

Wet Waste Hydrothermal Liquefaction and Biocrude Upgrading to Hydrocarbon Fuels: 2020 State of Technology

March 2021

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Summary

Data from Pacific Northwest National Laboratory's conversion hydrothermal liquefaction (HTL) program for wet waste was used to update the pathway techno-economic analysis (TEA) for the fiscal year 2020 State of Technology (2020 SOT). Figure S.1 shows the modeled minimum fuel selling price (MFSP) for the 2020 SOT, along with the 2018 and 2019 SOTs (Snowden-Swan et al. 2020) and the 2022 projected goal case set forth in the original design report (Snowden-Swan et al. 2017). These costs are for a HTL plant scale of 110 dry ton/day sludge feed and a larger centralized upgrading plant scale of 38 million gallons/year biocrude feed, commensurate with the design case. All costs are in 2016 dollars.

Corresponding cost breakdowns and technical parameters for each case are given in Appendix B. Options with and without ammonia stripping treatment of the HTL aqueous phase (AP) recycle stream are included in the analysis to account for municipalities where direct recycle of untreated HTL AP back to the wastewater treatment plant is feasible.

The modeled fuel blendstock minimum fuel selling price (MFSP) for the 2020 SOT is estimated at \$4.50/GGE and \$4.08/GGE for cases including and excluding ammonia stripping of the AP, respectively. This represents a reduction of \$0.61/GGE, or 12%, relative to the 2019 SOT (Snowden-Swan et al. 2020). Research progress on the HTL process includes an increase of reactor LHSV from 3.6 to 4.0 and a newly designed staged approach for sludge pumping and heating, resulting in a 1 cent and 26 cent reduction in modeled MFSP, respectively. The new heat exchanger configuration is less capital intensive than the previous SOT and provides a system design that is more scalable with regard to practical fabrication limitations. Further cost reduction for the exchangers may be possible with the use of core inserts to enhance tube velocity and heat transfer rates. Biocrude hydrotreating research progress improved weight hourly space velocity (WHSV) from 0.67 to 0.72 hr⁻¹ in the guard bed and from 0.39 to 1.02 hr⁻¹ in the main hydrotreating bed, a 7% and 162% improvement, respectively. Hydrotreating performance was not sacrificed at the higher throughput rates and catalyst activity remained stable over the run. The demonstrated increase in WHSVs reduced the modeled MFSP by \$0.34/GGE.

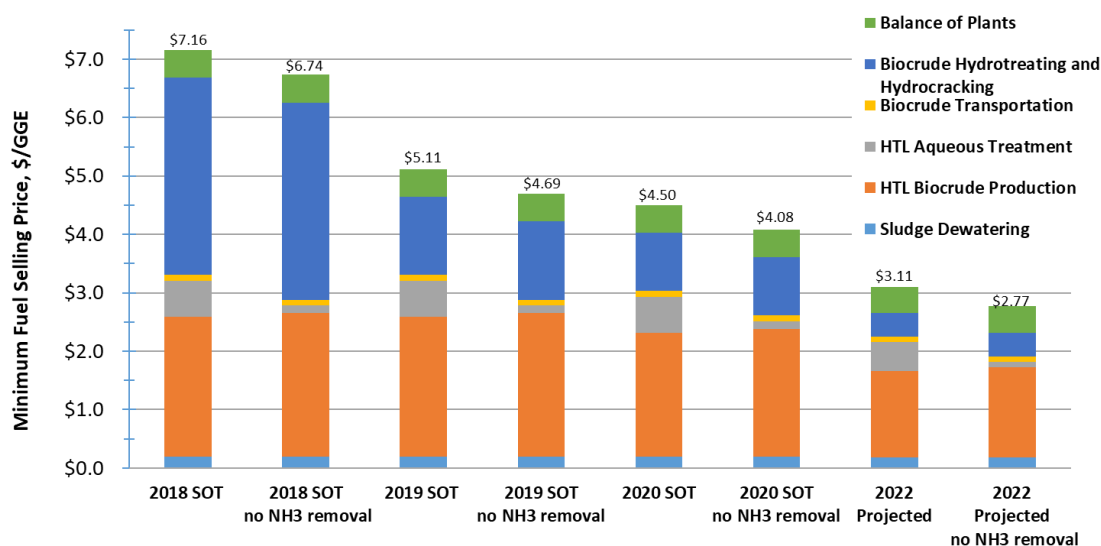


Figure S.1. Wet waste HTL and biocrude upgrading pathway cost allocations.

Wet waste feedstocks tested in this year's HTL and biocrude hydrotreating research include:

- 50/50 mix of primary/secondary sludge from Central Contra Costa Sanitary District (CCCSD), CA, without added lime
- 100% pure primary sludge from Great Lakes Water Authority (GLWA) in Detroit, MI
- Cow manure from a dairy operation, with and without inorganic catalyst
- Biosolids from anaerobic digestion of wastewater treatment sludge

The 50/50 sludge from CCCSD was run to compare with the same plant's sludge run in fiscal year 2019 (FY19) (Snowden-Swan et al. 2020), but without the added lime that is currently used to assist in their sludge incineration process. Biocrude yield was increased by 11% for the sludge without lime relative to the sludge including lime, even when run at a lower solids content. Pure primary sludge from GLWA was run in Pacific Northwest National Laboratory's engineering scale HTL system and produced relatively high biocrude yield (41% dry, ash-free basis), even at lower solids content (15.3 wt%) and high ash (26% dry basis). Cow manure was run with and without a small amount of additive to investigate the potential catalytic effect on product yields. Inclusion of additive appears to boost biocrude yield by 22% and shift overall conversion selectivity from lighter, water-soluble products (i.e., the AP yield) to the heavier biocrude phase. These results are encouraging because they show that similar yields to sludge (37-44% range) may be possible for the cow manure if fed at optimal solids content (20-25%). Biocrude yield from biosolids (solids from anaerobic digestion of sludge) was significantly lower than those seen with sludges or manures, likely due in part to high ash content in the biosolids (33% dry, ash-free basis) and therefore more biocrude adhering to the more abundant solids during separation. Through continuous testing of real-world sludge, manure, and fats/oils/grease samples from actual generators over the past several years, we have demonstrated continuous HTL processing viability for three out of four of the major high-volume underutilized wet waste feedstocks (DOE 2017). Food waste, the fourth major resource, is planned for testing in FY21.

Treatment testing of AP was conducted for three thermochemical methods, catalytic hydrothermal gasification, steam phase-catalytic reduction of wastewater and catalytic upgrading. Testing data was used to develop initial conceptual models and associated TEA at the SOT scale to provide initial high-level economics and sensitivity around the SOT MFSP for each of the proposed methods. When used in conjunction with ammonia stripping, the methods could provide an estimated 65-100% removal of chemical oxygen demand (COD) and an estimated 80-100% removal of ammonia nitrogen. Organic nitrogen removal for the methods tested and modeled ranges from 0-100%. The initial TEA indicated that these methods could add 57-74 cents per GGE and 8-14 cents per GGE for high and medium COD removal methods, respectively. Initial anaerobic digestion testing was also conducted on the AP. Results for gas production and COD reduction indicate that there are compounds in the AP that inhibit the AD organisms. Separate testing by Washington State University using a culture acclimated over several months suggests that AD may be effective for COD reduction. Further testing is needed to verify the feasibility of this method for AP treatment.

Future work to advance this waste-to-energy pathway toward the technical and cost targets includes demonstrating processability of 25% solids to HTL, demonstrating industrially relevant (1-2 year) hydrotreating catalyst life through extended time-on-stream operation, developing a comprehensive AP treatment strategy for conversion of organic and nitrogen, and identifying and characterizing regional

waste blending scenarios that could enhance economies of scale for HTL in urban and other areas of concentrated waste generation.

Acronyms and Abbreviations

AD	anaerobic digestion
AFDW	ash-free dry weight
ALK	alkalinity
AP	aqueous phase
BOD	biological oxygen demand
CCCS	Central Contra Costa Sanitary District
COD	chemical oxygen demand
TCOD	total COD
SCOD	soluble COD
CSTR	continuous stirred-tank reactor
DAF	dry, ash-free
FOG	fats, oils, and grease
FY	fiscal year
GGE	gasoline-gallon equivalent
GHG	greenhouse gas
GLWA	Great Lakes Water Authority
HTL	hydrothermal liquefaction
ICP	inductively coupled plasma
MBSP	minimum biocrude selling price
MFSP	minimum fuel selling price
PFR	plug-flow reactor
PNNL	Pacific Northwest National Laboratory
SOT	state of technology
TEA	techno-economic analysis
TKN	total Kjeldahl nitrogen
SKN	soluble Kjeldahl nitrogen
TOS	time-on-stream
TPD	U.S. ton/day
TS	total solids
TSS	total suspended solids
VFA	volatile fatty acids
VS	volatile solids
VSS	volatile suspended solids
WHSV	weight hourly space velocity
WRRF	wastewater treatment and water resource recovery facility

Contents

Summary	ii
Acronyms and Abbreviations	v
Contents	vi
1.0 Introduction.....	1
2.0 Conversion Model Overview	2
2.1 Heat Exchanger Re-Design.....	2
3.0 Experimental Results	6
3.1 Wet Waste Feedstock Composition	6
3.2 Wet Waste Hydrothermal Liquefaction	8
3.3 Aqueous Phase (AP) Treatment.....	11
3.3.1 Thermochemical Methods	11
3.3.2 Anaerobic Digestion	14
3.4 Biocrude Catalytic Hydrotreating	18
4.0 2020 SOT Modeled Costs	22
5.0 Future Work and Progression to 2022 Projected Case.....	26
6.0 References.....	29
Appendix A – Comprehensive List of Waste Feedstocks Testing Data	A.1
Appendix B – Technical Tables and Separate HTL Plant Economics.....	B.1
Appendix C – Life Cycle Inventory for Supply Chain Sustainability Analysis	C.1
Appendix D – Cost Factors and Financial Assumptions.....	D.1

Figures

Figure S.1. Wet waste HTL and biocrude upgrading pathway cost allocations.	ii
Figure 1. Sludge HTL and biocrude upgrading block diagram for the 2019 SOT.	2
Figure 2. 2019 SOT (A) and 2020 SOT (B) design for pumping and heating of sludge feed to HTL reactor.....	3
Figure 3. Viscosity of three 50/50 primary/secondary sludge feeds (all at 20% solids). The samples are WW-06: GLWA sludge as received; CC-20-A: Contra Costa County sludge autoclaved; CC-20-R: Contra Costa County sludge, as received. The solid lines show the experimental data. The data for CC-20-R were used in the heat-exchanger calculations. Three extrapolations are shown above the experimental upper limit of 160 °C. ‘H,’ ‘M’ and ‘L’ represent high, medium and low extrapolations, respectively.....	4
Figure 4. Sensitivity of exchanger effective area to viscosity curve extrapolation (low, medium and high curves in Figure 3).	5
Figure 5. PNNL continuous flow laboratory HTL reactor system.....	8
Figure 6. PNNL’s engineering scale HTL system.	9

Figure 7. Modeled process designs of CHG/ACU (A) and SCREW (B) methods for HTL AP upgrading.	12
Figure 8. Biogas production curves for AD treatability testing of AP (at 28X dilution).	17
Figure 9. COD reduction for AD treatability testing of AP (at 28X dilution).	18
Figure 10. Density of upgraded biocrude from the GLWA sludge generated in PNNL's HTL engineering scale unit (MHTLS13) as a function of TOS.	21
Figure 11. Boiling point distribution (ASTM D2887) for hydrotreated product from sludges, swine manure, and the process model.	21
Figure 12. Combined HTL and biocrude upgrading process cost allocations.	24
Figure 13. MBSP and cost allocation of wet waste HTL process with different AP treatment.	25
Figure 14. Waste blending sites identified in preliminary geospatial analysis for waste-to-fuel. Values given are potential dry tons/day of waste that could be collected at a \$50/tonne transportation cost, with the total from 45 service areas representing 82% of the total wet waste resource.	27
Figure 15. Additive impacts of potential performance improvements and plant scale on production cost.	28
Figure B.1. Hydrothermal liquefaction biocrude cost allocations.	B.5

Tables

Table 1. Updates to parameters and costing for the primary heat exchangers in the SOT.	4
Table 2. Ultimate and proximate analysis (wt%) of wet waste samples tested.	7
Table 3. Wet waste HTL testing results and model assumption	10
Table 4. Operating conditions and experimental measures of HTL AP treatment methods.	13
Table 5. Key modeling assumptions of AP treatment methods for cost estimation.	14
Table 6. Veolia Characterization of HTL aqueous phase (MHTLS-13).	15
Table 7. Anaerobic digestion testing results of HTL aqueous phase (diluted 28X).	16
Table 8. Wet waste biocrude hydrotreating experimental results and model assumptions.	20
Table 9. Economic results for 110 dry ton/day sludge HTL plant (with AP NH ₃ stripping).	22
Table 10. Economics for biocrude upgrading plant feeding 115,000 gal/day.	23
Table A.1. List of feedstocks tested to date in support of the HTL SOT and pathway development.	A.1
Table A.2. HTL performance data for waste feedstocks tested to date.	A.2
Table A.3. Hydrotreating performance data for waste feedstocks tested to date.	A.4
Table B.1. Processing area cost contributions and key technical parameters for the SOT and projected cases for the combined wet waste HTL and upgrading pathway.	B.1
Table B.2. Processing area cost contributions and key technical parameters for the SOT and projected cases for the separate wet waste HTL plant.	B.3
Table C.1. Hydrothermal liquefaction plant parameters for greenhouse gas and water analysis.	C.1
Table C.2. Upgrading plant parameters for greenhouse gas and water analysis.	C.2
Table D.1. Cost factors for direct and indirect project costs.	D.1

Table D.2. Financial assumptions for the economic analysis.	D.1
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1.0 Introduction

Each year, the U.S. Department of Energy Bioenergy Technologies Office (BETO) assesses progress in their research and development efforts toward sustainable production of renewable fuels (DOE 2016) through the annual state of technology (SOT) assessment. The SOT assessment evaluates the impact of the year's research progress on the modeled minimum fuel selling price (MFSP) for selected biofuel/bioprocess conversion pathways and measures the current state of the technology relative to defined goal case projections. Technical and cost targets for a projected goal case set for the year 2022 were previously established for the wet waste hydrothermal liquefaction (HTL) and biocrude upgrading pathway and summarized in a design report (Snowden-Swan et al. 2017). The 2019 SOT assessment showed a reduction of \$2.05/GGE in MFSP relative to the 2018 SOT resulting from research progress in biocrude hydrotreating catalyst performance (Snowden-Swan et al. 2020). This report summarizes the research and associated techno-economic analysis (TEA) for the pathway 2020 SOT. Methods and economic assumptions for the n^{th} plant analysis used for the TEA are consistent with the design report (Snowden-Swan et al. 2017), with the exception of updates in the modeled cost year (2016) and income tax rate (21%). Appendix D provides the full list of financial and economic assumptions used in the analysis.

2.0 Conversion Model Overview

Figure 1 shows the overall block flow diagram for the conversion of sludge from a wastewater treatment and water resource recovery facility (WRRF) via HTL and biocrude upgrading. The modeled scales for the WRRF/HTL plant and the centralized biocrude upgrading plant are 110 dry ton/day sludge and 38 million gal/yr biocrude feed, respectively, and are consistent with the design case and 2019 SOT (Snowden-Swan et al. 2017, 2020). The centralized upgrading plant processes 10 times the amount of biocrude generated from one 110 dry ton/day HTL plant. The overall process configuration is unchanged from the 2019 SOT assessment. While the overall process remains the same as the previous SOT, the HTL model has been updated with a new pumping and heating configuration. The details of the new design are presented in section 2.1.

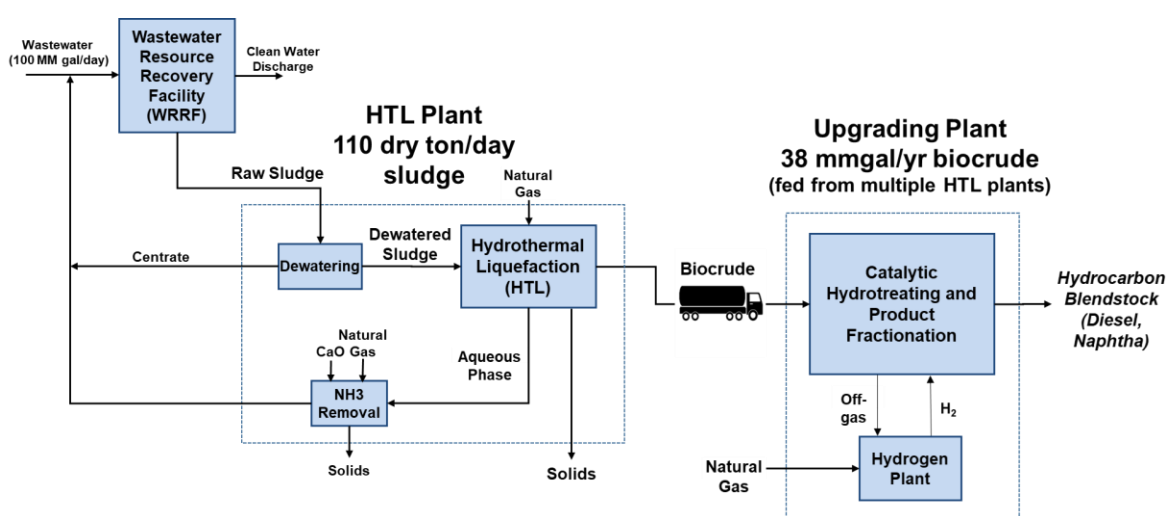


Figure 1. Sludge HTL and biocrude upgrading block diagram for the 2019 SOT.

