2.2.2.302

Enabling Hydrothermal Liquefaction for Fuels and Sewage Sludge Disposal

March 13, 2023

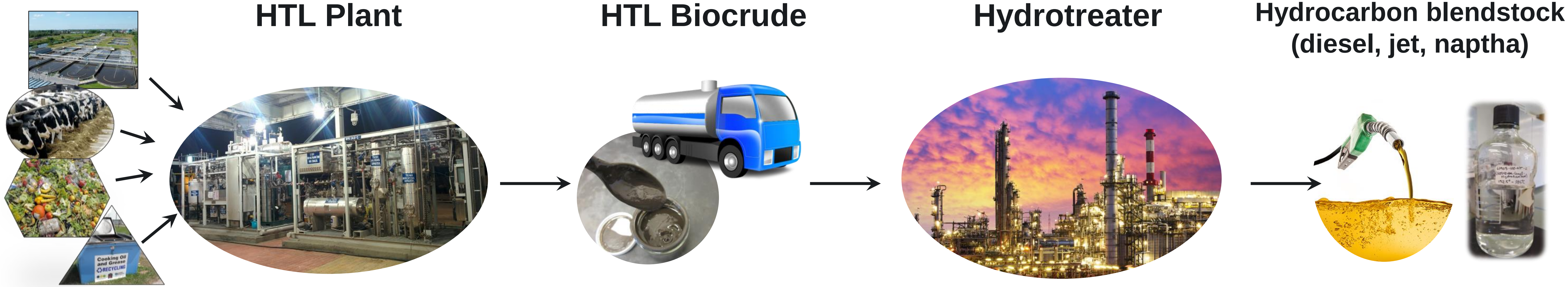
Organic Waste Conversion Technology Area

Mike Thorson

Pacific Northwest National Laboratory

Process Overview for Hydrothermal Liquefaction (HTL): **Transforming Wet Wastes to Liquid Fuels**

HTL Plant | **HTL Biocrude** | **Hydrotreater** | **Hydrocarbon blendstock (diesel, jet, naptha)**



HTL Conditions
330-350°C / 2900 psig / 10-30 min.
"Accelerating what the Earth does"

Stable biocrude
60% C-yield
Thermally stable

Hydrotreater conditions
400°C / 1500 psig
"Standard refinery unit-op"

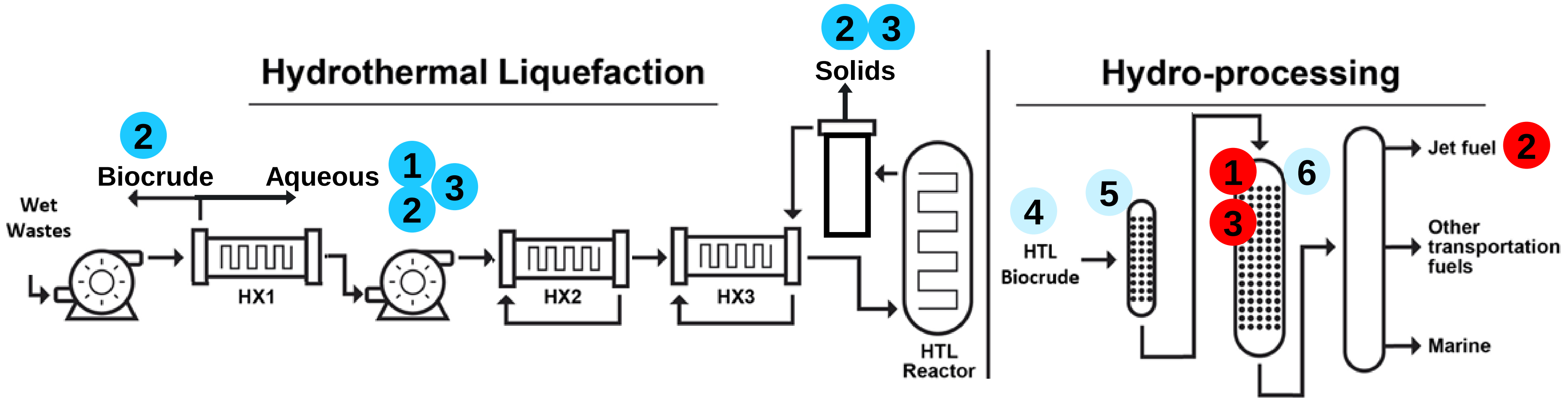
Fuel Blendstocks
(95%+ C-yield)
Predominantly high cetane diesel (70%)

- Conceptually simple (i.e., heated pipe), continuous process.
- High carbon yields to liquid hydrocarbons [~78% greenhouse gas (GHG) reduction].
- Tolerates dirty, wet feedstocks.

Benefit #1: **Potential for ~6 billion gallon/year of fuel in the U.S.**

Benefit #2: **Alternative disposal processes expensive (~$4/gal fuel produced).**

Project Overview: Addressing Barriers to Deployment



Key:
- This project
- Scale-up project
- HDN project

1. **Aqueous treatment:** Enable sustainable recycle.
2. **Per- and polyfluoroalkyl substances (PFAS):** Life cycle throughout HTL and upgrading.
3. **Nutrient recovery:** Recover phosphorus and nitrogen as fertilizer.
   - **Solids:** Ensure solids can be land applied.
4. **Low-grade feedstocks:** Expand feedstocks for regional wet waste processing (larger facilities). ✓

- **Improved HX:** Sustainable, cost-effective heat recovery.
- **Reactor Plugging:** Correlation to predict plugging.
- **Reliable Operation:** >500-hr time-on-stream w/o plug.
- **Scale-up testing:** Testing at scale.
- **Blow down system life:** Engineering robustness.

All hydroprocessing pathways:

5. **Catalyst life:** Achieve >1yr performance. ✓

- **Efficient guard bed:** Prevent plugs: ebullated/slurry reactor.

Sustainable aviation fuel (SAF):
- **Nitrogen content in fuel:**
  - Reduce the nitrogen in the fuel to <10 parts per million.
  - Understand the nitrogen-sulfur interactions on fuel thermal stability.
- Advance HTL towards SAF approval process

Diesel:
- **Blend level limits:** Isomerization for cold temperature properties.
- **Fuel Stability:** Understand and address fuel oxidation.

Big Picture: HTL is attractive for sludge disposal, but key barriers remain for deployment.
Research Focus: Targeted research de-risking HTL to convert wet wastes to fuels.

1 – Approach: Key to HTL Deployment is Addressing Uncertainty and Technical Challenges

Partnerships:

Commercialization Entities

ALOVIAM, PyroEco (Transforming waste to bioenergy), GLWA, Circlia Nordic, Genifuel, metrovancouver, Brown and Caldwell

Feedstock Providers

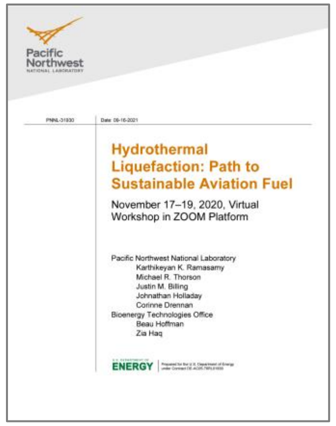
Central Contra Costa Sanitary District, Columbia Crest, Purdue University, GLWA, LambWeston, Joint Base Lewis-McChord, Darigold, Washington State University, WM Waste Management, Ice Harbor Brewing Company, metrovancouver

