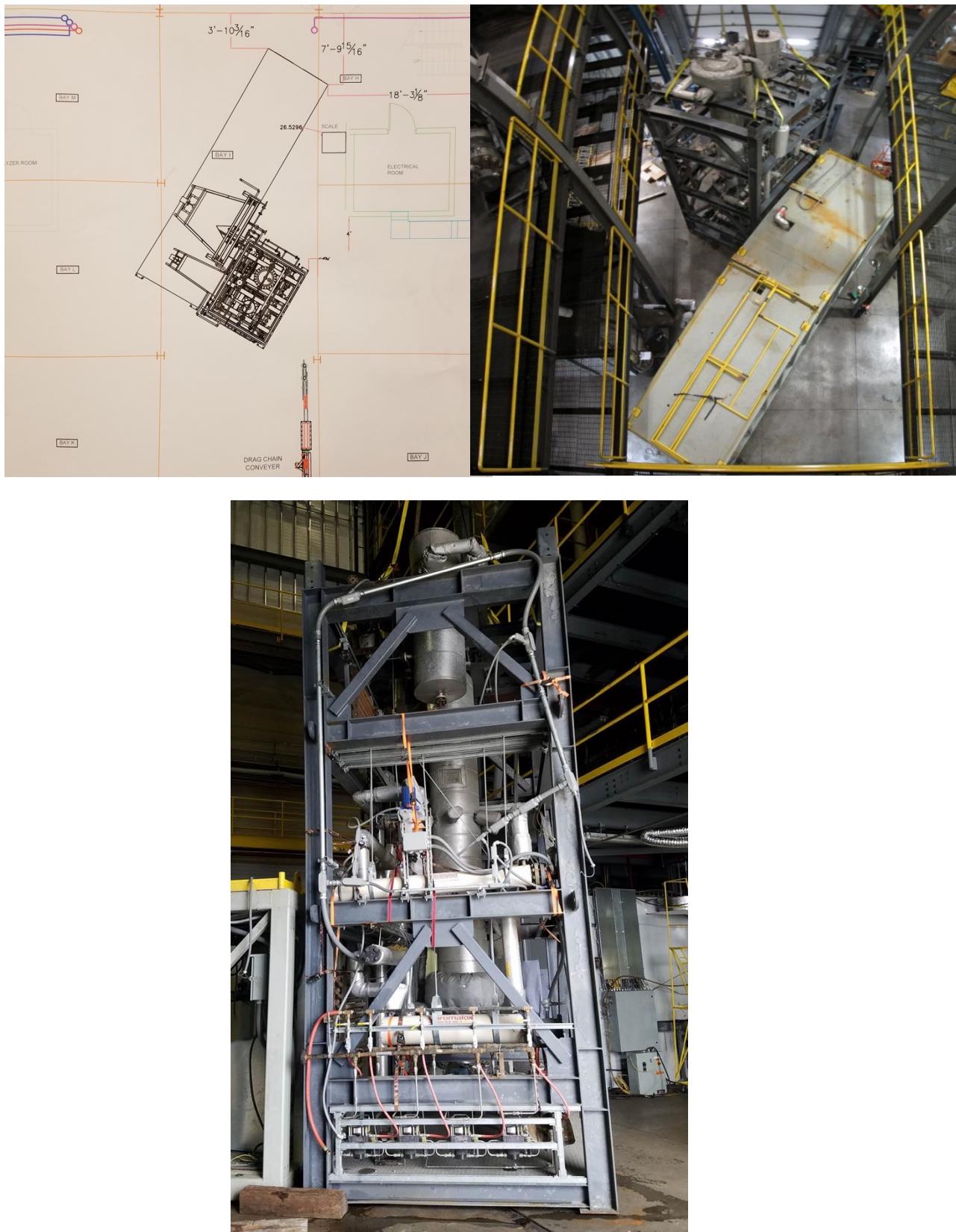


Figure 12. FT system installation at EERC, November 6, 2017.

FT operating results at EERC

The reactor was heated to the nominal operating temperature range of 230°C – 240°C in the day before receiving the syngas stream from the gasifier, and the catalyst charge was supplied a stream of ~10% hydrogen in nitrogen to reduce any catalyst that might have been exposed to air ingress during the move. A laser Raman gas analyzer (RLGA) on the tailgas did not detect any water vapor (-40°F dew point reading) indicating that there had been no oxidation of the catalyst during the move and reassembly.

The gasifier began heating late Sunday evening December 3, and began supplying gas to the FT system just before midnight Monday evening. Tuesday some minor operational issues were addressed including successful configuration of the radar interface detection instrument communications with the control system. The large product cooler fan stalled when the split sheave on the motor came loose and backed into the motor. This was repaired, adjusted and placed back into service. With the product interface instrument communicating the automated product removal system was still not functioning due to a pressure imbalance between the liquid and vapor lines feeding the separation vessel. Once that was corrected the product collection system functioned as designed. Tuesday there was a major winter storm with snow and high winds that closed the University and sent all non-essential EERC personnel home. This team continued working through the storm and the only impact of the storm on operation was related to a delayed delivery of liquid nitrogen.

The product collection rate appeared to be about 3 gallons per day (gpd). This was half the rate demonstrated using a single reactor tube of 4' length loaded with a 2kg charge of hybrid CoRu catalyst in MFEC earlier in this project. Had we seen the same catalyst productivity we would have expected an oil production rate in the range of 88-106 gpd. The Ceramatec run had a production rate of about 10 gpd, with a syngas flow rate estimated to be 25%-33% of the target flow and 20°C and 4 bar below the target operating temperature and pressure, which may have suggested a minor reduction in catalyst productivity. The catalyst activity did not improve during the run. Consultation with the catalyst developer at Chevron suggested we should increase the flow rates of fresh feed, and increase the operating temperature to 240°C. An additional explanation for the low productivity suggested by Chevron was the low CO concentration in the syngas. A comparison of syngas compositions from Ceramatec runs and EERC is shown in Table 1. Both the Ceramatec micro GC and the EERC RLGA showed to be well calibrated against standard gas mixes approximating syngas compositions.

Table 1: Syngas composition comparison.

Syngas Component	Ceramatec 2015 O ₂ enriched ATR	Ceramatec Oct 2017 Air ATR Natural Gas	EERC Dec 2017 Coal-Biomass Gasifier
N ₂	26.4%	47.4%	39.7%
H ₂	46.6%	30.6%	11.3%
CO	22.3%	13.2%	6.0%
CO ₂	4.1%	5.1%	37.5%
CH ₄	0.5%	3.6%	4.0%

The reactor temperature and syngas feed rates were increased as recommended, with no improvement of production rate. In fact, by Friday morning the rate had dropped to nothing. The highest production rates were seen Tuesday Dec 5, with fresh feed rates of 45-50 kg/h and recycle rates in the 90-100 kg/h range. The evening of Thursday Dec 7, to the early morning

hours of Friday Dec 8, (with a short syngas outage in that period) the rates were ramped to 100 kg/h fresh feed and 25kg/hr recycle. No telltale exotherm was noted in the reactor centerline temperatures except a 3-4°C response in the lowest thermocouple of the center tube and the instrumented outer ring tube. By morning any indication of activity in the lower bed was gone and the 18 bed thermocouple readings converged to the jacket temperature. Oil production had stopped and the inlet and exit syngas compositions were virtually identical. The catalyst activity was completely lost.

We discussed possible deactivation mechanisms with EERC and Intramicron engineers familiar with the system. Given the high CO₂/CO ratio and low syngas conversion it was argued that deactivation by carbon formation via Boudouard reaction was unlikely. The syngas was more oxidizing than reducing, however thermodynamic calculations by Intramicron showed that copper and cobalt could not be oxidized by carbon dioxide under these conditions. The two common catalyst regeneration procedures are oxidative and hydrogenation processes. There was not enough time or available hydrogen (or any basis to suggest) for an oxidative regeneration which would oxidize both catalyst and copper MFEC requiring re-reduction of both. While it was not expected that there was oxidation of the catalyst, wax produced by low temperature operation during startup at Ceramatec could in theory block pores in the catalyst and reduce activity. Hydrogenation is an effective means of removing wax by converting it to methane.

A feed stream of ~10% hydrogen in nitrogen was supplied to the hot reactor from 21:30 on Dec 8 to 16:30 on Dec 10, consuming about a dozen 200 SCF bottles of hydrogen. The RLGA showed a steady -40°F dew point in the tail gas, indicating no reduction reactions. Levels of CO₂ and CH₄ were under 0.5%, suggesting a slow desorption of CO₂ and hydrogenation of wax to methane. However on restarting FT operations overnight on Sunday Dec 10, no activity had been restored through the 3-day attempt at regeneration by hydrogenation. Syngas flow to the FT system was terminated mid-afternoon of Monday Dec 11.

The most stable and complete data from the EERC run was obtained Dec 5-6. An attempt at a mass balance and productivity-selectivity analysis was computed from this data set and is shown in Figure 13. The catalyst productivity was 0.06 ml C₅₊/g_{Cat}/h compared with 0.50 ml C₅₊/g_{Cat}/h from the same catalyst formulation tested at Ceramatec in April of 2015. Even with the lower catalyst activity the H₂ and CO conversions achieved were 50.0% and 45.5% respectively because of the low initial fresh feed flow rate and the low H₂ and CO concentration in the syngas. Methane selectivity was abnormally low at 2.2% vs. 18.9% for the hybrid catalyst in MFEC tested in April 2015. Carbon dioxide selectivity was -136% vs. -0.01% in 2015. It is not clear whether these radical changes in activity and selectivity can be attributed to a difference in catalyst or syngas conditions.

We requested the EERC analytical lab compare the product distribution of samples from the Ceramatec (October 26, 2017) and EERC runs. A sample taken early on Tues Dec 5 at EERC (Figure 15), compared closely to the Ceramatec sample (Figure 14) and was primarily paraffinic with some alpha-olefin as expected from a Co catalyst. However analysis of the total product collection showed a highly aromatic product with over 50% benzene and roughly 1/3 as alkylated aromatics as shown in Figure 16. The syngas preheat loop uses Therminol 59 heat transfer fluid which is an alkylated aromatic (Figure 17). Since we didn't commission this system at Ceramatec and it was in use at EERC the possibility of crossover in the syngas heat exchanger was studied and eliminated as a possibility. At the end of the run the syngas heat exchanger was pressurized to 160 psig and held pressure overnight. The full inventory of Therminol 59 was recovered from the preheat loop. The chromatograms in Figure 16 and Figure

17 are clearly different and are not likely explained by cracking of Therminol on the zeolite catalyst support even if it were shown that the heat exchanger was losing fluid to the syngas.

A request to discuss these product distribution results with the Chevron catalyst developer was made just as he was leaving the country until mid-January. The Ceramatec personnel have left for a new company and will follow up on his return and brief the NETL PM with any new insights to this problem.

Since only ~ 12 gallons of product were collected at the EERC and the product composition is not representative of an expected FT fuel from the hybrid catalyst we were not able to provide the proposed 10 gallons of distilled jet fraction fuel. One gallon of the 12-13 gallons of FT product collected during the Ceramatec run in October 2017 had been shipped to EERC to determine distillation parameters. This was distilled by EERC yielding 38% naphtha, 55% jet fraction and 7% heavy fraction as shown in Figure 18. The half-gallon of jet fraction separated by distillation shown in Figure 19 will be shipped from EERC to NETL as directed by the PM.

Time & date		12/6/17 09:00		0.08/057 R liter-atm/g/mol K		7.3 bbl/ton	
Temperature		222 °C coolant setpoint		28.32 Liters/ft ³		306.6 gal/ton	
Pressure		20.0 bar		294.26 kg/SCFM		3.26 kg/ft ³	
Fresh Syngas		33.388 SCFM reading		45.529 kg/hr Coriolis direct measure mass flow (30deg/hr displays as 22 SCFM)		0.862 g/mol	
Fresh Syngas		21.021 SCFM goal seek corrected to match mass flow at inlet (match B8 to B6)		45.529 kg/hr calc		0.411 mol/Sec	
Recycle Syngas		53.8315 SCFM		93.62 kg/hr Coriolis direct measure		0.411 mol/Sec	
Total		1415.0 SCFM		1.052 mol/Sec		1.84	
H2		Fresh in GC		In Normalized		Out GC	
H20		10.95		0.06465		6.21	
N2		0.03		0.003%		0.013%	
CO		38.4		0.132		0.162	
CO2		5.97		0.0246		3.57	
C1H4		37.53		0.1595		39.38%	
C2		4.14%		0.0170		4.61	
C2=		0.00%		0.0000		0.30%	
C3		0.00%		0.0000		0.00%	
C3=		0.00%		0.0000		0.11%	
C4		0.00%		0.0000		0.063	
C5		0.00%		0.0000		0.10%	
C5=		0.00%		0.0000		0.105	
C6		0.00%		0.0000		0.09%	
C7		0.00%		0.0000		0.105	
Total		96.696		0.4109		97.197	
1.89 H2/CO		0.583 C4/C3 from Apr 2015 runs		0.3645		1.05217	
0.625 C5/C3		0.625 C5/C3		0.625 C6/C3		1.46307	
0.262 C7/C3		0.262 C7/C3					
Collection time		24 hr		Actual Collection		Goal seek O58 varying oil and O59 varying water to zero mass balance	
Oil		18.54 kg		7.96		oil recovery	
Water		64.819 kg		37.58		water recovery	
Oil rate		0.772 kg/hr		54.6% actual water+oil / calculated			
Oil/water		22.2%		0.28860			
Cn ave estimate		9.8					
Produced Oil-CH ₄		2.20 Product H/C					
Produced H ₂ O		0.01508 mol/sec					
Produced H ₂ O Total		0.04164 mol/sec					
Non-N2 Total		12.64694		11.78078		0.96580	
8.07581		7.20964				8.17444	
Mass Balance							
Feed		atom mol/sec		atom mol/sec		atom mol/sec	
H		0.1614		0.16277		0.2593	
C		0.2011		0.215159		0.2011	
O		0.3436		0.49790		0.451519	
N		0.3264		4.57114		0.49790	
				0.3264		0.3264	
Gas Exit		atom fraction		atom fraction		atom fraction	
		g/sec		0.11652		0.26135	
				0.01508		0.2011	
				0.18108		0.451519	
				0.66628		0.49790	
				0.04164		0.57114	
				10.00%		100.0%	
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Figure 13. FT system mass balance calculation from EERC data December 6, 2017.

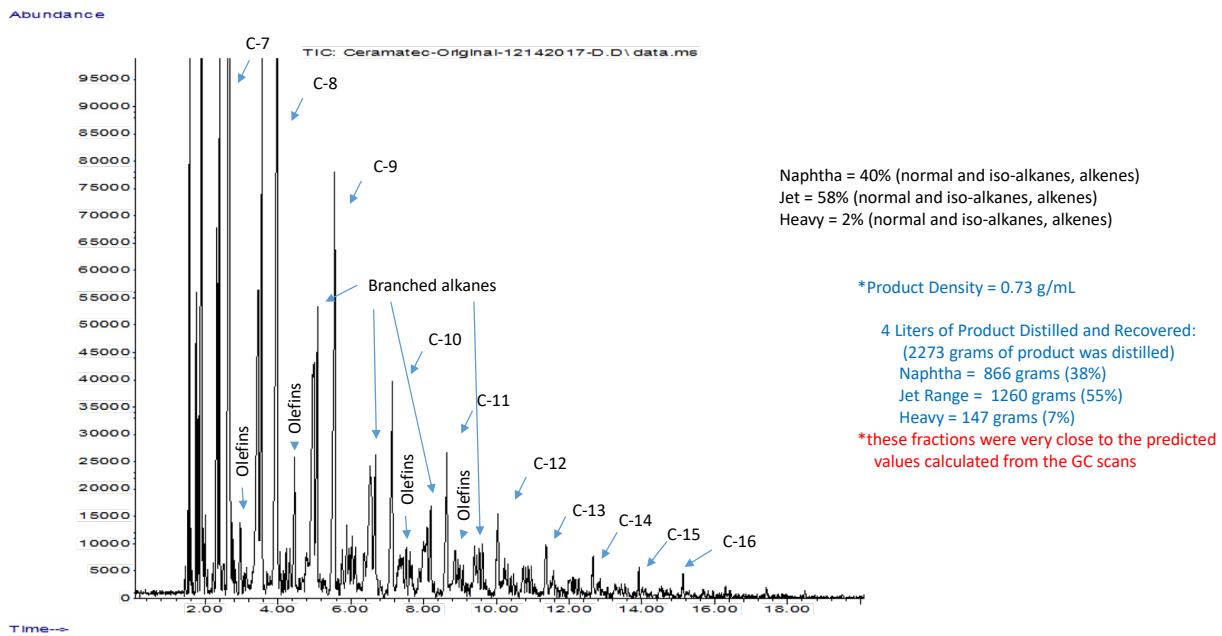


Figure 14. FT product distribution from Ceramatec run of October 25-26, 2017.

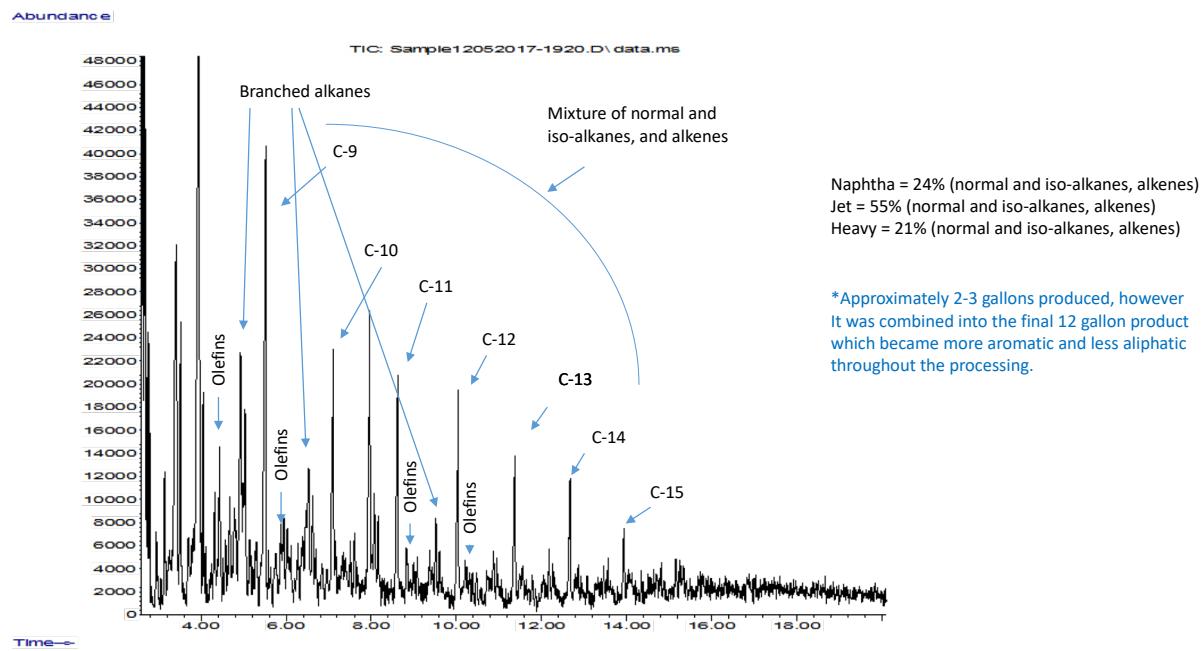


Figure 15. FT product distribution from EERC sample of December 5, 2017.

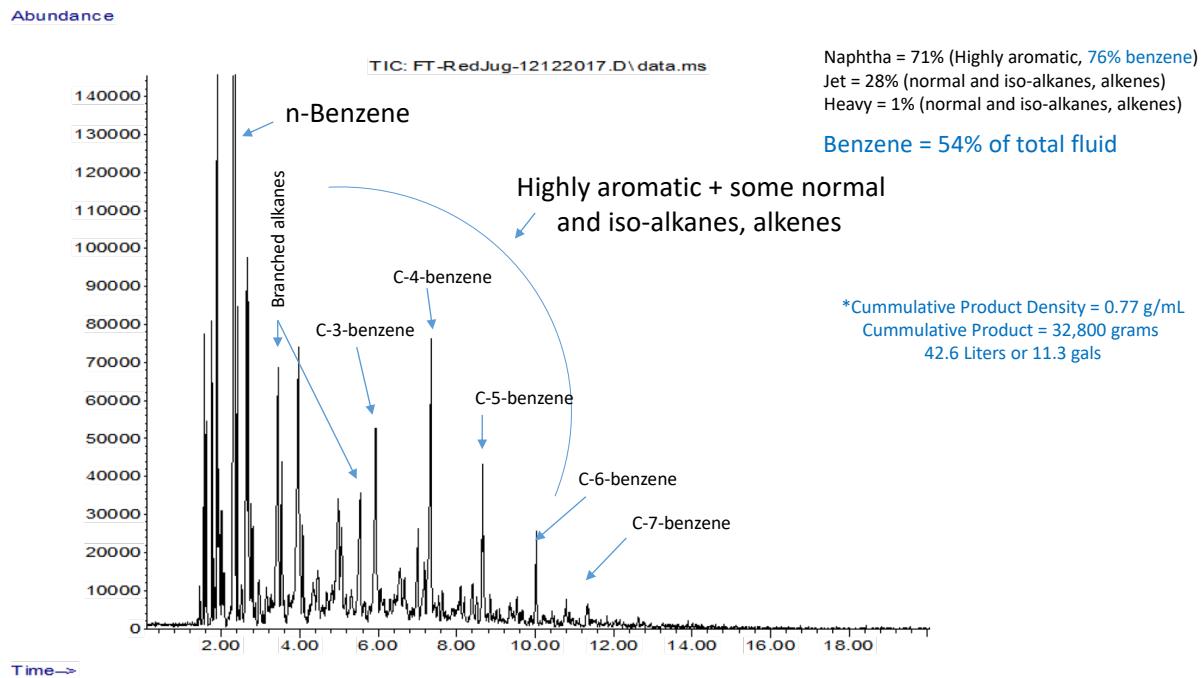


Figure 16. FT product distribution from EERC composite sample of December 12, 2017.