2.1 Heat Exchanger Re-Design

The 2019 SOT (Snowden-Swan et al. 2020) showed that approximately 50% of the capital cost for the HTL plant is from the heat exchangers used to heat process slurry to the reactor temperature (350°C). The high cost of the exchangers stems from the high viscosity of the slurry feedstock, which leads to low Reynolds numbers and a large effective area requirement. The high operating pressure of the HTL process also leads to thick tube and shell walls. A detailed comparative design analysis of several alternative arrangements was performed to investigate more practical and potentially economically advantageous designs for operation at high pressure. Figure 2 shows flowsheets comparing the 2019 SOT and the new (2020 SOT) designs for the heating and pumping section of the HTL plant. There are two major differences in the new design compared to the previous one. First, the new design splits the heating into 2 stages, where the slurry is heated to 500°F (260°C) at 1000 psia in the 1st stage (HX-100), then pumped to 3069 psia and heated to 656°F (347°C) in the 2nd stage (HX-101). The advantage of having two stages is that the majority of the slurry heating can be performed at the lower tube pressure. A high temperature pump (rotary lobe type) is used to pump the heated slurry to the HTL reactor pressure of 3069 psia (Berglin et al. 2012). The second change in the design is that heat transfer fluid is employed as an intermediate carrier and therefore the shell side pressures on all exchangers are low (165 psia). Reduced

pressure on the tube side of the 1st stage heat exchangers and on the shell side in both the 1st and 2nd stage lead to reduced tube and shell thicknesses and therefore reduced cost.

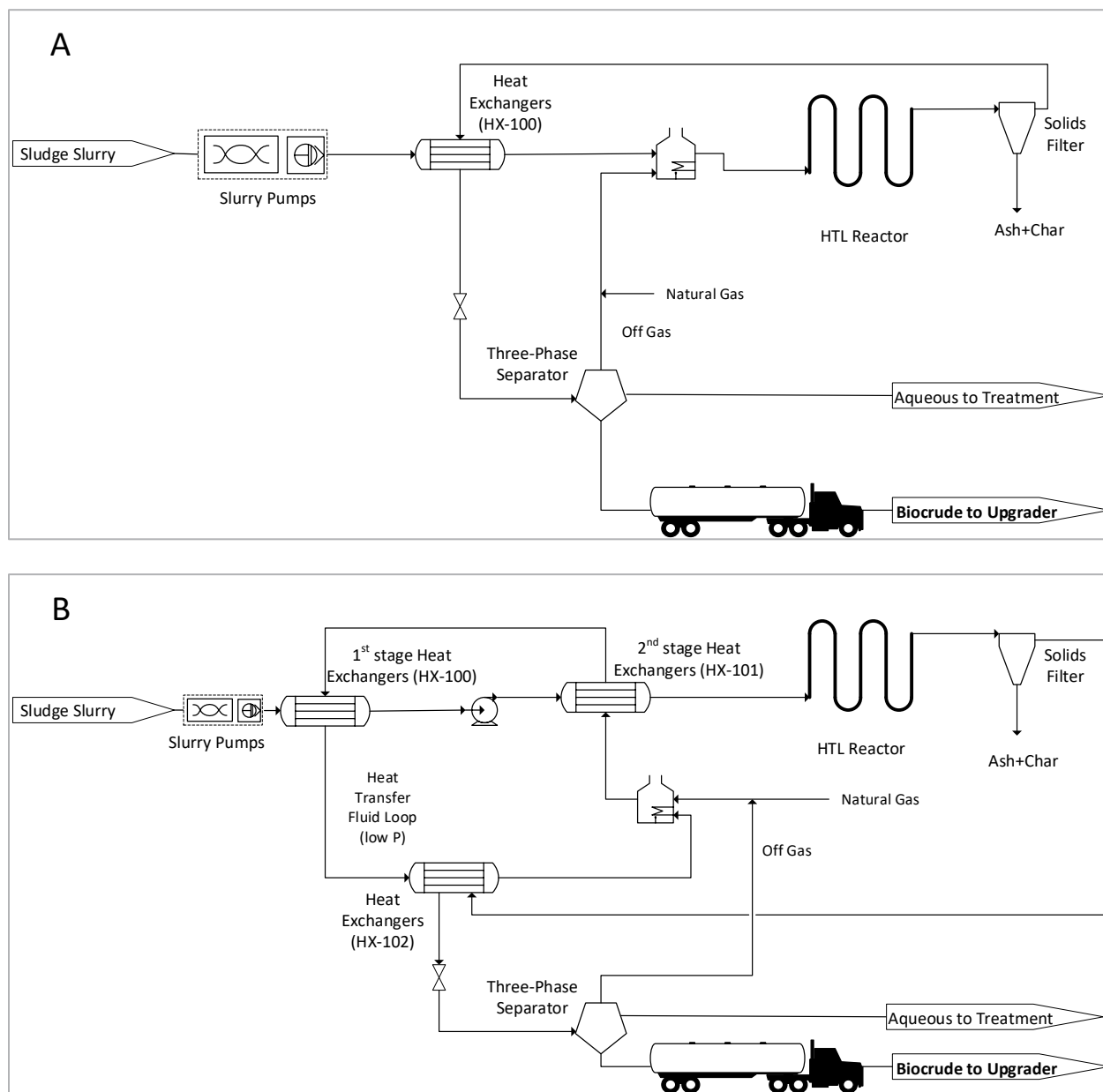


Figure 2. 2019 SOT (A) and 2020 SOT (B) design for pumping and heating of sludge feed to HTL reactor.

Sludge rheology data was generated in-house and used to improve the fidelity of the exchanger designs and sizing. Figure 3 shows the viscosity curves for three 50/50 primary/secondary wastewater treatment sludge samples: WW-06 was sludge from Great Lakes Water Authority (GLWA), as received; CC-20-A was Contra Costa County sludge, autoclaved; and CC-20-R was Contra Costa County sludge, as received. All samples contained 20% solids and were tested at a shear rate of 50s^{-1} . The data for CC-20-R were used in the SOT baseline heat exchanger calculations. Due to pressure limitations of the viscometer, the

maximum temperature tested was 71°F (160°C). Extrapolation of the viscosity curve was performed to provide an estimate over the entire temperature regime, with a range of high, medium and low viscosity estimates. The range of extrapolations is expected to cover the range from conservative to optimistic conditions. The medium extrapolation was used for the SOT and sensitivity analysis was conducted with the high and low estimates.

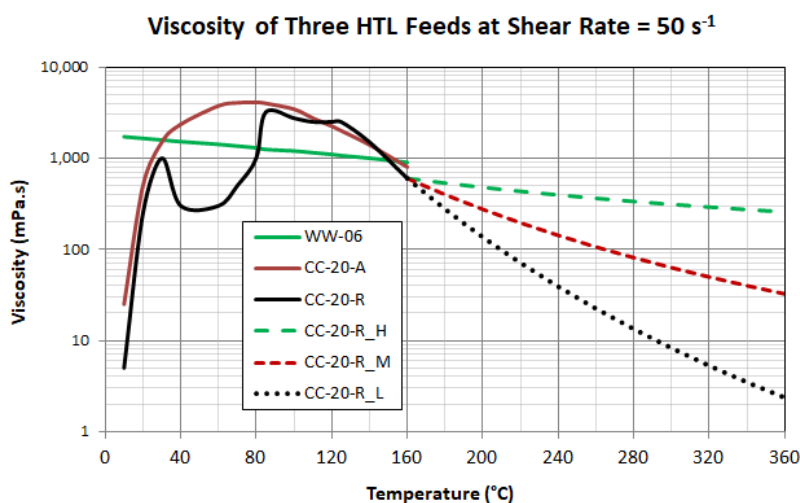


Figure 3. Viscosity of three 50/50 primary/secondary sludge feeds (all at 20% solids). The samples are WW-06: GLWA sludge as received; CC-20-A: Contra Costa County sludge autoclaved; CC-20-R: Contra Costa County sludge, as received. The solid lines show the experimental data. The data for CC-20-R were used in the heat-exchanger calculations. Three extrapolations are shown above the experimental upper limit of 160 °C. ‘H,’ ‘M’ and ‘L’ represent high, medium and low extrapolations, respectively.

Table 1 shows the updates in design parameters and costing of heat exchangers for the 2020 SOT relative to the 2018/2019 SOT. It is important to note that the assumed overall heat transfer coefficient (U) used to size the exchangers was also updated for the current SOT, which resulted in larger effective areas. However, the overall improvements in the design counteracted this impact to effectively reduce the exchanger capital cost by \$2.9 million. Importantly, the new design also provides a more realistic configuration that can be fabricated at scale using conventional pipe schedules for both shells and tubes, as indicated by vendor feedback.

Table 1. Updates to parameters and costing for the primary heat exchangers in the SOT.

	2018/2019 SOT Model	2020 SOT Model (new split configuration with hot oil on the shell)		
	High-P HX (HX-100)	Low-P HX (HX-100)	High-P HX (HX-101)	HX-102
Design Pressure (psia)	3074 (tube) 2969 (shell)	1000 (tube) 164 (shell)	3069 (tube) 165 (shell)	2900 (tube) 180 (shell)
Assumed U, Btu/hr/ft ² /°F	50	17	27	76
LMTD, °F	122	61	30	77
Area, ft ²	3,277	19,329	12,213	3,953
Installed Cost, \$MM (2016)	12.2	3.1	4.5	1.7

Total Installed Cost, \$MM (2016)	12.2	9.3
Source of Costing	Knorr 2015	New configuration (Fig.2) and HTRI design informed by sludge viscosity testing data

Figure 4 depicts a sensitivity analysis around the heat exchanger effective area for the low, medium and high viscosity extrapolations shown in Figure 3. This uncertainty in the extrapolated region above 71°F (160°C) has the most potential impact on the calculated area of HX-101, with the required effective area varying by nearly 70%, which translates to an approximate 45% change in cost using a scaling exponent of 0.7. Planned modifications to the viscometer in 2021 will enable additional feedstock rheology testing over the full temperature regime to provide improved fidelity of the heat exchanger sizing estimations. Along with this data, a new data program for incorporating viscosity curves and enhancing the model will help tighten the designs. In addition, technology enhancements to reduce exchanger effective area and cost will be explored. Examples include the use of tube inserts and corrugated tubes to improve heat transfer rates.

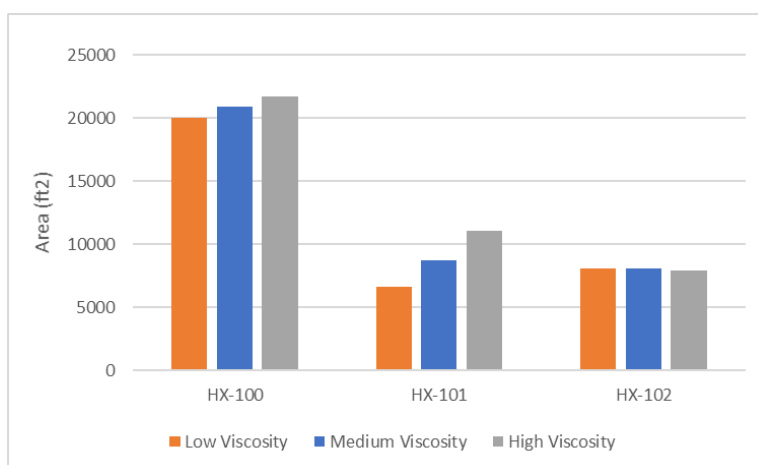


Figure 4. Sensitivity of exchanger effective area to viscosity curve extrapolation (low, medium and high curves in Figure 3).

3.0 Experimental Results

The primary experimental results used in the SOT analysis include 1) wet waste compositional analysis; 2) wet waste HTL processing; 3) aqueous phase (AP) treatment; and, 4) hydrotreating of resulting biocrudes. The experimental data and discussion of how they were used in the analysis are presented in the following sections.

3.1 Wet Waste Feedstock Composition

Wet waste feedstocks tested in FY20 include the following:

- 50/50 mix of primary/secondary sludge from Central Contra Costa Sanitary District (CCCSD), CA, without added lime
- 100% pure primary sludge from Great Lakes Water Authority (GLWA) in Detroit, MI
- Cow manure from a dairy operation, with and without inorganic catalyst
- Biosolids from anaerobic digestion of wastewater treatment sludge

The 50/50 sludge from CCCSD was run to compare with the same plant's sludge run in FY19 (see WW09 in Table A.1, Appendix A), but without the added lime that is currently used to assist in their sludge incineration process. The sludge from GLWA is from their WRRF primary treatment (solids settling) step. The biosolids sample was from a Texas WRRF (provided by Aloviam), and the cow manure was provided by Washington State University. Table 2 shows the ultimate and proximate analysis for the feedstocks tested. Analysis for the GLWA sludge, on which the design case (Snowden-Swan 2017) and SOT are based, is also listed for comparison. Note the cow manure was collected from a controlled feeding operations dairy and is representative of bulk waste that would be collected at larger scale. A comprehensive list of wet waste feedstocks tested to date in support of the development of this pathway is also given in Appendix A. To date, three of the four major underutilized wet waste feedstocks generated (wastewater solids, manure, food waste, and fats/oils/grease [FOG]) have successfully been processed using real-world samples from actual waste generators. Testing of food waste is planned for FY21.

The modeled 2020 SOT feedstock composition remains unchanged to maintain consistency with the design case at this time. It is conceivable and desirable that in the future, wastes could efficiently be collected in areas of the country where generation is concentrated, thereby improving economies of scale for the HTL conversion plant. Many WRRFs are already collecting regional food wastes and FOG for co-digestion with sludge in their anaerobic digestion units. Initial geospatial resource analysis and siting (Seiple 2019) suggests that “hot spots” of generation, primarily located in metropolitan areas and intense animal farming areas (e.g., Midwest U.S.), actually account for the majority of the wet waste resource. More details on the waste blending resource analysis are given in Section 5. Testing of additional feedstocks provides critical information to inform the feasibility of regional waste blending scenarios that help move toward an overall strategy of capturing as much of these underutilized wastes as possible and enabling a more circular economy.

Table 2. Ultimate and proximate analysis (wt%) of wet waste samples tested.

	WW06 50/50 Sludge GLWA (Dry)	WW06 50/50 Sludge GLWA (DAF)	MHTLS13 Primary Sludge GLWA (Dry)	MHTLS 13 Primary Sludge GLWA (DAF)	WW14 Biosolids (Dry)	WW14 Biosolid s (DAF)	WW17 CCCS D Sludge (No Lime) (Dry)	WW17 CCCS D Sludge (No Lime) (DAF)	WW19A ^a Cow Manure (Dry)	WW19A ^a Cow Manure (DAF)	WW19B ^a Cow Manure (Dry)	WW19B ^a Cow Manure (DAF)	2020 SOT and 2022 Models (Dry)	2020 SOT and 2022 Models (DAF)
C	41.1	52.0	42.3	52.5	34.3	47.6	44.8	52.7	43.9	50.6	43.1	50.3	46.8	52.1
H	5.8	7.3	6.2	7.7	4.7	6.5	6.1	7.1	5.7	6.6	5.7	6.7	6.5	7.2
O	26.1	33.0	26.9	33.4	26.4	36.1	27.4	32.3	34.0	39.4	33.8	39.4	29.7	33.1
N	5.0	6.3	4.2	5.2	5.3	7.4	6.1	7.1	2.6	3.0	2.6	3.0	5.7	6.3
S	1.0	1.3	1.0	1.2	1.6	2.3	0.7	0.8	0.5	0.6	0.5	0.6	1.2	1.3
Ash	26.1		25.6		32.6		17.1		15.9		16.7		15.0	
P	1.9		1.9		2.0		1.9		0.7		0.7		1.9	
Carb	16.7	22.8	26.7	34.9	17.5	30.5	30.8	38.2	60.3	70.0	NM	NM	Not modeled	
Fat	22.6	30.8	20.6	27.0	11.6	19.3	14.2	17.6	10.1	11.8	NM	NM	Not modeled	
Protein	34.1	46.4	29.0	38.0	29.6	51.0	37.6	46.7	15.7	18.2	NM	NM	Not modeled	
FAME	11.9	16.2	15.4	20.2	5.5	13.0	9.5	11.5	5.8	6.7	NM	NM	Not modeled	
Ash	26.6		23.7		41.4		17.4		13.8		NM			

(a) WW19-A and WW19-B were run without and with inorganic homogeneous catalyst, respectively.