Refinery / Catalyst Partners

Kern Energy (We Fuel the Future), Topsoe

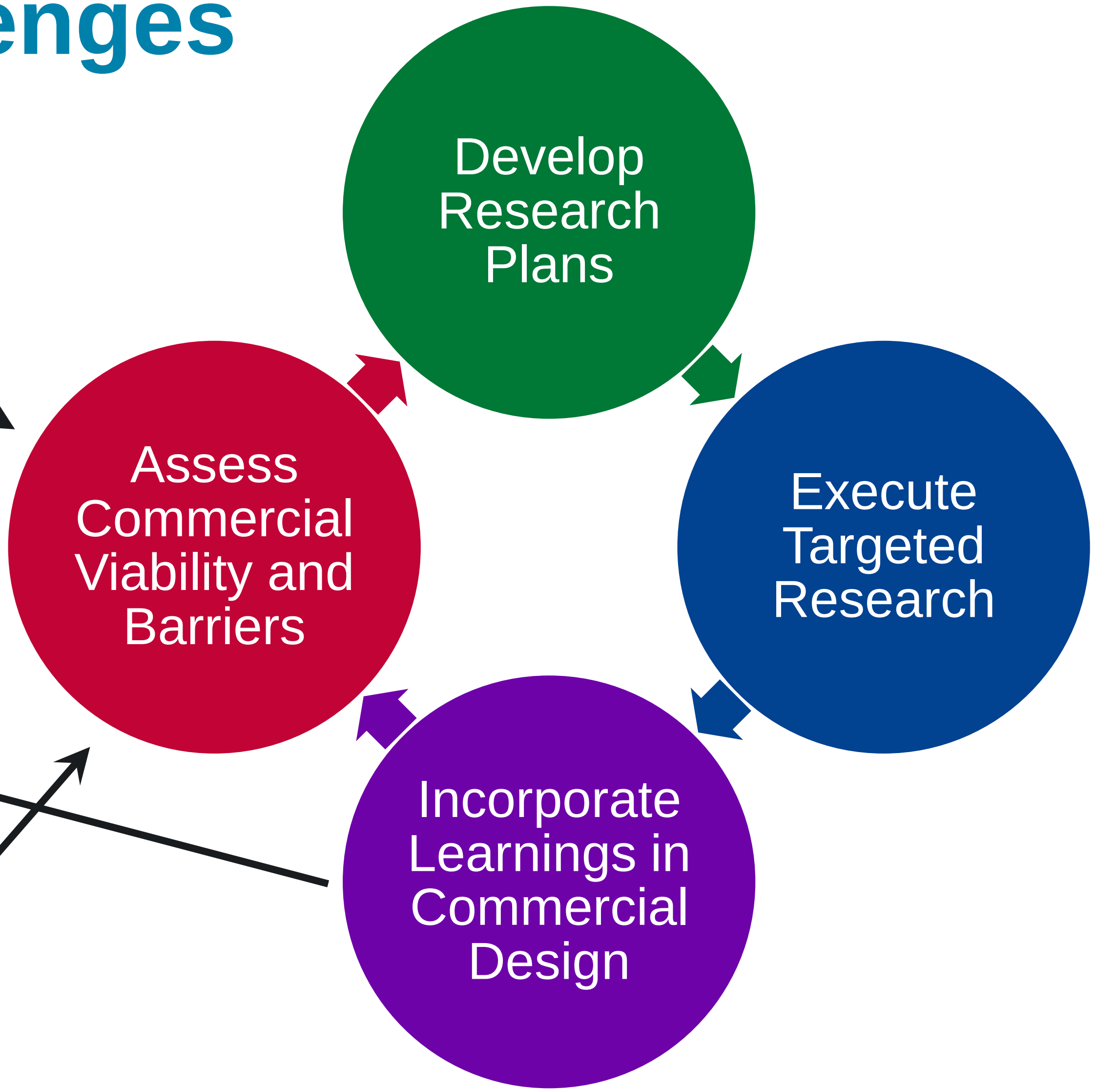
International Collaborators

HyFlexFuel, Water Environment Federation (the water quality people), IEA Bioenergy (Technology Collaboration Programme), Aarhus University, WPI



Monitor commercialization efforts

Steeper Energy, Arbios Biotech, Licella



Continual feedback loop key to address real-world barriers with implementable solutions and implement learnings with commercialization entities.

Pacific Northwest NATIONAL LABORATORY

1 – Approach: Develop SMART Research Plans

Develop Research Plans

	Task	Risk / Opportunity	Objective	
FY21, FY22	Biocrude Upgrading	Jet fuel applicability	Achieve alpha and beta testing of SAF product	✓
		Stable process	>2000 hours of stable hydrotreating performance with on-spec fuel.	✓
	Feedstock R&D	Impact of solids content	Improve HTL yield >2% by increasing HTL solids content (20 to 25wt%).	✓
		Regional impacts	Test regional feedstock HTL blend to increase the design HTL plant size 9X.	✓
FY23+	Nutrient Recovery	Phosphorous recovery	Demonstrate extraction of 75% of phosphorous from residual solids.	✓
	Tracking Forever Chemicals	Effective treatment	Verify at least 50% cumulative destruction of five PFAS compounds.	On track
		Impact of entire process	Track PFAS destruction through HTL, hydroprocessing, mild oxidation, and struvite extraction.	On track
	Aqueous Treatment	Real-world application	Assess aqueous product by waste recovery resource facility (WRRF) to ensure recyclability.	On track
		Effective treatment	Demonstrate >50% carbon and COD reduction.	✓
		Cost-effective	Demonstrate a cost-advantaged mild oxidation process (vs CHG).	On track
		Process stability	>2000 hours continuous operation while meeting partner WRRF specifications.	On track
	Diversity, Inclusion	Effective team	All task leads complete Diversity, Inclusion, and Belonging training.	✓
		Impact students	Hire at least one student from an underrepresented group in STEM	✓
		Impact next generation	Reach one classroom/year through outreach.	✓

Focused on addressing risk/challenges with defined objectives and milestones.

1 – Approach: **Execute Targeted Research Aligned to Commercial Embodiment**

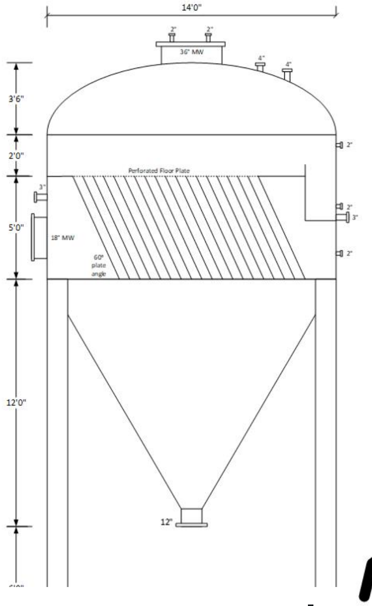
Critical success metrics:

- Cost-effective **HTL aqueous stream disposal.**
- Quantitate **destruction of forever chemicals.**
- Effective **residual solids treatment for disposal.**

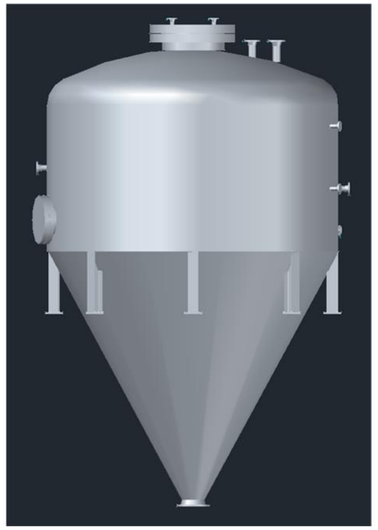
Execute Targeted Research → **Incorporate Learnings in Commercial Design**

Addressing FY21 peer review feedback:
Don't trade one waste problem for another, more challenging one.