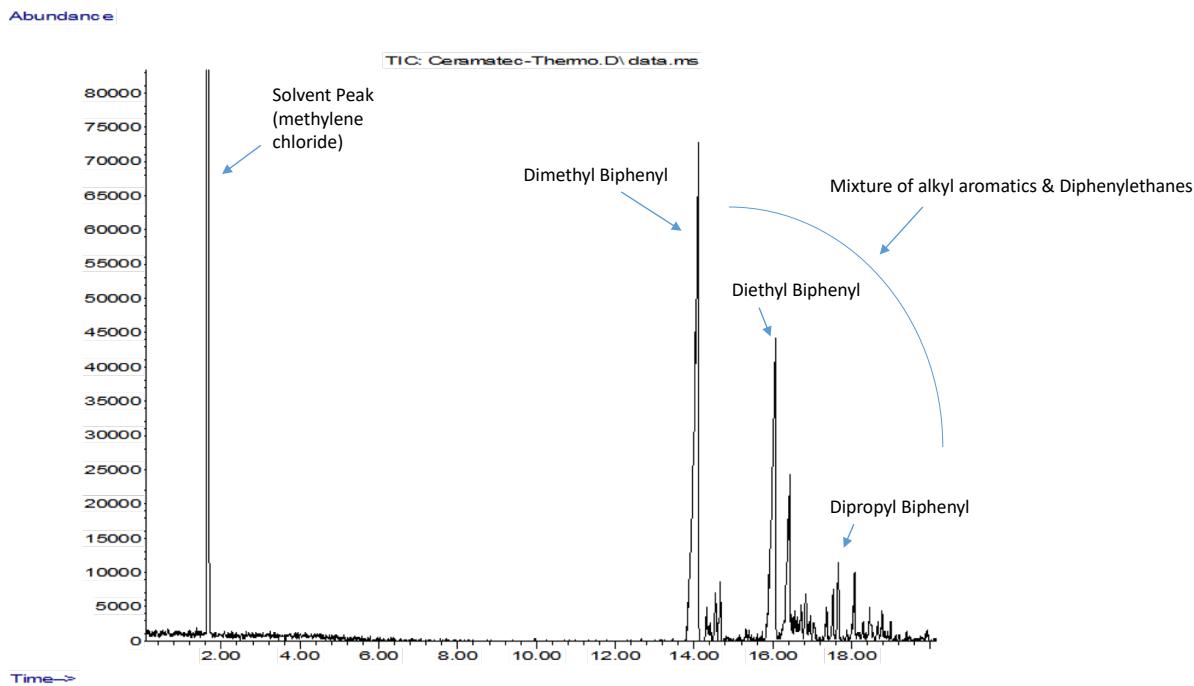


Figure 17. Analysis of Therminol 59 heat transfer fluid to eliminate cross-over contamination hypothesis.

Distilled Jet-Range Hydrocarbon Fraction

(distilled from FT sample produced at Ceramatec)

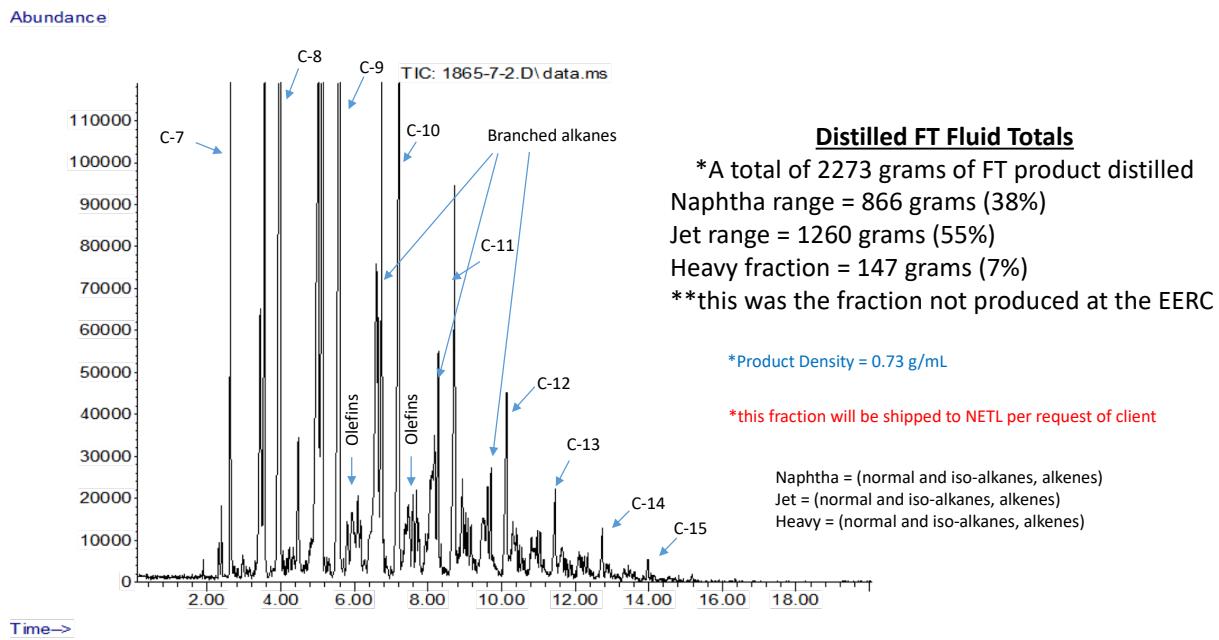


Figure 18. Analysis of distilled Jet-range product from Ceramatec run of Oct 25-26, 2017.

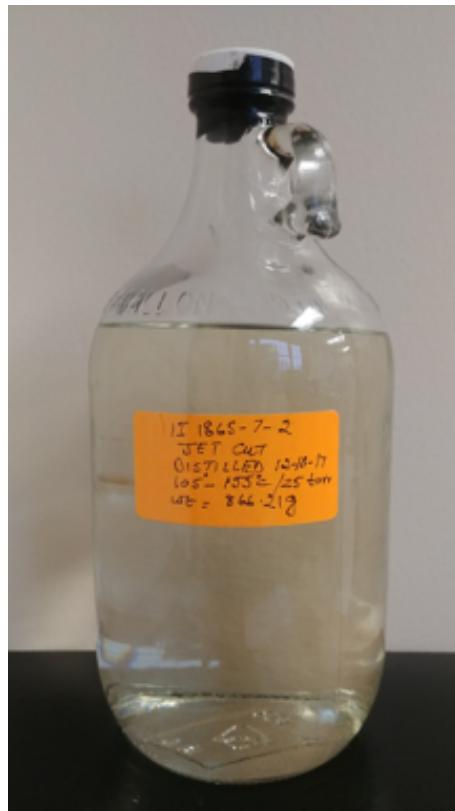


Figure 19. Distilled Jet-range product from Ceramatec run of Oct 25-26, 2017.

Plan for Next Period

Even though a contract modification earlier this year extended the contract period of performance to March 31, 2018, all planned work has been completed and reported. No work or spending on this program is planned for the duration of the contract. Discussions will be held between the Ceramatec PI (at his new company) and the NETL PM on how to best resolve the questions arising from this project.

Products

Related Publications, Conference Papers, and Presentations:

None this quarter

CHANGES/PROBLEMS

No Report

SPECIAL REPORTING REQUIREMENTS

This program has no special reporting requirements at this time.

BUDGETARY INFORMATION

Table 1. Cost Plan Status – FY2015

Baseline Reporting Quarter	Budget Period 1							
	Q1		Q2		Q3		Q4	
	10/1/14– 12/31/14		1/1/15-3/31/15		4/1/15-6/30/15		7/1/15-9/30/15	
	Q1	Cumulative Total	Q2	Cumulative Total	Q3	Cumulative Total	Q4	Cumulative Total
Baseline Cost Plan								
Federal Share	\$242,349	\$242,349	\$242,349	\$484,698	\$242,349	\$727,048	\$242,349	\$969,397
Non-Federal Share	\$127,850	\$127,850	\$127,850	\$255,700	\$127,850	\$383,550	\$127,850	\$511,399
Total Planned	\$370,199	\$370,199	\$370,199	\$740,398	\$370,199	\$1,110,597	\$370,199	\$1,480,796
Actual Incurred Cost								
Federal Share	\$125,680.40	\$125,680.40	\$138,113.50	\$263,793.90	\$168,700.91	\$432,494.81	\$375,520.60	\$808,015.41
Non-Federal Share	167237.35	167237.35	205809.22	373046.57	\$0	\$373,047	0	\$373,047
Total Incurred Costs	\$372,710	\$372,710	\$268,301	\$641,011	\$741,699	\$995,925	\$375,521	\$1,371,446
Variance								
Federal Share	\$116,669	\$116,669	\$104,236	\$220,905	\$73,648	\$294,553	(\$133,171)	\$161,381
Non-Federal Share	(\$39,388)	(\$39,388)	(\$77,959)	(\$117,347)	\$127,850	\$10,503	\$127,850	\$138,353
Total Variance	(\$2,511)	(\$2,511)	\$101,898	\$99,387	(\$371,500)	\$114,672	(\$5,322)	\$109,350

Table 2. Cost Plans Status – FY2016

Baseline Reporting Quarter	Budget Period 2							
	Q5		Q6		Q7		Q8	
	10/1/15– 12/31/15		1/1/16-3/31/16		4/1/16-6/30/16		7/1/16-9/30/16	
	Q1	Cumulative Total	Q2	Cumulative Total	Q3	Cumulative Total	Q4	Cumulative Total
Baseline Cost Plan								
Federal Share	\$523,871	\$1,493,268	\$523,871	\$2,017,138	\$523,871	\$2,541,009	\$523,871	\$3,064,880
Non-Federal Share	\$116,471	\$627,871	\$116,471	\$744,342	\$116,471	\$860,813	\$116,471	\$977,285
Total Planned	\$640,342	\$2,121,138	\$640,342	\$2,761,480	\$640,342	\$3,401,822	\$640,342	\$4,042,164
Actual Incurred Cost								
Federal Share	\$ 465,059.56	\$ 1,273,074.97	\$ 431,584.06	\$ 1,704,659	\$ 460,343.05	\$ 2,165,002	\$ 445,662.13	\$ 2,610,664
Non-Federal Share	23279	\$396,326	\$186,693	\$583,019		\$583,019		\$583,019
Total Incurred Costs	\$465,059	\$ 1,669,400.54	\$ 431,584.06	\$ 2,287,677.98	\$ 460,343.05	\$ 2,748,021.03	\$ 445,662.13	\$ 3,193,683
Variance								
Federal Share	\$58,811	\$220,193	\$92,287	\$312,479	\$63,528	\$376,007	\$ 78,208.54	\$454,215
Non-Federal Share	\$93,192	\$231,545	(\$70,222)	\$161,323	\$116,471	\$277,795	\$116,471	\$394,266
Total Variance	\$175,283	\$451,738	\$208,758	\$473,802	\$179,999	\$653,801	\$ 194,679.89	\$848,481

Table 3. Cost Plans Status – FY2017

Baseline Reporting Quarter	Budget Period 3							
	Q9		Q10		Q11		Q12	
	10/1/16– 12/30/16		1/1/17-3/31/17		4/1/17-6/30/17		7/1/17-9/30/17	
	Q1	Cumulative Total	Q2	Cumulative Total	Q3	Cumulative Total	Q4	Cumulative Total
Baseline Cost Plan								
Federal Share	\$358,139	\$3,423,019	\$358,139	\$3,781,158	\$358,139	\$4,139,297	\$358,139	\$4,497,436
Non-Federal Share	\$130,449	\$1,107,734	\$130,449	\$1,238,184	\$130,449	\$1,368,633	\$130,449	\$1,499,082
Total Planned	\$488,588	\$4,530,753	\$488,588	\$5,019,341	\$488,588	\$5,507,929	\$488,588	\$5,996,518
Actual Incurred Cost								
Federal Share	\$ 430,717.00	\$3,041,381	\$ 265,225.95	\$3,306,607	\$607,141	\$3,913,748	\$181,368	\$4,095,116
Non-Federal Share		\$583,019	\$427,264	\$1,010,283	\$499,204	\$1,509,487	\$274,439	\$1,783,926
Total Incurred Costs	\$ 430,717.00	\$ 3,624,400.16	\$ 692,490.16	\$4,316,890	\$1,106,345	\$5,423,235	\$455,807	\$5,879,042
Variance								
Federal Share	\$ (72,578.00)	\$381,637	\$92,913	\$474,550	(\$249,002)	\$225,548	\$176,771	\$402,320
Non-Federal Share	\$130,449	\$524,715	(\$296,815)	\$227,900	(\$368,754)	(\$140,854)	(\$143,990)	(\$284,844)
Total Variance	\$57,871	\$906,353	(\$203,902)	\$702,451	(\$617,756)	\$84,694	\$32,782	\$117,476

A separate quarterly cost status update will not be provided for Q13, only the final end of project SF-425.

Appendix A: Intramicron Final Report

PROJECT INFORMATION

Federal Agency	U.S. Department of Energy
Funding Program	National Energy Technology Laboratory
Award Number	DE-FE0023863
CFDA Number	81.089
Start Date	October 1, 2014
Program Title	Technology for GHG Emission Reduction and Cost-Competitive Mil-Spec Jet Fuel Production using CTL

QUARTERLY REPORT

1. ACCOMPLISHMENTS:

a. PROJECT GOALS

A summary of the milestones for the entire project have been compiled (Table 1). The major activity of the reporting period was the demonstration of the sulfur skid.

Table 1: Project Milestones and Planned Completion Dates.

Milestone Title/Description	Planned Completion Date	Actual Completion Date	Comments
Fischer Tropsch Synthesis			
Load test reactor to Ceramatec containing Chevron's Catalyst	12/31/2014	1/7/2015	
Determine media composition final design reactor tubes	9/30/2015	9/30/2015	
Finish media preparation for final design reactor tubes	3/31/2016	6/24/2016	
MFEC loaded into final design reactor tubes	6/30/2016	11/14/2016	
Provide support for demonstration and modeling effort	9/30/2017	8/24/2015	Provided Aspen modeling specifications to integrate models on VMG Sim.
Desulfurization			
Complete system modification for high-pressure IM-DTS testing	3/31/2015	3/31/2015	
Complete initial IM-DTS evaluations with syngas and pressure	6/30/2015	7/02/2015	
Evaluate IM-DTS with composition from CWS gasification tests	9/30/2015	2/12/2017	
System design complete	12/31/2015	11/01/2016	

P&ID and PFD for desulfurization	3/31/2016	1/13/2017	
Desulfurization system fabricated and tested	6/30/2016	12/11/17	System is fabricated and assembled, testing at IntraMicron is complete; Final tests will occur at EERC.
Provide testing support to Ceramatec	10/1/2016	11/5/16	
Installation support at EERC	5/31/2016	11/29/17	Installation complete at EERC
Provide support to Ceramatec for demonstration and modeling effort	9/30/2017	12/4/17	

b. DETAILED ACCOMPLISHMENTS

Sulfur Skid Demonstration: Summary

IntraMicron's desulfurization skid performed beyond expectations during the demonstration at Energy & Environmental Research Center (EERC). The feed gas for the demonstration was produced by the EERC's transport reactor development unit (TRDU). The TRDU used both pure coal and a coal/biomass blend to produce syngas during the demonstration. Syngas produced by the TRDU is contaminated with steam, tars, ash, and hydrogen sulfide. IntraMicron's sulfur skid converted 90% of the hydrogen sulfide into elemental sulfur from both the coal and coal/biomass blend gas. Figure 1 and Figure 2 are pictures of the sulfur skid installed at EERC.

Figure 1: IM Desulfurization Skid Installed at EERC. Figure removed to protect proprietary information.

Figure 2: IM Desulfurization Skid Installed at EERC. Figure removed to protect proprietary information

Sulfur Skid Demonstration: Design and Installation

The sulfur skid easily integrated into EERC's process thanks to the unit's versatility. EERC did not need to hand over control to IntraMicron of any of the valves governing the flowrate of gas into the sulfur skid. The skid was fed on a slip stream between the TRDU and syngas compressor, Figure 3, so maintaining control over these valves was important to EERC for the process's overall performance. As EERC tweaked control valves to keep their process running the flowrate to the sulfur skid changed. The flowrate to the sulfur skid fluctuated between 500 and 1200 slpm, but the sulfur skid's performance was not hampered by the fluctuations in the gas feed.

The sulfur skid has three basic modes of operation. First is syngas bypass mode. This mode is used to bring the reactor up to temperature during start-up or to keep the system at temperature if the syngas feed is disrupted. Syngas flows into the skid through stream 6 and out of the skid through stream 8 in Figure 3 during this mode of operation, but syngas does not flow through the OSR reactor, condenser, and other equipment on the sulfur skid. The inlet and outlet syngas valves XV-01 and XV-06 are closed, and the bypass valves HV-01 and HV-02 are open. The syngas flow through the sulfur skid during bypass

is shown in Figure 4 and Figure 6. Nitrogen is used to maintain gas flow and purge the system of syngas during bypass mode. The nitrogen flows out through XV-05 to the 5lb stack shown in Figure 5. The second mode is run mode to the thermal oxidizer. In this mode the inlet and outlet syngas valves are open, and the bypass valves are closed. Nitrogen is off, and a controlled amount of air is injected to the OSR reactor. Syngas leaving the skid is directed to the thermal oxidizer through stream 8 in Figure 3. The third mode of operation is run mode to the FT skid. No changes to the sulfur skid's valve positions are needed to switch from the second mode to the third. EERC personnel oversaw re-directing the skid's treated gas to stream 7 from stream 8 in Figure 3. The sulfur skid used all three modes of operation during the demonstration.

Figure 3: Energy & Environmental Research Center PFD. Figure removed to protect proprietary information.

Figure 4: PFD 1. Figure removed to protect proprietary information

Figure 5: PFD 2. Figure removed to protect proprietary information

Figure 6: PFD 3. Figure removed to protect proprietary information

Sulfur Skid Demonstration: Operation Summary

Frequent interruptions of the TRDU's operation forced the sulfur skid to start-up and revert to bypass mode six times during the demonstration. The interruptions in syngas flow were never due to the sulfur skid having any kind of failure or need to shut down. The length of each run and the reason for entering bypass mode is broken down in

Table 2. During the final run the treated syngas was blended back into the feed stream for the FT skid, but during the other runs the syngas was directed to the thermal oxidizer.

Table 2: Time on treating syngas and reasons for interruptions

Run	Start	Stop	Hours	Reason for Stopping Syngas Feed
1	December 5 th	December 7 th	52	Coal Plug. TRDU stopped gasification
2	December 7 th	December 8 th	17	EERC controls computer lost power. TRDU stopped gasification
3	December 8 th	December 8 th	4	Coal feed was turned off to replace the feed hopper level indicator. TRDU stopped gasification
4	December 8 th	December 8 th	2	Major coal plug. TRDU stopped gasification
5	December 9 th	December 10 th	26	EERC N2 tank ran low. TRDU stopped gasification
6	December 10 th	December 11 th	12	IntraMicron reached its demonstration goals

Sulfur Skid Demonstration: Technical Results

The desulfurization reactor was loaded with IntraMicron's Oxidative Sulfur Removal (OSR) catalyst, a COS hydrolysis catalyst, and a silica guard bed to prevent liquid water and tars from reaching the catalyst bed and damaging it. Figure 7Figure 3 is a diagram of the loaded reactor.

Figure 7: Diagram of Desulfurization Reactor Loading.
Figure removed to protect proprietary information.

The desulfurization demo ran for 112.5 hours. For 102.5 of those hours, the average syngas flowrate was 1000 slpm. During most of the demonstration, the GHSV through the OSR catalyst bed was 1679 hr^{-1} . When syngas was tested in the lab-scale desulfurization reactor, the optimal GHSV through the OSR bed was 5390 hr^{-1} . Syngas desulfurization was performed at several different GHSVs in the lab-scale pressurized and atmospheric reactors, but the best conversion to elemental sulfur was achieved at 5390 hr^{-1} . With the catalyst charge used in the demonstration reactor, the highest H₂S conversion to elemental sulfur was 81%. The sulfur distribution for the best lab-scale pressurized results are shown in Figure 8.