DAF = dry, ash-free

3.2 Wet Waste Hydrothermal Liquefaction

Figure 5 shows a schematic of the sludge HTL experimental bench-scale system at Pacific Northwest National Laboratory (PNNL). The capacities of the system's stirred vessel reactor and plug-flow reactor (PFR) are 600 mL and 550 mL, respectively, with a flow rate of 2-4 L/hour. PNNL's engineering-scale HTL testing unit, shown in Figure 6, has a similar configuration but with a capacity approximately five times that of the bench scale system (12-16 L/hour). Testing with CCCSD 50/50 primary/secondary sludge (WW17), biosolids (WW14) and cow manure (WW19) were run in the bench scale unit, while testing of the GLWA sludge (MHTLS-13) was run in a pure plug flow configuration in the engineering scale system.

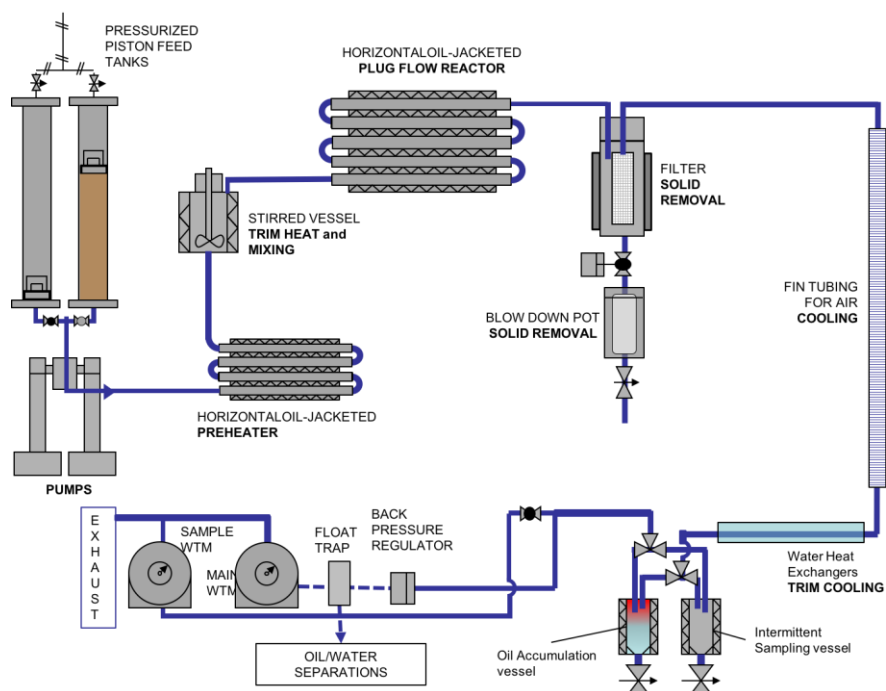


Figure 5. PNNL continuous flow laboratory HTL reactor system.



Figure 6. PNNL's engineering scale HTL system.

Experimental HTL testing conditions and results are given in Table 3, along with the parameters used for the modeled SOT and projected cases. The 2020 SOT feedstock and biocrude yields have not changed from the 2019 SOT assumptions (Snowden-Swan et al. 2020). Testing results show that the biocrude yield from GLWA primary sludge run in the engineering scale system (MHTLS13) is slightly lower than that achieved with the primary/secondary sludge from GLWA originally run in 2017 (WW06). However, the yield was relatively high (41% DAF basis) given that it was run at lower feed solids content and high ash (26% dry basis). Primary sludge showed higher yields than secondary sludge in previous HTL work (Marrone 2016), so this result is not surprising. Yield from CCCSD sludge without lime (WW17) was boosted from 37% to 41% (11% increase) relative to the CCCSD sludge including lime (see Appendix A, Table A.1, WW09), even with a lower feed solids content.

Cow manure was run without (WW19A) and with (WW19B) an additive ($< 0.5\%$) to investigate the potential catalytic effect on product yields. Addition of catalyst appears to increase biocrude yield by 22% (from 32 to 39%) and to shift overall conversion selectivity from lighter, water soluble products (i.e., the AP yield) to the heavier biocrude phase. These results are encouraging because they show that similar yields to sludge (37-44% range) may be possible for HTL of cow manure if run at optimal solids content (20-25%). Biocrude yield from biosolids (WW14) is significantly lower than sludge or manure. This is likely due in part to high ash content (32%) in the biosolids and therefore higher levels of biocrude adhering to the solids during separation, as seen with the higher solids yield (20% ash-free dry basis) relative to other feedstocks. Note that all four of the new feedstocks were processed at less than optimal solids contents ($< 20\%$ solids) due to the limitations of the bench and engineering scale pumping systems. For this reason and to maintain consistency with the 50/50 primary/secondary feedstock assumed for the design case (Snowden-Swan et al. 2017), the 2020 SOT feedstock and yield assumed are consistent with the GLWA performance results (WW06 in Table 3). Note that pumping of 20-25% solids content slurry is expected to be well within the capabilities of commercial scale slurry pumps (Berglin et al. 2012).

Table 3. Wet waste HTL testing results and model assumption

Operating Conditions and Results	50/50 Sludge (GLWA) WW06	Primary Sludge (GLWA) MHTLS13	AD Biosolids WW14	50/50 Sludge-no lime (CCCCSD) WW17 SS-1	Cow Manure WW19A	Cow Manure WW19B	2020 SOT Model	2022 Projected Model
Temperature, °F (°C)	656 (347)	662 (350)	649 (343)	653 (345)	646 (341)	639 (337)	656 (347)	656 (347)
Pressure, psia (MPa)	2979 (20.5)	2940 (20.3)	2840 (19.6)	2840 (19.6)	2940 (20.3)	3000 (20.7)	2979 (20.5)	2979 (20.5)
Feed solids, wt%								
Ash included	20%	15.3%	16.7%	14.6%	15%	15%	20%	25%
Ash-free basis	15%	11.4%	11.3%	12.1%	12.3%	12.1%	17%	21%
Liquid hourly space velocity, vol./h per vol. reactor	3.6 ^(d)	4.0	3.5	3.5	3.5	3.5	3.6	6
Equivalent residence time, min.	17	15	17	17	17	17	17	10
Product yields ^(a) (dry, ash-free sludge), wt%								
Oil (biocrude)	44%	41%	31%	41%	32%	39%	44%	48%
Aqueous	31%	33%	35%	36%	42%	30%	29%	25%
Gas	16%	19%	14%	19%	22%	29%	16%	16%
Solids	9%	7%	20%	4%	3%	3%	12%	11%
Carbon yields								
Oil (biocrude)	58%	51%	42%	55%	49%	53%	65%	72%
Aqueous	24%	30%	31%	30%	29%	25%	21%	18%
Gas	8%	9%	8%	10%	13%	15%	10%	10%
Solids	10%	9%	20%	5%	10%	7%	5%	1%
HTL dry biocrude analysis, wt%								
C	78.5%	78.5%	76.3%	75.9%	76.5%	76.5%	78.3%	78.3%
H	10.7%	10.8%	9.4%	9.8%	9.2%	9.0%	10.8%	10.8%
O	4.7%	5.8%	6.3%	8.5%	9.6%	9.8%	4.8%	4.8%
N	4.8%	4.2%	5.1%	5.0%	3.9%	4.1%	4.9%	4.9%
S	1.2%	0.6%	1.8%	0.6%	0.4%	0.3%	1.2%	1.2%
P	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	Not modeled ^(b)	Not modeled ^(b)
Ash	0.06%	0.1%	1.0%	0.2%	0.4%	0.2%	0.0%	0.0%
HTL dry biocrude H:C ratio	1.6	1.7	1.5	1.5	1.4	1.4	1.6	1.6
HTL biocrude dry higher heating value ^(c) , Btu/lb (MJ/kg)	16,900 (39.5)	17,000 (39.6)	15,980 (37.2)	15,970 (37.1)	15,700 (36.5)	15,600 (36.4)	17,100 (39.7)	17,100 (39.7)
HTL biocrude moisture, wt%	4.4%	3.5%	7.3%	7.0%	4.5%	4.8%	4.0%	4.0%
HTL biocrude wet density @ 77°F (25°C) (g/ml)	0.98	0.95 ^(g)	1.01 ^(f)	Pending	1.03 ^(g)	1.04 ^(g)	0.98	0.98
Aqueous phase chemical oxygen demand (mg/L)	61,300	53,800	53,000	66,100	61,800	59,800	62,700	61,100

(a) Recovered after separations.

(b) Phosphorus partitioning is not directly modeled in Aspen because of the small quantity, most of which reports to the solid phase.

(c) Calculated using Boie's equation (Boie 1953).

(d) The experimental system includes a continuous stirred-tank reactor (CSTR) followed by a PFR. The CSTR helps prevent overheating of the feed.

(e) Runs A and B are without and with homogeneous catalyst in feed.

(f) Measured at 104°F (40°C)

(g) Measured at 140°F (60°C)

(i) WW runs were in the bench-scale system and MHTLS-13 was run in the engineering scale system.

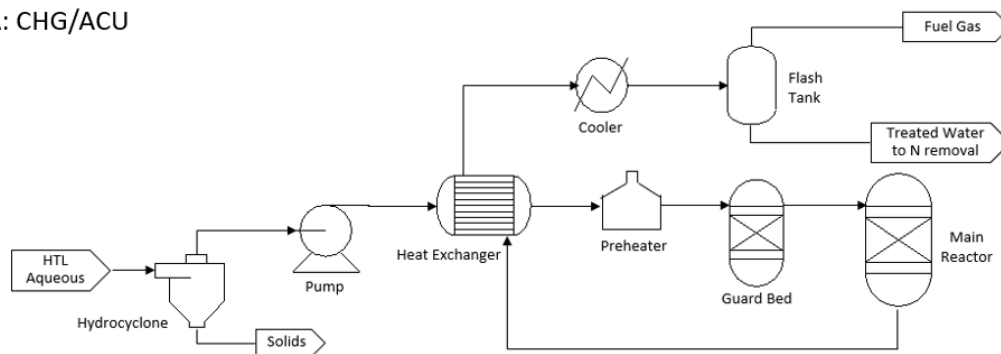
3.3 Aqueous Phase (AP) Treatment

3.3.1 Thermochemical Methods

For the modeled 2020 and previous SOTs, the AP from the HTL reactor is assumed to be treated with ammonia stripping and then recycled to the WRRF. While this is one possible method for treating the AP, it may not be the most optimal for several reasons. Firstly, pH adjustment is necessary to shift ammonia to the gas phase, which is achieved with the addition of lime. Lime has high greenhouse gas emissions and also results in the generation of lime sludge as a waste. Second, because the AP contains high chemical oxygen demand (COD), significant levels of organic are removed along with ammonia in the air stripping process, which leads to an impure ammonia stream. Thus, a salable nutrient by-product is not feasible and the stripped ammonia and organics must be destroyed in a thermal oxidation unit, which requires natural gas for heat and represents wasted carbon and nitrogen. And lastly, significant COD remains in the AP after stripping, which could potentially impact the WRRF's biological operations, depending on the compounds still remaining. These aspects all affect the environmental sustainability of the pathway and therefore improved methods for treating the AP are under investigation.

Treatment testing of AP in 2020 focused on COD removal as a first step prior to ammonia stripping, to reduce potential risk from HTL components recycled to the WRRF and to enable recovery of a more pure ammonia stream as a by-product of the process. Three thermochemical methods, catalytic hydrothermal gasification (CHG), steam phase-catalytic reduction of wastewater (SCREW) and AP catalytic upgrading (ACU), were tested. The testing data was used to develop conceptual models and associated TEA at the SOT scale to provide initial high-level economics and sensitivity around the SOT MFSP for each of the CHG, ACU, and SCREW options. The conceptual process flowsheets are shown in Figure 7. Note that process configurations and TEA for these options represent the scaled up systems, which do not exactly match the laboratory scale experimental setups. In the laboratory systems, solids in the untreated AP from HTL were separated in a big source container by settling. The laboratory SCREW system is a once-through system, which does not include H₂ recovery and recycling units. All testing methods were run with AP from HTL of CCCSD primary/secondary sludge (MHTLS-07), containing a COD of 65,000 mg/L, and approximate C, S and N contents of 2%, 0.6% and 0.7%, respectively. A total suspended solids (TSS) content of 0.84 mg/L was assumed, which was measured in anaerobic digestion testing (Section 3.3.2). In the process models, the laboratory data for reductions in COD, C, N, and S were used, while the measured gas-phase product selectivity was slightly adjusted to satisfy the overall elemental balance. Further work is necessary to validate the process assumptions.

A: CHG/ACU



B: SCREW

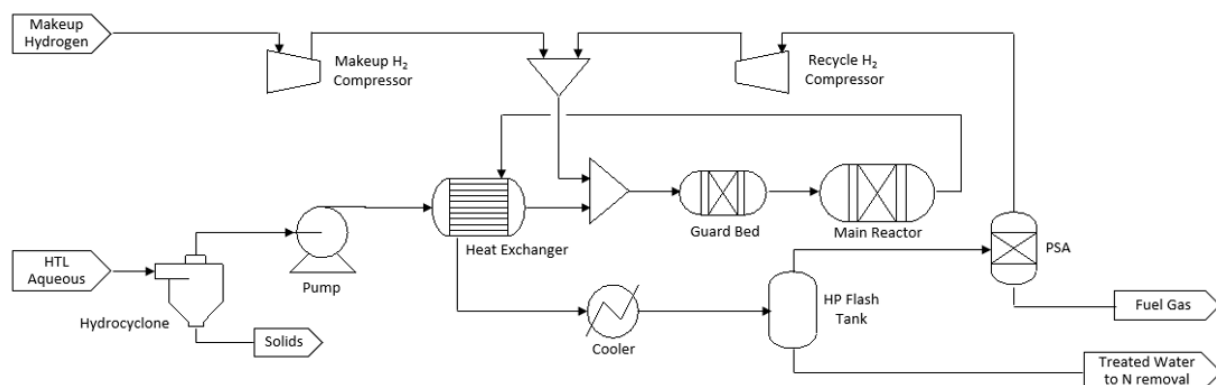


Figure 7. Modeled process designs of CHG/ACU (A) and SCREW (B) methods for HTL AP upgrading.

In all modeled processes, the AP product from the HTL reactor is first sent to a hydrocyclone to remove residual suspended solids to protect downstream operations. A hydrocyclone was selected because it is capable of separating fine particles and has been widely used in wastewater treatment plants for solid-liquid separation (Bayo et al., 2015) and the oil industry for oil-water separation (Young et al., 1994). In the CHG process, Ru/C catalyst was used to promote gasification reactions to convert organics in the HTL AP into fuel gas (containing 2 vol% H₂, 21 vol% CO₂, and 72 vol% CH₄) at elevated temperature and pressure (350 °F and 3050 psig). The main products of the CHG process are fuel gas and treated water. The fuel gas can be used to supply plant heat and steam. More details about the CHG process can be found in Jones et al. (2014). In the ACU process, ZnZr catalyst was used to promote the conversion of organics in the AP into CO₂, CH₄, light alkenes and ketones (with carbon selectivity of 24%, 15%, 20% and 6%, respectively). The process design of the ACU process is similar to the CHG process, with the exception that the ACU process is operated near ambient pressure. The pump in the ACU process is only needed to overcome pressure drops from the hydrocyclone, reactors and pipeline. In the SCREW process, the HTL AP is hydrotreated with excess H₂ in a fixed bed reactor filled with NiMo/C catalyst at 752°F (400 °C) and 500 psig. The main products are colorless clean water and high value gases (30.7 wt% CO₂, 9.7 wt% CH₄, and 59.6 wt% C₂₊ gases) that can be used as utilities. The treated water from the CHG and SCREW processes, containing near zero volatile organics but considerable NH₃, is sent to a NH₃ stripper to produce high purity NH₃, which is then converted into (NH₄)₂SO₄ and sold as by-product to improve process economics. For the ACU process, the treated water, which still contains organic N species, is sent either to a NH₃ stripper (STR) to remove almost all NH₃ and produce by-product, or to a zeolite based temperature swing adsorption (TSA) unit to remove about 80% NH₃ and organic N species, which can then be oxidized. Both options were modeled due to uncertainty as to which option would be the most effective. Additional work is necessary to determine the optimal method for nitrogen removal for the

ACU method. Note that this zeolite adsorption approach is a new concept currently under development, aiming to provide cost effective alternatives to NH₃ stripping for treating both NH₃ and organic N species.