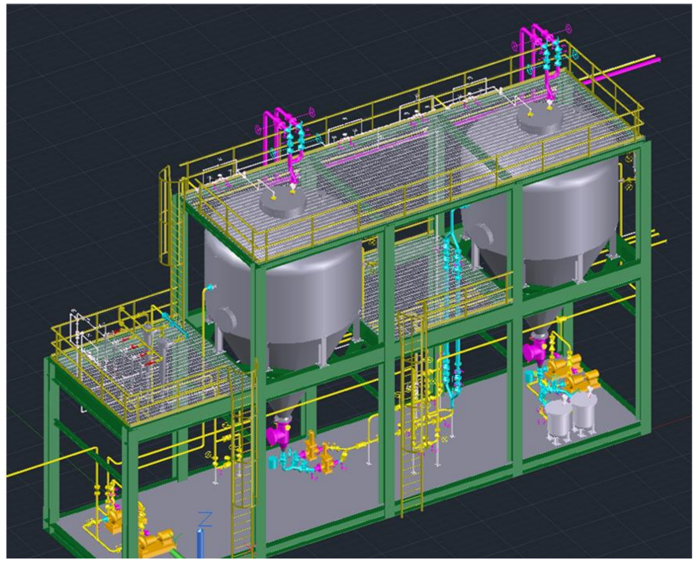
Bench Experiments



Conceptual Scale-up

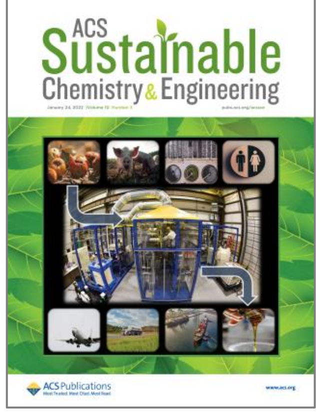


Equipment Design



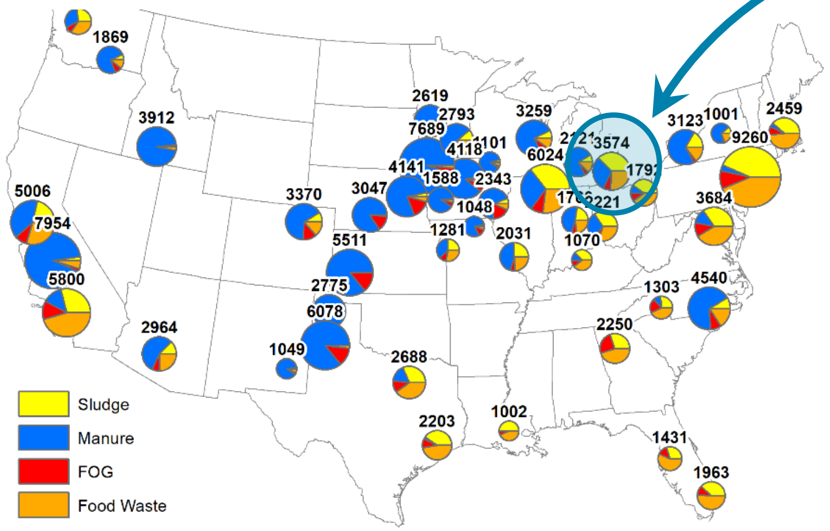
Process Integration

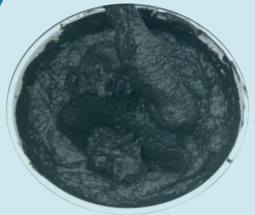
2 – Progress and Outcomes:
Regional Wet-Waste Blends = Excellent Feedstocks



Objectives:

- Assess using regional wet-waste blends to support 9x increase in HTL plant size (2021 milestone).
- Assess impact of increasing feedstock solids content up to 25%.

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Sludge (50%) | Food (40%) | FOG (10%)

→  → 

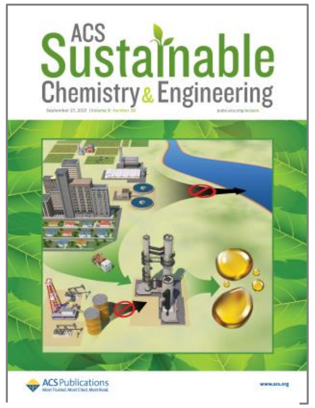
Product | Biocrude

Key outcomes:

1. Resource-informed, real-world regional blends have high yields and outstanding quality.[1,2]
2. Increasing solids content from 15 to 25 wt% increased biocrude yield (46% vs 44%).[1]
3. Funded competitive project with Great Lakes Water Authority assessing HTL for their site.

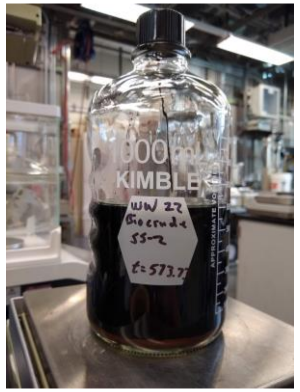
[1]Cronin, D., et al. (2021). Comparative Study on the Continuous Flow Hydrothermal Liquefaction of Various Wet-Waste Feedstock Types. *ACS Sustainable Chemistry & Engineering*.
[2]Snowden-Swan, et al. (2021). Wet Waste Hydrothermal Liquefaction and Biocrude Upgrading to Hydrocarbon Fuels: 2020 State of Technology (No. PNNL-30982). PNNL, Richland, WA.

2 – Progress and Outcomes:
Demonstrate Stable, Scalable Hydrotreating



Assessed extended time-on-stream hydrotreating of biocrudes from mixed fuels.