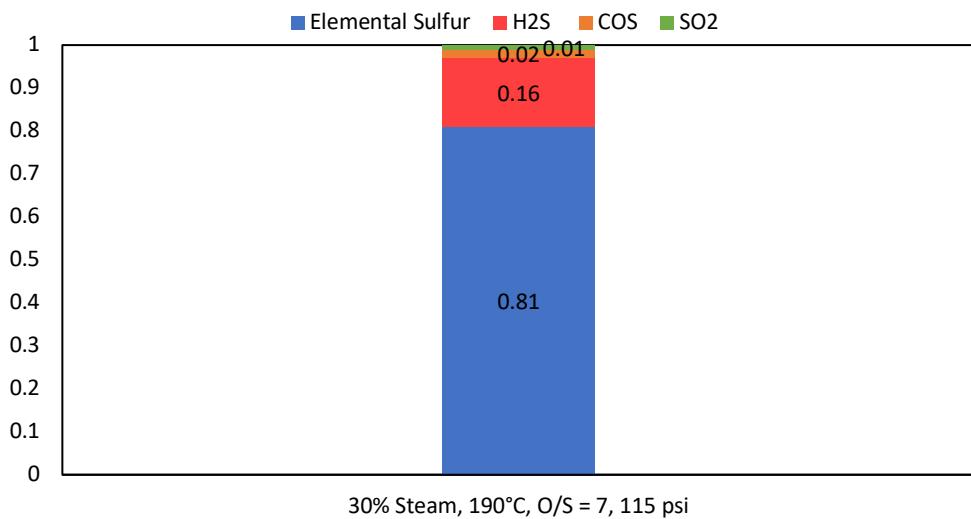


Figure 8: Best case lab-scale results. GHSV = 5390 hr^{-1}

On average, the inlet H₂S concentration during the 102.5 hours of 1000 slpm syngas flow was 3330 ppm. For the first day and a half of the demonstration, the GC results were unreliable due to a plumbing issue. When the GC issues were resolved, the chromatogram results were confirmed by testing the inlet and outlet H₂S concentration of the gas with Dräger tubes, as shown in Figure 9. When the Dräger tubes were used to test the gas, the inlet H₂S concentration was 3500 ppm, and the outlet H₂S concentration was 37 ppm. The overall conversion to elemental sulfur was 92%, with the majority of the sulfur remaining in the gas being SO₂.

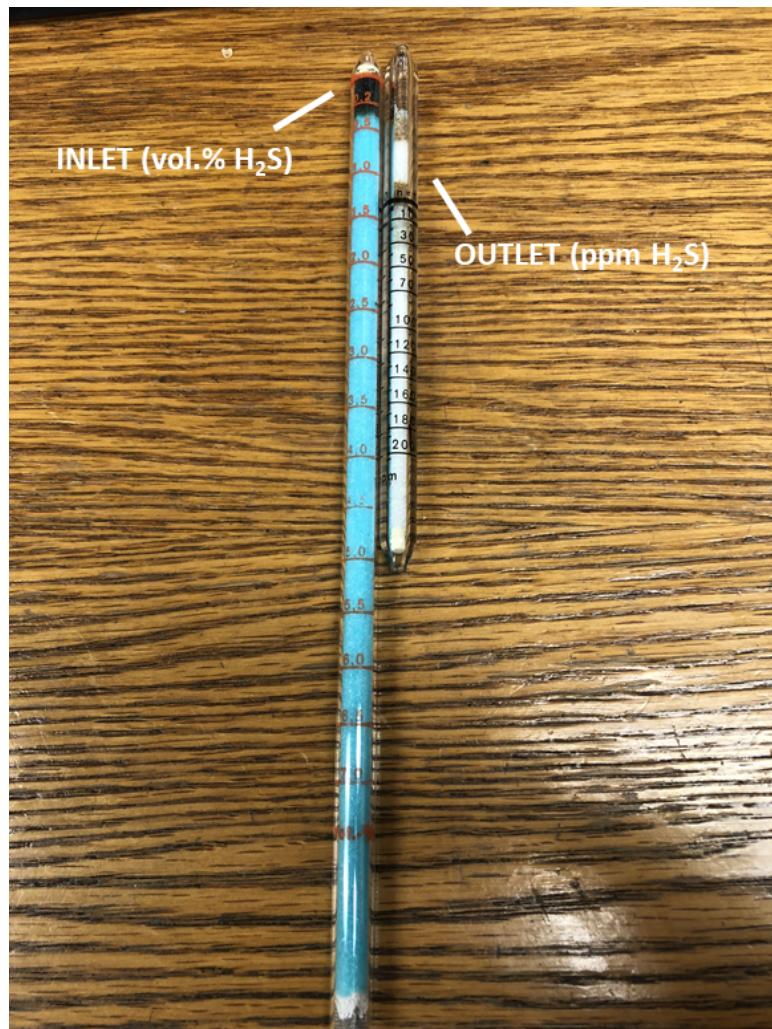


Figure 9: Results of Dräger tube H-2S concentration tests for inlet syngas (left) and outlet syngas (right).

The syngas flowrate, inlet H₂S concentration, and conversion to elemental sulfur are plotted against the demonstration time in Figure 10. IntraMicron's desulfurization skid began processing syngas at 9:50 am on December 5, 2017. The plot in Figure 10 begins at Hour 38.5, 12:20 am on December 7, 2017, which is when the GC issues were resolved, and the first reliable chromatograms were produced and confirmed with the results of the Dräger tube tests.

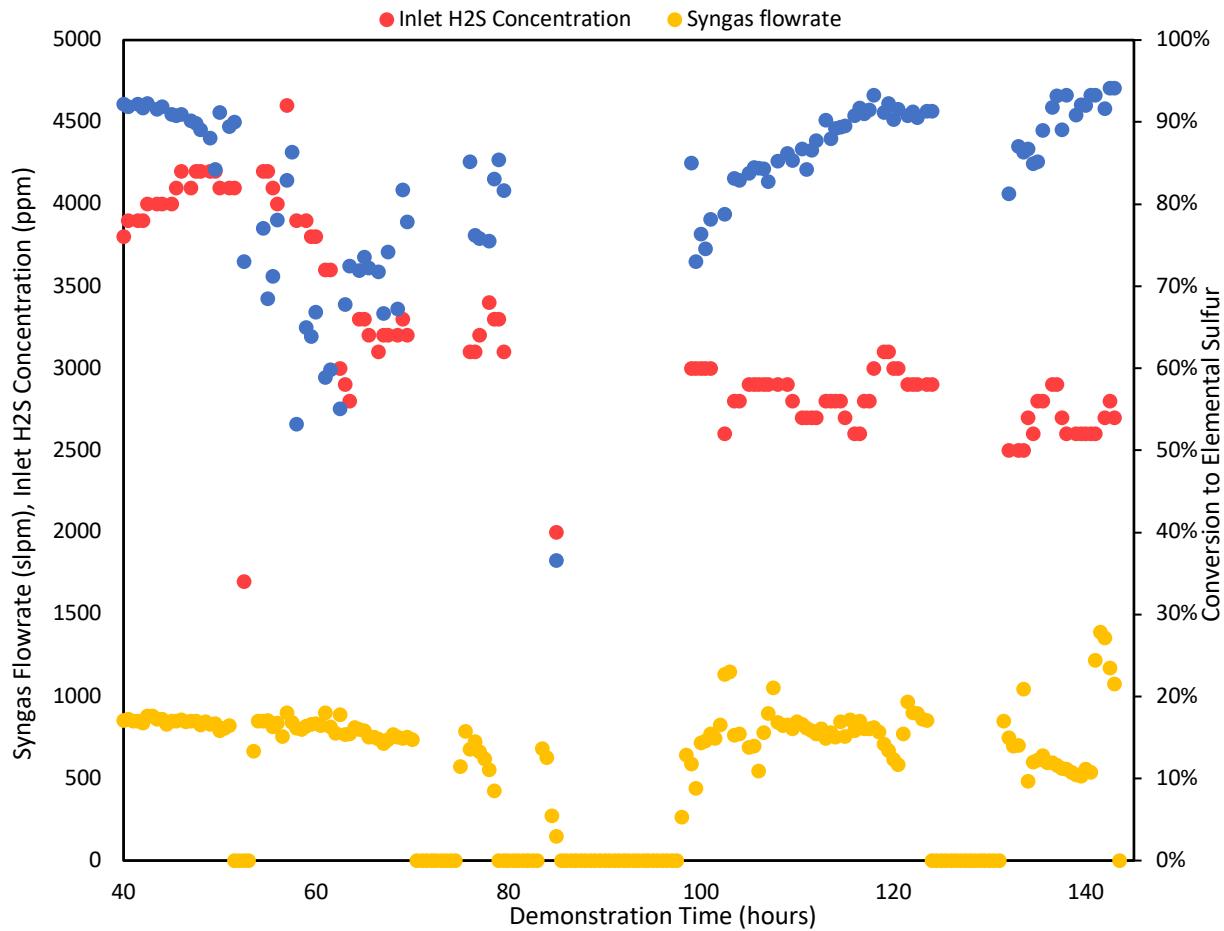


Figure 10: Syngas flowrate, inlet H2S concentration, and conversion to elemental sulfur vs. time.

At that point, the conversion to elemental sulfur had already reached 92%. It would remain stable in the low 90% range until 1:20 pm on December 7 when the syngas flow to the skid stopped because of a coal feed plug. At that point, the desulfurization skid ran on N_2 instead of syngas with no air flow until the plug was cleared and syngas flow was returned to the IM skid at 3:20 pm. This was EERC's first gasifier shutdown during the run. After this shutdown, the conversion to elemental sulfur significantly decreased and failed to return to a stable condition. This is likely because EERC began preparing to switch from a 100% coal feed to a coal/biomass blended feed to the gasifier just as the conversion to elemental sulfur started to reach 90% again. After the switch to the blended feed, which was accompanied by a 25% decrease in inlet sulfur concentration and an increase in inlet gas temperature, the conversion to elemental sulfur dropped again, but began to slow increase over the next day as the bed temperature decreased, as shown in Figure 11. Three more shutdowns occurred due to coal plugs and the loss of EERC's TRDU data acquisition system within the next 2 days. After each of these shutdowns, the conversion to elemental sulfur was similar to the result before the respective shutdown. Only after starting back up after the 13-hour shutdown on December 9, did the conversion begin to increase steadily again. The conversion to elemental sulfur returned to 90+% after 15 hours on stream. After the next shutdown, the conversion dipped again, but quickly increased and then remained stable as the syngas flow to the skid, and GHSV, increased. For the final two hours of the 112.5 hour run, the syngas flowrate was 1780 slpm, and the GHSV was 2717 hr^{-1} . For 4.5 hours before the flowrate increase, the conversion to elemental sulfur was stable

between 91% and 94%; after the increase, the conversion stayed in that range. As in the lab, increasing the GHSV 150% had little effect on the overall conversion to elemental sulfur. The effect of the oxygen to sulfur ratio (O/S) on the overall conversion was also minimal, as shown in Figure 11. The catalyst bed temperature, however, did affect the yield to elemental sulfur.

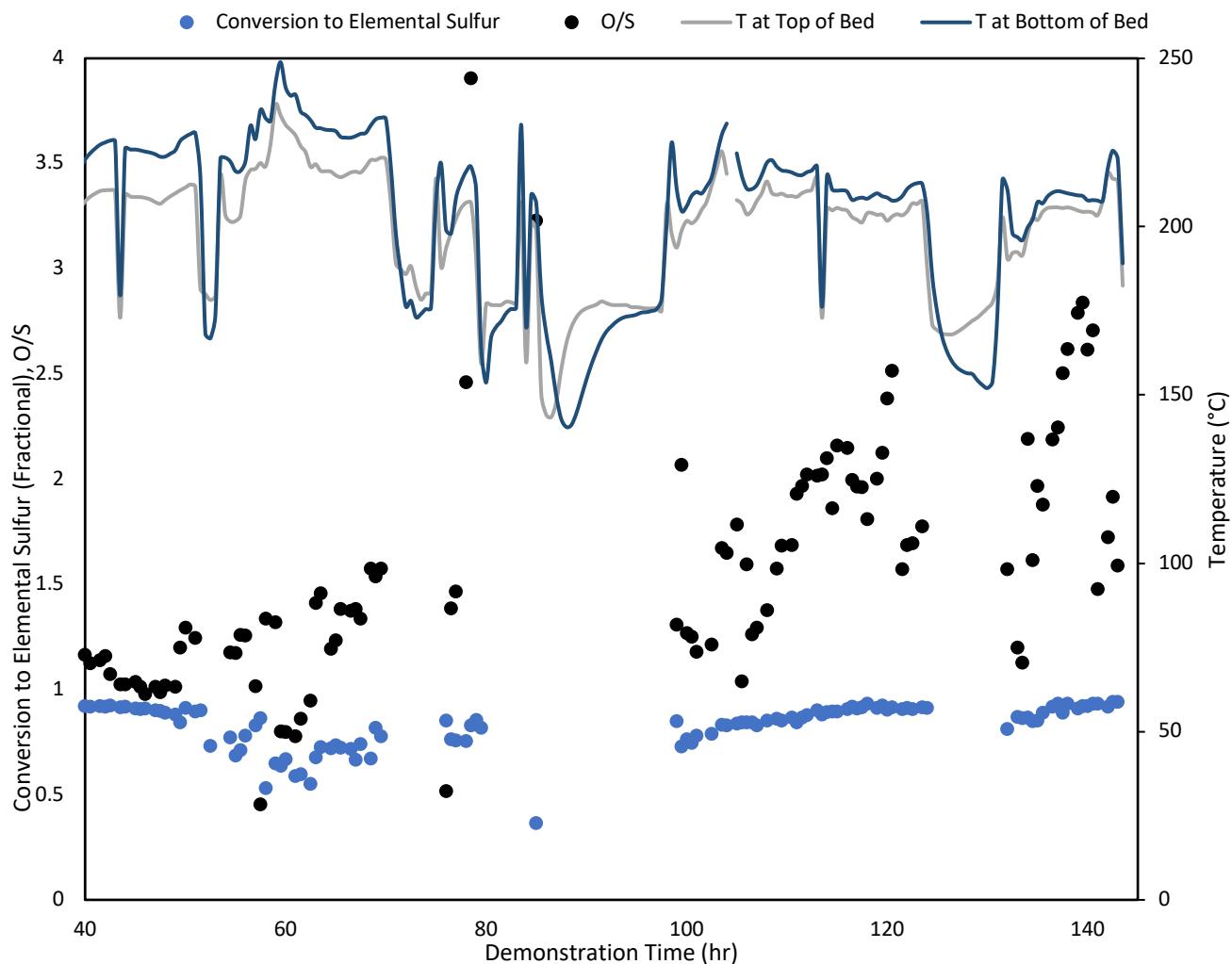


Figure 11: Conversion, O/S, and bed temperature vs. time. Note: The large dips in bed temperature correspond to gasifier shutdowns. Nitrogen ran through the system during shutdowns instead of hot syngas.

The period of lowest conversion coincides with the highest bed temperature spike. The bed temperature increased to 250°C at the bottom of the bed during the hours before the switch to the coal/biomass blended feed due to an increase in the inlet syngas temperature. As shown in Figure 12, the increase in bed temperature corresponded with an increase in the outlet H₂S concentration. The yield to SO₂ and COS remained comparatively stable throughout the run and were each capped below 5%.

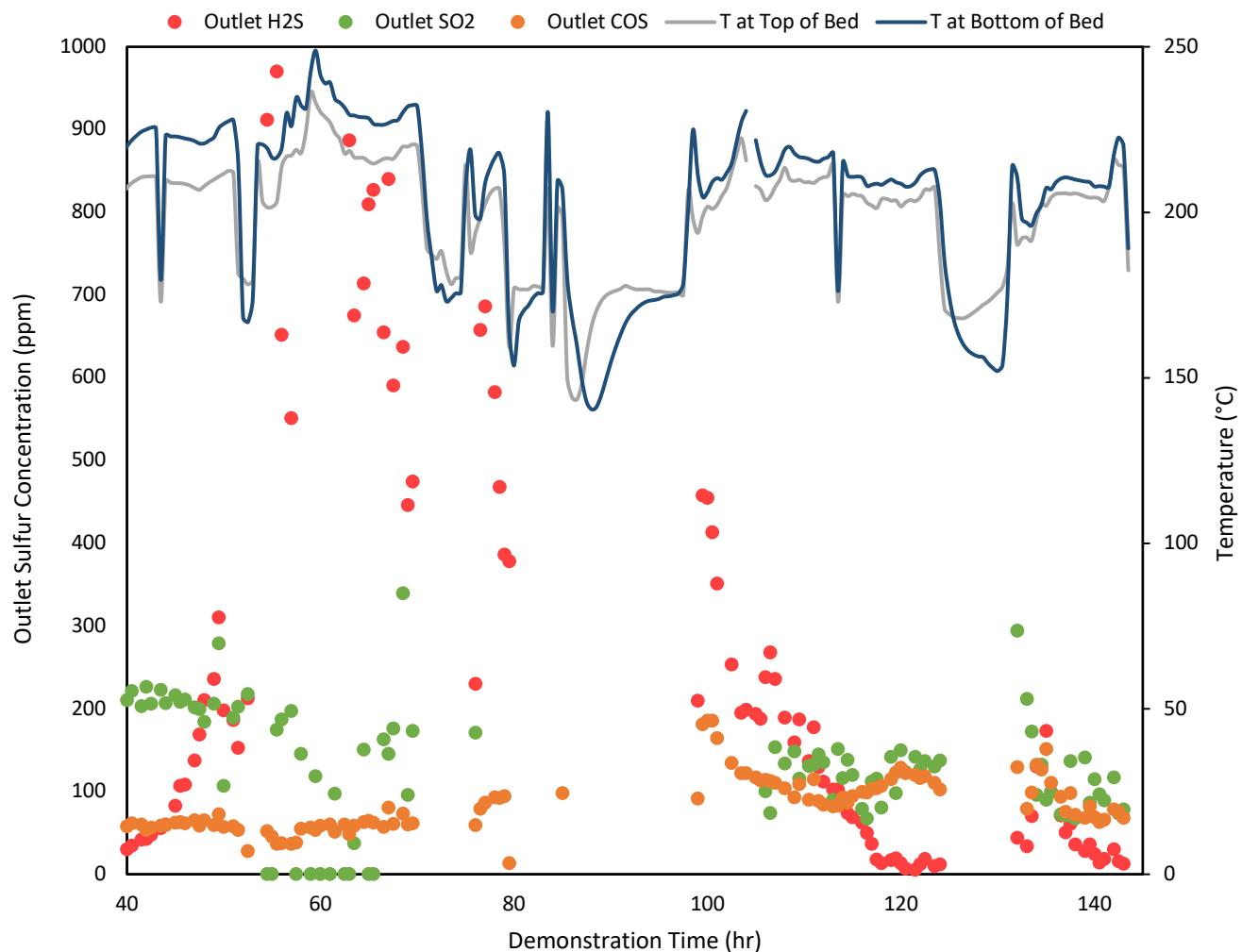


Figure 12: Outlet sulfur concentration and bed temperature vs. time. Note: The large dips in bed temperature correspond to gasifier shutdowns. Nitrogen ran through the system during shutdowns instead of hot syngas.

Despite the inconsistencies in the syngas inlet H_2S concentration and temperature and the multiple gasifier shutdowns, the OSR catalyst bed continued to convert H_2S into elemental sulfur, and the yield to elemental sulfur was much higher than the target of 65% for 107 hours of the 112.5 hour demonstration run. The yield to elemental sulfur was higher than the yield achieved in the lab with the same catalyst charge at the same operating pressure. In the lab, the temperature of the catalyst bed is uniform. During the demonstration, however, the temperature gradient from the top of the bed to the bottom of the bed ranged from 5°C to almost 20°C.

The OSR catalyst has proven itself robust. The catalyst was able to return to optimal performance after several shutdowns. The desulfurization system converted over 90% of the H_2S in coal-gasified and coal/biomass-gasified syngas to elemental sulfur in the presence of high steam and tar contents and despite fluctuations in temperature, H_2S concentration, O/S, and syngas flowrate.

c. What opportunities for training and professional development has the project provided?
IntraMicron's Co-Op Engineers have gained valuable experience working on a project that spans multiple disciplines. The young engineers have had to exercise skills that go beyond what is learned in the classroom to make a project on paper come to life. They are leaning how to effectively scale up a process and are continuing to learn research and development and manufacturing skills.

d. How have the results been disseminated to communities of interest?
Results have not yet been disseminated.

PRODUCTS

- a) Publications, conference papers, and presentations**
Nothing to report.
- b) Website(s) or other Internet site(s)**
Nothing to report.
- c) Technologies or techniques**
Nothing to report.
- d) Inventions, patent applications, and/or licenses**
Nothing to report.
- e) Other products**
Nothing to report.

IMPACT

- a) What is the impact on the development of the principal discipline(s) of the project?**
Nothing to report.
- b) What is the impact on other disciplines?**
Nothing to report.
- c) What is the impact on the development of human resources?**
Nothing to report.
- d) What is the impact on physical, institutional, and information resources that form infrastructure?**
Nothing to report.
- e) What is the impact on technology transfer?**
Nothing to report.
- f) What is the impact on society beyond science and technology?**
Nothing to report.
- g) What dollar amount of the award's budget is being spent in foreign country(ies)?**
None.