Table 4 summarizes the operating conditions and key experimental data for each treatment option tested. As shown, all methods tested can significantly reduce the AP COD, and therefore produce high purity NH₃ for by-product production. The CHG and SCREW methods provide nearly 100% COD reduction at relatively severe operating conditions and/or consumption of H₂, which can potentially lead to high treatment cost. The ACU process, on the other hand, does not provide the level of COD reduction as CHG and SCREW (as indicated by the measured COD, C, N, and S reduction), but can be operated at near ambient pressure with much less capital investment. Therefore, the ACU method can be considered as a low-cost, mid-level COD reduction option for HTL AP treatment.

Table 4. Operating conditions and experimental measures of HTL AP treatment methods.

Method	CHG	SCREW	ACU
Operating conditions			
Temperature, °F (°C)	662 (350)	752 (400)	752 (400)
Pressure, psia (bar)	3065 (211)	515 (36)	15 (1)
Liquid hourly space velocity, vol./h per vol. reactor	0.54	0.5	0.27
Catalyst			
Type	Ru/C	NiMo/C	ZnZr
Regeneration [Frequency]	Acetone wash, Hydrogen reduction [every 5 days]	Not needed	Not needed
Guard bed [Frequency]	Not needed	C [every 10 days]	C [every 10 days]
Reactor Performance (experimentally measured if not specified in footnotes)			
Main products	Fuel gas (2 vol% H ₂ , 21 vol% CO ₂ , and 72 vol% C H ₄)	CO ₂ , C ₁ -C ₅ light gases	CO ₂ , CH ₄ , light alkenes, ketones
COD reduction, %	99.8	97.7	Not measured
H ₂ consumption, g/100g feed		0.39 ⁽¹⁾	
C reduction, %	100	85	85
Organic N reduction, %	100	18	0
S reduction, %	100	93	0

(1) Estimated based on elemental balance.

The process design and experimental data presented in Figure 7 and Table 4 were used to develop process and cost models to evaluate the economic performance of the above three methods for HTL AP treatment. Note that for the ACU method, two process designs were considered with different approaches to treat nitrogen-rich components. Table 5 summarizes additional assumptions that were used for cost estimation of each process design. The initial testing and modeling results indicate that these methods could provide an estimated 80-100% ammonia nitrogen removal and 0 -100% organic nitrogen removal from the AP. Note that catalyst lives for these methods represent optimistic conditions that are possible through further research.

Table 5. Key modeling assumptions of AP treatment methods for cost estimation.

	CHG	SCREW	ACU-STR	ACU-TSA
Catalyst price, \$/kg	128	38	2	2
Catalyst life, year	1	1	1	1
Additional N treatment method used	NH ₃ stripping, (NH ₄) ₂ SO ₄ production	NH ₃ stripping, (NH ₄) ₂ SO ₄ production	NH ₃ stripping, (NH ₄) ₂ SO ₄ production	Zeolite-based TSA, THROX
Organic N reduction, %	100	18	0	80
NH ₃ in treated water w/o additional N treatment, wt% ⁽¹⁾	1.01	0.98	0.94	0.94
NH ₃ reduction, %	100	100	100	80
By-product end use				
Fuel gas	Burned	Burned	Burned	Burned
N-rich components	(NH ₄) ₂ SO ₄	(NH ₄) ₂ SO ₄	(NH ₄) ₂ SO ₄	Oxidized
COD reduction, %	Measured	Measured	65 ⁽²⁾	70 ⁽²⁾

(1) Estimated from reactor model.

(2) Estimated from the elemental composition of the treated water.

3.3.2 Anaerobic Digestion

Batch anaerobic digestion (AD) testing was conducted by Veolia to investigate the feasibility of combined AD and anaerobic ammonium oxidation (anammox) to reduce AP carbon and nitrogen levels. An initial AD step was determined to be required to reduce COD in the AP to levels amenable for the anammox bacteria. Testing was performed on AP from HTL of GLWA primary sludge (MHTLS-13, see Table 3). Characterization of the AP is shown in Table 6. All analyses were carried out in the Veolia lab except for oil and grease and BOD₅ measurements, which were done by Test America. High rate anaerobic wastewater treatment systems are designed to treat soluble components in wastewater. The wastewater retention time in the AD reactor is normally not long enough to allow for significant breakdown of solids. The TSS concentration in wastewaters that are most suitable for high rate AD should be less than 10% of the total COD (TCOD) concentrations. In this case, the TSS of the AP sample is quite low at 84 mg/L, well under 10% of the TCOD concentration. With ammonia higher than 2500 mg/L, ammonia inhibition and/or toxicity can start to be a problem.

Healthy anaerobic bacteria (referred to here as AD sludge) require certain amounts of macronutrients, nitrogen (N) and phosphorus (P). These nutrients ensure proper AD sludge activity and growth. A COD:N:P ratio of 1000:5:1 is recommended for wastewaters similar to the HTL AP. The TCOD of the AP was 48950 mg/l. From this, the recommended amounts of N and P are 244 mgN/l and 49 mgP/l. The AP characterization results indicate that this wastewater is deficient in phosphorus. Therefore, we expect that additional P would be required in a full scale plant. For the Veolia treatability test, phosphorus was added in with the micronutrient blend.

The COD to biological oxygen demand (BOD₅) ratio of this sample was 2.42. The COD to BOD₅ ratio is typically 1.6 - 2.0 for easily biodegradable wastewaters originating from industrial applications. The 2.42 ratio for the AP indicates that the COD is not as easily biodegradable as other types of wastewater. A higher COD to BOD₅ ratio typically indicates that the final of COD reduction will be depressed.

Sulfur, in the form of H_2S , in high concentrations can be inhibitory or toxic to anaerobic bacteria. Sulfate and sulfite are converted to H_2S in the anaerobic reactor. The sulfate concentration in the AP sample was 228 mg/l. This ratio of SO_4 to COD would not be toxic for the AD sludge. Sulfur is also a nutrient required by the AD sludge. At least 15 mgS/l are required to ensure healthy AD sludge. Therefore, sulfur will not have to be added to the system as part of the micronutrients. The SO_4 will also be converted to H_2S and HS^- in the anaerobic reactor, and will be present in the biogas, as well as the liquid effluent.

FOG in the influent to high rate anaerobic reactors should be kept to a minimum. Veolia suggests that the influent FOG concentration be less than 75 mg/l to ensure good system performance. There was no FOG detected in the AP sample. Hence, FOG should not cause any problems for an anaerobic digester.

Table 6. Veolia Characterization of HTL aqueous phase (MHTLS-13).

Parameters	Units	Undiluted AP
pH	s.u.	8.45
Acetic Acid	meq/L	23.86
Propionic Acid	meq/L	3.44
Butyric Acid	meq/L	1.14
Total VFA	meq/L	4.58
ALK	meq/L	182
TCOD	mg/L	48950
SCOD	mg/L	48200
TS	mg/L	6970
VS	mg/L	5785
TSS	mg/L	84
VSS	mg/L	84
TKN	mg N/L	4594
SKN	mg N/L	3698
NH ₃	mg N/L	2810
NO ₂	mg N/L	0.688
NO ₃	mg N/L	87.5
Total P	mgPO ₄ /L	15.6
Soluble P	mgPO ₄ /L	11.2
Ortho-P	mgPO ₄ /L	5.58
SO ₄	mg/L	228
Chloride	mg/l	122
Oil & Grease	mg/L	<2.5
BOD ₅	mg/L	20200

Typically, the maximum allowable reactor influent COD concentration in full-scale AD plants is 8000 mg/L, therefore the AP sample was diluted prior to testing. A first set of tests was performed with sample diluted to 7500 mg/L COD (a dilution of 6.5X). Results at this dilution were unfavorable, indicating poor COD reduction and microbial inhibition. A second set of tests was then conducted at a 4X higher dilution than the initial testing (1760 mg/L COD) to potentially bring the inhibitory compounds into a range that the organisms could tolerate.

Testing was conducted in a 2.0-liter glass bench reactor. Three hundred fifty (350) milliliters of active AD sludge from an operating high rate Veolia system was used to seed the reactor, resulting in a starting F:M (food to microorganism ratio) of about 0.13 gTCOD/gVSS (volatile suspended solids). Micronutrients and phosphorus were added to the reactor. The pH was adjusted to 7.2 s.u. and sodium bicarbonate was

added for buffering. The reactor was sealed, and the contents were gently and intermittently mixed. The temperature was maintained at 95°F (35°C) using a water bath.

The biogas produced is primarily methane with some carbon dioxide and is bubbled through a bottle containing a caustic solution. The carbon dioxide in the biogas is absorbed by the alkaline solution, so only the remaining methane is measured in by the measuring counter which is connected to a computer. The volume of methane generated during the study is continually recorded during the study. During the course of the digestion the pH is checked once per day to ensure it is within optimal range for the biomass, 6.8-7.6 s.u. The pH did not significantly drift in either direction and no further adjustment was required. The TCOD and soluble COD (SCOD) are measured at the beginning and end of each test run. SCOD is measured from the filtrate of a glass fiber filter (1.5 micron). The volatile fatty acids (VFA) are also measured at the end of each test run.

The results of the second sets of tests at the higher dilution (28X diluted from original sample) are shown in Table 7. In this test, four consecutive runs were completed. At the end of the first run the contents of the reactor are allowed to settle and the effluent is decanted off. For the second run, the remaining microbial biomass is fed fresh AP (diluted) sample and nutrients in the same proportions as the first run and the procedure is repeated for subsequent runs. Removal of TCOD and SCOD from the AP over the four tests averaged 30%. The final effluent was sent out for BOD testing, which showed 466 mg/L of BOD remaining in the effluent. The calculated overall BOD degradation is 72% at this lower digester loading rate. The BOD reduction is calculated by multiplying the TCOD/BOD ratio of the wastewater with the measured TCOD in the last run to get a calculated starting BOD. The final BOD was a direct measurement of the effluent. The COD reduction trended down in each run, but appears that it may stabilize rather than go to zero (as happened in the first treatability test). The gas yield is higher than the expected theoretical value of 0.35 LCH₄/gTCOD_r due to the increased contribution of endogenous respiration. The effluent VFA concentration is low in these tests, indicating that the methane formers are not severely inhibited. It appears that a constituent(s) in the AP is preventing significant COD reduction, even at the high dilution rate.

Table 7. Anaerobic digestion testing results of HTL aqueous phase (diluted 28X).

Test	Units	Run1B	Run 2B	Run 3B	Run 4B
Run Time	hours	73	132	108	132
AD Sludge	g VSS	28	28	28	28
Slg Activity	gSCODr/gVSS/d	0.03	0.03	0.03	0.03
F:M	gTCOD/gVSS	0.13	0.20	0.26	0.29
F:M	gSCOD/gVSS	0.12	0.18	0.25	0.27
TCOD in	mg/L	1760	2850	3620	4090
TCOD out	mg/L	1070	1890	2680	3270
TCOD reduction	%	39	34	26	20
SCOD in	mg/L	1710	2520	3560	3720
SCOD out	mg/L	1070	1780	2540	2770
SCOD reduction	%	37	29	29	26
Methane	ml	686	824	849	885
Eff VFA	meq/L	0.35	0.42	0.42	0.45
Gas Yield	LCH ₄ /gTCOD _r	0.50	0.43	0.45	0.54
Gas Yield	LCH ₄ /gSCOD _r	0.54	0.56	0.42	0.47

The biogas production curves for the four treatability tests at 28X dilution are shown in Figure 8. Ideally, for a readily digestible wastewater, the gas production curves for a run series would all follow roughly the same shape, with slight gas production improvements over time as the biomass acclimates to the AP. But, for the AP treatability test, the biogas curves look worse in each successive run, confirming the low COD destruction results, shown in Figure 9.

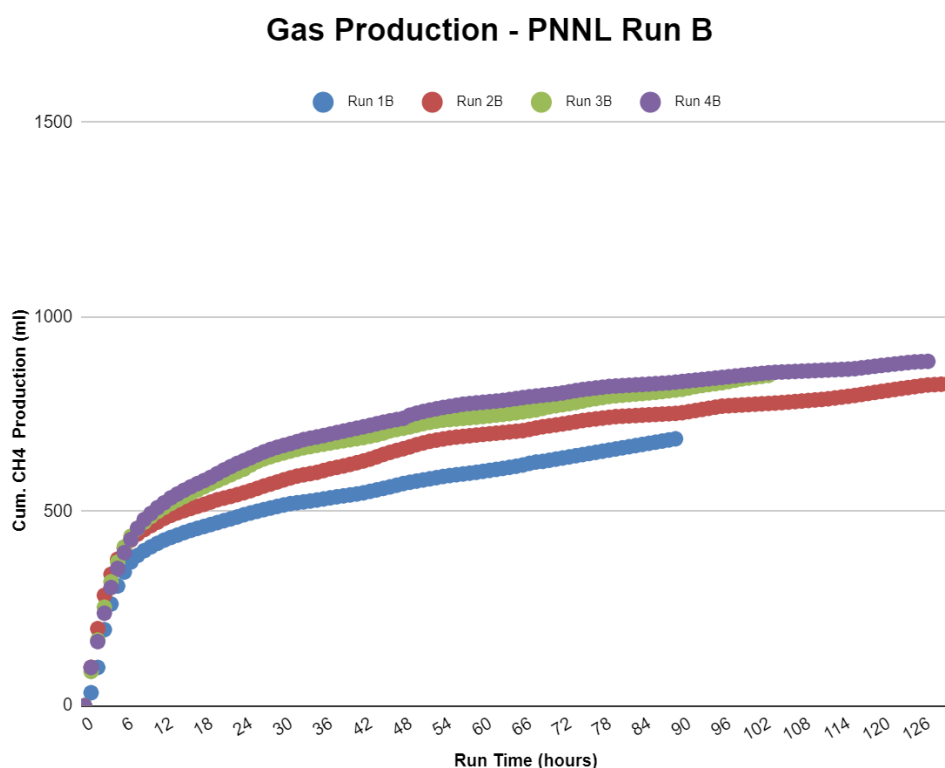


Figure 8. Biogas production curves for AD treatability testing of AP (at 28X dilution).

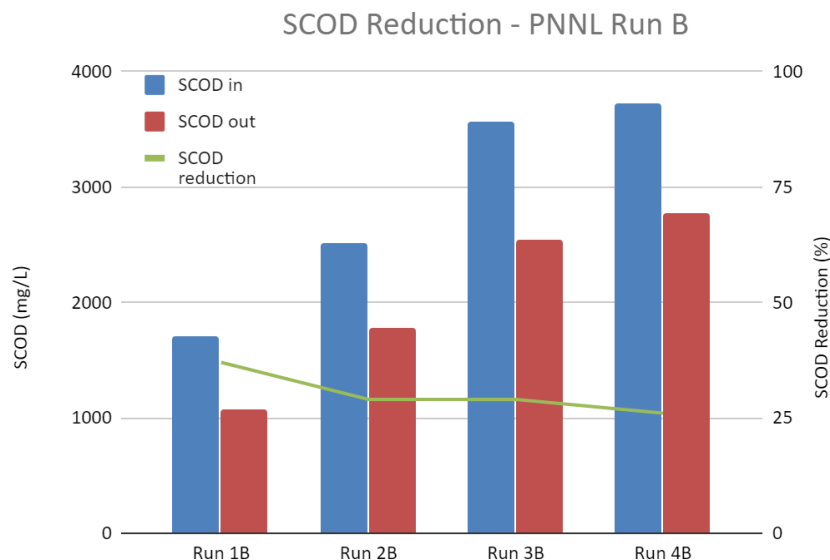


Figure 9. COD reduction for AD treatability testing of AP (at 28X dilution).

Overall, the results from the treatability testing show that at a normal system loading with Veolia AD sludge, there appears to be a compound or compounds in the wastewater that severely inhibit the anaerobic biomass from digesting available COD. At a low digester loading rate in Run B, the inhibition was somewhat mitigated (compared to the first run at a 4X higher AP concentration) to the point that the biomass was still active. However, the overall destruction of COD was still quite low. Some byproducts of the HTL process, such as quaternary amines, may be the cause of the inhibition. Even a few mg/L of some quaternary amines can completely kill an anaerobic digester. Since there is a fairly large amount of BOD in the AP, aerobic digestion could potentially be considered, although there are challenges. Activated sludge growth will be high, and operational costs will likely be high to be able to supply enough air to the system for the amount of oxygen needed.

In FY18, testing of continuous AD of aqueous byproduct from HTL of sewage sludge was completed by Washington State University under subcontract to PNNL. In this testing, clarified decant from dairy manure was used as a nutrient base for the growth of the anaerobic microbial consortia. The initial inoculum was obtained from a local digester in the lab that used feedlot manure as substrate. The AD reactor was fed with the clarified manure decant to establish a stable consortia, and then HTL AP was slowly added into the feed at stepwise increases over many months. At 15% of full strength of HTL AP, a COD reduction of 35% achieved in the AD reactor, operated at hydraulic detention time of 20 days, and at 37°C. This performance was achieved after about 130 days of acclimation. At these conditions, a the methane yield of 95 mL/gVS was achieved. Future work on AD of HTL AP will benefit from adaptation/acclimation of the biological consortium and advanced AD reactor design.