Biocrude

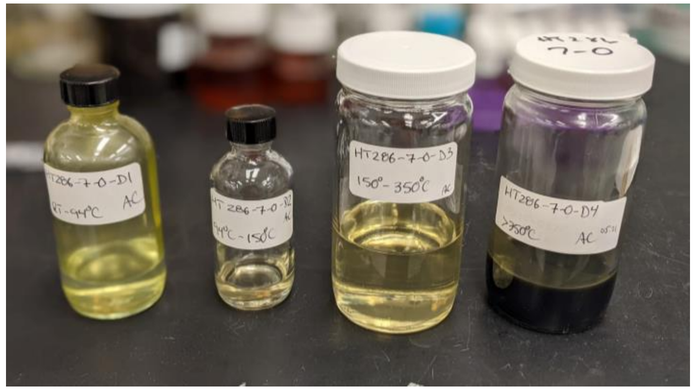


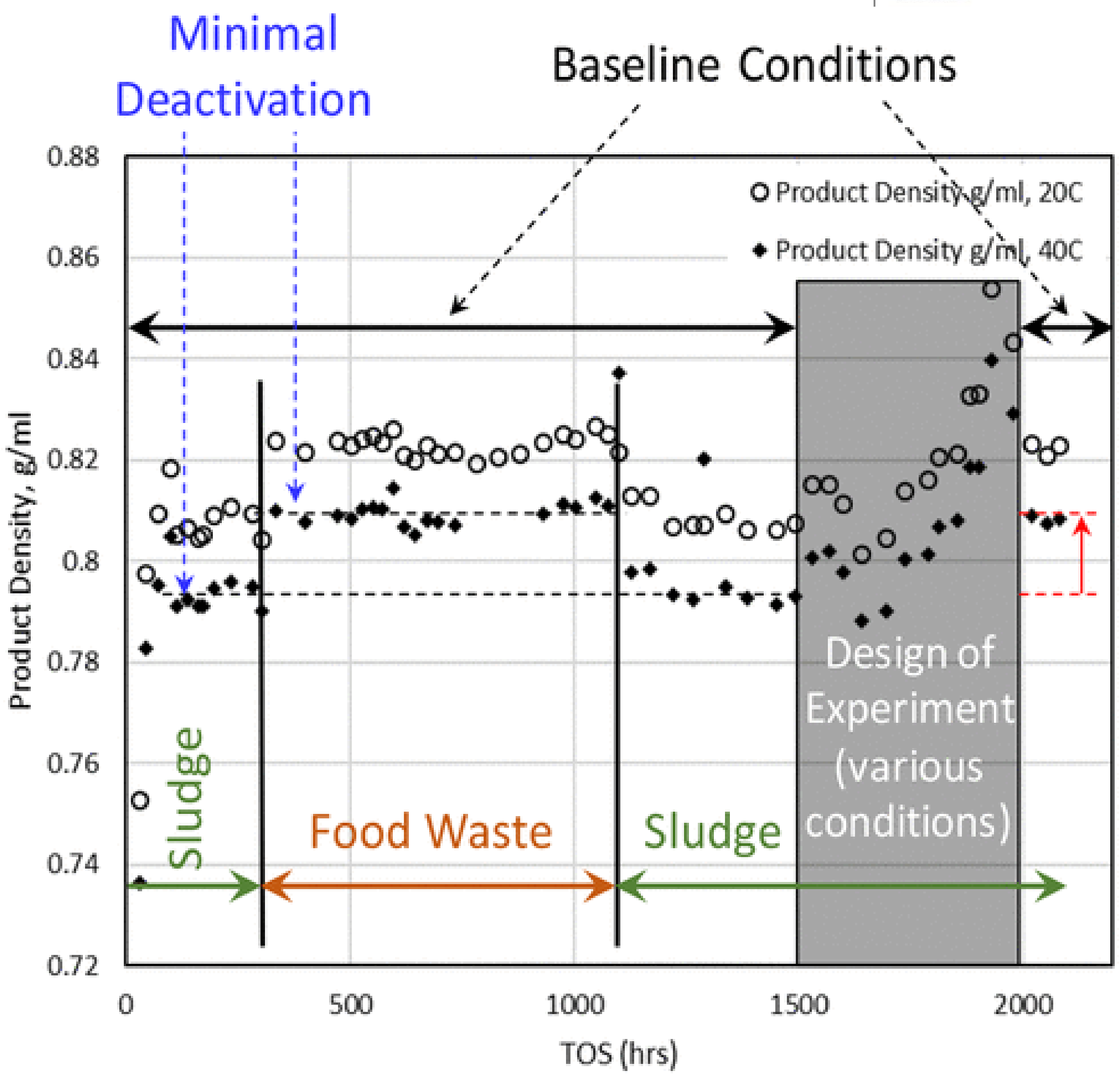
→

Upgraded

→

Distilled





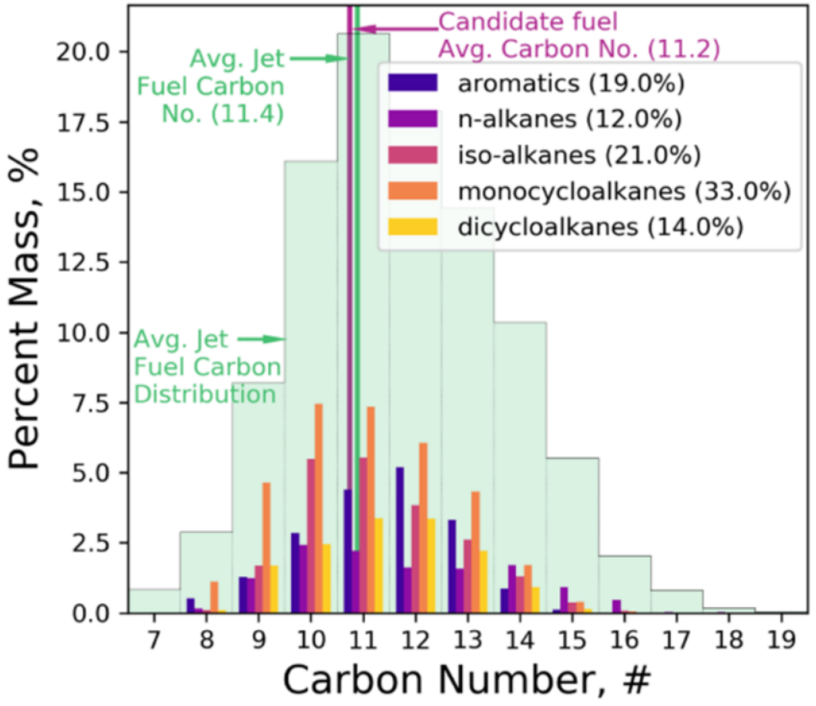
Key outcome:

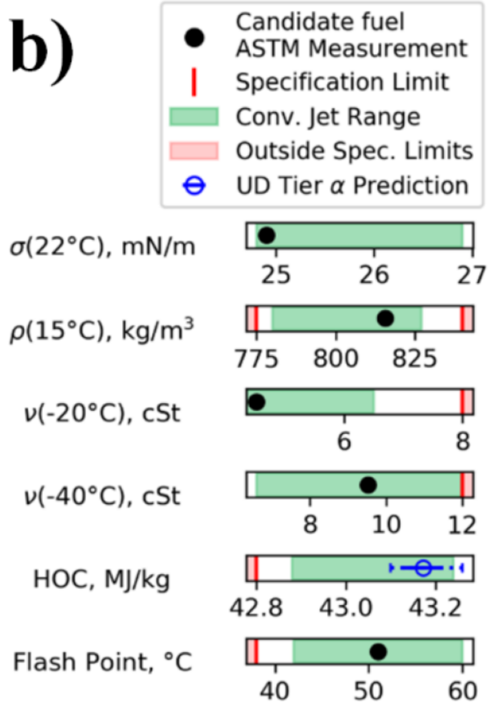
- Stable continuous hydrotreating: 1500+ hours
  - World record!
  - Industrially relevant catalyst lifetimes.

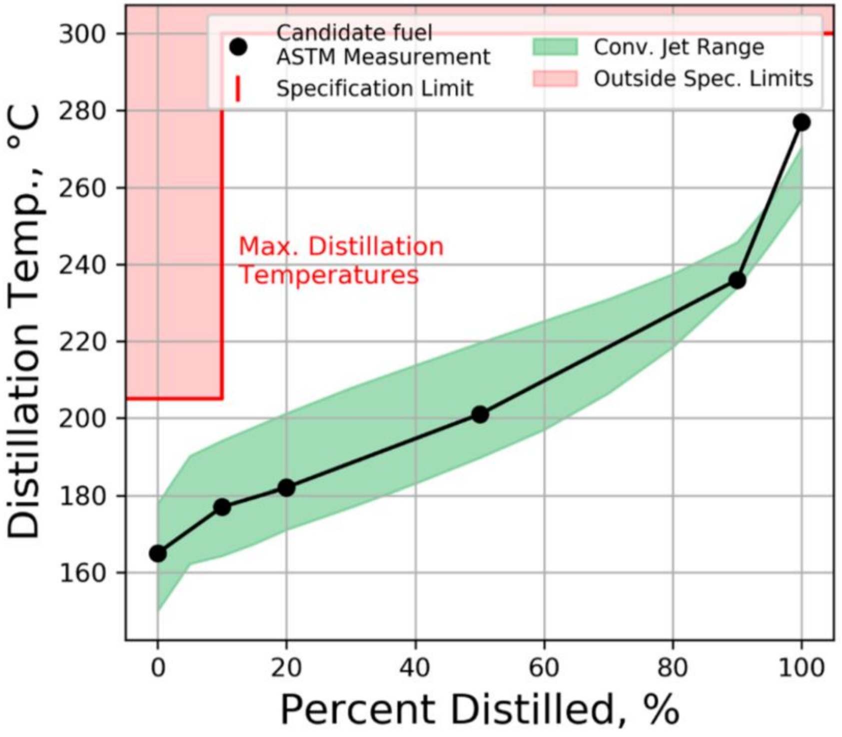
[1]Cronin, D. J., Subramaniam, S., Brady, C., Cooper, A., Yang, Z., Heyne, J., ... & Thorson, M. R. (2022). Sustainable Aviation Fuel from Hydrothermal Liquefaction of Wet Wastes. *Energies*, 15(4), 1306.
[2]Kallupalayam Ramasamy, K., Thorson, M. R., Billing, J. M., Holladay, J. E., Drennan, C., Hoffman, B., & Haq, Z. (2021). *Hydrothermal Liquefaction: Path to Sustainable Aviation Fuel* (No. PNNL-31930). Pacific Northwest National Lab.(PNNL), Richland, WA (United States).
[3]Subramaniam, S., Santosa, D. M., Brady, C., Swita, M., Ramasamy, K. K., & Thorson, M. R. (2021). Extended Catalyst Lifetime Testing for HTL Biocrude Hydrotreating to Produce Fuel Blendstocks from Wet Wastes. *ACS Sustainable Chemistry & Engineering*, *9*(38), 12825-12832.
[4]Thorson, M. R., Santosa, D. M., Hallen, R. T., Kutnyakov, I., Olarte, M. V., Flake, M., ... & Swita, M. (2021). Scaleable Hydrotreating of HTL Biocrude to Produce Fuel Blendstocks. *Energy & Fuels*, *35*(14), 11346-11352.