Appendix B: EERC Final Report

December 29, 2017

Mr. Joseph Hartvigsen
Senior Engineer
Ceramatec, Inc.
2425 South 900 West
Salt Lake City, UT 84119

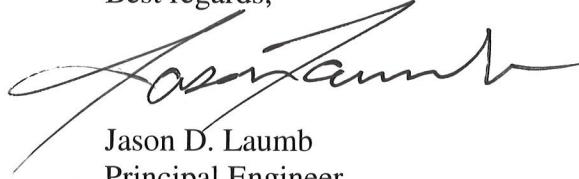
Dear Mr. Hartvigsen:

Subject: Final Report Entitled "Gasification Support for Ceramatec"; EERC Fund 21334

Please find attached the subject final report prepared by the Energy & Environmental Research Center (EERC).

If you have any questions or require clarification of any point, please contact me by phone at (701) 777-5114 or by e-mail at jlaumb@undeerc.org.

Best regards,



Jason D. Laumb
Principal Engineer
Coal Utilization Group Lead

JDL/kal

Attachment

GASIFICATION SUPPORT FOR CERAMATEC

Final Report

(for the period of September 14, 2016, through December 31, 2017)

Prepared for:

Joseph Hartvigsen

Ceramatec, Inc.
2425 South 900 West
Salt Lake City, UT 84119

Prepared by:

Jason D. Laumb

Energy & Environmental Research Center
University of North Dakota
15 North 23rd Street, Stop 9018
Grand Forks, ND 58202-9018

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GASIFICATION SUPPORT FOR CERAMATEC

INTRODUCTION

The Energy & Environmental Research Center (EERC) is pleased to submit this final performance report in support of the project entitled “Gasification Support for Ceramatec.” The EERC was tasked with providing clean syngas to support testing of a Fischer–Tropsch technology and sulfur removal technology provided by Ceramatec, Inc., and IntraMicron (IM). The goal was to provide coal–biomass-derived syngas for a 10-day period, resulting in a 10-gallon sample of a jet cut fuel and 100 hours of operation on the sulfur removal skid.

ACCOMPLISHMENTS AND DELIVERABLES

Gasifier Operation and Syngas Delivery

The EERC’s transport integrated gasification (TRIG) system operated in gasification mode for 89.8 hours on lignite and 87.7 hours on a lignite–biomass blend, for a total of 177.5 hours of syngas production. Table 1 is a chronology of TRIG operation, including disruptions in gasification. During the course of the test campaign, several events caused a break in syngas production. Most were related to fuel feed issues; in some cases, a small obstruction in the fuel feed line was cleared almost immediately and barely noticed in the gas composition; in other cases, feed was reestablished within a few minutes, resulting in a temporary decrease in combustible components in the gas.

Some fuel feed incidents lasted longer: specifically, the afternoon of December 7, the evening of December 8 into the morning of December 9, and the morning of December 13. On the afternoon of December 8, the coal feeder was intentionally stopped and the feeder depressurized to replace a level indicator in the fuel hopper.

Two other gaps in syngas production occurred. The morning of December 8, the TRIG data acquisition system tripped, resulting in about a 3-hour restart. The afternoon of December 10, the system was taken into combustion mode while waiting for a truck to refill the centerwide nitrogen tank. In each case, operators of downstream systems were notified, and actions were taken to minimize impact on downstream systems, such as taking systems off line or shutting off oxygen to the TRIG to eliminate the risk of oxygen breakthrough on downstream components.

Two fuels were used for these tests. The TRIG was started up on December 3 using a low-sodium lignite. This fuel was used through December 7 at 2300. At that time, a blend of lignite and biomass (wood) was used, in a biomass/coal ratio of 30/70 (by weight). The biomass blend was used through 1200 on December 13, at which time the system was switched back to lignite. Fuel feed rate for lignite gasification averaged 425 lb/hr for the biomass blend; the average feed rate was 406 lb/hr.

Table 1. Chronology of TRIG Operation

Date	Time	Activity
12/3/2017	2100	Start coal feed in combustion mode
12/4/2017	0836	Start gasification
	2120	Start syngas flow to National Center for Hydrogen Technology® (NCHT®)
12/6/2017	1902–1911	Coal feed plug
12/7/2017	1305–1404	Coal feed plug
	2300	Start feeding biomass blend
	2300–0015	Coal feed plug
12/8/2017	0804	Lost TRIG data acquisition system
	1100	Back in gasification
	1700–1726	Fuel feed off to replace feed hopper level indicator
	2220–0600	Fuel feed plug
12/9/2017	0600–0854	Combustion mode – heating up after fuel plug
	0854	Gasification mode
12/10/2017	1300–1950	Combustion mode – system nitrogen low
	1112–1115	Fuel feed plug
	2303	Open pipeline bypass to NCHT
	2353	Start IM process flow into TRIG (instead of thermal oxidizer)
12/11/2017	0033	Ceramatec taking syngas with IM flow
	0646	IM switching process flow back to thermal oxidizer
	0922	IM shutting down for the week
	1400	Ceramatec stopped taking syngas
	1640–1645	Fuel feed plug
12/12/2017	0128–0130	Fuel feed plug
12/13/2017	0846–0919	Fuel feed plug
	1207	Started feeding lignite
	1630	Transitioning to air-blown gasification
	1711	Combustion mode to burn carbon out of system
	1755	Fuel feed off

Syngas Composition

While syngas was being produced, gas composition was consistent. A water–gas shift (WGS) reactor was used to produce a H₂/CO ratio of 2.1 to 2.3. A portion of the syngas flow was bypassed around the WGS bed to allow for more accurate control of the ratio. Table 2 shows the average syngas composition for both the lignite and the blend, measured at the outlet of the sulfur-polishing bed. Nitrogen is high because nitrogen is used to purge pressure taps, refill coal and ash lock hoppers, and backpulse the candle filters in the particulate collection device.

Table 2. Syngas Composition, %

	Lignite	Biomass Blend
N ₂	34.64	34.49
CO	8.90	8.35
CO ₂	33.04	34.84
H ₂	19.14	17.34
CH ₄	2.05	3.26
H ₂ /CO	2.15	2.08

The difference in syngas quality between the two fuels was not dramatic. The lignite syngas had higher concentrations of both CO and H₂, while the biomass blend produced higher concentrations of CO₂ and CH₄.

IM Skid Operation

Upon arrival, this skid was reassembled and placed in the EERC's TRIG tower. After all of the plumbing and electrical hookups were completed, the system was pressure-tested and inspected. A standard operating procedure (SOP) was generated for skid operation.

The goal for the IM sulfur removal skid was to obtain 100 hours of operation on coal/biomass derived syngas. During the 100-hours of operation, sulfur removal levels varied from 60% to over 90%. The skid provided by IntraMicron has been removed from the TRIG tower and shipped back to IntraMicron.

Ceramatec Fischer-Tropsch Skid

Upon arrival this skid was reassembled and placed in the EERC's NCHT Fuels of the Future building. After all of the plumbing and electrical hookups were completed, a catalyst reduction was performed with house nitrogen and bottled hydrogen. A SOP was also created for safe skid operation.

The Fischer-Tropsch skid was provided coal-biomass-derived syngas for approximately a 10-day time frame. The average syngas composition is provided in Table 2. Liquid production from the skid was much lower than the anticipated 2 barrels per day. As such only 12 gallons of liquids was produced. It is not yet known by the project team why liquid production was low. A second catalyst reduction was performed during the test campaign to increase catalyst productivity. Catalyst productivity decreased after the second reduction. This skid has been successfully removed from the Fuels of the Future building, placed outside, covered with tarps, and awaits final disposition instructions.

Liquid Distillation

Approximately 12 gallons of liquids was produced by the skid. However, the liquids produced at the EERC were highly aromatic in nature and not representative of liquids typically produced by this catalyst. As a result, it was determined by Ceramatec that the distillation will

proceed with a 3.2-liter sample that was produced at the Ceramatec facility. The EERC's batch distillation column was utilized to produce a jet cut from these liquids. The product was distilled, resulting in approximately 1.7 liters of a jet fraction. Gas chromatography–mass spectroscopy (GC–MS) analysis was performed on the 12 gallons. The jet fraction was placed in an approved shipping container and awaits shipment to the U.S. Department of Energy.

MEETING ESTABLISHED GOALS

The goals of this project were threefold:

- Provide coal–biomass-derived syngas for a 10-day period of time.
- Achieve 100 hours of operation on the IM technology.
- Produce 10 gallons of a jet cut fuel.

The first two goals were met by the project team. The last goal of producing 10 gallons of a jet cut fuel was not met. It is unclear as to why the catalyst productivity was low and shifted toward aromatics during the testing.

ACTUAL OR ANTICIPATED PROBLEMS/RISKS

Several challenges were encountered during the 10-day testing campaign. However, many of the challenges were immediately addressed and resulted in very low down time with the testing skids. The challenges and methods to mitigate are summarized in Table 3.

Table 3. Challenges Encountered During Testing

Challenge	Impact	Solution
Fuel Feed Plugging	Low	Dislodge plug with rod or blast of house nitrogen.
Low Nitrogen Levels	Low	Place skids in standby while awaiting delivery of nitrogen. Monitor tank regularly.
Catalyst Liquid Production Low	High	Tried second reduction. Varied hydrogen to CO ratio.
Highly Aromatic Product	High	Distilled product from shakedown
Fuel Feed Sensor Malfunction	Low	Replace sensor.
Nozzle to Thermal Oxidizer Plugged	Low	Treat wastewater with filter barrels to remove tar.
Data Acquisition System Failure	Low	Closely monitor uninterruptible power supply battery level.

RELEVANT PUBLICATIONS

None.

NEW INVENTIONS OR TECHNOLOGIES

None.

1.0 PROJECT INFORMATION

Federal Agency	U.S. Department of Energy (DOE)
Funding Program	National Energy Technology Laboratory (NETL)
Award Number	DE-FE0023863
CFDA Number	81.089
Start Date	October 1, 2014
Program Title	Technology for GHG Emission Reduction and Cost-Competitive Mil-Spec Jet Fuel Production Using CTL

2.0 QUARTERLY REPORT REQUEST

The quarterly report covering the CTL program activities from October 1 through December 31, 2017, is due for submission to NETL on January 31, 2018. This memo outlines the subaward quarterly reporting requirements for the program as mandated by DOE.. *If any of the sections below do not apply to your research at this time, please mark them as "Nothing to Report."*

1 ACCOMPLISHMENTS: Mandatory

a) Major goals of the project

Ceramatec, Inc., requested that the Energy & Environmental Research Center (EERC) assist with participation in a coal-to-liquids (CTL) project as a gasification partner for an existing Ceramatec contract with DOE NETL regarding production of jet fuel from coal or coal-biomass blends.

The project is funded by the U.S. Air Force but managed through DOE NETL. The overall objective of the program is to produce a synthetic fuel from coal or coal-biomass blends that is suitable for use in aircraft (Jet A or JP-5). A subsidiary objective is to produce the synthetic fuel with less carbon emissions than are emitted by processing natural petroleum. Since this is unlikely to be possible with the primary input being coal, the program allows the use of up to 49% of the energy to be input from biomass. Ceramatec is constructing a nominal two (2)-barrel-per-day Fischer-Tropsch (FT) reactor, and IntraMicron, Inc., is constructing a sulfur removal system consistent with cleaning the amount of synthesis gas required for a reactor of that size.

The EERC scope of work includes fuel preparation/allocation, gasification of coal-biomass blends, analysis of produced liquids, and distillation of the liquids into a final product.

In order to support the overall project goal of delivering 10 gallons of Jet A liquids, the EERC is performing the following tasks.

Task 1 – Site Planning and Preparation

The EERC will prepare gasification equipment as well as technology demonstration space to support installation of the sulfur reactor and FT skid provided by Ceramatec. While many of the components to support this work are already at the EERC, it is expected some slight modifications will be needed. Several up-front planning meetings will be necessary with the entire project team to ensure proper project integration, including scheduling and time lines.

Task 2 – Installation of Skid-Mounted Equipment

The EERC will support the project team in the installation and integration of the sulfur removal skid, FT reactor system, and associated equipment. It is anticipated that the FT skids will be installed in the Fuels of the Future building at the EERC, while the IntraMicron sulfur removal system will be located in the gasification systems tower. As part of the installation process, a rigorous hazardous operations (HAZOP) review will be conducted with the entire project team. The EERC anticipates using review processes agreed upon by the project team and the EERC safety office. EERC engineers, machinists, and technology development operators will be available to support this effort. Prior to equipment arriving on the EERC campus, a trip will be made to Ceramatec's facilities to view the skids in operation in order to inform the HAZOP review and EERC installation efforts.

Task 3 – Subsystem Testing

EERC staff will support the testing of Ceramatec's sulfur removal system as well as the FT reactor. The EERC's transport reactor development unit (TRDU) gasification system will provide the necessary syngas at a nominal hydrogen/carbon monoxide ratio of 2:1 as specified in the information package provided by Ceramatec. The TRDU can be operated in oxygen-blown mode in order to minimize nitrogen dilution, producing a syngas stream at a rate of approximately 250 or 400 scfm in air-blown mode. The pressure and temperature of the syngas will be adjusted as necessary with heat exchange equipment and in-house syngas compressors. Excess syngas will be routed to a thermal oxidizer already installed at the EERC.

The coal to be used will be a North Dakota lignite sourced by the EERC. Biomass material will be sourced and secured by the EERC; however, discussions will take place with the project team prior to finalizing a source. The EERC has the capability to prepare the necessary solid fuels for this work. The TRDU has been successfully operated on a wide range of coal–biomass blends.

Additional work with the Lignite Energy Council (LEC) will involve gasifier operation on North Dakota lignite syngas, corrosion coupon testing, and slipstream impurity removal tests. Both projects will benefit from mutual sharing of data and information as well as sharing of syngas resources.

To ensure that 100 hours of operation is attained, the EERC proposes a 10-day or two 5-day TRDU testing campaigns. Additionally, the EERC will supply a sulfur removal system that can be used in reserve if challenges are experienced with the vendor-provided skid. The backup system will not only protect the FT catalyst from excess sulfur but will also prevent shutdown during a testing campaign.

Task 4 – Liquid Distillation and Analysis

Existing EERC distillation equipment, previously described, can be used to create the Jet A fraction desired. In addition, EERC laboratories will be used to analyze liquid fuel samples throughout the subsystem testing and distillation tasks. This will ensure proper delivery of a Jet A fraction.

Task 5 – System Decommissioning

Upon completion of the subsystem testing effort, equipment will be decommissioned. This effort will be conducted in concert with Ceramatec and other project team members to ensure successful removal of the equipment. The FT skid and sulfur removal system will be disconnected and removed, and the

high-bay area will be restored to its original state of readiness for subsequent use.

Task 6 – Management and Reporting

This task will support management and reporting efforts at the EERC. A detailed report describing gasifier operations, syngas composition, heat/material balances, and lessons learned will be provided upon completion of the liquid fuels testing. It is also anticipated that the EERC will be involved in at least one face-to-face project review meeting and multiple teleconferences.

b) Accomplishments this Quarter

Task 1 – Site Planning and Preparation

- Final piping connections were made for both skids.
- Safety review
 - Final HAZOP meetings were held and standard operating procedures were created for each subsystem.
 - Several actions items were identified, and Ceramatec and EERC employees made necessary changes for safe operation.

Task 2 – Installation of Skid-Mounted Equipment

The Ceramatec and IntraMicron skids were delivered to the EERC in November (6th and 3rd respectively) of 2017. Both skids were placed indoors immediately and final electrical/plumbing connections were made. Each skid underwent a final electrical safety inspection and was pressure-tested prior to operation.

Task 3 – Subsystem Testing

Both subsystems were provided coal/biomass-derived syngas from December 3 to December 13, 2017. Several minor interruptions in syngas delivery were encountered during the test. Most interruptions were due to fuel feed plugs. Over 100 hrs of operation were achieved on the IntraMicron skid, while the Ceramatec skid produced 12 gallons of liquid fuel.

Task 4 – Liquid Distillation and Analysis

An initial analysis of the liquids produced during the testing indicated a high degree of aromatic compounds, which is not common for this type of catalyst. As a result, liquids produced during commissioning at Ceramatec were used in the distillation task. Approximately 1.7 liters of a jet fuel cut were obtained from approximately 3.2 liters of liquid fuel. The fuel is awaiting transfer to DOE.

Task 5 – System Decommissioning

Decommissioning activities began immediately following the testing campaign. Both skids were disconnected and removed from EERC buildings. The IntraMicron skid was shipped back to IntraMicron on December 26, 2017. The Ceramatec skid has been placed on blocks and covered with tarps.

Task 6 – Management and Reporting

- Meetings
 - The EERC has conducted no fewer than one internal design team meeting a week, dependent upon need.
 - ♦ Conference calls with project partners have been convened.
 - ♦ E-mails and phone conversations have occurred as needed.
 - ♦ Document exchange has been initiated between partners.

- Reporting
 - The EERC has completed a project final report that briefly summarizes activities at the EERC. The report has been forwarded to Ceramatec.

2 PRODUCTS: Mandatory

What has the project produced?

Nothing to report.

- a) **Publications, conference papers, and presentations**
 - **Journal publications.**
Nothing to report.
 - **Books or other non-periodical, one-time publications.**
Nothing to report.
 - **Other publications, conference papers and presentations.**
Nothing to report.
- b) **Website(s) or other Internet site(s)**
Nothing to report.
- c) **Technologies or techniques**
Nothing to report.
- d) **Inventions, patent applications, and/or licenses**
Nothing to report.
- e) **Other products**
A 1.7-liter sample of jet fuel.

3 IMPACT: Optional

What is the impact of the project? How has it contributed?

- a) **What is the impact on the development of the principal discipline(s) of the project?**
Nothing to report.
- b) **What is the impact on other disciplines?**
Nothing to report.
- c) **What is the impact on the development of human resources?**
Nothing to report.
- d) **What is the impact on physical, institutional, and information resources that form infrastructure?**
Nothing to report.
- e) **What is the impact on technology transfer?**
Nothing to report.
- f) **What is the impact on society beyond science and technology?**
Nothing to report.
- g) **What dollar amount of the award's budget is being spent in foreign country(ies)?**
\$0.00

Appendix C: SES Final Report

This serves as the SES draft final report for the project.

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Summary

An innovative CO₂ mitigation technology called cryogenic carbon capture (CCC) requires less energy and costs less than alternatives. This post-combustion technology cools flue gas to CO₂ desublimation temperatures (−100 to −135 °C), separates the solid CO₂ that forms from the flue gas—from the remaining light gases, warms the cold products by cooling the incoming gases in a recuperative heat exchanger, compresses the solid/liquid CO₂ to final pressures of 100–200 bar, and delivers a compressed liquid CO₂ stream and an atmospheric-pressure, light-gas stream, both near the initial stream temperature. Detailed analysis estimate overall energy and economic costs about 50% lower than most competing processes that involve air separation units (ASUs), solvents, or similar technologies. In addition, the CCC process enjoys several ancillary benefits, including (a) it is a minimally invasive bolt-on technology, (b) it provides highly efficient removal of most pollutants (Hg, SO_x, NO₂, HCl, etc.), (c) it recovers flue gas moisture which typically exceeds the process cooling water demand, and (d) it enables grid-level, highly efficient and cost effective energy storage. This paper outlines the process details and economic and energy comparisons relative to other well-documented alternatives.

This process has been applied to three syngas applications in the context of the project to generate transportation fuels from biomass or other solid fuel sources. This document outlines a detailed analysis of energy and cost demands for a standard flue gas and specific energy estimates for three different syngas applications, concluding with the syngas analysis most suited to the most recent version of the current project. The cost estimates provided here are based on detailed comparisons with flue gas treating, which have well-documented cost and energy numbers. The syngas application does not have comparable detailed costs for comparison, and the cost estimates for the syngas applications depend primarily on the costs of the other portions of the process which are detailed by other partners of this project. The energy costs of this technology are documented in detail here, together with a sensitivity analysis.

Introduction

Energy and related environmental issues span national and regional boundaries and influence many generations. Their solutions require comparable scope and duration. Many people envision a future dominated by non-fossil energy generation, a dramatic departure from the current infrastructure. However, solutions to the most daunting energy issues cannot wait for a fossil-free energy future. There is a critical need for an energy supply and utilization infrastructure based on currently available processes that provides for current needs while greatly reducing the environmental consequences to future generations [1]. These global and multi-generational environmental concerns include traditional and climate change pollutant emission, energy-driven water demand, resource depletion, greater and more effective use of renewable energy, and economics. The CCC technology addresses most of these issues.

Fossil fuels provide about 85% of US and global energy. **Error! Reference source not found.** illustrates that the recent past and projections for at least the next generation indicate that fossil fuels will continue to dominate global energy use, even accounting for recent declines in the North America and Europe. Most of the renewable energy replacements for fossil fuel have enjoyed impressive cost reductions in the recent years and often compete well with fossil fuels. However, they primarily address

power generation and are non-dispatchable, with predicted availabilities that extend only into the very short term. It is difficult to imagine a near-term, fossil-free infrastructure. Fossil fuels also dominantly contribute to global climate change. For these reasons, many climate change experts identify carbon capture and storage (CCS) as not just a possible, but an essential energy technology[add iea and related references].