3.4 Biocrude Catalytic Hydrotreating

Biocrude from the GLWA sludge (MHTLS13) was hydrotreated in the fixed-bed bench scale system described previously (Snowden-Swan et al. 2020). The process consists of an initial step whereby the feed is first flowed over a fixed guard bed (CoMo catalyst) to remove the majority of inorganics (through hydrodemetalization and filtering) and then a second packed bed (NiMo catalyst) where most of the deoxygenation and denitrogenation of the biocrude occurs. The reactor is packed with catalyst extrudates to ensure identical pore diffusion limitations will be observed at both lab and commercial scales. Inert

fine particles are co-packed with the catalyst to ensure the catalyst is fully wetted and has ideal plug flow (these will only be issues at the lab scale as the higher superficial velocity at a commercial scale eliminates these issues).

Table 8 gives the reactor conditions and product results from biocrude derived from GLWA sludge (MHTLS13), along with results from the previous runs with biocrude derived from GLWA sludge (Snowden-Swan et al. 2017) and the 2020 SOT and the 2022 goal case models for comparison. Also, results from hydrotreated swine manure-derived biocrude (WW15) from the FY19 SOT (Snowden-Swan et al. 2020) are included as they were not available at the time the FY19 SOT was issued. Feedstock composition and HTL performance for swine manure were presented in the 2019 SOT report and the data is again presented in Appendix B of this report. Reactor throughput rate has a significant impact on economics both for operating and capital cost reasons, and for this reason, work in 2020 targeted increased space velocity. Relative to the 2019 SOT, weight hourly space velocity (WHSV) was increased from 0.65 to 0.72 hr⁻¹ in the guard bed and from 0.39 to 1.03 hr⁻¹ in the main hydrotreating bed, an 11% and 164% improvement for the guard and main beds, respectively. The performance was not sacrificed at the higher throughput rates, as is shown by the essentially equivalent results for hydrogen consumption, product yields, and product oil compositions for the MHTLS13 run compared to previous runs. In addition, the deactivation rate was slow, as shown by the density curve in Figure 10. For these reasons, catalyst life for the 2020 SOT is maintained at 552 hours, consistent with that demonstrated for the 2019 SOT.

Table 8. Wet waste biocrude hydrotreating experimental results and model assumptions.

Component	WW06 (GLWA 50/50 sludge) (HT-62005-60)	WW15 (Swine Manure)	MHTLS13 (GLWA Primary Sludge) HT282/HT283	2020 SOT Model	2022 Projected Model
Temperature, °F (°C)	752 (400)	752 (400)	752 (400)	752 (400)	752 (400)
Pressure, psia	1540	1515	1562	1540	1515
Guard bed catalyst sulfided?	CoMo/alumina Yes	CoMo/alumina Yes	CoMo/alumina Yes	CoMo/alumina Purchased presulfided	CoMo/alumina Purchased presulfided
Main bed catalyst sulfided?	CoMo/alumina Yes	NiMo/alumina Yes	NiMo/alumina Yes	NiMo/alumina Purchased presulfided	CoMo/alumina Purchased presulfided
Guard bed WHSV, wt./hr per wt. catalyst	0.46	0.42	0.72	0.72	1.3
Main bed WHSV, wt./hr per wt. catalyst	0.29	0.42	1.03	1.03	0.75
HTL biocrude feed rate, ml/h	5.6	2.16	130 (main bed)	Commercial scale	Commercial scale
Time-on-stream (catalyst life)	302 hours	133 hours	112 hours	552 hours	2 years
Chemical H ₂ consumption, wt/wt HTL biocrude (wet)	0.046	0.043	0.050	0.046	0.044
Product yields ^(a) , lb/lb dry biocrude (vol/vol wet biocrude)					
Hydrotreated oil	0.82 (0.99)	0.85	0.83	0.82 (0.97)	0.84 (0.97)
Aqueous phase	0.14 (0.13)	0.13	0.16	0.14	0.13 (0.19)
Gas	0.08	0.06	0.04	0.10	0.07
Product oil, wt%					
C	85.6	85.7	84.7	85.3	85.3
H	14.6	12.9	14.3	14.1	14.1
O	1.0	<0.5	0.22	0.6	0.6
N	<0.05	1.60	0.84	0.04	0.04
S	7-10 ppm	<0.03	<0.3	0.0	0.0
Product oil, H:C	2.1	1.8	2.0	2.0	2.0
Aqueous carbon, wt%	0.10	Not measured	Not measured	0.6	0.2
Gas analysis, volume%					
CO ₂ , CO	0	0	3	0	0
CH ₄	51	45	19	39	33
C ₂ +	49	55	78	35	38
NH ₃	Not measured	0	Not measured	23	26
NH ₄ HS	Not measured	0	Not measured	3	3
Total acid number, feed (product)	59 (<0.01)	Not calculated	Not calculated	Not calculated	Not calculated
Viscosity@104°F (40°C), cSt, feed (product)	400 (2.7)	1040 (5.6)	165 (3.7)	Not calculated	Not calculated
Density@104°F (40°C), g/ml, feed (product)	0.98 (0.79)	0.96 (0.84)	0.95 (0.81)	0.98 (0.79)	0.98 (0.79)

(a) Yield after phase separation.

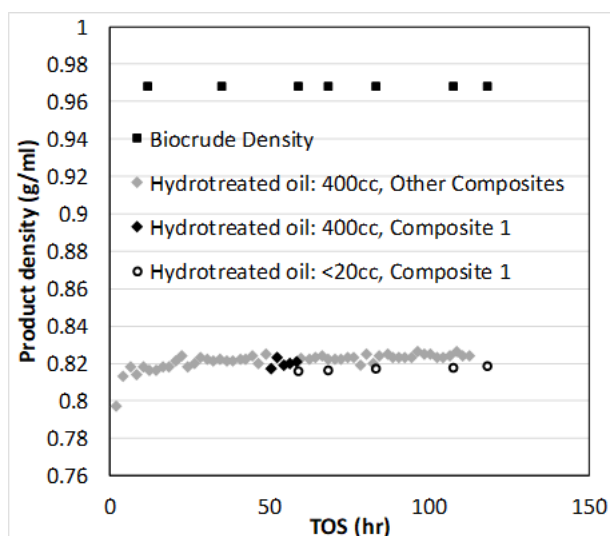


Figure 10. Density of upgraded biocrude from the GLWA sludge generated in PNNL's HTL engineering scale unit (MHTLS13) as a function of time-on-stream (TOS).

Figure 11 shows boiling point curves from simulated distillation (ASTM Method D2887) of the hydrotreated product from the primary sludge-derived biocrude (MHTLS13) and swine manure-derived biocrude (WW15) along with results from the previous GLWA primary/secondary sludge run (WW06), and the modeled product (matched to WW06 data) for comparison.

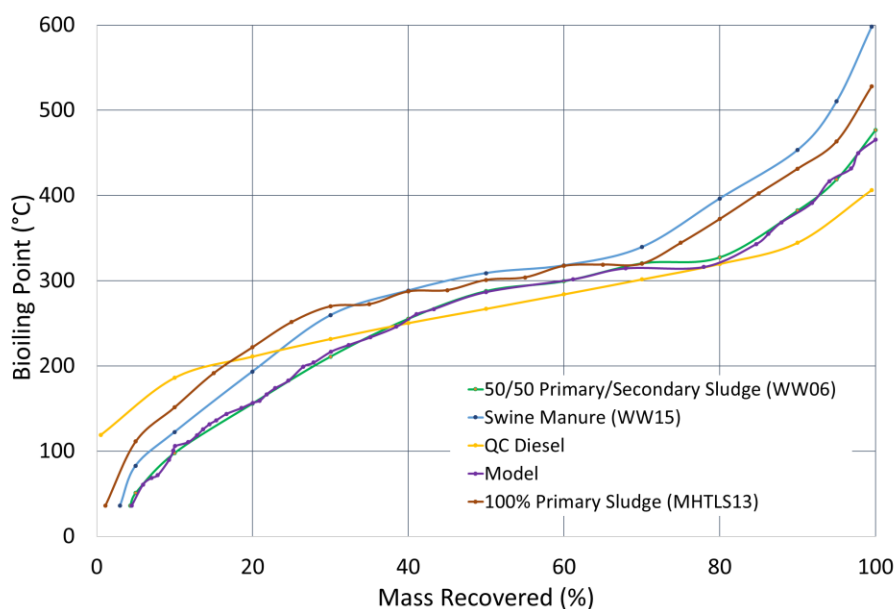


Figure 11. Boiling point distribution (ASTM D2887) for hydrotreated product from sludges, swine manure, and the process model.

4.0 2020 SOT Modeled Costs

Table 9 lists the major economic results for the HTL plant for the 2020 SOT. Costs for the 2018 SOT, 2019 SOT, and 2022 projected (goal) cases are also given for comparison. All costs are in 2016 dollars. The HTL plant scale processes 110 dry ton/day of sludge feed and produces 10,404 gal/day of biocrude. Cases shown include the baseline ammonia stripping treatment for the AP. Appendix B gives the HTL cost breakdown for cases excluding AP ammonia stripping to represent plants that would not need treatment of the AP prior to recycling back to the WRRF. The alternative treatment options assessed in section 3.3 are still in early phases of research and screening and therefore have not been incorporated into the SOT MFSP at this time. The main updates in the HTL costs are the redesign of the sludge heat exchangers (see Section 2.1) and a modest increase in the HTL reactor LHSV (see Section 3.2), which reduced the minimum biocrude selling price (MBSP) for the HTL plant by 24 cents and 1 cent per GGE, respectively. The reduction in MBSP resulting from the new heating and pumping design is due to savings in both capital (20 cents/GGE) and operating costs (4 cents/GGE). Note that the 2018 and 2019 SOT cases were not back casted with the updated heat transfer coefficient (see Section 2.1). In addition to the cost savings, a critical benefit of the new configuration is that it represents a more practical design for scale-up given the high operating pressures, as indicated by feedback from vendors.

Table 9. Economic results for 110 dry ton/day sludge HTL plant (with AP NH₃ stripping).

	2018 and 2019 SOT	2020 SOT	2022 Projected SOT
Capital Costs, \$ million			
Installed costs			
Sludge feedstock dewatering	1.3	1.3	1.3
HTL biocrude production	19.5	16.9	12.3
HTL aqueous phase recycle treatment	2.8	2.8	2.3
Balance of plant	0.6	0.6	0.6
Total installed capital cost	24.2	21.6	16.5
Fixed capital investment	45.7	40.8	31.3
Total capital investment (TCI)	48.1	42.9	32.9
Operating Costs, \$/GGE biocrude (\$ million/yr)			
Variable operating cost			
Avoided sludge disposal cost	0	0	0
Natural gas	0.11 (0.4)	0.07 (0.3)	0.09 (0.4)
Chemicals	0.20 (0.7)	0.20 (0.7)	0.18 (0.7)
Electricity	0.17 (0.6)	0.17 (0.6)	0.11 (0.4)
Fixed costs	0.88 (3.2)	0.83 (3.1)	0.67 (2.7)
Capital depreciation	0.41 (1.5)	0.38 (1.4)	0.25 (1.0)
Average income tax	0.12 (0.5)	0.11 (0.4)	0.08 (0.3)
Average return on investment	1.15 (4.3)	1.02 (3.8)	0.74 (3.0)
MBSP, \$/gal biocrude	3.27	3.00	2.27
MBSP, \$/GGE biocrude	3.04	2.79	2.11

Table 10 lists the primary economic results for the biocrude upgrading plant. The upgrading plant processes 114,729 gal/day of biocrude feed and produces 109,248 gal/day of fuel blendstock (27,888 gal/day naphtha and 81,360 gal/day diesel). The MFSP for the upgrading plant includes \$0.10/GGE (gasoline-gallon equivalent) for transporting the biocrude 100 miles to the upgrading facility (Snowden-

Swan et al. 2017). The main update to the upgrading plant model is increased WHSV for the hydrotreater guard bed and main reactor, as demonstrated in the experimental research (see Section 3.4). This resulted in a reduction of \$0.34/GGE in the conversion cost for the upgrading plant from \$1.74/GGE for the 2019 SOT to \$1.40/GGE for the 2020 SOT. Catalyst life was maintained at 552 hr, the catalyst time-on-stream demonstrated for the 2019 SOT. Note that economic results given in Table 9 and Table 10 are dependent on plant scale, which is set at 110 ton/day sludge feed for the HTL plant and 38 mmgal/yr biocrude feed for the upgrading plant, commensurate with the original design case (Snowden-Swan et al. 2017). The 2022 projected costs differ slightly from the costs presented in the original design case due to updates made in the modeled year and income tax rate (see Appendix D).

Table 10. Economics for biocrude upgrading plant processing ~115,000 gal/day.

	2018 SOT	2019 SOT	2020 SOT	2022 Projected
Capital Costs, \$ million				
Installed costs				
Hydrotreating	46.7	41.9	37.9	31.6
Hydrocracking	6.1	6.1	6.1	6.2
Hydrogen plant	26.3	26.3	26.3	25.6
Steam cycle	1.7	1.7	1.7	1.5
Balance of plant	6.2	6.2	6.2	6.1
Total installed capital cost	87.0	82.2	78.2	71.0
Indirect costs	60.9	57.5	54.7	49.6
Fixed capital investment	162.5	153.4	145.8	132.3
Total capital investment (TCI)	173.7	164.0	155.9	141.5
Operating Costs, \$/GGE (\$ million/yr)				
Biocrude feedstock ^a , including transport	3.37 (127.6)	3.37 (127.6)	3.10 (125.8)	2.32 (89.6)
Natural gas	0.04 (1.4)	0.04 (1.4)	0.04 (1.4)	0.05 (1.7)
Catalyst	2.80 (105.9)	0.84 (31.9)	0.54 (20.5)	0.01 (0.5)
Wastewater disposal	0.002 (0.1)	0.002 (0.1)	0.002 (0.1)	0.002 (0.1)
Electricity and water makeup	0.02 (0.9)	0.02 (0.9)	0.02 (0.9)	0.02 (0.9)
Fixed costs	0.27 (10.2)	0.26 (9.9)	0.25 (9.6)	0.24 (9.1)
Capital depreciation	0.143 (5.4)	0.14 (5.1)	0.13 (4.9)	0.002 (4.4)
Average income Tax	0.05 (1.9)	0.04 (1.6)	0.04 (1.5)	0.04 (1.4)
Average return on investment	0.47 (17.7)	0.40 (15.0)	0.37 (14.0)	0.43 (16.7)
MFSP, \$/GGE fuel blendstock ^a	7.16	5.11	4.50	3.11
MFSP, \$/GGE (conversion cost only)	3.79	1.74	1.40	0.79
MFSP, \$/gal diesel ^a	7.67	5.48	4.82	3.33
MFSP, \$/gal naphtha ^a	7.07	5.05	4.44	3.06

a. Price reflects cost of biocrude production from HTL process for case including ammonia stripping of aqueous phase.

Figure 12 illustrates the annual modeled MFSP from the 2018, 2019, and 2020 SOTs and the projected 2022 goal case for the combined wet waste HTL and biocrude upgrading process pathway. Results for the separate HTL plant are given in Appendix B. The modeled 2020 SOT MFSP is \$4.50/GGE and \$4.08/GGE for the scenarios with and without ammonia removal from the HTL AP, respectively. The overall MFSP has been reduced by \$2.66/GGE since the initial SOT and a reduction of \$1.39/GGE is needed to reach the 2022 goal. Further research to improve HTL and hydrotreating performance is needed to meet the 2022 target. In addition, development of regional waste blending scenarios that can help economies of scale for both the HTL and upgrading plants is needed. Additional detail on the plans for progression to the goal case are given in Section 5. The complete list of processing area costs and key technical parameters and targets for the SOT and projected cases are given in Appendix B. Appendix C gives the life cycle inventory of inputs and outputs for the HTL and upgrading plants that is needed for

the pathway the Supply Chain Sustainability Analysis (Cai et al., 2018, 2020). Carbon and energy efficiencies for the pathway are also presented in Appendix C.