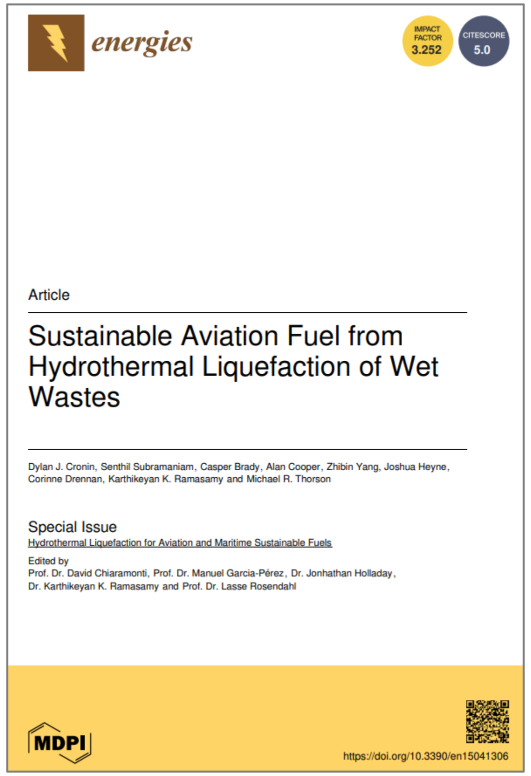
2 – Progress and Outcomes: **HTL of Wet Wastes Produces a High-Quality SAF**

- ~25% of upgraded fuel in jet range.
- Similar mix of cycloalkanes, n-alkanes, iso-alkanes, aromatics to traditional jet.
- Positive Tier α and β jet fuel properties.[1]
- Main concern is nitrogen in the jet fuel.








energies

Article

Sustainable Aviation Fuel from Hydrothermal Liquefaction of Wet Wastes

Dylan J. Cronin, Senthil Subramaniam, Casper Brady, Alan Cooper, Zhibin Yang, Joshua Heyne, Corinne Drennan, Karthikeyan K. Ramasamy and Michael R. Thorson

Special Issue
Hydrothermal Liquefaction for Aviation and Maritime Sustainable Fuels

Edited by
Prof. Dr. David Chiaramonti, Prof. Dr. Manuel Garcia-Pérez, Dr. Jonathan Holladay, Dr. Karthikeyan K. Ramasamy and Prof. Dr. Lasse Rosendahl

MDPI

https://doi.org/10.3390/en15041306

Key outcome: Meets all Tier α and β SAF specs.

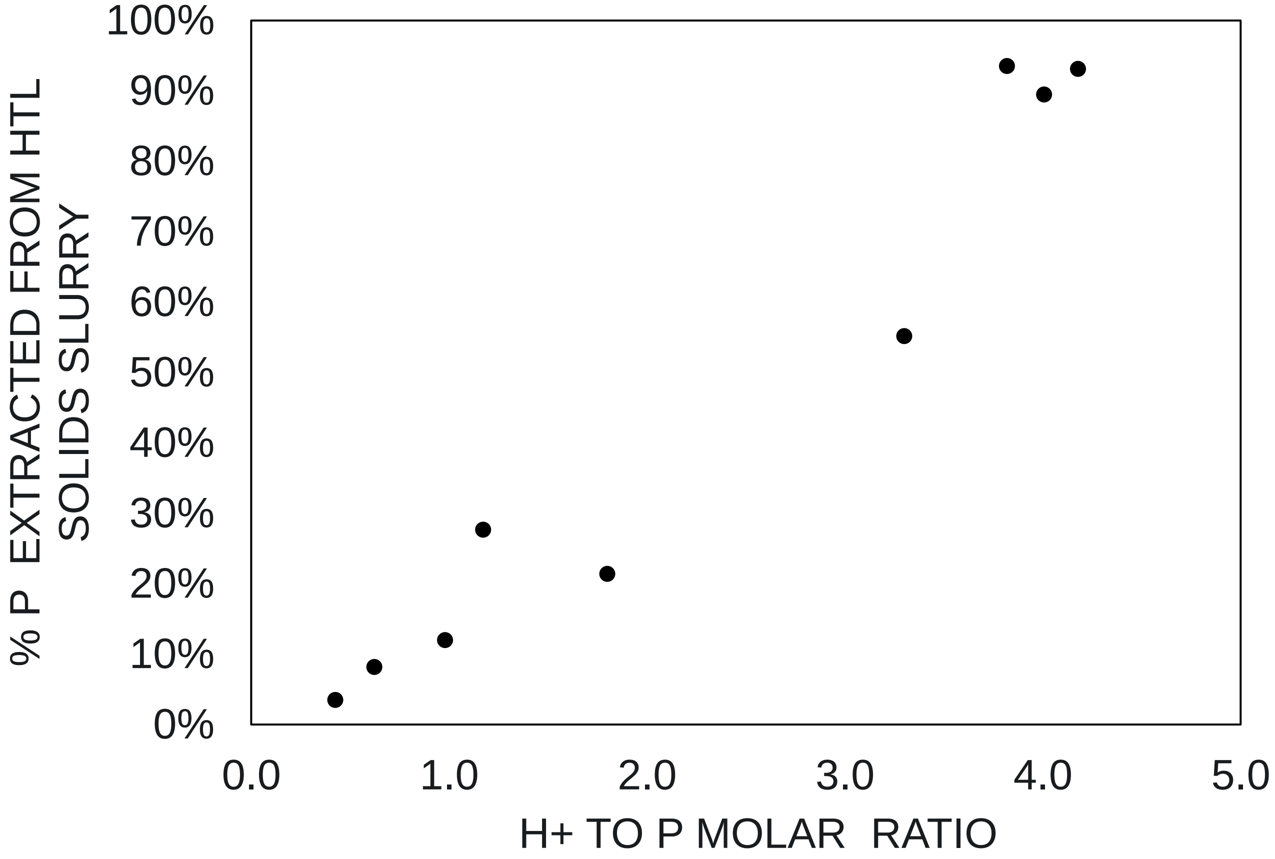
[1]Cronin, D. J., Subramaniam, S., Brady, C., Cooper, A., Yang, Z., Heyne, J., ... & Thorson, M. R. (2022). Sustainable Aviation Fuel from Hydrothermal Liquefaction of Wet Wastes. *Energies*, 15(4), 1306.

2 – Progress and Outcomes: Recovering Phosphorus and Nitrogen as Fertilizer – New Task in FY23

Objective: Develop process to extract valuable fertilizer co-products from HTL side stream.

- Concentrated, phosphorus-containing fertilizer.
- Reduction in NH_3N content in HTL aqueous.

Purified phosphorous product



Early progress:

- >75% extraction of phosphorus from HTL solids as an impure struvite.
- Removal of ~50% NH_3N from HTL aqueous.

2 – Progress and Outcomes: **Tracking Destruction of Forever Chemicals – New Task in FY23**

Background: PFAS pollution is an environmental concern.