A recently patented process for CO₂ removal from flue gas [2] [3] [4] portends a substantial decrease in both cost and energy consumption compared to solvent- and oxygen-based systems. The current standard carbon capture process to which new processes are compared is an amine CO₂ capture system. This technology involves flowing the flue gas through an absorber column where the CO₂ is removed from the flue gas via absorption into the amine which is dissolved in water. This liquid stream, which is then composed of predominantly water with small amounts of dissolved amine and CO₂, is then heated in a desorption column in order to vaporize the CO₂ as a relatively pure gas (contaminants would include water and a small amount of amine). This gaseous CO₂ stream is then further compressed to nominally 150 bar. The two main sources of the parasitic load in an amine process are the cyclical heating and cooling of the water/amine stream, and the compression of the CO₂ gas from a gas that is nominally at atmospheric pressure to a liquid at 150 bar. The heating is accomplished via low pressure steam, which is directed into the amine process rather than providing power via utilization in the low pressure section of the steam turbine. By comparison, the main sources of the parasitic load in the CCC process are the power required to run the refrigeration loops and the power required to run the forced draft fan to overcome process pressure drop. The CCC process utilizes optimized heat integration in order to minimize the amount of cyclical heating and cooling of process streams. Additionally, the CCC process compresses a solid/liquid CO₂ stream whereas the amine compresses a gaseous CO₂ stream, both from nominally atmospheric pressure to about 150 bar. Solids and liquid compression requires a very small fraction of the energy required for gaseous compression. Figure 1 illustrates the sources of energy demand from amine and CCC.

Amine				
	Heat Exchange	Desorber	Compression	Refrigeration
Energy Demand	Small	Intermediate	Intermediate	None
CCC				
	Heat Exchange	Desorber	Compression	Refrigeration
Energy Demand	Small	None	Very Small	Intermediate

Figure 1 - Comparison of the primary energy demands in an air-separation-based unit and in the cryogenic carbon capture process

CCC integration strategies

While the CCC process requires essentially no changes to the upstream process, there are several integration steps that provide significant ancillary benefit compared to existing systems. Most of these involve either removing existing process steps or very simple low-grade heat and cooling integrations. One of the core ancillary benefits of CCC is that it can replace current state-of-the-art pollutant removal technologies and capture all criteria pollutants including Hg and air toxics, with the notable exception of CO, more efficiently than current best available control technologies. Using available vapor pressure data and correlations, Figure 2 shows the capture level of key pollutants over the range of temperatures required by the CCC technology. Table 3 gives the exact predicted capture of these pollutants at the temperature corresponding to 90% CO₂ capture.

Since CCC is able to capture pollutants so effectively, it has the capacity to replace current capital equipment such as flue gas desulfurization (FGD) units for SO_x reduction and selective catalytic reduction (SCR) units for NO_x reduction in any greenfield installation and will bring any retrofitted plants well past current standards for pollutant levels of the stated compounds. The ability to replace the FGD unit also allows for additional integration that can vastly improve power plant efficiency. Current

greenfield coal-fired power plants utilize steam to heat the boiler feed water back to the temperature at which it enters the coal boiler. If there is no FGD unit, the flue gas exiting the bag house in a power plant that utilizes CCC technology must nevertheless be cooled to ambient temperatures. Rather than using additional cooling water, this cooling can be performed in a countercurrent heat exchanger with the boiler feed water, a stream that benefits from the low-grade heat that can be extracted from the flue gas. This increases the flow rate of steam through the low-pressure turbine by decreasing the load of the heat recuperators, and improves the cycle efficiency of the power plant. The energy penalty and cost of CCC can also decrease via the removal of the FGD and implementation of boiler feed water heating. The specific effect of these integrations will be addressed in detail later in this paper.

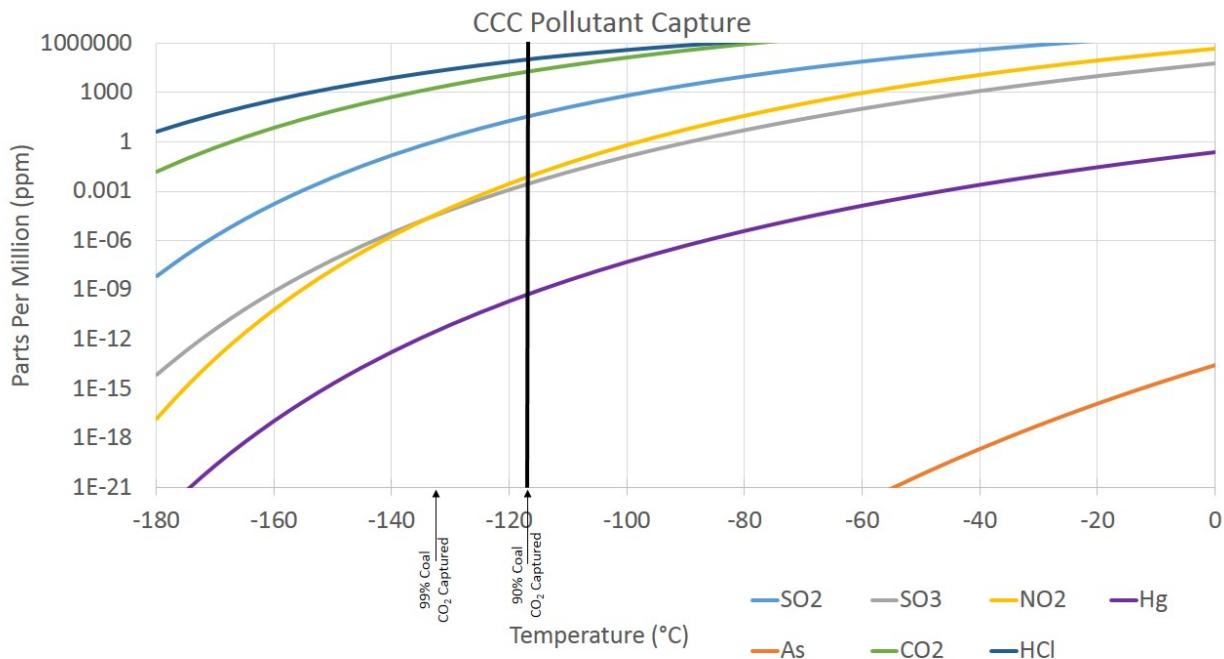


Figure 2 - Pollutant Capture at CCC temperatures

Table 1 - Pollutant levels at 90% CO₂ capture in parts per million

Pollutant	ppm
SO ₂	32.73
SO ₃	0.002
NO ₂	0.007
Hg	4.83E-10
As	7.31E-37
CO ₂	17882

An additional integration that can be performed is to utilize the cooling capacity of the dry light gas stream that exits the CCC process. This stream can be utilized to create a large amount of chilled water. Utilizing this integration allows for the CCC process to meet its own chilled water demand.

Processing Modeling and Validation Improvements

Baseline CCC ECL™ Techno-Economic Modeling

SES has performed detailed cost and energy analyses regarding the CCC ECL™ technology. For all costing calculations, SES utilized the Excel-based costing program Power Systems Financial Model (PSFM) developed by NETL, which program NETL used to develop the reported cost estimates for the amine system. We have utilized this model with the same assumptions as are present in the September 2013 release of the Cost and Performance Baseline for Fossil Energy Plants [22]. This allows SES to estimate the cost of electricity (COE) and energy penalty without introducing error caused by differing assumptions in the financial models, ancillary costs, and contingencies. We compare CCC with two case studies published and periodically updated by NETL. These are NETL Case B12A, which presents a greenfield 550 MW net coal-fired supercritical pulverized coal (SC PC) power plant without carbon capture, and NETL Case B12B, which presents a greenfield 550 MW net coal-fired SC PC power plant with 90% CO₂ capture achieved via amine absorption. The CCC simulations here also assume a 550 MW_e net SC PC power plant.

Updating the CCC ECL™ Techno-Economic Modeling

To expand and update the techno-economic analysis of the CCC ECL™ process, we have continued to optimize both the software used to model the process as well as adjusted the refrigerant profiles and compositions to reduce the energy penalty. These adjustments stem from research into improved alternatives to the refrigerant loops used to cool various portions of the CCC ECL™ process.

Improved In-House Software

As part of the task to complete the techno-economic analysis, SES has implemented two new unit operations in its in-house simulation software. We plan to test both units in the near-term on the skid-scale process, and we expect to use these units on larger pilot demonstration designs and full-scale installation designs.

The first of these units, a distillation column, has been fully implemented within the software. The implementation is based on an equilibrium, rather than a transport-driven, model. SES is currently building a distillation column for use on the existing skid-scale system to purify the liquid stream of CO₂ from the screw press and melter. The column's design was based on simulations using the new Distillation Column unit operation. Currently, distillation is the preferred method of purification moving forward to the pilot and full-scale systems, so the newly implemented unit will be invaluable in simulations to estimate the economics of future CCC systems and processes.

A sample output from a seven-stage distillation column simulation is shown in Figure 3.

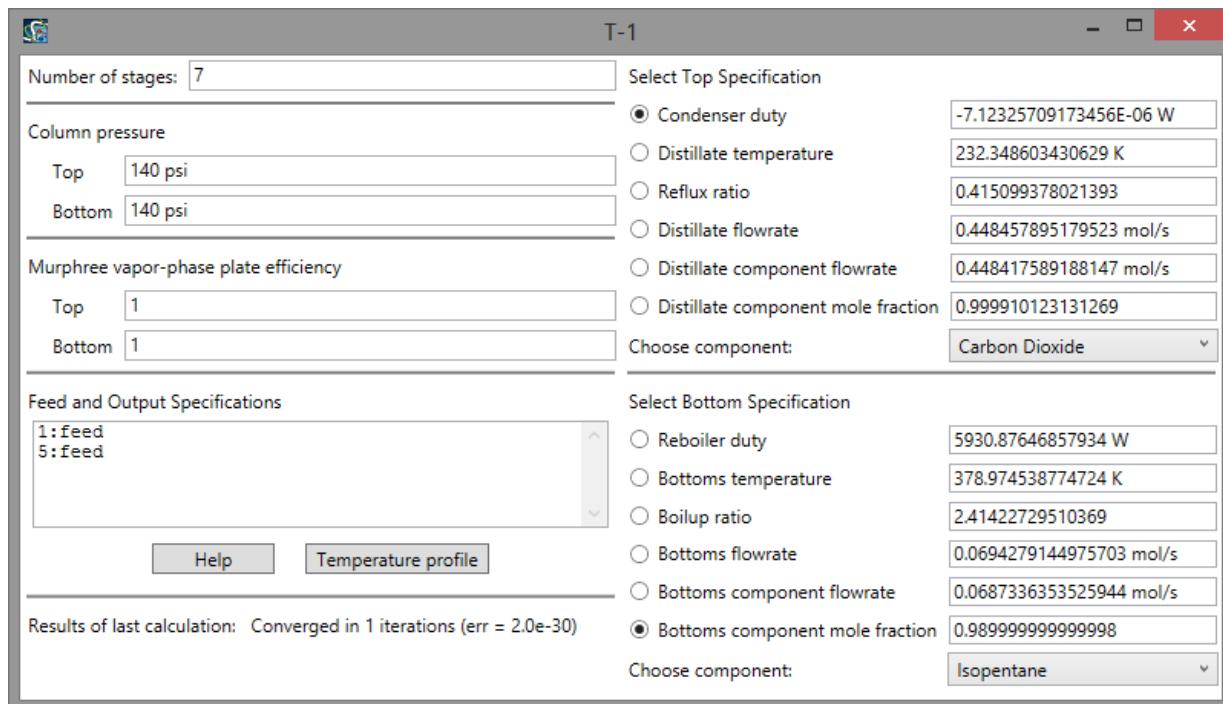


Figure 3. Example output from the new Distillation Column unit operation from a simulation of purifying CO_2 in the skid-scale process.

We have also implemented a Spray Tower unit operation that simulates both counter- and co-current spray towers. Unlike the Distillation Column model, this model is transport-driven, making it substantially more complex. A sample of the output from this simulator is shown in Table 2. We have integrated the model into our full simulation package to enable general, process-wide simulation.

Table 2. Partial output from SES' standalone Spray Tower simulator.

Position (m)	Tg (K)	Y _{CO2-g}	Y _{HC-g} (ppm)	V _g (m/s)	T _{ps} (K)
-0	167.5043	0.03211541	0.199351	1.007273	140.2413
-0.2857143	168.9253	0.04803211	0.1915751	1.013042	144.8199
-0.5714286	169.9967	0.06190523	0.2024531	1.017381	148.5724
-0.8571429	170.8725	0.07741574	0.2067055	1.020924	152.1862
-1.142857	171.5905	0.09459024	0.1985785	1.023825	155.8012
-1.428571	172.2049	0.1131736	0.1680003	1.026306	159.1241
-1.714286	172.6765	0.1326922	0.1004149	1.028212	162.013
-2	173	0.15	0	1.029532	163.1909

The simulations continue to be updated with improvements that have been developed during experimental testing. The most notable of these improvements is the development of the novel drying techniques from Task 2. The direct-contact dryer described above has major ramifications on both the parasitic load and capital cost of the pilot-scale and full-scale processes. We have utilized the data from Task 2 to simulate a direct-contact dryer on the full-scale process to be able to have a direct comparison between direct-contact drying and a fixed-bed dryer.

Independent Techno-Economic Analysis

Additionally, collaborative work with EPRI to establish their own independent techno-economic analysis has made significant progress. Our collaborative work has resulted in their development of their own heat and mass balance. A copy of their work is included in this report.

Energy Performance Comparisons

One of the key aspects of any carbon capture and storage (CCS) technology is the parasitic load that must be absorbed by the power plant for the separation and pressurization of CO₂. The efficiency of the separation and pressurization steps ends up being manifest in the real-world cost of generated electricity. A power plant with a higher parasitic load requires more fuel to achieve the same output. The plant itself, and all of its components, must be larger in order to handle these larger fuel flow rates. Additionally, the amount of gas that then needs to be processed increases, thus requiring that both the equipment and total loading for the CCS technology be larger.

It becomes important at this juncture to establish a baseline against which the CCC technology will be measured. The National Energy Technology Laboratory (NETL) of the United States Department of Energy (DOE), has published a report entitled “Cost and Performance Baseline for Fossil Energy Plants Volume 1a: Bituminous Coal (PC) and Natural Gas to Electricity” [22]. This report contains detailed mass and energy balances for 6 power plant configurations, each labeled with a case number. In addition to mass and energy balances, each case contains detailed estimates for capital expenditures, operating costs, consumables and fuel costs, etc. These numbers are then amortized to create a reported cost of electricity, allowing for a financial comparison between the different power plant options on the same terms. Cases B12A and B12B of this report concern themselves with a supercritical (SC) pulverized coal (PC) power plant, and will be used as the baseline for comparison in this paper. Case B12A is the detailed study of a greenfield SC PC power plant installation without carbon capture, providing a baseline for energy and cost comparisons. Case B12B is the detailed study of a greenfield SC PC power plant installation with carbon capture. The CCS technology chosen for the Case B12B study is an amine CO₂ capture system that captures 90% of the inlet CO₂. In both cases, the net power generation capacity of the power plant is 550 MW. All mass balance, energy balance, and cost numbers are quoted from or based on the Revision 3, July 2015 version of the report [22]. In order to stay as close as possible to the report, all CCC simulations were carried out at 90% capture, although the CCC process can easily cope with capture efficiencies at and above 99%.

There are multiple ways to measure the energy penalty associated with a CCS technology. This work will analyze the energy penalty of CCC and compare it to the published amine system in terms of the processed output CO₂ stream and in terms of the effect on the power plant. The first of these is presented in the forms of electric gigajoules required per metric ton of CO₂ produced (GJ/tonne). This number is instructive in that it provides a scalable value that can then be applied to other similar flue gas streams. The effect on the power plant is presented in terms of the percent increase in the net plant high heating value (HHV) heat rate. This was chosen because it is a key parameter stated in the NETL report and used in costing simulations. The net plant HHV heat rate is the amount of thermal energy (based on the HHV of the fuel) input per unit of electricity output, and is presented in the NETL report in

units of BTU/kWh. This number is instructive because it takes into account the effect of decreasing the total amount of fuel required to achieve the same net power plant output.

Energy Performance of NETL Cases B12A and B12B

As previously stated, Case B12A of the NETL report is a non-capture SC PC power plant. As such, the associated energy penalty in terms of processed CO₂ is 0.00 GJ/tonne since there is no CO₂ capture. The net HHV heat rate for Case B12A is 8,379 BTU/kWh. The energy penalty of Case B12B is not explicitly stated in the report in terms electric energy per unit of processed CO₂. The reason for this is because a large amount of the energy penalty associated with absorption technologies is through redirection of steam to heat the amine during the desorption process. The potential energy that could have been extracted by a steam turbine represents the electric energy penalty of this heat redirection. While this number is not stated in the report, it can easily be calculated assuming that the turbine efficiencies of Case B12A and Case B12B remain constant. Doing so results in an energy penalty of 1.05 GJ/tonne for Case B12B. The net HHV heat rate is given explicitly in the report as 10,508 BTU/kWh, which indicates a parasitic load of 20.28%.

Energy Performance of CCC

Detailed thermodynamic process simulations of the CCC process have been performed in order to quantify the energy penalty associated with utilizing the CCC technology. These simulations have been independently verified by, among others, American Air Liquide, General Electric, Booz Allen Hamilton, and the Pacific Northwest National Laboratory. For the process simulations detailed in this report, turbomachinery isentropic efficiencies, including the refrigerant compressors, were assumed to be 90%. All pump efficiencies were assumed to be 85%.

The energy penalty associated with CCC is due almost exclusively to the work needed to power the compressors and pumps in the process. There is also a small load associated with the steam required for running the distillation column. This has been quantified in terms of electric power (MW_e), which is the equivalent power that could be extracted from this stream if it were run through the low pressure turbine. However, the combined work of the refrigeration compressors and flue gas blower account for greater than 93% of the overall energy penalty. Table 3 contains an itemized list of the parasitic loads in the CCC process with an initial inlet gas matching that reported in the NETL report (i.e. a net 550 MW_e plant).

Table 3 - List of Parasitic Loads for CCC

Energy Source	Work (MW _e)
Flue Gas Compression	8.4
Refrigerant Compression	89.5
Separations Compression	0.3
Condensed Phase Pumping	4.1
Steam Redirection	2.6
Total	105.0

The real benefit of the CCC numbers can be seen in the established energy metrics stated above. In terms of the processed outlet stream, the energy penalty of CCC is 0.841 GJ/tonne. Its calculated net HHV heat rate is 10,011 BTU/kWh, corresponding to a parasitic load of 16.30%. The parasitic load of the CCC process as-is, without any additional integration, is approximately three quarters of the corresponding parasitic load of the amine process.

Energy Performance of Integrated CCC

As stated above, the CCC technology allows for further integration by removal of capital equipment for pollutant remediation and utilization of the low-grade heat available in the flue gas. Additionally, removal of the FGD unit does allow for greater utilization of the low-grade heat, as detailed above. Utilizing “Exhibit 3–47 Case B12A heat and mass Balance, supercritical steam cycle” from the NETL report allowed for precise calculation of the energy required for reheating the boiler feed water and the extra power that is generated by the larger steam flow rate through the low-pressure turbine. Utilization of the heat extracted from the flue gas allows for approximately 47% of the boiler feed water to be heated to 298 °F (147.8 °C), the temperature at which it enters the de-aerator before further pressurization and heating. This means that 47% of the steam which was previously extracted and used for heating is now able to proceed through the entirety of the turbine. This additional steam accounts for an additional 5.5% increase in the produced power of the low-pressure turbine that would not be otherwise available. In the case of an integrated CCC process, this amounts to approximately 14.3 MW of additional electricity to offset the energy penalty. The additional power results in a lower amount of fuel required to meet the net 550 MW requirement, and further decreases the size and power requirements of the CCC equipment. As every power plant is unique, not every plant will be able to utilize the entire benefit of the CCC pollutant capture value proposition. Therefore, each integration has been considered independently. These independent cases are:

1. CCC with steam cycle integration and including all pollutant removal equipment
2. CCC with removal of the FGD unit but without steam cycle integration and inclusion of the other pollutant removal equipment
3. CCC with the removal of the SCR unit but without steam cycle integration and inclusion of the other pollutant removal equipment
4. CCC with the removal of the mercury mitigation equipment but without steam cycle integration and inclusion of the other pollutant removal equipment
5. CCC with full cumulative benefits of all integration, e.g. steam cycle integration and no additional pollutant removal equipment beyond CCC

The energy penalty of the SCR and mercury removal are almost negligible (40 kW and 22 kW for Case B12A, respectively), although their capital cost and operating cost are large. There is some parasitic load associated with the operation of the FGD (~3 MW), and its value has been included in the calculations based on the stated values of the previously cited NETL report. Table 4 presents a summary of the energy penalties for these differing integration strategies.