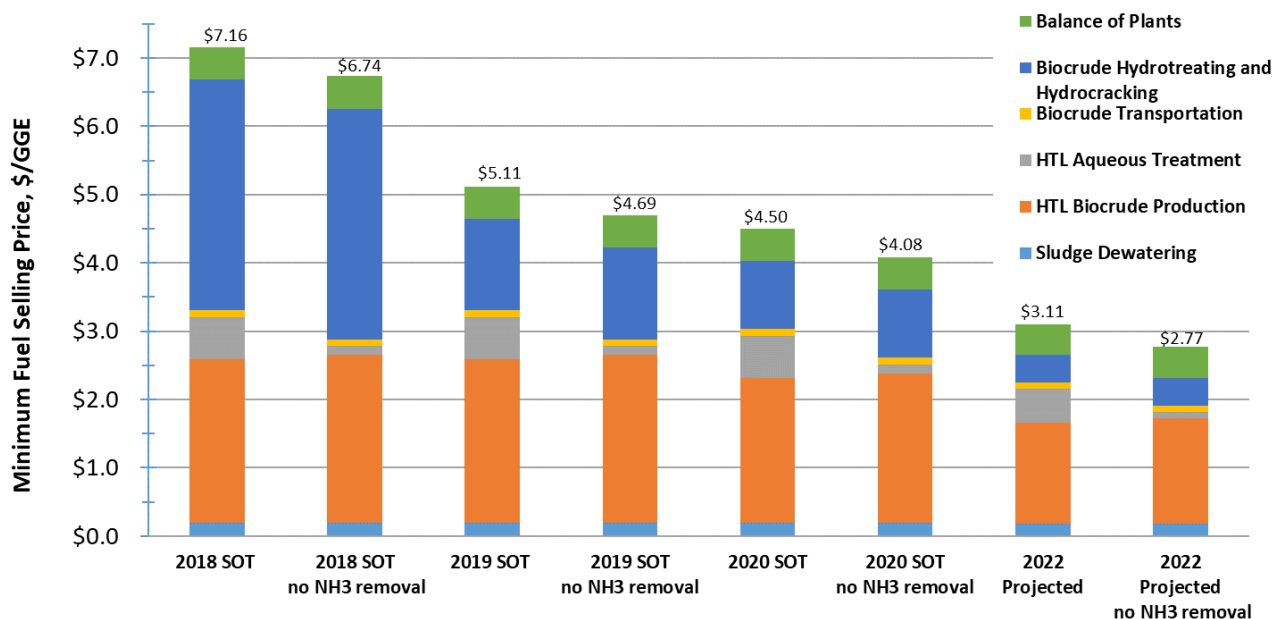


Figure 12. Combined HTL and biocrude upgrading process cost allocations.

In addition to the process improvements reflected in the SOT, this year's feedstock testing has shown that HTL can process dairy manure and three of the four major high-volume wet wastes (wastewater solids, manures, FOG and food waste) have now been characterized and successfully tested in PNNL's continuous systems.

A sensitivity analysis was performed to evaluate the potential impact of the different AP thermochemical treatment options tested (Section 3.3.1) on the MBSP of the wet waste HTL process and compared with the 2020 SOT baseline (\$2.79/GGE biocrude). Note that the analysis for these options is high-level and preliminary for the purpose of providing an initial screening and identification of cost drivers for the options tested. The TEA results are presented in Figure 13. As shown, the ACU method provides a much lower capital cost than CHG and SCREW due to its low operating pressure, which makes it the most cost effective AP treatment option among all three methods investigated in this work. Even though both ACU designs can provide lower MBSP, their projected COD reductions are much lower than CHG and SCREW because of their limited capability of treating S and N rich components and relatively lower C conversion. SCREW has the highest capital cost because of the addition need for a PSA unit for H₂ separation and recovery and its relatively high operating pressure. CHG has the highest variable cost primarily because of the relatively expensive catalyst and the complexity of catalyst regeneration. Both ACU options have very low variable cost due to the use of less expensive catalyst. SCREW has a relatively high variable cost because of its H₂ consumption. ACU-STP has a lower MBSP than ACU-TSA because of the sale of (NH₄)₂SO₄ as by-product. Overall, AP treatments offering high COD reduction may add about \$0.57/GGE (SCREW) to \$0.74/GGE (CHG) to the MBSP. The ACU option offers mid-level COD reduction and may add about \$0.08/GGE to \$0.14/GGE to the MBSP.

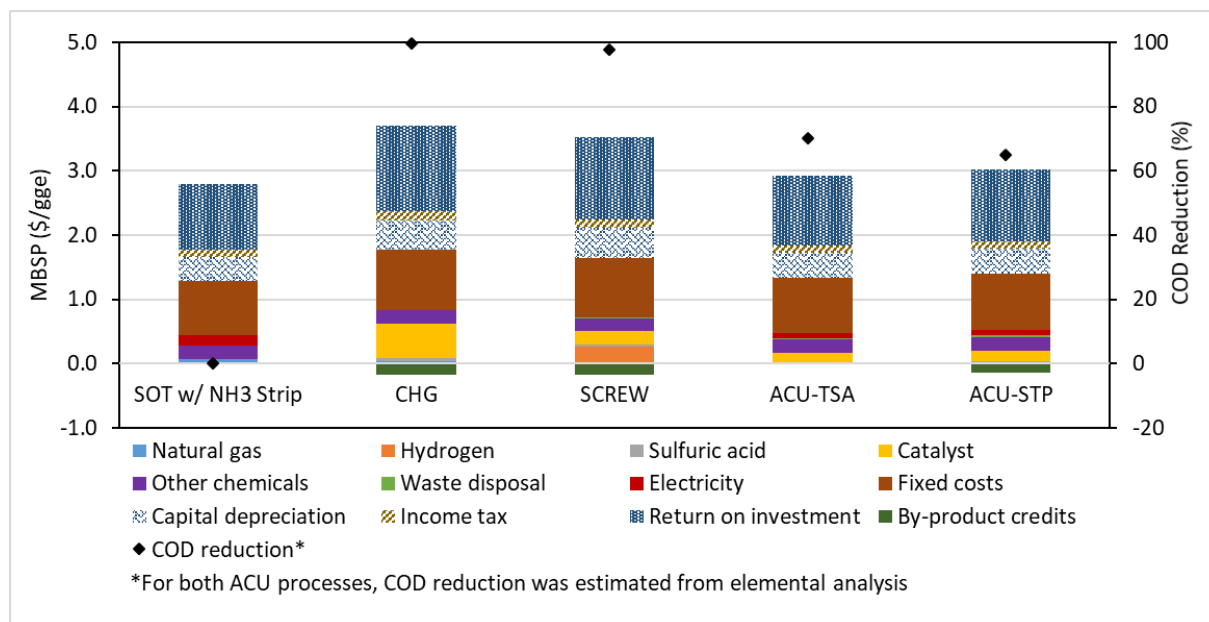


Figure 13. MBSP and cost allocation of wet waste HTL process with different AP treatment.

5.0 Future Work and Progression to 2022 Projected Case

Significant progress was made in FY20 to reduce the modeled cost of the SOT by \$0.61 per GGE of fuel blendstock, demonstrate HTL and biocrude upgrading of several large impact wet waste feedstock, and provide a new heating and pumping configuration that has improved scalability at industrial scale. In addition, initial testing of several thermochemical methods and anaerobic digestion treatment of HTL AP was carried out. High-level TEA sensitivity analysis indicates that the thermochemical methods tested could add 57-74 cents per GGE and 8-14 cents per GGE to the HTL plant MBSP for high and medium COD removal options, respectively. Initial anaerobic digestion testing indicates that there are compounds in the AP that inhibit the AD organisms, but separate testing by Washington State University using an adapted culture show more promising results. Further testing is needed to verify the feasibility of AD for AP treatment. Future research necessary to advance the pathway toward the 2022 goal case will focus on the following areas.

HTL: Increasing feed solids content naturally reduces capital and operating costs associated with processing carrier feed water and can also improve yields through better oil/water phase separation. The current solids content set for the SOT is 20% for wastewater sludge, which has been demonstrated with the GLWA sludge. The target solids content was set at 25% for the design case as this level of solids should be possible with specialized slurry pump technologies designed for wastewater treatment and other industries (Berglin et al. 2012). Fuel production cost savings resulting from increasing feed solids from 20% to 25% solids is estimated at approximately 25 cents/GGE. An increase in biocrude yield from the SOT of 44% to the target of 48% is estimated to reduce MFSP by an additional 25 cents/GGE. Attempts to run 25% solids sludge in our existing system configurations have proven challenging due to the limitations of equipment at the bench/engineering scale. However, system modifications/adjustments may be possible to enable higher solids pumping and will be investigated. Testing of the new pumping and heating configuration is needed to validate the new design. Also, enhancements to the heat exchangers to improve line velocities and/or turbulence with tube corrugations or core inserts could lead to reduced areas and further reduction in capital costs. Further investigation is needed to test the feasibility of using these technologies with wet waste slurries.

Biocrude Catalytic Upgrading: Further advancements in biocrude hydrotreating performance are critical to reduce modeled MFSP and drive the SOT toward the 2022 target. With the progress made in FY20 to increase reactor WHSV, future research will focus on demonstrating increased catalyst TOS to further reduce catalyst consumption and cost for the modeled plant. A 1000-hour biocrude hydrotreating run is planned in FY21 to provide a basis for estimating catalyst performance and life at commercial scale. A catalyst lifetime of 1 year for the guard bed and hydrotreater bed is expected to reduce the upgrading cost by 52 cents per GGE to \$0.88/GGE. If this can be achieved, the total contribution of catalyst cost toward MFSP will be minimized to about 4 cents/GGE and capital cost contributions will dominate at that point.

Aqueous Phase Treatment: Nitrogen and COD removal from the AP may be necessary to mitigate negative impact to a WRRF's treatment train. Testing of thermochemical technologies for removal of COD show some promise. Initial TEA indicate that these methods could add approximately 57-74 cents per GGE and 8-14 cents per GGE for high and medium COD removal methods, respectively. Removal of COD prior to ammonia stripping enables recovery of an ammonia by-product from the AP, reduces natural gas consumption used in the thermal oxidation of the stripped ammonia, and results in a cleaner water stream for recycle to the headworks or reuse elsewhere. AP treatment technologies for removal of nitrogen and COD will continue to be investigated to develop the most environmentally sustainable and economical methods. Close collaboration with wastewater industry and universities is an important aspect of future work.

Transition to Waste Blend Scenarios:

Plant scale is a key economic driver for the pathway, as shown in the design case (Snowden-Swan et al. 2017). Follow-on analysis to the waste blending study performed by Seiple et al. (2019) identified concentrated regions, or “hot spots,” of wet waste generation in the U.S. Shown in Figure 14, preliminary results indicate that 82% of the total wet waste resource, including wastewater solids, manure, food waste, and FOG, could be collected at sites over a 1000 dry ton/day scale at a transportation cost of \$50/tonne. Where the maximum waste collection radius for the analysis is 155 mi (250 km), the weight averaged waste collection radius for the depicted hot spots is 106 mi (170 km). These results suggest that regional collection at much larger and more economically feasible scales than the 110 ton/day baseline used in the design case may be plausible for a significant portion of the wet waste feedstock resource. A preliminary estimate of the impact on MFSP of regional collection and processing is included in the next paragraph. These preliminary results will be updated in FY21 to complete a full geospatial and siting analysis for regional waste blending scenarios and integrated into the SOT analysis.

45 Service Areas ≥ 1000 dry Mg/d account for 51.3 dry Tg/y (82% of total inventory)
Scenario: Compete; USD 50 max; 250-km max

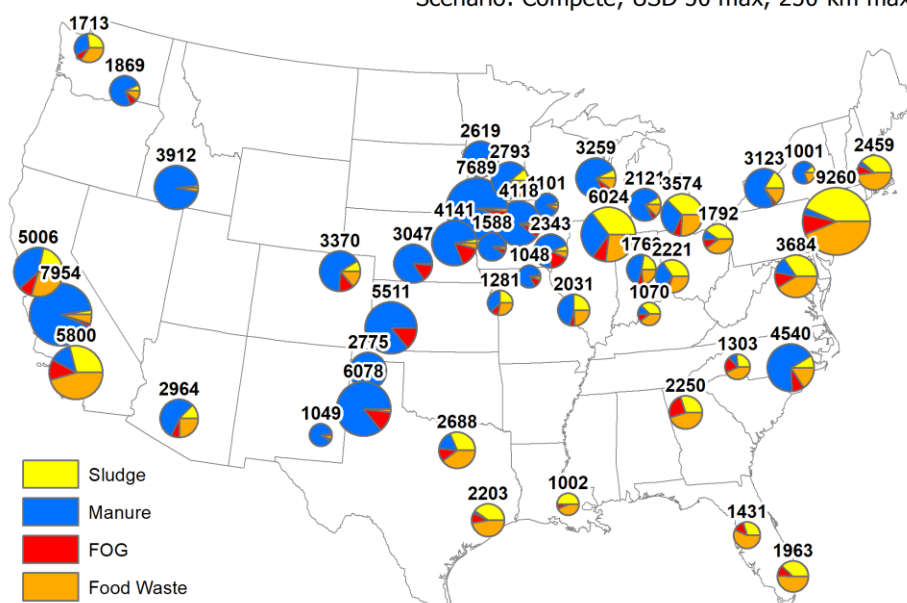


Figure 14. Waste blending sites identified in preliminary geospatial analysis for waste-to-fuel. Values given are potential dry tons/day of waste that could be collected at a \$50/tonne transportation cost, with the total from 45 service areas representing 82% of the total wet waste resource.

Impact of Research Improvements:

Figure 15 shows the estimated aggregated impact on the SOT MFSP of the planned areas of improvement discussed above, including a HTL feed solids content of 25%, biocrude yield of 48%, 43% decrease in HTL heat exchanger capital cost (estimated from the use of core insert technology), and biocrude hydrotreating catalyst lifetime of 1 year (“HT Cat Life 1 yr” in figure). As shown, these improvements reduce the MFSP to \$3.24/GGE for the baseline HTL scale (110 dry ton/day, “110 TPD” in figure). Also shown is the affect of increasing the HTL plant scale to 1000 dry ton/day (“1000 TPD” in figure) by collecting and transporting regional wet wastes. This case assumes waste feedstock is transported at 20% total solids content and at the \$50/dry tonne (\$45/dry ton) cost estimated from the initial hot spot analysis (Figure 14). Note that this scenario does not include a waste tipping fee or avoided sludge treatment cost. It also assumes that the biocrude upgrader is co-located with the HTL plant (and WRRF) and therefore does not include the cost for transporting biocrude from the HTL plant to the upgrading plant that is otherwise included in the SOT. At the larger HTL plant scale and including the impact of research

improvements, the estimated MFSP is \$2.55/GGE. With these improvements, the MFSP is within range of BETO's 2030 cost goal of \$2.5/GGE (DOE 2020). The whiskers shown on the 1000 TPD bars represent a range of \$40-60/dry ton waste transportation cost. Cost varies by about +/-9 cents per GGE from the base case of \$50/dry ton transportation cost. Approximately 65% and 91% of the wet waste resource can be collected at the \$40/ton and \$60/ton costs, respectively (not shown).

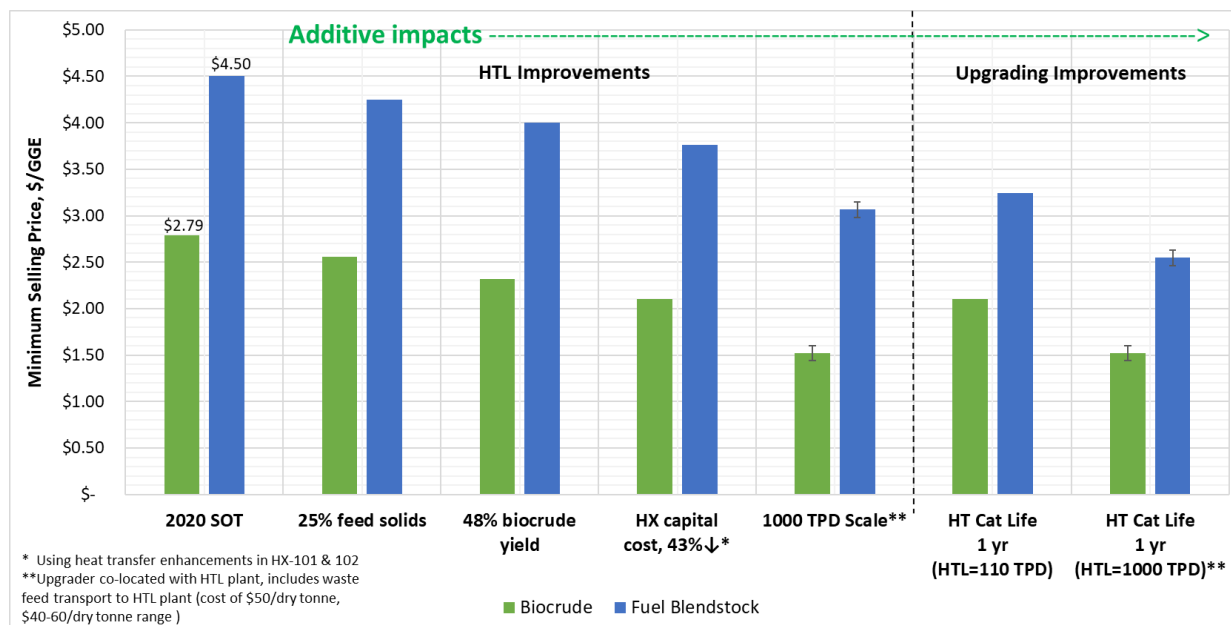


Figure 15. Additive impacts of potential performance improvements and plant scale on production cost.