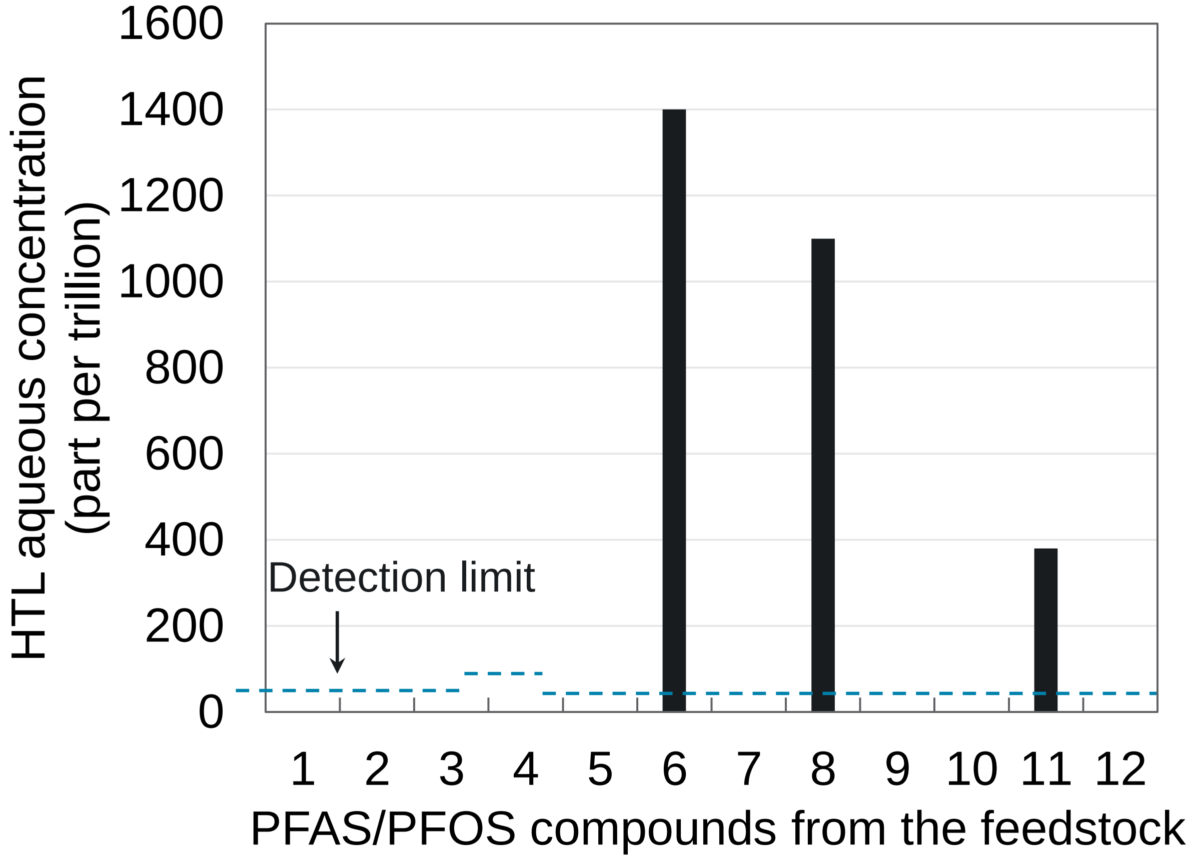
- U.S. Environmental Protection Agency (EPA) Guidance (2016): "Established the health advisory levels at **70 parts per trillion.**"
- EPA (2022): "Propose to designate two PFAS as hazardous substances under CERCLA, or Superfund."

Objectives: Study the **impact of HTL processing on forever chemicals.**

- Track PFAS/perfluorooctane sulfonic acid (PFOS) from the feedstock via all processes: HTL, aqueous oxidation, struvite recovery, and hydrotreating.

Early Progress:

- Demonstrated high level of PFAS/PFOS destruction in HTL.
- Identified major PFAS/PFOS compounds of concern in HTL aqueous stream.
- Demonstrated >10,000x reduction in PFAS/PFOS chemicals through hydrotreating (3 spiked samples)

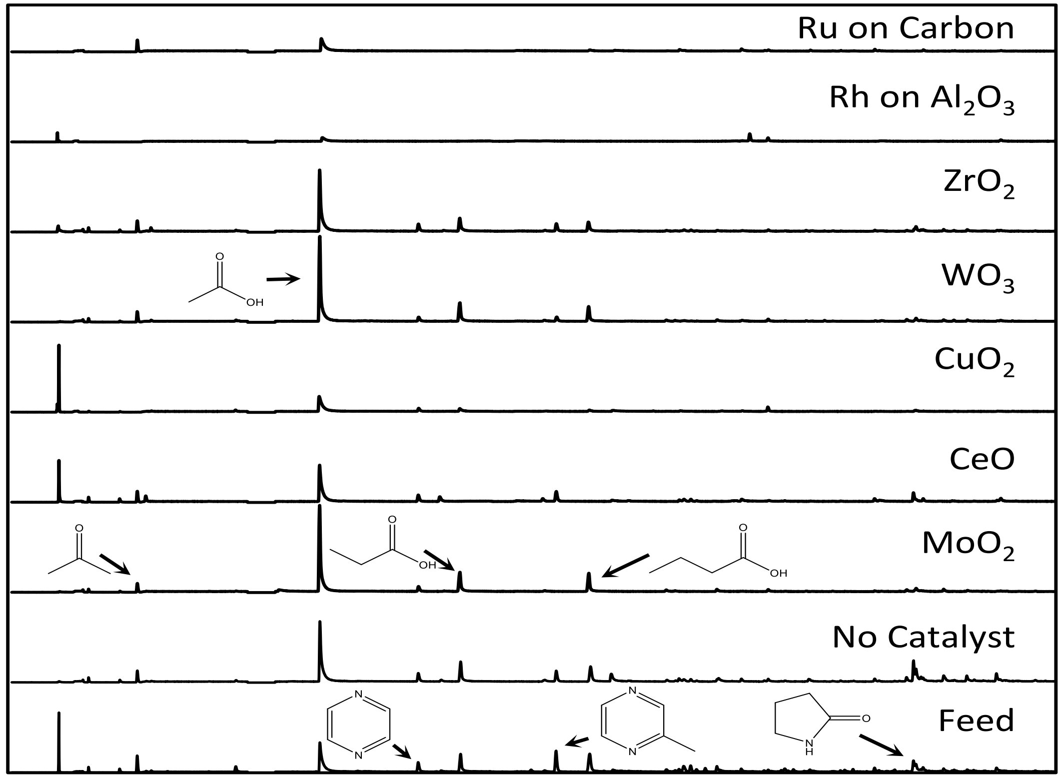


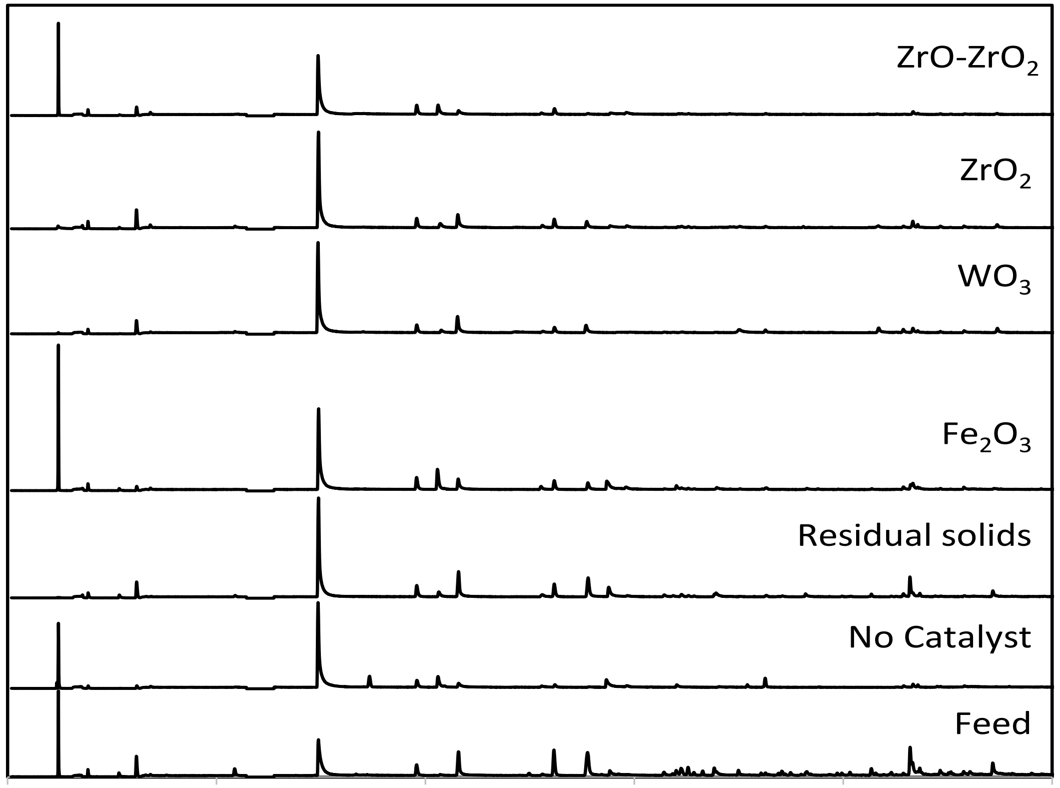
2 – Progress and Outcomes: Mild Oxidation to Enable Recycle of HTL Aqueous to a WRRF

Motivation: HTL aqueous phase is full of recalcitrant compounds, limiting recycle to WRRFs.