Table 4 - Summary of Energy Penalty of CCS technologies

	Case B12A	Case B12B	CCC	CCC Steam Integration	CCC no FGD	CCC no SCR	CCC no Hg Removal	CCC Full Integration
Power Needed (GJ/tonne CO₂)	0.000	1.047	0.841	0.673	0.799	0.840	0.841	0.630
HHV Heat Rate (BTU/kWh)	8379	10508	10011	9638	9915	10009	10010	9548
Parasitic Load	0.00%	20.28%	16.30%	13.06%	15.48%	16.29%	16.29%	12.24%

One important note regarding Case B12B is that the results are based purely on simulations and do not incorporate any data from actual installations. The Boundary Dam Power Station in Saskatchewan uses the Cansolv amine process that is used for Case B12B. Their reported parasitic load is approximately 30%, which is a 50% increase in parasitic load from the numbers reported in Case B12B [23]. Additional techno-economic carbon capture reviews also suggest that the findings of Case B12B might be optimistic regarding the energy requirements of amine carbon capture (Table 5) [24]. These techno-economic studies come from the US, Europe, China, and Australia, with similar assumptions as those used in the NETL study (e.g., amine-based processes, flue gas pre-processing to enable amine CCS, compression of the final CO₂ stream). While the parasitic load varies based on report assumptions, the power needed is fairly constant across the board, and it is about 35% more than the reported value from Case B12B.

Table 5 - Energy requirements for amine-based CCS technologies, based on reports from the US, Europe, China, and Australia.

	CMU	EPRI	TNO	TPRI	CSIRO
Power Needed (GJ/tonne CO₂)	1.42	1.41	1.52	1.44	1.42
Base Plant HHV Heat Rate (BTU/kWh)	8676	8979	7982	8257	8868
Plant with Capture HHV Heat Rate (BTU/kWh)	11402	12342	11586	11439	12053
Parasitic Load	23.91%	27.25%	31.11%	27.82%	26.42%

Cost Comparisons

Cost comparisons were performed in a similar manner to the energy penalty calculations, using the NETL reports as an outline for all financial assumptions. Additionally, the NETL-developed Excel-based costing program, Power Systems Financial Model (PSFM), was used for all cost of electricity (COE) calculations. PSFM was also used to develop the reported cost estimates for Case B12A and Case B12B. This has allowed for all COE estimates to be performed without introducing error caused by differing assumptions in the financial models, ancillary costs, and contingencies.

Cost of Electricity (COE) of the NETL Cases

All cost details can be found in the reports cited above. The COE for Case B12A, as reported by NETL, is 82.3 \$/MWh. The corresponding COE for Case B12B, including the transportation, storage, and monitoring (TS&M) of the processed CO₂, is 142.8 \$/MWh, which corresponds to an increase of 73.5% compared with Case B12A. This increase takes into account the extra fuel and increased size of the base power plant needed to achieve the net 550 MW requirement. Additional cost increases can be attributed to the increased operating and maintenance requirements of the larger plant, including the CCS portion, and an increase in contingencies based around the inherent risks involved with CCS implementation.

Cost of CCC

Staying as true to possible to the costing assumptions available, and utilizing real-world cost estimates and vendor quotes for capital cost totals, the COE for a greenfield power plant installation utilizing CCC as a capture technology was also calculated. This includes increases in fuel, operating, maintenance, and capital costs for scaling up the power plant due to the parasitic load in addition to the cost of the CCC process equipment itself. The resulting COE for CCC is 119.7 \$/MWh, which corresponds to an increase of 45.4%, a little over half the increase in cost of Case B12B.

If the extra benefits of an integrated CCC process are taken into account, then the COE calculation further improves. The pollutant mitigation units each represent a very large avoided capital cost, and the decrease in fuel requirements, process chemicals, and corresponding decrease in process equipment size also represent a significant portion of cost savings. A full summary of each scenario is included in Figure 4 and it is apparent that in every instance, CCC outperforms Case B12B and other comparable amine-based CCS techno-economic estimates (Table 8). However, what becomes even more apparent is that the ability to retrofit an existing plant with low energy penalty becomes almost paramount to any CCS installation. For both a single plant retrofit and the retrofit scenario, the cost of electricity for a 550 MW plant is actually lower than for a greenfield SC PC coal-fired power plant, 11.1% less expensive in the case of a single retrofit and 1.9% less expensive for the retrofit scenario. This points out a very important, real-world implication of CCS: a large fraction of current day coal-fired power plants have no capital costs on their current books, meaning the full cost of electricity is due to operation, maintenance, and fuel costs.

Table 6 below. The cost of CO₂ avoided and CO₂ captured is also included in Figure 5 and Table 7. The cost of CO₂ avoided is calculated according to the following equation:

$$Avoided\ Cost = \frac{(COE_{with\ removal} - COE_{reference})\$/MWh}{(CO_2\ Emissions_{reference} - CO_2\ Emissions_{with\ removal})\text{tonne}/MWh}$$

The Importance of Retrofit on Cost Analysis

Although this project focuses on a new FT process and a greenfield installation, the CCC process is minimally invasive in the remaining process and can be retrofit to any process. Other competing technologies, such as amine absorption, would require either substantial rebuilding and rerouting of steam and other lines or require a dedicated steam/heat generation system in addition to the retrofit. Most other CO₂ removal technologies, such as oxyfuel or chemical looping, have almost no retrofit

capability. This section of the report summarizes the retrofit approach and includes retrofit cost analysis with the greenfield analyses.

Utilizing the same assumptions and the same model as the other costing scenarios presented in this work, estimates of the COE for retrofitting the CCC technology have been calculated. Retrofitting allows for the energy market to meet enforced carbon restrictions by leveraging existing capital resources rather than having to decommission and replace existing plants. In the United States, the vast majority of capital of existing power plants has been paid off and has no effect on current COE. The ability to leverage this existing capital resource sets CCC apart from the other core CCS technologies. These numbers have been generated assuming that the boiler feed water integration is in place, which would only necessitate a small change in the current piping of the steam system at any existing power plant.

Cost numbers for the CCC portion of the retrofit costs are assumed to be the same as those at a greenfield installation. Energy penalty calculations indicate that a brand new power plant of the same size as that required for a 550 MW net plant with CCC would produce 657 MW if no carbon capture system were in place. Therefore, retrofitting this hypothetical existing 657 MW plant with CCC would represent a capital cost investment of the CCC portion of the process and the expanded cooling water system. This would reduce the net power output of the existing plant by 107 MW, resulting in a 550 MW plant. This means that for approximately every 5 plants of this size that are retrofitted, a brand new plant would have to be built to replace the lost capacity.

The COE of a single retrofit plant was calculated by first scaling the costs of Case B12a to a 657 MW net plant. The capital cost portion of the COE was then removed to simulate a fully paid off plant. All operation, maintenance, and fuel costs were left untouched. The cost of a greenfield CCC plant, and its expanded cooling water requirements, were added to this cost to create a CCC single retrofit case. Since this does not fully account for the loss in capacity, we have also included a CCC retrofit scenario, which was calculated by averaging the COE of 5 of these hypothetical retrofit plants with the COE of 1 brand new installation to make up for the lost power. A summary of the results of these simulations appears below in Figure 4, along with the results from all greenfield scenarios (It is apparent that in every instance, CCC outperforms Case B12B and other comparable amine-based CCS techno-economic estimates (Table 8). However, what becomes even more apparent is that the ability to retrofit an existing plant with low energy penalty becomes almost paramount to any CCS installation. For both a single plant retrofit and the retrofit scenario, the cost of electricity for a 550 MW plant is actually lower than for a greenfield SC PC coal-fired power plant, 11.1% less expensive in the case of a single retrofit and 1.9% less expensive for the retrofit scenario. This points out a very important, real-world implication of CCS: a large fraction of current day coal-fired power plants have no capital costs on their current books, meaning the full cost of electricity is due to operation, maintenance, and fuel costs.

Table 6). It is also important to note than the retrofit numbers do not include any of the integration benefits previously discussed.

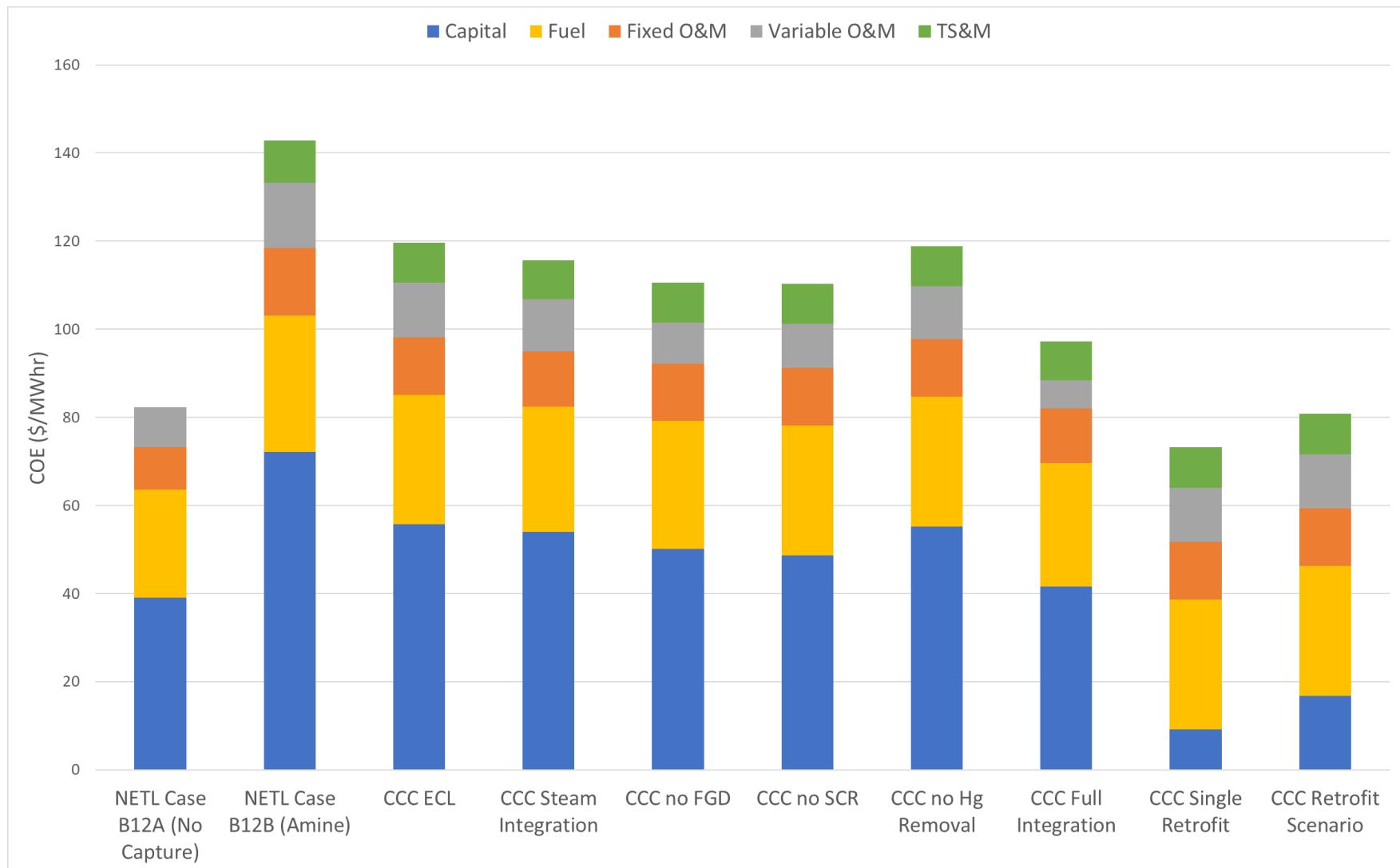


Figure 4 - COE of different case studies broken into component parts.

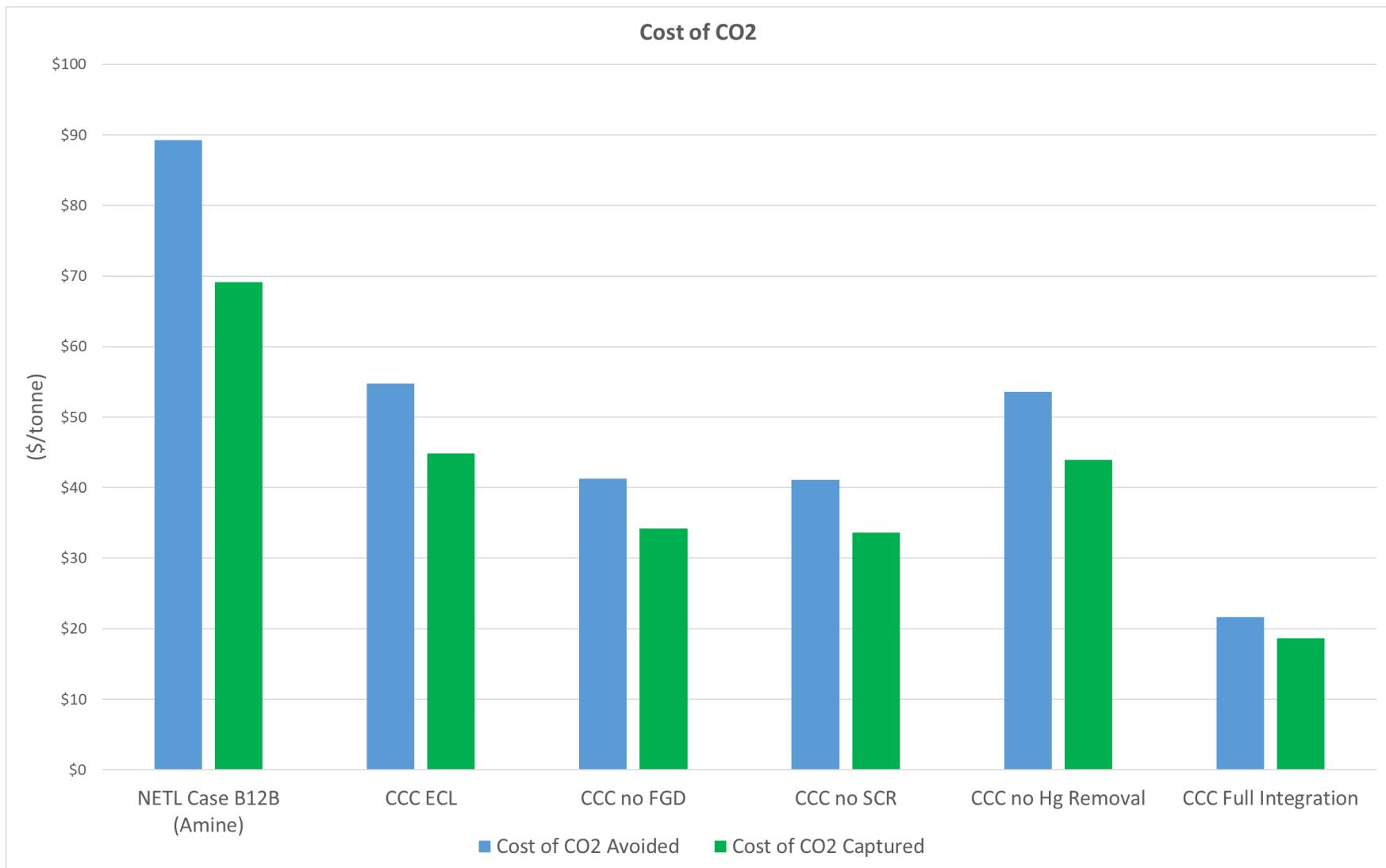


Figure 5 - Cost of CO₂

It is apparent that in every instance, CCC outperforms Case B12B and other comparable amine-based CCS techno-economic estimates (Table 8). However, what becomes even more apparent is that the ability to retrofit an existing plant with low energy penalty becomes almost paramount to any CCS installation. For both a single plant retrofit and the retrofit scenario, the cost of electricity for a 550 MW plant is actually lower than for a greenfield SC PC coal-fired power plant, 11.1% less expensive in the case of a single retrofit and 1.9% less expensive for the retrofit scenario. This points out a very important, real-world implication of CCS: a large fraction of current day coal-fired power plants have no capital costs on their current books, meaning the full cost of electricity is due to operation, maintenance, and fuel costs.

Table 6 - Cost comparison between NETL Cases B12A and B12B as well as estimates for the CCC process, both as a greenfield installation and as a retrofit installation.

	Case B12A	Case B12B	CCC ECL	CCC Steam Int.	CCC no FGD	CCC no SCR	CCC no Hg Removal	CCC Full Int.	CCC Single Retrofit	CCC Retrofit Scenario
COE (\$/MWh)	82.30	142.80	119.67	115.63	110.52	110.34	118.90	97.17	73.21	80.78
TS&M Costs (\$/MWh)	0.00	9.60	9.15	8.82	9.06	9.15	9.15	8.74	9.15	9.15
Fuel Costs (\$/MWh)	24.60	30.90	29.43	28.33	29.14	29.43	29.43	28.07	29.43	29.43
Variable Costs (\$/MWh)	9.10	14.70	12.35	11.83	9.28	10.02	12.04	6.36	12.35	12.35
Fixed Costs (\$/MWh)	9.60	15.40	13.06	12.58	12.94	13.06	13.06	12.46	13.06	13.06
Capital Costs (\$/MWh)	39.00	72.20	55.68	54.07	50.09	48.68	55.22	41.54	9.22	16.79
Difference from Case B12A	0.00%	73.51%	45.40%	40.50%	34.28%	34.07%	44.47%	18.07%	-11.05%	-1.85%

Table 7 - Cost of CO₂ comparison for greenfield cases.

	Case B12B	CCC ECL	CCC Steam Integration	CCC no FGD	CCC no SCR	CCC no Hg Removal	CCC Full Integration
Cost of CO₂ Avoided (\$/tonne)	89.30	54.73	48.48	41.28	41.07	53.60	21.60
Cost of CO₂ Captured (\$/tonne)	69.17	44.83	41.46	34.18	33.64	43.91	18.67

The same additional reviews of amine performance as cited above in the energy penalty discussion also performed economic estimates of the cost of electricity. These are included here in Table 8.

Table 8 – Increase in COE for amine-based CCS installations [24] and CCC.

	NETL	CMU	EPRI	TNO	TPRI	CSIRO	CCC
Reference Non-capture Plant COE (\$/MWh)	82.3	59.1	73.4	43.9	42.0	53.4	82.3
Amine-based CCS Plant COE (\$/MWh)	142.8	99.2	121.1	79.2	62.0	114.5	119.7
Increase in COE (\$/MWh)	60.5	40.1	47.7	35.3	20.0	61.1	37.4
Difference from Base Case	73.5%	67.9%	65.0%	80.4%	47.6%	114.4%	45.4%

According to the January 2013 Electric Power Annual released by the United States Energy Information Administration [21], the most recent data show an average cost of only 35.1 \$/MWh for 2011—the simulated year of the NETL study—in generation costs related to operation, maintenance, and fuel costs for fossil steam generated electricity. While it is no doubt daunting to project the cost of electricity increasing by 81.9%, reality holds an even more daunting truth. If retrofits are not an option and the full capacity of existing power plants must be replaced with those capable of carbon capture, the real cost of electricity generation to the utility skyrockets from around 35.1 ¢/kWh to 142.8 \$/MWh, an increase of 307%. In the low-cost regions of the country, where average generation costs are about 20 \$/MWh, this represents an increase greater than 600%. Under these circumstances, a single CCC retrofit would increase the COE by 85% (26% of this increase is due to transportation, storage, and monitoring). While this is still a very large increase, it pales in comparison to the alternative of having to replace lost capacity with new installations or significant rebuilds.

Conclusion

The current energy infrastructure still relies heavily on fossil fuel resources for the vast majority of the world's energy demands. A transitional energy infrastructure is necessary to mitigate the adverse effects of fossil-based energy, such as global climate change. For these reasons, carbon capture and sequestration has become a critical technical challenge. Cryogenic carbon capture represents a viable alternative to current technologies. Its advantages include:

- (a) a lower parasitic load, ~25-50% lower than established metrics for absorption technology, depending on the reference study;
- (b) lower cost of electricity, approximately 60% of the increase calculated using established metrics for absorption technology;
- (c) minimally invasive bolt-on technology capable of being retrofitted to existing plants;
- (d) scalability to largest commercial emitters, such as coal-fired power plants;

- (e) ancillary pollutant removal and the inherent integration benefits associated therewith;
- (f) ability to capture CO₂ at well above the industry standard 90% capture efficiency; and
- (g) equipment and unit operations already familiar to power plant personnel.

By virtually every established metric, CCC outperforms competing carbon capture technologies and represents a potentially paradigm-shifting solution to the current questions facing the energy industry.

Feed Gas Specifications

These simulations include two different initial feed conditions, one that is typical of syngas from a coal or biomass gasifier (Table 9) and a second based on Ceramatec feedback and is used in some of their designs (Table 10). The two compositions differ in several important ways. The Ceramatec feed gas includes a H:C ratio (about 3.44) well over twice as high as the gasifier syngas stream (1.46), includes much higher molecular nitrogen mole fraction (0.48 vs. 0.02), contains no trace impurities such as H₂S, NH₃, or HCN, has a much higher molecular H₂ to CO ratio (8.0 vs. 0.61) and has a higher initial CO₂ mole fraction (0.04 vs. 0.09). The simulations use both of these conditions.