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Appendix A– Comprehensive List of Waste Feedstocks Testing Data

Table A.1. List of feedstocks tested to date in support of the HTL SOT and pathway development.

	WW0 6 50/50 Sludge GLW A (Dry)	WW06 50/50 Sludge GLWA (DAF)	WW09 50/50 Sludge CCCS (Dry)	WW09 50/50 Sludge CCCS (DAF)	WW10 CCCS Sludge/FOG (80/20) (Dry)	WW10 CCCS Sludge/FOG (80/20) (DAF)	WW15 Swine Manur e (Dry)	WW15 Swine Manure (DAF)	MHTLS 13 Primary Sludge GLWA (Dry)	MHTLS 13 Primary Sludge GLWA (DAF)	WW14 Biosolids (Dry)	WW14 Biosolids (DAF)	WW17 CCCS Sludge (No Lime) (Dry)	WW17 CCCS Sludge (No Lime) (DAF)	WW19A ^b Cow Manure (Dry)	WW19A ^b Cow Manure (DAF)	WW19B ^b Cow Manure (Dry)	WW19B ^b Cow Manure (DAF)	2020 SOT and 2022 Models (Dry)	2020 SOT and 2022 Models (DAF)
C	41.1	52.0	43.3	51.1	49.5	58.5	47.6	53.7	42.3	52.5	34.3	47.6	44.8	52.7	43.9	50.6	43.1	50.3	46.8	52.1
H	5.8	7.3	6.3	7.4	6.9	8.2	6.3	7.1	6.2	7.7	4.7	6.5	6.1	7.1	5.7	6.6	5.7	6.7	6.5	7.2
O	26.1	33.0	30.2	35.6	24.6	29.0	30.9	34.8	26.9	33.4	26.4	36.1	27.4	32.3	34.0	39.4	33.8	39.4	29.7	33.1
N	5.0	6.3	4.5	5.3	3.1	3.7	3.4	3.8	4.2	5.2	5.3	7.4	6.1	7.1	2.6	3.0	2.6	3.0	5.7	6.3
S	1.0	1.3	0.6	0.5	0.5	0.6	0.6	0.6	1.0	1.2	1.6	2.3	0.7	0.8	0.5	0.6	0.5	0.6	1.2	1.3
Ash	26.1		16.7 ^(a)		17.2		12.5		25.6		32.6		17.1		15.9		16.7		15.0	
P	1.9		2.5		2.2		1.4		1.9		2.0		Not ready		0.7		0.7		1.9	
Carb	16.7	22.8	37.2	46.1	45.2	55.2		50.1	26.7	34.9	17.5	30.5	30.8	38.2	60.3	70.0	NM	NM		Not modeled
Fat	22.6	30.8	6.5	8.0	15.0	18.3		24.7	20.6	27.0	11.6	19.3	14.2	17.6	10.1	11.8	NM	NM		Not modeled
Protein	34.1	46.4	36.7	45.4	21.6	26.4		25.2	29.0	38.0	29.6	51.0	37.6	46.7	15.7	18.2	NM	NM		Not modeled
FAME	11.9	16.2	13.7	17.0	26.5	32.3		16.6	15.4	20.2	5.5	13.0	9.5	11.5	5.8	6.7	NM	NM		Not modeled
Ash	26.6		19.2		18.1				23.7		41.4		17.4		13.8		NM			

(a) (b) CCCSD currently treats their wastewater with lime to help incineration process. Ash content without lime is estimated at 14%.

(c) WW19-A and WW19-B were run without and with inorganic homogeneous catalyst, respectively.

DAF = dry, ash-free

Table A.2. HTL performance data for waste feedstocks tested to date.

Operating Conditions and Results	50/50 Sludge (GLWA) WW06	50/50 Sludge (CCCSD) WW09	80/20 Sludge/FOG (CCCSD) WW10	Swine Manure WW15	50/50 Sludge (GLWA) MHTLS13	AD Biosolids WW14	50/50 Sludge-no lime (CCCSD) WW17 SS-1	Cow Manure WW19A	Cow Manure WW19B	2020 SOT Model	2022 Projected Model
Temperature, °F (°C)	656 (347)	655 (346)	653 (345)	653 (345)	662 (350)	649 (343)	653 (345)	646 (341)	639 (337)	656 (347)	656 (347)
Pressure, psia (MPa)	2979 (20.5)	2845 (19.6)	2895 (20.0)	2840 (19.6)	2940 (20.3)	2840 (19.6)	2840 (19.6)	2940 (20.3)	3000 (20.7)	2979 (20.5)	2979 (20.5)
Feed solids, wt%				24.9%							
Ash included	20%	17.4%	16.8%	21.8%	15.3%	16.7%	14.6%	15%	15%	20%	25%
Ash-free basis	15%	14.5%	13.9%		11.4%	11.3%	12.1%	12.3%	12.1%	17%	21%
Liquid hourly space velocity, vol./h per vol. reactor	3.6 ^(d)	3.6 ^(d)	3.7 ^(d)	3.5 ^(d)	4.0	3.5	3.5	3.5	3.5	3.6	6
Equivalent residence time, min.	17	17	16	17	15	17	17	17	17	17	10
Product yields ^(a) (dry, ash-free sludge), wt%											
Oil (biocrude)	44%	37%	50%	49%	41%	31%	41%	32%	39%	44%	48%
Aqueous	31%	34%	26%	21%	33%	35%	36%	42%	30%	29%	25%
Gas	16%	23%	19%	25%	19%	14%	19%	22%	29%	16%	16%
Solids	9%	5%	5%	5%	7%	20%	4%	3%	3%	12%	11%
Carbon yields											
Oil (biocrude)	58%	52%	60%	59%	51%	42%	55%	49%	53%	65%	72%
Aqueous	24%	29%	26%	22%	30%	31%	30%	29%	25%	21%	18%
Gas	8%	12%	9%	13%	9%	8%	10%	13%	15%	10%	10%
Solids	10%	6%	5%	7%	9%	20%	5%	10%	7%	5%	1%
HTL dry biocrude analysis, wt%											
C	78.5%	77.6%	77.9%	71.3%	78.5%	76.3%	75.9%	76.5%	76.5%	78.3%	78.3%
H	10.7%	9.9%	10.9%	10.0%	10.8%	9.4%	9.8%	9.2%	9.0%	10.8%	10.8%
O	4.7%	6.8%	7.2%	13.4%	5.8%	6.3%	8.5%	9.6%	9.8%	4.8%	4.8%
N	4.8%	5.2%	3.6%	4.3%	4.2%	5.1%	5.0%	3.9%	4.1%	4.9%	4.9%
S	1.2%	0.4%	0.3%	0.6%	0.6%	1.8%	0.6%	0.4%	0.3%	1.2%	1.2%
P	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%		

Operating Conditions and Results	50/50 Sludge (GLWA) WW06	50/50 Sludge (CCCSO) WW09	80/20 Sludge/FOG (CCCSO) WW10	Swine Manure WW15	50/50 Sludge (GLWA) MHTLS13	AD Biosolids WW14	50/50 Sludge-no lime (CCCSO) WW17 SS-1	Cow Manure WW19A	Cow Manure WW19B	2020 SOT Model	2022 Projected Model
Ash	0.06%	0.07%	0.05%	0.28%	0.1%	1.0%	0.2%	0.4%	0.2%	Not modeled ^(b) 0.0%	Not modeled ^(b) 0.0%
HTL dry biocrude H:C ratio	1.6	1.5	1.7	1.7	1.7	1.5	1.5	1.4	1.4	1.6	1.6
HTL biocrude dry higher heating value, Btu/lb (MJ/kg)	16,900 (39.5) ^(c)	16,400 (38.0) ^(c)	16,900 (39.3) ^(c)	15,200 (35.3) ^(c)	17,000 (39.6)	(37.2) ^(c)	15,970 (37.1)	15,700 (36.5)	15,600 (36.4)	17,100 (39.7)	17,100 (39.7)
HTL biocrude moisture, wt%	4.4%	4.0%	3.2%	5.0%	3.5%	7.3%	7.0%	4.5%	4.8%	4.0%	4.0%
HTL biocrude wet density @ 25°C (g/ml)	0.98	0.99	0.95	0.96	0.95 ^(g)	1.01 ^(f)	Not ready	1.03 ^(g)	1.04 ^(g)	0.98	0.98
Aqueous phase chemical oxygen demand (mg/L)	61,300	75,200	77,800	95,400	53,800	53,000	66,100	61,800	59,800	62,700	61,100

(a) Recovered after separations.

(b) Phosphorus partitioning is not directly modeled in Aspen because of the small quantity, most of which reports to the solid phase.

(c) Calculated using Boie's equation (Boie 1953).

(d) The experimental system includes a continuous stirred-tank reactor (CSTR) followed by a PFR. The CSTR helps prevent overheating of the feed.

(e) Runs A and B are without and with homogeneous catalyst in feed.

(f) Measured at 40°C

(g) Measured at 60°C

(i) WW runs were in the bench-scale system and MHTLS-13 was run in the engineering scale system.

Table A.3. Hydrotreating performance data for waste feedstocks tested to date.

Component	WW06 (GLWA sludge) (HT-62005- 60)	WW09 (CCCSD sludge) HT-62006-86	WW10 (CCCSD sludge/FOG) HT-62006-86	WW15 (Swine Manure)	MHTLS13 GLWA HT282/HT283	2020 SOT Model	2022 Projected Model
Temperature, °F (°C)	752 (400)	752 (400)	752 (400)	752 (400)	752 (400)	752 (400)	752 (400)
Pressure, psia	1540	1535	1535	1515	1562	1540	1515
Guard bed catalyst sulfided?	CoMo/alumin a Yes	CoMo/alumina Yes		CoMo/alumina Yes	CoMo/alumina Yes	CoMo/alumina Yes	CoMo/alumin a Purchased presulfided
Main bed catalyst sulfided?	CoMo/alumin a Yes	NiMo/alumina Yes		NiMo/alumina Yes	NiMo/alumina Yes	NiMo/alumina Yes	CoMo/alumin a Purchased presulfided
Guard bed WHSV, wt./hr per wt. catalyst	0.46	0.68	0.65	0.42	0.72	0.72	1.3
Main bed WHSV, wt./hr per wt. catalyst	0.29	0.39	0.38	0.42	1.03	1.03	0.75
HTL biocrude feed rate, ml/h	5.6	7.3		2.16		130 (main bed)	Commercial scale
Time-on-stream (catalyst life)	302 hours	552 hours		133 hours		112 hours	2 years
Chemical H ₂ consumption, wt/wt HTL biocrude (wet)	0.046	0.058	0.051	0.043	0.050	0.046	0.044
Product yields ^(a) , lb/lb dry biocrude (vol/vol wet biocrude)							
Hydrotreated oil	0.82 (0.99)	0.84	0.82	0.85	0.83	0.82 (0.97)	0.84 (0.97)
Aqueous phase	0.14 (0.13)	0.13	0.17	0.13	0.16	0.14	0.13 (0.19)
Gas	0.08	0.08	0.06	0.06	0.04	0.10	0.07
Product oil, wt%							
C	85.6	85.0	84.8	85.7	84.7	85.3	85.3
H	14.6	14.3	15.1	12.9	14.3	14.1	14.1

O	1.0	<0.5	<0.5	<0.5	0.22	0.6	0.6
N	<0.05	0.73	0.07	1.60	0.84	0.04	0.04
S	7-10 ppm	0.03	0.14	<0.03	<0.3	0.0	0.0
Aqueous carbon, wt%	0.10	Not measured	Not measured	Not measured	Not measured	0.6	0.2
Gas analysis, volume%							
CO ₂ , CO	0	5	4	0	3	0	0
CH ₄	51	9	33	45	19	39	33
C ₂ +	49	86	63	55	78	35	38
NH ₃	Not measured	Not measured	Not measured	0	Not measured	23	26
NH ₄ HS	Not measured	Not measured	Not measured	0	Not measured	3	3
Total acid number, feed (product)	59 (<0.01)	Not measured	Not measured	Not calculated	Not calculated	Not calculated	Not calculated
Viscosity@40°C, cSt, feed (product)	400 (2.7)	Not measured	166 (3.7)	1040 (5.6)	165 (3.7)	Not calculated	Not calculated
Density@40°C, g/ml, feed (product)	0.98 (0.79)	0.99 (0.81)	0.95 (0.79)	0.96 (0.84)	0.95 (0.81)	0.98 (0.79)	0.98 (0.79)

(a) Yield after phase separation.

Appendix B – Technical Tables and Separate HTL Plant Economics

Table B.1. Processing area cost contributions and key technical parameters for the SOT and projected cases for the combined wet waste HTL and upgrading pathway.

Processing Area Cost Contributions & Key Technical Parameters	Metric	2018 SOT with NH3 removal	2018 SOT no NH3 removal	2019 SOT with NH3 removal	2019 SOT no NH3 removal	2020 SOT with NH3 removal	2020 SOT no NH3 removal	2022 Projected with NH3 removal	2022 Projected no NH3 removal
Fuel selling price	\$/GGE	\$7.16	\$6.74	\$5.11	\$4.69	\$4.50	\$4.08	\$3.11	\$2.77
Conversion Contribution	\$/GGE	\$7.06	\$6.64	\$5.01	\$4.59	\$4.4	\$3.98	\$3.01	\$2.67
Performance Goal	\$/GGE							\$3	\$3
Production Diesel	mm gallons/yr	27	27	27	27	27	27	28	28
Production Naphtha	mm gallons/yr	9	9	9	9	9	9	9	9
Diesel Yield (AFDW sludge basis)	gal/US ton sludge	79	79	79	79	79	79	89	89
Naphtha Yield (AFDW sludge basis)	gal/us ton sludge	27	27	27	27	27	27	30	30
Natural Gas Usage (AFDW sludge basis)	scf/US ton sludge	4,951	3,898	4,951	3,898	3,717	2,664	4,914	3,861
Feedstock									
Total Cost Contribution	\$/GGE fuel	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0
Feedstock Cost (dry sludge basis)	\$/US ton sludge	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0
Sludge Dewatering									
Total Cost Contribution	\$/GGE fuel	\$0.20	\$0.20	\$0.20	\$0.20	\$0.20	\$0.20	\$0.18	\$0.18
Capital Cost Contribution	\$/GGE fuel	\$0.10	\$0.10	\$0.10	\$0.10	\$0.10	\$0.10	\$0.09	\$0.09
Operating Cost Contribution	\$/GGE fuel	\$0.10	\$0.10	\$0.10	\$0.10	\$0.10	\$0.10	\$0.09	\$0.09
Sludge HTL									
Total Cost Contribution	\$/GGE fuel	\$2.40	\$2.45	\$2.40	\$2.45	\$2.12	\$2.18	\$1.49	\$1.55
Capital Cost Contribution	\$/GGE fuel	\$1.46	\$1.46	\$1.46	\$1.46	\$1.27	\$1.27	\$0.83	\$0.83
Operating Cost Contribution	\$/GGE fuel	\$0.94	\$0.99	\$0.94	\$0.99	\$0.84	\$0.91	\$0.66	\$0.72
HTL Biocrude Yield (dry)	lb/lb sludge	0.44	0.44	0.44	0.44	0.44	0.44	0.48	0.48
Liquid Hourly Space Velocity (LHSV)	vol/h/vol	3.6	3.6	3.6	3.6	4.0	4.0	6.0	6.0