Approach: Mild oxidation can clean up aqueous phase.

- Completed screening, semi-batch, and CSTR demonstration of technology.





Key accomplishments:

- Numerous catalysts (e.g., HTL solids) effective for mild oxidation.
- Oxidizes nitrogen organics to carboxylic acids (>2x increase in acids).

3 – Impact: **HTL solves two crucial challenges to society: Sustainable Aviation Fuel and Sewage Sludge Disposal**

Value #1: Low GHG fuels

- Potential of 6 billion gal/year of fuel (U.S.).
- 78% reduction in GHG.

Value #2: Sludge disposal

- Sludge disposal represents ~50% of wastewater costs.
- Provides destruction of forever chemicals.
- Costs expected to continue to increase as land application becomes illegal.

3.1 M gal fuel/yr[2]
$12.4M/yr.

Two potential revenue streams
Example:100 dry tons/day plant

HTL provides a disposal solution in addition to sustainable fuel production.

Offset sludge disposal costs[1]:
$7.3-14.6M /yr.

[1]Basis of disposal costs: $200-400/dry ton or $40/wet ton @ 10-20 wt% solids, [2]Assumed fuel value of $4/gal.

3 – Impact: **HTL Addresses Environmental and Social Justice Issues Worldwide**

Need processes to cost effectively destroy PFAS.

UPI UPI.com

Maine passes first PFAS biosolids ban, taking stand against forever chemicals

BANGOR, Maine, May 4 (UPI) -- Maine has become the first state to ban fertilizer using sewage sludge containing "forever chemicals"...

May 4, 2022

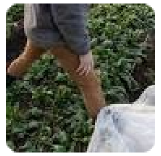
NPR

EPA Moves To Regulate Toxic PFAS Chemicals

PFAS chemicals have been linked to illnesses, including cancer, but there remain a lot of questions about how they affect people's health and in...

The Washington Post

States take on PFAS 'forever chemicals' with bans and lawsuits



Colorado, for example, passed a law this year covering many products, ... And California passed laws this year to ban PFAS in cosmetics and...

Nov 5, 2022

The Portland Press Herald

Latest impact of PFAS contamination: Rising sewer rates

This spring, Maine banned the reuse of sewage sludge as fertilizer ... And those costs are only expected to rise as disposal rules get more...

Jul 10, 2022

Maine bans use of sewage sludge on farms to reduce risk of PFAS poisoning

Sludge used as crop fertilizer has contaminated soil, water, crops and cattle, forcing farmers to quit

ENVIRONMENT

Colorado has been spreading biosolids with "forever chemicals" on farms, records show. How dangerous is it?

Environmental groups say there is no safe level for toxic PFAS chemicals in drinking water or on farm land. State regulators say they are studying it.

Fertilizer recovery can reduce foreign dependance.

Fortune

Fertilizer prices just hit a record high sparking fears of global starvation and the worst food insecurity level since World War II

Russian fertilizers are everywhere · A global food supply crisis.

Mar 21, 2022

WSJ Wall Street Journal

Fertilizer Prices Surge as Ukraine War Cuts Supply, Leaving ...

That, together with the impact of the West's financial sanctions against Moscow, means fertilizer exports from Russia—the world's largest...

Mar 24, 2022

FORTUNE

FINANCE · FERTILIZER

'A perfect storm for the whole food system right now': One of the world's largest fertilizer companies warns that every country—even those in Europe—is facing a food crisis

BY TRISTAN BOVE

January 26, 2023 at 10:53 AM PST

BBC

Putin is weaponising food, says boss of fertiliser giant Yara

17 January · Comments

High sludge disposal costs are born by local communities.

Metro Vancouver's new sewage treatment plant near YVR Airport to cost over $10 billion

Kenneth Chan | Jul 8 2021, 3:53 pm

Summary: Targeted Research to Accelerate HTL Deployment to Convert Wet Wastes to Fuels

Approach	• Close partnership with industry and technoeconomic analysis and resource assessment teams to (a) prioritize research and (b) provide industrially viable solutions. • SMART milestones to ensure successful impact.

Progress and Outcomes	• Assessed key levers impacting conversion costs (solids content, regional blends).
	• Advanced hydroprocessing of biocrude to produce SAF:
	  • >2000 hours of stable hydroprocessing
	  • Excellent Tier α and β properties of SAF fraction.
	• Progress towards FY23 tasks and milestones:
	  • Initial assessment of PFAS compounds in HTL aqueous and solids stream
	  • >75% extraction of phosphorous
	  • Development of mild oxidation process for HTL aqueous cleanup.

Impact	• Produce SAF. • Disposing of sludge (including PFAS removal) and extracting fertilizer.
Future Outcomes	• Aqueous treatment: Use mild oxidation to enable recycle of aqueous stream. • PFAS: Assess PFAS partitioning and destruction throughout HTL and upgrading. • Nutrient recovery: Recover phosphorus and nitrogen as fertilizer while enabling land application.

Quad Chart Overview

Timeline

- Original project start date: Oct 1, 2019
- Project renewal start date: Oct 1, 2022
- Project end date: Sept 30, 2025

	FY 22	Total Award
DOE Funding	10/1/2021-9/30/2022 $360,000	$1,200,000 (10/1/2022-9/30/2025)
Project Cost Share*	$0	$0

TRL at Project Start: 2
TRL at Project End: 4

Project Goal

Develop mild oxidation to enable recycle of the aqueous stream, develop a process for concentrated phosphorous extraction, and track forever chemical destruction through HTL.

End of Project Milestone

Develop an aqueous and solids treatment process (mild oxidation and struvite precipitation) that meets solids disposal requirements and aqueous recycle requirement at partner WRRFs.

Funding Mechanism

Lab Call

Project Partners

- Great Lakes Water Authority
- MetroVancouver
- Aloviam
- Circlia Nordic

Acknowledgements

- Beau Hoffman, Technology Manager, Bioenergy Technologies Office

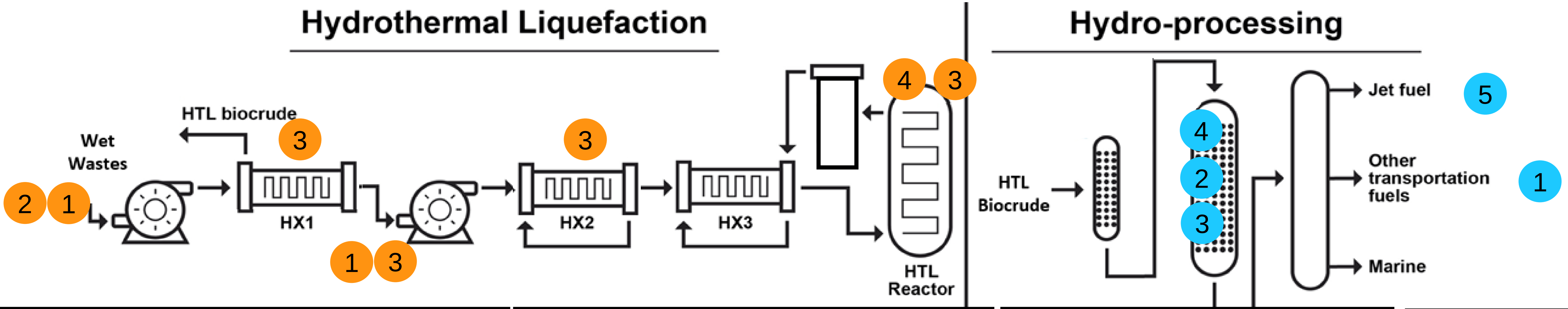
Experimental Team:
- Mike Thorson
- Andy Schmidt
- Uriah Kilgore
- Todd Hart
- Sam Fox
- Miki Santosa
- Senthil Subramanian
- Igor Kutnyakov
- Matt Flake
- Mariefel Olarte
- Lisa Middleton Smith