Table 9 Base Common Gasifier Syngas Composition

Component	Mole Fraction
N ₂	0.0178
CO ₂	0.0891
CH ₄	0.0178
H ₂ O	0.1000
H ₂	0.2939
CO	0.4809
H ₂ S	4.00E-04
NH ₃	1.00E-04
HCN	5.00E-05

Table 10 Base Ceramatec Syngas Composition

Component	Mole Fraction
N ₂	0.476
CO ₂	0.040
CH ₄	0.008
H ₂ O	0.001
Isopentane	0.000
Ar	0.006
H ₂	0.321
CO	0.148
C ₂₊	9.7e-4

CCC Simulation Results

Aspen Plus simulations with these two stream as feedstocks indicate the ability of CCC to separate the undesirable components from the streams. Aspen Plus is not capable of rigorously simulating the CCC process because it and all other commercial process simulators of which we are aware cannot form or destroy solids in heat exchangers or other unit operations except for a Gibbs reactor. However, several Gibbs reactors in series approximation the SES-patented desublimating heat exchanger, which is how the Aspen simulations treat this process. Aspen Plus simulations enjoy reputation for rigor and a widespread user base, so this type of workaround in Aspen provides us a way to communicate with others about the process performance. Actual process design relies SES-in-house process simulation software that is routinely benchmarked against external standards, including Aspen calculations.

The results of processing these streams with CCC appear in Table 11 and Table 12. These particular results include 97% capture for the gasifier stream and 90% capture for the Ceramatec stream. There are several issues to note, as indicated in the subsections below.

Table 11 Base Common Gasifier Syngas Composition after CCC. Compare with inlet stream in Table 9

Component	Mole fraction	% Removed
N ₂	0.021742	0
CO ₂	0.010576	97
CH ₄	0.021861	0
H ₂ O	1.27E-05	100
H ₂	0.358616	0
CO	0.587109	0
H ₂ S	7.85E-05	93
NH ₃	4.69E-06	98
HCN	4.13E-09	100

Table 12 Base Ceramatec Syngas Composition after CCC. Compare with inlet stream in Table 10

Component	Mole Fraction	% Removed
N ₂	0.495	0
CO ₂	0.004	90
CH ₄	0.008	0
H ₂ O	0.000	100
Isopentane	1.18E-05	
Ar	0.006	0
H ₂	0.334	0
CO	0.154	0
C ₂₊	0.000	100

CO₂ Removal

The CCC process effectively removes CO₂ without removing the important process gases of CO and H₂. These two simulations illustrate 90% and 97% CO₂ capture. Experiments with combustible gas feed streams performed previously and outside the scope of this project demonstrate that these simulated

results are highly accurate, even in process transients. The CO₂ first forms a solid as the process gas stream cools, then separates from the remaining gas stream, and then forms a pressurized liquid as the purified CO₂ stream melts under pressure and during heat recovery.

Other Process Gases

CCC removes no H₂ or CO from the stream. This remains true even at extremely high CO₂ removal rates (99.999%). CO and H₂ are essentially inert gases in this process, as are N₂, Ar, and O₂. However, the process removes essentially all of any initial H₂O.

At these low pressures, CH₄ also acts as an inert, the CCC process captures increasing amounts of CH₄ with increasing pressure and decreasing temperature, the latter leading to increasing CO₂ capture fraction.

Pollutants and Catalyst Poisons

The CCC process removes most trace pollutants and potential catalyst poisons. CCC removes over 90% of the trace pollutants and potential catalyst poisons. The exit pollutant concentrations and removal percentages appear in Table 11 for the simulation that contain H₂S, NH₃, and HCN. The nature of the CCC process is such that the exit concentrations of these pollutants remain the same regardless of their inlet concentrations but the exit concentrations decrease quite rapidly with decreasing temperature (or increasing CO₂ capture). None of the initial gas compositions included other potential poisons such as heavy metals, but CCC removes Hg and all other heavy metals to near zero concentration.

Energy Demand

The energy demand for these processes depends strongly on the initial CO₂ concentration, somewhat on pressure and capture percentage, and weakly or not at all on most other parameters. To illustrate these dependencies, we completed a parametric analysis of the process stream over the following range of conditions:

- CO₂ capture: 95%, 96%, 97% (base), 98%, and 99%
- Pressure: 16 psi (base), 175, 200, and 290 psi,
- CO₂ inlet composition: of 4, 5, 6, 8, 9 (base), 12, and 14 %.

The CO₂ composition changed by adding CO₂ to or removing CO₂ from the stream such that the CO₂ varied and the remaining gas flow rates remained the same but mole fractions changed because of the change in the CO₂ flowrate. These perturbations occurred both individually and simultaneously for a total of 48 simulations. Typical results (for the baseline case) appear as Table 11. The simulations indicated the amount of energy required for the removal in units of MJ/kg CO₂ collected.

These simulations show the energy demand as a function of composition, pressure, and capture efficiency. Figure 6 and Figure 7 show the energy penalties at fixed pressure for different conditions. Figure 8 shows the energy penalty for all combinations. The trends evident in the figures show that the energy penalty:

1. decreases rapidly with an increase in CO₂ inlet percentage,
2. increases with a decrease in pressure, and
3. increases with an increase in capture percentage.

The system is much more sensitive to inlet CO₂ percentage than it is to pressure or capture percentage over these ranges.

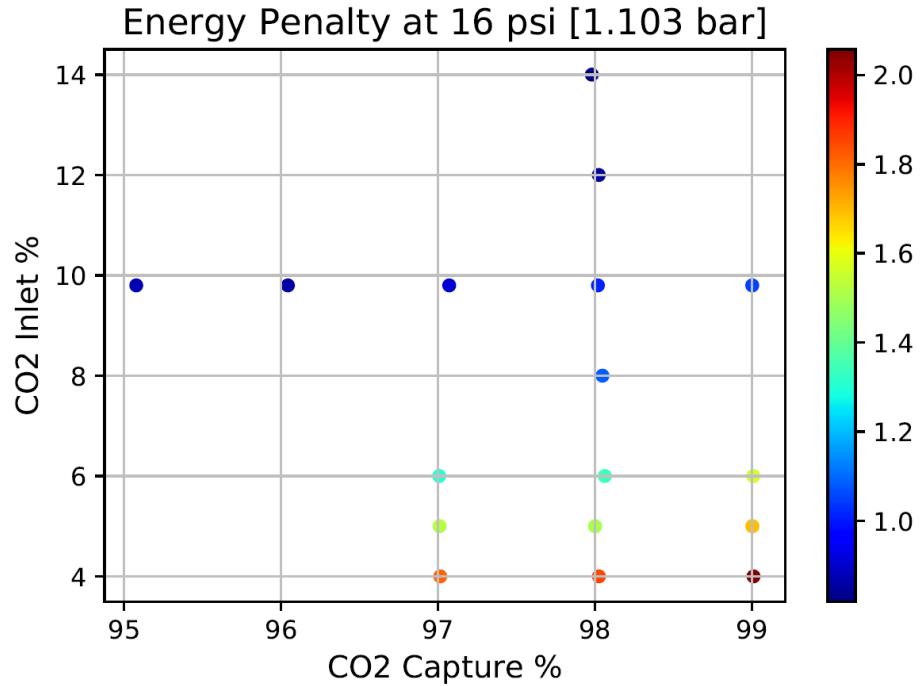


Figure 6 Energy Penalties for 16 psi Inlet. Energy Penalty Color Bar Corresponds to MJ/kg

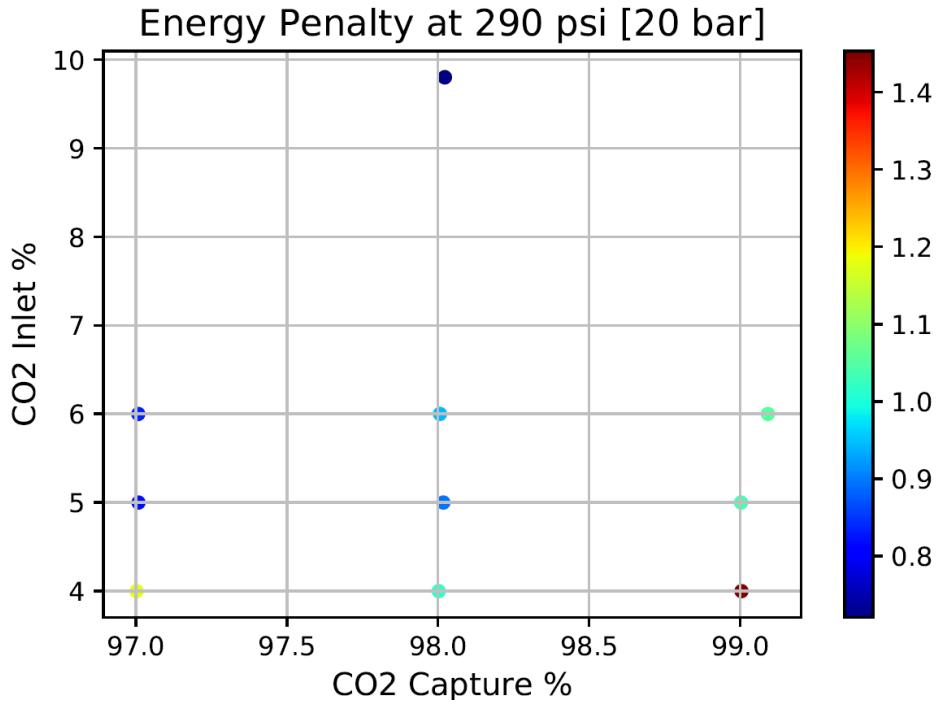


Figure 7 Energy Penalties for 290 psi Inlet. Energy Penalty Color Bar Corresponds to MJ/kg

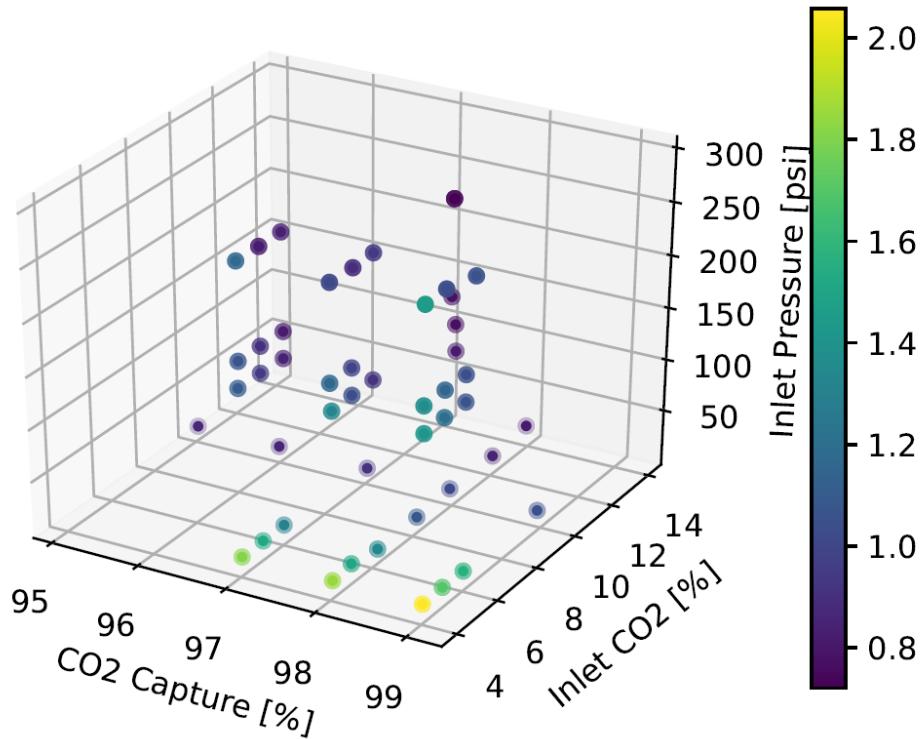


Figure 8 Energy Penalties for Different Conditions. Energy Penalty Color Bar Corresponds to MJ/kg

Final Syngas Treatment Analysis

The other project members provided the process flow diagram and stream table shown in Figure 9 and Figure 10, respectively. There are several places in the process where CCC could usefully be applied and it has the potential to reduce or eliminate the need for some of these steps. However, this analysis focuses on treating Stream 24, the inlet to the Fischer Tropsch reactor, to reduce the CO₂ content dramatically and thereby decrease the CO₂ footprint of this process to less than that of traditional processing of petroleum fuels.

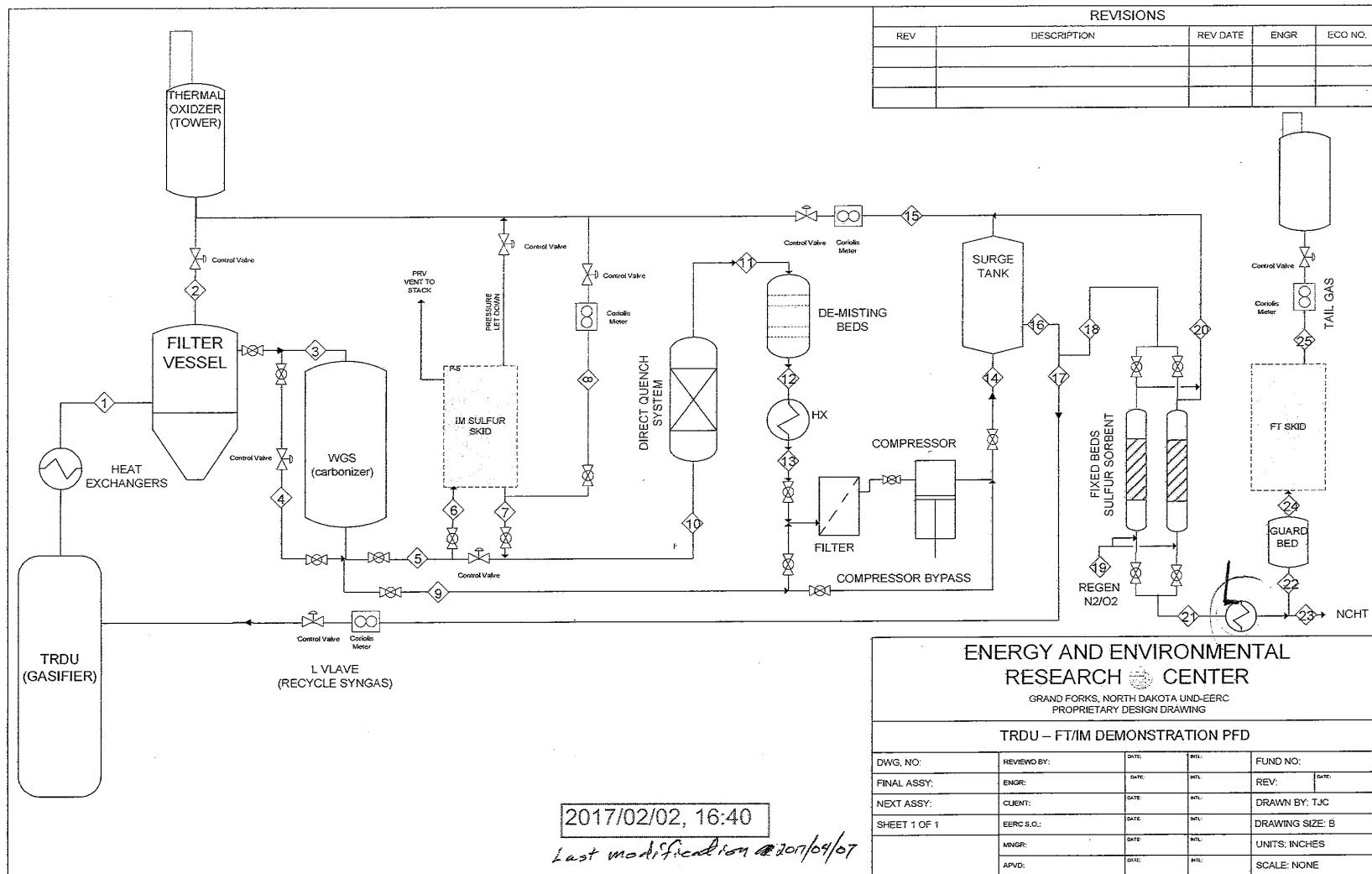


Figure 9 Most recent process configuration obtained from EERC.

PFD#	1	2	3	4	5	6	7	8*	9*	10	11	12
	HGFV Inlet	TRDU exit to TOX	HGFV to WGS	WGS Bypass	Post WGS and Bypass	IM Skid inlet	IM Skid return	IM Skid Flow to TOX	Compressor Bypass	Seive Tower Inlet	Quench Exit	Reheat HX Inlet
Temp. (°F)	660	610	600	600	570	550	72	72	530	515	80	72
Press. (psig)	150	145	145	145	140	140	135	135	140	130	120	115
Flow Rate (SCFH)	24000	7400	5600	11000	16600	2400	2400	2400	16600	16600	12500	12500
Pipe Size (in)			2	2	1	1	2	2	2	1-1/2	1-1/2	1
Schedule/ Material			sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I

N2 (vol%)	23	23	23	23	23	23	23	23	23	30	30
H2 (vol%)	14	14	14	14	19	19	19	19	19	25	25
CO (vol%)	11	11	11	11	9	9	9	9	9	12.5	12.5
CO2 (vol%)	20	20	20	20	22	22	22	22	22	30	30
CH4 (vol%)	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	2.4	2.4
H2O (vol%)	28	28	28	28	23	23	23	23	23	1.5	1.5
H2S (ppm)	3000	3000	3000	3000	3000	0	0	2400	2400	3100	3100
Tars (ppm)	2500	2500	2500	2500	2500	2500	2500	2500	2500	Trace	Trace
Condensate (lbm/hr)	0	0	0	0	0	0	0	0	0	200	0

PFD#	13	14	15	16	17	18	19**	20**	21	22	23***	24	25
	Reheat Hx Outlet	Post Compressor	Surge Tank to TOX	Surge Tank Outlet	L-Vlave Recycle	Fixed Bed Inlet	Fixed Bed Regen	Fixed Regen Exhaust	Fixed Bed Outlet	Condenser HX's Outlet	Syngas to NCHT	FT Inlet	FT Tail Gas
Temp. (°F)	250	475	425	425	400	400	72	1100	600	65	65	65	??
Press. (psig)	112	405	400	400	395	395	5	3	390	375	350	360	??
Flow Rate (SCFH)	12500	12500	300	12200	5000	7200	1000	1000	7200	7200	200	7000	??
Pipe Size (in)	1	1	1	1	1	1	1	1	1	1	1/2 Tube	1	??
Schedule/ Material	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	sch40/316I	.065 wall 316ss	sch40/CS	sch40/316I

N2 (vol%)	30	30	30	30	30	97	97	30	30	30	30	??
H2 (vol%)	25	25	25	25	25	0	0	25	25	25	25	??
CO (vol%)	12.5	12.5	12.5	12.5	12.5	0	0	12.5	12.5	12.5	12.5	??
CO2 (vol%)	30	30	30	30	30	0	0	30	30	30	30	??
CH4 (vol%)	2.4	2.4	2.4	2.4	2.4	0	0	2.4	2.4	2.4	2.4	??
H2O (vol%)	1.5	1.5	1.5	1.5	1.5	0	0	2.2	1.5	1.5	1.5	??
H2S (ppm)	3100	3100	3100	3100	3100	0	~2%	< 2	< 2	< 2	< 10 ppb	??
Tars (ppm)	Trace	Trace	Trace	Trace	Trace	0	0	Trace	Trace	Trace	Trace	??
Condensate (lbm/hr)	0	0	0.5	0	0	0	0	0	2.5	0	0	??

* Alternate gas flow path (No flow during normal operation)

**Fixed beds regenarted off line, 2 to 3% O2 used for desulfurization of sorbent

*** Slip steam to NCHT only during syngas combustion tests

Figure 10 Stream table corresponding to the process flow diagram of Figure 9

Stream Selection

The water content, temperature, pressure and stream recycling led to the choice of stream 24 (FT Inlet) as the best candidate for treatment. The stream properties are listed in Table 13 with composition listed in Table 14.

Table 13 Properties of Stream 24

Temperature (°F)	65
Pressure (psig)	360
Flow Rate (SCFH)	7000

Table 14 Composition of Stream 24. All values are Volume % unless specified otherwise

N ₂	30
H ₂	25
CO	12.5
CO ₂	30
CH ₄	2.4
H ₂ O	1.5
H ₂ S (ppb)	<10
Tars (ppm)	Trace
Condensate (lb _m /hr)	0

The Cryogenic Carbon Capture with External Cooling Loop (CCC-ECL) system was used to capture both 90 and 99% of the inlet CO₂. Results are shown in Table 15 and Table 16. As seen, the incremental energy cost for capturing 99% of the stream is small and this is probably the best design point for the system.

Table 15 Properties of Treated Streams

Capture Percent	90%	99%
Temperature (°F)	59	59
Pressure (psig)	350	350
Flow Rate (SCFH)	5032	4846
Energy Penalty (MJ/kg CO ₂)	0.490	0.495
Energy Cost (kW)	13.35	14.82

The resulting composition of the final streams under these two conditions appear in Table 4.

Table 16 Composition of Stream 24. All values are Mole % unless specified otherwise. Values are rounded

Capture Percent	90%	99%
N ₂	41.2	42.7
H ₂	34.3	35.6
CO	17.1	17.8
CO ₂	4.1	0.4
CH ₄	3.3	3.4

H ₂ O	0	0
Iso-Pentane (ppm)	170	14
H ₂ S (ppb)	<10	<10
Tars (ppm)	0	0
Condensate (lb _m /hr)	0	0

The CCC-ECL system efficiently removes the CO₂ from this inlet stream with minimal losses to any other component and minimal energy demand. The pressure loss is minimal and, if a compressor is needed, would be less than 200 W of added energy penalty.