Processing Area Cost Contributions & Key Technical Parameters	Metric	2018 SOT with NH3 removal	2018 SOT no NH3 removal	2019 SOT with NH3 removal	2019 SOT no NH3 removal	2020 SOT with NH3 removal	2020 SOT no NH3 removal	2022 Projected with NH3 removal	2022 Projected no NH3 removal
Preheaters Capital Cost (installed)	\$MM	12	12	12	12	9	9	6	6
HTL Water Recycle Treatment									
Total Cost Contribution	\$/GGE fuel	\$0.61	\$0.13	\$0.61	\$0.13	\$0.62	\$0.13	\$0.49	\$0.09
Capital Cost Contribution	\$/GGE fuel	\$0.21	\$0.00	\$0.21	\$0.00	\$0.21	\$0.00	\$0.16	\$0.00
Operating Cost Contribution	\$/GGE fuel	\$0.40	\$0.13	\$0.40	\$0.13	\$0.41	\$0.13	\$0.33	\$0.09
Balance of Plant - HTL									
Total Cost Contribution	\$/GGE fuel	\$0.06	\$0.07	\$0.06	\$0.07	\$0.07	\$0.07	\$0.07	\$0.07
Capital Cost Contribution	\$/GGE fuel	\$0.04	\$0.04	\$0.04	\$0.04	\$0.05	\$0.05	\$0.04	\$0.04
Operating Cost Contribution	\$/GGE fuel	\$0.02	\$0.02	\$0.02	\$0.02	\$0.02	\$0.02	\$0.03	\$0.03
Biocrude Transport	\$/gge fuel	\$0.10	\$0.10	\$0.10	\$0.10	\$0.10	\$0.10	\$0.10	\$0.10
Biocrude Upgrading to Finished Fuels									
Total Cost Contribution	\$/GGE fuel	\$3.38	\$3.38	\$1.34	\$1.34	\$1.00	\$1.00	\$0.40	\$0.40
Capital Cost Contribution	\$/GGE fuel	\$0.40	\$0.40	\$0.34	\$0.34	\$0.30	\$0.30	\$0.25	\$0.25
Operating Cost Contribution	\$/GGE fuel	\$2.97	\$2.97	\$1.01	\$1.01	\$0.70	\$0.70	\$0.15	\$0.15
Hydrotreating Mass Yield on dry Biocrude	lb/lb biocrude	0.82	0.82	0.82	0.82	0.82	0.82	0.84	0.84
Guard Bed Weight Hourly Space Velocity (WHSV)	wt/h/wt	0.46	0.46	0.67	0.67	0.72	0.72	1.30	1.30
Guard Bed Catalyst Lifetime	years	0.03	0.03	0.06	0.06	0.06	0.06	1	1
Hydrotreater Weight Hourly Space Velocity (WHSV)	wt/h/wt	0.29	0.29	0.39	0.39	1.02	1.02	0.75	0.75
Hydrotreater Catalyst Lifetime	years	0.03	0.03	0.06	0.06	0.06	0.06	2	2
Balance of Plant - Upgrading									
Total Cost Contribution	\$/GGE fuel	\$0.42	\$0.42	\$0.40	\$0.40	\$0.40	\$0.40	\$0.39	\$0.39
Capital Cost Contribution	\$/GGE fuel	\$0.26	\$0.26	\$0.24	\$0.24	\$0.24	\$0.24	\$0.22	\$0.22
Operating Cost Contribution	\$/GGE fuel	\$0.16	\$0.16	\$0.16	\$0.16	\$0.16	\$0.16	\$0.17	\$0.17

Processing Area Cost Contributions & Key Technical Parameters	Metric	2018 SOT with NH3 removal	2018 SOT no NH3 removal	2019 SOT with NH3 removal	2019 SOT no NH3 removal	2020 SOT with NH3 removal	2020 SOT no NH3 removal	2022 Projected with NH3 removal	2022 Projected no NH3 removal
Models: Case References		Sludge HTL 2018 SOT final.bkp;Sludge HTL Biocrude Upgrading 2018 SOT.bkp				Sludge HTL 2020 SOT final-base-split-hotoil_v2.bkp; Sludge HTL Biocrude Upgrading 2020 SOT.bkp		Sludge HTL Goal Case 8-17-2017 FINAL 110 TPD 1.bkp;WW-06 Bio-Oil Upgrading 10X 110 TPD.bkp	

Table B.2. Processing area cost contributions and key technical parameters for the SOT and projected cases for the separate wet waste HTL plant.

Processing Area Cost Contributions & Key Technical Parameters	Metric	2018 SOT with NH3 removal	2018 SOT no NH3 removal	2019 SOT with NH3 removal	2019 SOT no NH3 removal	2020 SOT with NH3 removal	2020 SOT no NH3 removal	2022 Projected with NH3 removal	2022 Projected no NH3 removal
HTL Biocrude selling price	\$/GGE	\$3.04	\$2.65	\$3.04	\$2.65	\$2.79	\$2.40	\$2.11	\$1.79
Conversion Contribution, Biocrude	\$/GGE	\$3.04	\$2.65	\$3.04	\$2.65	\$2.79	\$2.40	\$2.11	\$1.79
Production Biocrude	mm GGE/yr	4	4	4	4	4	4	4	4
Production Biocrude	mm gallons/yr	3	3	3	3	3	3	4	4
Biocrude Yield (AFDW sludge basis)	gal/US ton sludge	111	111	111	111	111	111	123	123
Natural Gas Usage (AFDW sludge basis)	scf/US ton sludge	3,760	2,707	3,760	2,707	2,527	1,474	3,303	2,250
Feedstock									
Total Cost Contribution	\$/GGE fuel	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0
Feedstock Cost (AFDW sludge basis)	\$/US ton sludge	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0
Sludge Dewatering									
Total Cost Contribution	\$/GGE biocrude	\$0.18	\$0.18	\$0.18	\$0.18	\$0.18	\$0.18	\$0.17	\$0.17
Capital Cost Contribution	\$/GGE biocrude	\$0.09	\$0.09	\$0.09	\$0.09	\$0.09	\$0.09	\$0.08	\$0.08
Operating Cost Contribution	\$/GGE biocrude	\$0.09	\$0.09	\$0.09	\$0.09	\$0.09	\$0.09	\$0.08	\$0.08
Sludge HTL									
Total Cost Contribution	\$/GGE biocrude	\$2.23	\$2.28	\$2.23	\$2.28	\$1.97	\$2.03	\$1.41	\$1.47
Capital Cost Contribution	\$/GGE biocrude	\$1.36	\$1.36	\$1.36	\$1.36	\$1.18	\$1.18	\$0.79	\$0.79
Operating Cost Contribution	\$/GGE biocrude	\$0.87	\$0.92	\$0.87	\$0.92	\$0.78	\$0.84	\$0.62	\$0.68

Processing Area Cost Contributions & Key Technical Parameters	Metric	2018 SOT with NH3 removal	2018 SOT no NH3 removal	2019 SOT with NH3 removal	2019 SOT no NH3 removal	2020 SOT with NH3 removal	2020 SOT no NH3 removal	2022 Projected with NH3 removal	2022 Projected no NH3 removal
HTL Biocrude Yield (dry)	lb /lb sludge	0.44	0.44	0.44	0.44	0.44	0.44	0.48	0.48
Liquid Hourly Space Velocity (LHSV)	vol/h/vol	3.6	3.6	3.6	3.6	4.0	4.0	6.0	6.0
Preheaters Capital Cost (installed)	\$MM	12	12	12	12	9	9	6	6
HTL Water Recycle Treatment									
Total Cost Contribution	\$/GGE biocrude	\$0.57	\$0.12	\$0.57	\$0.12	\$0.58	\$0.12	\$0.46	\$0.08
Capital Cost Contribution	\$/gge biocrude	\$0.19	\$0.00	\$0.19	\$0.00	\$0.20	\$0.00	\$0.15	\$0.00
Operating Cost Contribution	\$/GGE biocrude	\$0.37	\$0.12	\$0.37	\$0.12	\$0.38	\$0.12	\$0.32	\$0.08
Balance of Plant									
Total Cost Contribution	\$/GGE biocrude	\$0.06	\$0.06	\$0.06	\$0.06	\$0.06	\$0.06	\$0.06	\$0.07
Capital Cost Contribution	\$/GGE biocrude	\$0.04	\$0.04	\$0.04	\$0.04	\$0.04	\$0.04	\$0.04	\$0.04
Operating Cost Contribution	\$/GGE biocrude	\$0.02	\$0.02	\$0.02	\$0.02	\$0.02	\$0.02	\$0.02	\$0.03
Models: Case References		Sludge HTL 2018 SOT final 2016\$.bkp				Sludge HTL 2020 SOT final-base-split-hotoil_v2.bkp		Sludge HTL Goal Case 8-17-2017 FINAL 110 TPD 1.bkp	

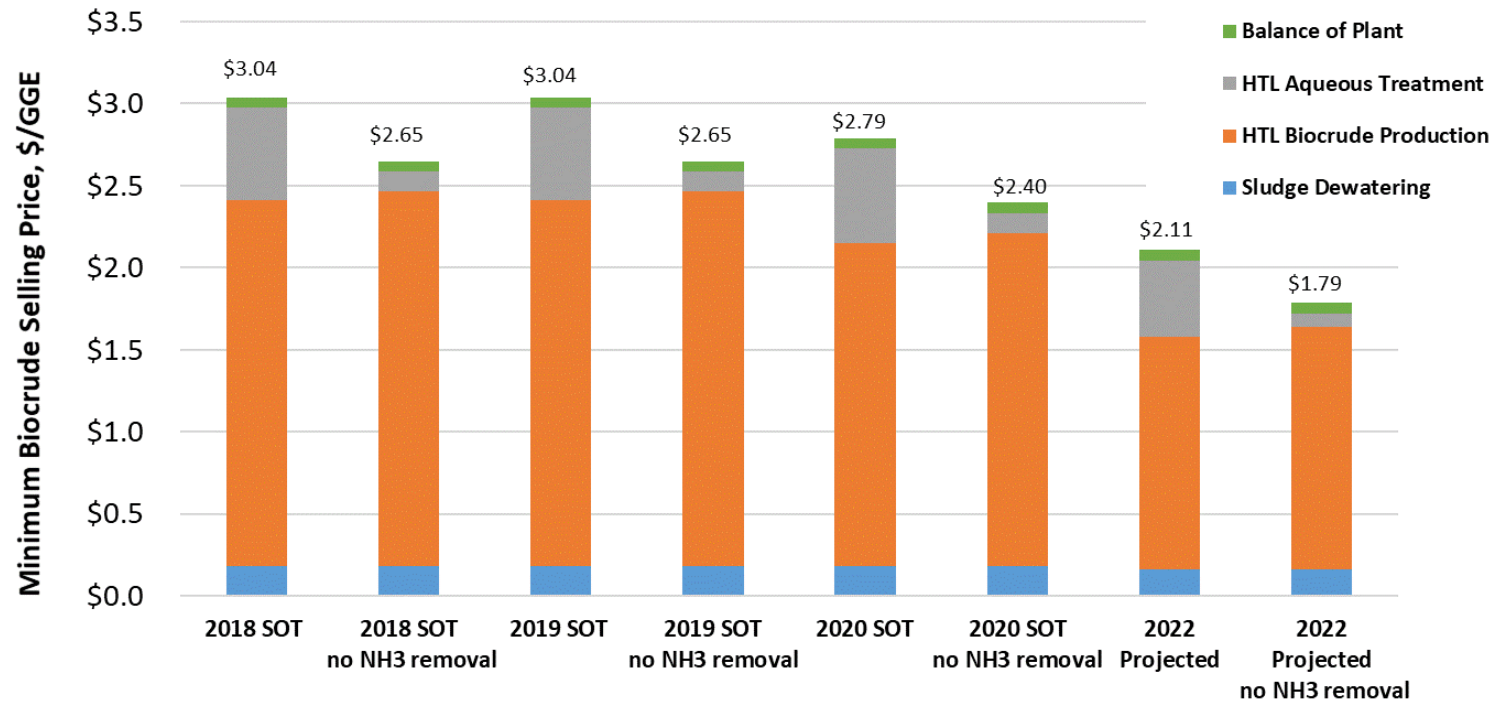


Figure B.1. Hydrothermal liquefaction biocrude cost allocations.

Appendix C – Life Cycle Inventory for Supply Chain Sustainability Analysis

Table C.1 and Table C.2 list the life cycle inventory for the hydrothermal liquefaction (HTL) and upgrading plants, respectively, that are provided to Argonne National Laboratory for Supply Chain Sustainability Analysis.

Table C.1. Hydrothermal liquefaction plant parameters for greenhouse gas and water analysis.

HTL Plant	2018/2019 SOT with NH ₃ Removal	2018/ 2019 SOT without NH ₃ Removal	2020 SOT with NH ₃ Removal	2020 SOT without NH ₃ Removal	2022 Projected with NH ₃ Removal	2022 Projected without NH ₃ Removal
Sludge Properties						
Solids content, %	20	20	20	20	25	25
Ash content (dry basis), %	15.02	15.02	15.02	15.02	15.02	15.02
Biocrude Properties						
Moisture content, %	4	4	4	4	4	4
Density, lb/gal	8.15	8.15	8.15	8.15	8.15	8.15
Lower heating value, Btu/gal	124,993	124,993	124,955	124,955	124,990	124,990
Inputs						
Sludge, lb/hr (dry basis)	9,167	9,167	9,167	9,167	9,167	9,167
Natural gas, lb/hr	625	450	420	245	549	374
Electricity, kW (HTL process)	297	264	328	295	181	148
Electricity, kW (at WRRF for chemical oxygen demand)	849	849	849	849	637	637
Dewatering polymer, lb/hr	24	24	24	24	24	24
Quicklime (CaO), lb/hr	994	0	994	0	994	0
Cooling water makeup, lb/hr	190	190	190	190	210	210
Outputs						
Biocrude, lb/hr	3,533	3,533	3,533	3,533	3,896	3,896
Aqueous phase, lb/hr	34,694	34,694	34,694	34,694	26,023	26,023
Wet solids, ^(a) lb/hr	5,681	5,681	5,681	5,681	5,522	5,522
Solids from HTL aqueous treatment	2,091	0	2,091	0	2,091	0
Carbon Efficiency						
Biocrude C / Feed C	65.4%	65.4%	65.3%	65.3%	72.1%	72.1%
Biocrude C / (Feed + NG) C	59.2%	60.9%	60.7%	62.5%	65.5%	67.5%
Energy Efficiency (LHV)^(b)	60.5%	63.2%	63.5%	66.5%	68.8%	72.0%
Energy Efficiency (LHV)^(c)	62.5%	65.4%	65.7%	68.9%	70.6%	73.9%
(a) 59% and 60% moisture for SOT and projected case, respectively.						
(b) Including extra electricity at WRRF for chemical oxygen demand (and including biomass energy)						
(c) Excluding extra electricity at WRRF for chemical oxygen demand (and including biomass energy)						

SOT	=	state of technology
WRRF	=	wastewater treatment and water resource recovery facility
NG	=	natural gas

Table C.2. Upgrading plant parameters for greenhouse gas and water analysis.

Upgrading Plant	2018 SOT	2019 SOT	2020 SOT	2022 Projected
Fuel Product Properties				
Diesel density, lb/gal	6.66	6.66	6.66	6.66
Diesel lower heating value, Btu/gal	124,394	124,394	124,423	124,410
Naphtha density, lb/gal	6.13	6.13	6.13	6.12
Naphtha lower heating value, Btu/gal	114,650	114,650	114,652	114,478
Inputs				
Biocrude, lb/hr	38,961	38,961	38,961	38,961
Natural gas, lb/hr	2,182	2,182	2,182	2,678
Electricity, kW	1,673	1,673	1,673	1,637
Cooling tower chemical, lb/hr	0.4	0.4	0.4	0.4
Boiler chemical, lb/hr	0.3	0.3	0.3	0.3
Hydrotreating catalyst, lb/hr	811	317	184	3.0
Hydrocracking catalyst, lb/hr	0.3	0.3	0.3	0.3
Hydrogen plant catalyst, lb/hr	0.4	0.4	0.4	0.4
Cooling water makeup, lb/hr	25,069	25,069	25,050	23,485
Boiler feedwater makeup, lb/hr	11,022	11,022	11,022	10,479
Outputs				
Diesel, lb/hr	22,577	22,577	22,583	23,206
Naphtha, lb/hr	7,124	7,124	7,119	7,140
Wastewater, lb/hr	22,773	22,773	22,460	21,503
Carbon Efficiency				
Fuel C / Biocrude C	87.0%	87.0%	87.0%	88.9%
Fuel C / (Biocrude + NG) C	82.5%	82.5%	82.5%	83.2%
Energy Efficiency (LHV)	85.5%	85.5%	85.5%	85.9%

Appendix D – Cost Factors and Financial Assumptions

Table D.1. Cost factors for direct and indirect project costs.

Direct Costs	
Item	% of Total Installed Cost (TIC)
Buildings	4.0%
Site development	10.0%
Additional piping	4.5%
Total Direct Costs (TDC)	18.5%
Indirect Costs	
Item	% of TDC
Prorated expenses	10%
Home office & construction fees	20%
Field expenses	10%
Project contingency	10%
Startup and permits	10%
Total Indirect Costs	60%
Working Capital	5% of FCI
Land	HTL: 6 acres @ \$15,000/acre Upgrading: 6% of Total Purchased Equipment Cost

Table D.2. Financial assumptions for the economic analysis.

Assumption Description	Assumed Value
Internal rate of return (IRR)	10%
Plant financing debt/equity	60% / 40% of total capital investment (TCI)
Plant life	30 years
Income tax rate	21%
Interest rate for debt financing	8.0% annually
Term for debt financing	10 years
Working capital cost	5.0% of fixed capital investment (excluding land)
Depreciation schedule	7-years MACRS ^(a) schedule
Construction period	3 years (8% 1 st yr, 60% 2 nd yr, 32% 3 rd yr)
Plant salvage value	No value
Start-up time	6 months
Revenue and costs during start-up	Revenue = 50% of normal Variable costs = 75% of normal Fixed costs = 100% of normal
On-stream factor	90% (7,920 operating hours per year)
(a) Modified accelerated cost recovery system	

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