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Appendix D: Analysis of F-T Jet Fuel Fraction at AFRL/RQTF

Analysis of EERC/Ceramatec F-T Jet Fuel

Tim Edwards, AFRL/RQTF, 18 May 2018

2 L of jet fuel was received from EERC on 5/9/2018. The fuel was labeled “Fischer-Tropsch-Derived Jet Fuel Range Hydrocarbons, Distilled 12-18-17”. It was assigned the AFRL/RQTF internal ID number of POSF 13313. A number of analyses were performed on the fuel to determine (within the constraints of the fuel volume) its conformance to the ASTM D7566 alternative jet fuel specification and its similarity to other F-T jet fuels and other approved alternative jet fuels. For example, significant physical property data on three F-T jet fuels was included in the Research Report supporting the first Annex to ASTM D7566. The data for these fuels will be used in this report – Sasol IPK (POSF 7629), Syntroleum S-8 (POSF 5018), and Shell F-T kerosene (POSF 5172). The major properties of interest in this report are distillation, freeze point/low T viscosity, flash point (these three define the fuel as a “kerosene”), composition, and thermal stability. The approved F-T jet fuels in general were predominantly iso-paraffins, although the Shell F-T jet (purchased by AF from Bintulu F-T plant) was a fairly narrow cut with a much higher proportion of n-paraffins than the others. This fuel was available from Shell but was not marketed as a jet fuel. The Syntroleum and Shell fuels were GTL fuels, while the Sasol IPK was a CTL fuel using Sasol’s higher-temperature process. Data for a conventional Jet A fuel (POSF 10325) with very typical jet fuel properties is also included.

Composition

The overall hydrocarbon class breakdown by GCxGC is shown in Table 1, along with a typical jet fuel and several other F-T jet fuels. As can be seen, the composition of the EERC/Ceramatec fuel is similar to that of other F-T jet fuels. Sasol’s process tends to create a highly-branched iso-paraffinic FT product, while the EERC/Ceramatec jet fuel more closely resembles the Syntroleum S-8 (GTL) fuel. All of these F-T fuels are notable for the low aromatic and cycloparaffin content relative to conventional Jet A fuel. The GCxGC data also gives the distribution across carbon numbers for these classes. The result for iso-paraffins is shown in Figure 1. The GCxGC data is included in the Appendix.

Table 1 – overall hydrocarbon class breakdown for various jet fuels.

Composition, mass %	13313 EERC	IPK 7629	S-8 5018	Shell 5172	Jet A 10325
Iso-paraffins	69.0	98.4	75.9	55.1	29.5
N-paraffins	21.5	0.3	23.4	44.0	20.0
Cycloparaffins	9.3	0.6	0.6	0.6	31.9
Aromatics	0.3	0.6	0.2	0.3	18.9
Equivalent formula	C10.4H22.6	C10.8H23.6	C11.8H25.6	C10.4H22.7	C11.4H22.1

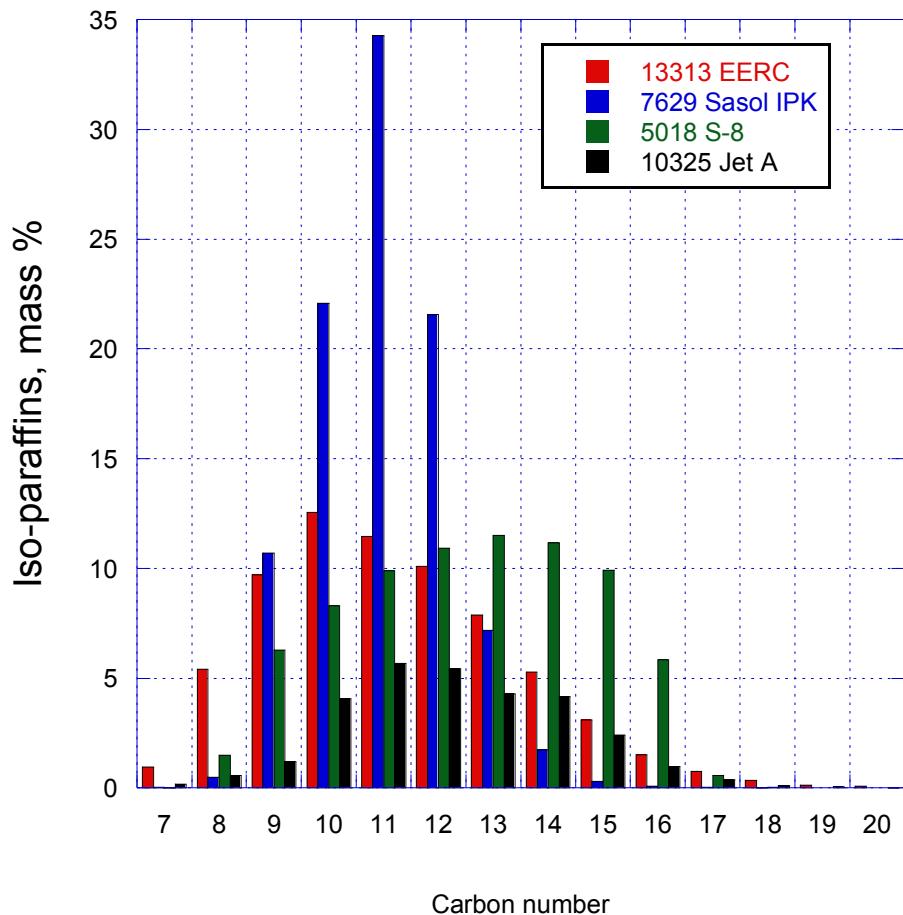


Figure 1 – Distribution of iso-paraffins across carbon number for various jet fuels.

Distillation

Related to the spread across the molecular weight/carbon number range, the distillation range can also be assessed by ASTM D86 and ASTM D2887 distillation. ASTM D86 is a venerable technique that is equivalent to a relative crude single stage distillation, with the advantage of data for most jet fuel batches being available. ASTM D2887 is a gas chromatographic technique that gives results similar to a true boiling point curve. The ASTM D2887 curves for the EERC Ceramatec fuel and several other fuels of interest are shown in Figure 2. The EERC fuel is broadly distributed across the boiling range, similar to the Jet A and S-8 fuels. The Shell FT fuel was relatively flat-boiling, with some industry discomfort with the T90 – T10 range. The EERC fuel has some lower-boiling material not found in the other jet fuels. This affects the flash point, as discussed below. The D86 distillation data (included in Table 2 below) is shown in Figure 3. Again, the EERC fuel is well-distributed across the boiling range, with the presence of some lower-boiling material relative to other jet fuels.

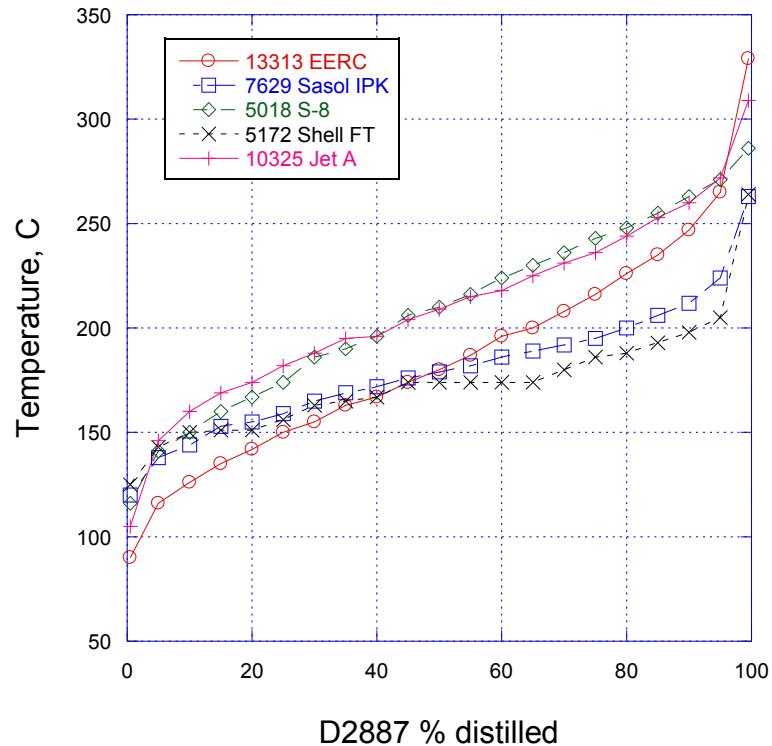


Figure 2 – ASTM D2887 distillation data

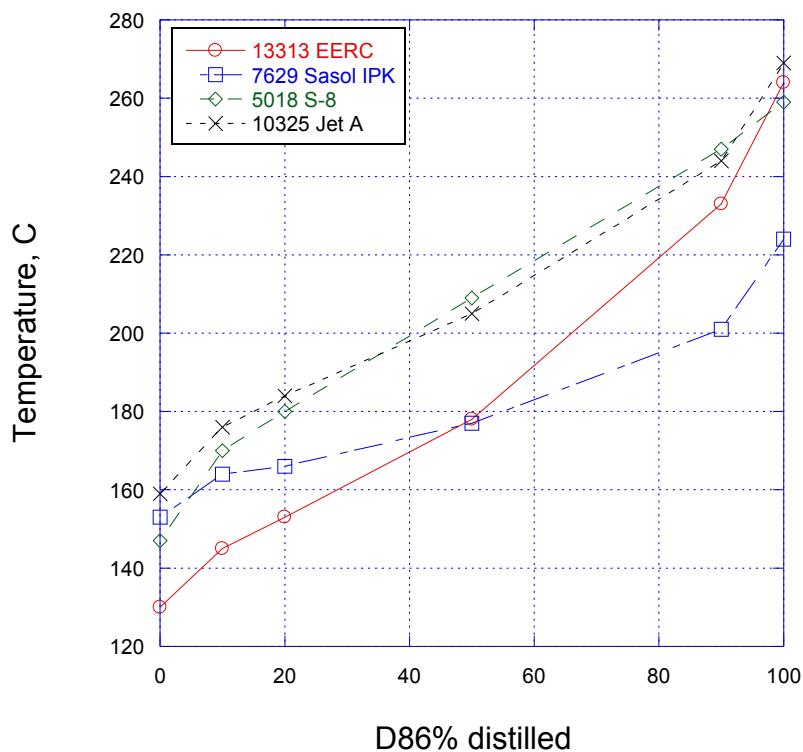


Figure 3 – ASTM D86 distillation data

Specification Properties

Several specification tests were performed on the EERC/Cermatec fuel, with results shown in Table 2. As anticipated from the distillation data, the flash point of the fuel is slightly below the specification limit, easily remedied by changing the distillation cut point slightly. The low temperature properties of the EERC fuel are excellent. The density of the EERC/Cermatec fuel is similar to the other F-T jet fuels. The thermal-oxidative stability of the EERC fuel also appears to well exceed specification requirements.

Table 2. Specification test summary

Property	ASTM D1655 Limits	13313	7629	5018	Jet A 10325
Flash point, C (ASTM D93)	>38	32	42	48	48
Freeze point, C (ASTM D2386)	<-40 (Jet A)	-64.4	<-75	--49	-51
Viscosity at -20 C, cSt (ASTM 5972)	<8	2.8 (5.0 at -40 C)	3.4	4.7	4.6
Distillation ASTM D86					
IBP		130	153	147	159
10%	<205 C	145	164	170	176
20%		153	166	180	184
50%		178	177	209	205
90%		233	201	247	244
FBP	<300 C	264	224	259	269
Density (D4052)	0.775-0.84	0.758	0.761		0.803
Thermal stability at 260 C					
Tube deposit	Visual rating VTR <3; ETR< 85 nm	ETR 6 nm	VTR 1	VTR 1	VTR 1
Filter DP	<25 mm Hg	0	0	0	0

Conclusion

The EERC/Cermatec F-T jet fuel appears to be a high-quality jet fuel blending component, similar to the Syntroleum S-8 GTL jet fuel. It appears that a small adjustment in the distillation cut point would bring the fuel into full compliance with ASTM D7566 Annex A1 requirements.

Appendix-

GCxGC compositional results for EERC/Cermatec and Syntroleum S-8 F-T fuels

GCxGC Summary			GCxGC Summary		
Hydrogen content (weight %)		15.4	Hydrogen content (weight %)		15.4
Average Molecular Wt (g/mole)		148	Average Molecular Wt (g/mole)		168
POSF-13313			POSF-5018		
	Weight %	Volume %		Weight %	
Aromatics			Aromatics		
Alkylbenzenes			Alkylbenzenes		
benzene (C06)	<0.01	<0.01	benzene (C06)	<0.01	
toluene (C07)	<0.01	<0.01	toluene (C07)	<0.01	
C2-benzene (C08)	0.04	0.03	C2-benzene (C08)	0.02	
C3-benzene (C09)	0.02	0.02	C3-benzene (C09)	0.03	
C4-benzene (C10)	0.05	0.05	C4-benzene (C10)	0.03	
C5-benzene (C11)	0.04	0.03	C5-benzene (C11)	0.03	
C6-benzene (C12)	0.03	0.03	C6-benzene (C12)	0.02	
C7-benzene (C13)	0.01	0.01	C7-benzene (C13)	0.02	
C8-benzene (C14)	0.01	0.01	C8-benzene (C14)	0.01	
C9-benzene (C15)	0.01	<0.01	C9-benzene (C15)	<0.01	
C10+-benzene (C16+)	<0.01	<0.01	C10+-benzene (C16+)	<0.01	
Total Alkylbenzenes	0.23	0.19	Total Alkylbenzenes	0.16	
Diaromatics (Naphthalenes, Biphenyls, etc.)			Diaromatics (Naphthalenes, Biphenyls, etc.)		
diaromatic-C10	<0.01	<0.01	diaromatic-C10	<0.01	
diaromatic-C11	<0.01	<0.01	diaromatic-C11	<0.01	
diaromatic-C12	<0.01	<0.01	diaromatic-C12	<0.01	
diaromatic-C13	<0.01	<0.01	diaromatic-C13	<0.01	
diaromatic-C14+	<0.01	<0.01	diaromatic-C14+	<0.01	
Total Diaromatics	<0.01	<0.01	Total Alkylnaphthalenes	<0.01	
Cycloaromatics (Indans, Tetralins,etc.)			Cycloaromatics (Indans, Tetralins,etc.)		
cycloaromatic-C09	<0.01	<0.01	cycloaromatic-C09	<0.01	
cycloaromatic-C10	0.04	0.03	cycloaromatic-C10	<0.01	
cycloaromatic-C11	<0.01	<0.01	cycloaromatic-C11	<0.01	
cycloaromatic-C12	<0.01	<0.01	cycloaromatic-C12	<0.01	
cycloaromatic-C13	<0.01	<0.01	cycloaromatic-C13	<0.01	
cycloaromatic-C14	<0.01	<0.01	cycloaromatic-C14	<0.01	
cycloaromatics-C15+	<0.01	<0.01	cycloaromatics-C15+	<0.01	
Total Cycloaromatics	0.05	0.04	Total Cycloaromatics	0.03	
Total Aromatics	0.29	0.24	Total Aromatics	0.20	
Paraffins			Paraffins		
iso-Paraffins			iso-Paraffins		
C07 & lower -isoparaffins	0.86	0.94	C07 & lower -isoparaffins	0.01	
C08-isoparaffins	5.09	5.40	C08-isoparaffins	1.48	
C09-isoparaffins	9.37	9.71	C09-isoparaffins	6.28	
C10-isoparaffins	12.30	12.54	C10-isoparaffins	8.29	
C11-isoparaffins	11.48	11.44	C11-isoparaffins	9.89	
C12-isoparaffins	10.09	10.08	C12-isoparaffins	10.93	
C13-isoparaffins	8.06	7.88	C13-isoparaffins	11.50	
C14-isoparaffins	5.45	5.28	C14-isoparaffins	11.16	
C15-isoparaffins	3.22	3.10	C15-isoparaffins	9.92	
C16-isoparaffins	1.59	1.52	C16-isoparaffins	5.84	
C17-isoparaffins	0.80	0.76	C17-isoparaffins	0.56	
C18-isoparaffins	0.36	0.34	C18-isoparaffins	0.02	
C19-isoparaffins	0.15	0.14	C19-isoparaffins	<0.01	
C20-isoparaffins	0.07	0.07	C20-isoparaffins	<0.01	
C21-isoparaffins	0.03	0.03	C21-isoparaffins	<0.01	
C22-isoparaffins	0.02	0.02	C22-isoparaffins	<0.01	
C23-isoparaffins	<0.01	<0.01	C23-isoparaffins	<0.01	
C24-isoparaffins	<0.01	<0.01	C24-isoparaffins	<0.01	
Total iso-Paraffins	68.95	69.25	Total iso-Paraffins	75.88	

n-Paraffins			n-Paraffins		
n-C07 & lower	1.79	1.94	n-C07 & lower	0.03	
n-C08	4.29	4.52	n-C08	1.37	
n-C09	4.34	4.47	n-C09	3.04	
n-C10	3.60	3.65	n-C10	4.00	
n-C11	2.65	2.65	n-C11	4.24	
n-C12	1.96	1.93	n-C12	3.87	
n-C13	1.41	1.37	n-C13	3.10	
n-C14	0.83	0.80	n-C14	2.26	
n-C15	0.35	0.34	n-C15	1.32	
n-C16	0.13	0.12	n-C16	0.14	
n-C17	0.05	0.05	n-C17	<0.01	
n-C18	0.03	0.03	n-C18	<0.01	
n-C19	0.01	0.01	n-C19	<0.01	
n-C20	<0.01	<0.01	n-C20	<0.01	
n-C21	<0.01	<0.01	n-C21	<0.01	
n-C22	<0.01	<0.01	n-C22	<0.01	
n-C23	<0.01	<0.01	n-C23	<0.01	
Total n-Paraffins	21.47	21.92	Total n-Paraffins	23.37	
Cycloparaffins			Cycloparaffins		
Monocycloparaffins			Monocycloparaffins		
C07 & lower monocycloparaffins	0.28	0.27	C07 & lower monocycloparaffins	<0.01	
C08-monocycloparaffins	1.72	1.62	C08-monocycloparaffins	0.12	
C09-monocycloparaffins	2.97	2.77	C09-monocycloparaffins	0.16	
C10-monocycloparaffins	1.92	1.74	C10-monocycloparaffins	0.12	
C11-monocycloparaffins	1.06	0.98	C11-monocycloparaffins	0.07	
C12-monocycloparaffins	0.41	0.38	C12-monocycloparaffins	0.02	
C13-monocycloparaffins	0.44	0.40	C13-monocycloparaffins	<0.01	
C14-monocycloparaffins	0.16	0.14	C14-monocycloparaffins	<0.01	
C15-monocycloparaffins	0.06	0.05	C15-monocycloparaffins	<0.01	
C16-monocycloparaffins	<0.01	<0.01	C16-monocycloparaffins	<0.01	
C17-monocycloparaffins	<0.01	<0.01	C17-monocycloparaffins	<0.01	
C18-monocycloparaffins	<0.01	<0.01	C18-monocycloparaffins	<0.01	
C19+-monocycloparaffins	<0.01	<0.01	C19+-monocycloparaffins	<0.01	
Total Monocycloparaffins	9.03	8.36	Total Monocycloparaffins	0.50	
Dicycloparaffins			Dicycloparaffins		
C08-dicycloparaffins	<0.01	<0.01	C08-dicycloparaffins	<0.01	
C09-dicycloparaffins	0.05	0.05	C09-dicycloparaffins	<0.01	
C10-dicycloparaffins	0.05	0.04	C10-dicycloparaffins	0.01	
C11-dicycloparaffins	0.03	0.03	C11-dicycloparaffins	0.01	
C12-dicycloparaffins	0.05	0.05	C12-dicycloparaffins	0.01	
C13-dicycloparaffins	0.03	0.02	C13-dicycloparaffins	<0.01	
C14-dicycloparaffins	0.03	0.02	C14-dicycloparaffins	<0.01	
C15-dicycloparaffins	0.01	0.01	C15-dicycloparaffins	<0.01	
C16-dicycloparaffins	<0.01	<0.01	C16-dicycloparaffins	<0.01	
C17+-dicycloparaffins	<0.01	<0.01	C17+-dicycloparaffins	<0.01	
Total Dicycloparaffins	0.26	0.22	Total Dicycloparaffins	0.05	
Tricycloparaffins			Tricycloparaffins		
C10-tricycloparaffins	<0.01	<0.01	C10-tricycloparaffins	<0.01	
C11-tricycloparaffins	<0.01	<0.01	C11-tricycloparaffins	<0.01	
C12-tricycloparaffins	<0.01	<0.01	C12-tricycloparaffins	<0.01	
Total Tricycloparaffins	<0.01	<0.01	Total Tricycloparaffins	<0.01	
Total Cycloparaffins	9.29	8.59	Total Cycloparaffins	0.55	
Average Molecular Formula - C	10.4		Average Molecular Formula - C	11.8	
Average Molecular Formula - H	22.6		Average Molecular Formula - H	25.6	