Analysis Team:
- Yuan Jiang
- Shuyun Li
- Yunhua Zhu
- Aye Meyer
- Lesley Snowden-Swan

Waste Resource Team:
- Tim Seiple
- Andre Coleman

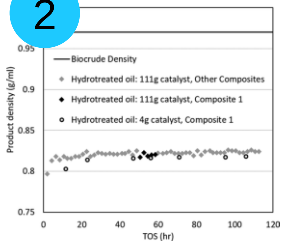
Additional Slides

Addressed numerous barriers towards commercialization (subset here)



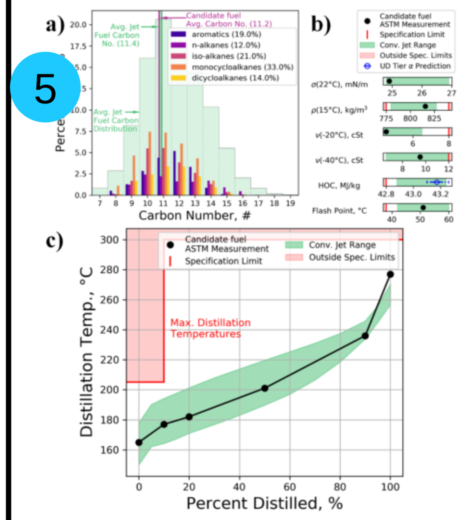
1 Demonstrated yield and capital cost improvements with increased solids content[4]

4 ID'd real world challenges with HTL of plastics 4

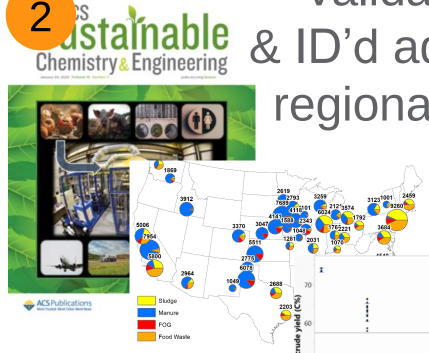


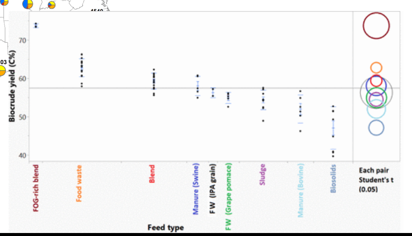
2 Scalable hydrotreating >20x scale-up[5]

Excellent Tier α and β SAF Properties[1] & ID'd key challenges[1,2]



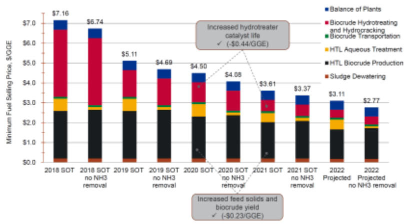
2 Validated viability & ID'd advantage of regional wet waste processing[4]



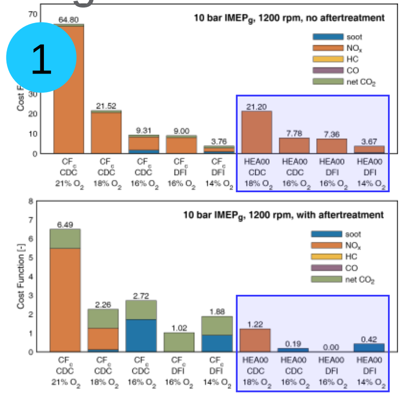


Quarterly GPRA updates to BETO (FY21)

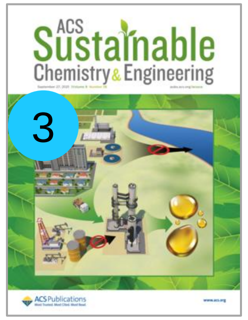
3 Significant SOT cost reductions[6]

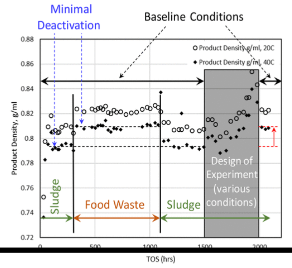


Excellent diesel engine test results



Stable hydrotreating >2000 hr![3]





[1]Cronin, D. J., Subramaniam, S., Brady, C., Cooper, A., Yang, Z., Heyne, J., ... & Thorson, M. R. (2022). Sustainable Aviation Fuel from Hydrothermal Liquefaction of Wet Wastes. *Energies*, 15(4), 1306.
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[3]Subramaniam, S., Santosa, D. M., Brady, C., Swita, M., Ramasamy, K. K., & Thorson, M. R. (2021). Extended Catalyst Lifetime Testing for HTL Biocrude Hydrotreating to Produce Fuel Blendstocks from Wet Wastes. *ACS Sustainable Chemistry & Engineering*, *9*(38), 12825-12832.
[4]Cronin, D., Schmidt, A. J., Billing, J., Hart, T. R., Fox, S. P., Fonoll, X., ... & Thorson, M. R. (2021). Comparative Study on the Continuous Flow Hydrothermal Liquefaction of Various Wet-Waste Feedstock Types. *ACS Sustainable Chemistry & Engineering*.
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[6]Snowden-Swan, L. J., Billing, J. M., Thorson, M. R., Schmidt, A. J., Jiang, Y., Santosa, D. M., ... & Taylor, M. A. (2021). Wet Waste Hydrothermal Liquefaction and Biocrude Upgrading to Hydrocarbon Fuels: 2020 State of Technology (No. PNNL-30982). PNNL, Richland, WA

Responses to Previous Reviewers' Comments

Feedback: The reviewers expressed interest in "Understand[ing] any issues with disposal of solids" … "and any recycle stream to the WRRF" & "Liability/utility of waste/byproduct streams will be important"

Response: We agree that the disposal or treatment strategy for full-scale integration within a WRRF is needed for the HTL aqueous and solids streams. PNNL is developing multiple promising commercially viable treatment solutions for the aqueous streams including catalytic hydrothermal gasification, anaerobic digestion, wet air oxidation, and other processes to clean up the stream. The ultimate goal is for all or part of the aqueous stream to be recycled to the headworks of WRRF. While the solids stream may meet landfill or land applications specifications, we believe there is an opportunity to improve the overall process yield by extracting residual oil from the solids stream.

Feedback: "It is not clear what the approach to actually extending the catalyst lifetime is"

Response: To extend the hydrotreater catalyst life, we used a guard bed to remove the Fe and other metals (hydrodemetallization) from the biocrude and thereby, reduce catalyst poisoning in the main hydrotreater due to metal deposition. We evaluated two guard-bed configurations, a slurry bubble column and a trickle-bed hydrotreater to reduce the metal content <10ppm before the main hydrotreater. The use of a guard bed enabled us to achieve a modeled hydrotreater catalyst life of 1 year by doing long-term catalyst lifetime testing for over 2000 hours.

Publications, Patents, Presentations, Awards, and Commercialization (since FY21 Review)

Publications:

1. Subramaniam S., Kilgore, U.K., Fox, S.P., Cronin, D.J., Guo, M.F., Schmidt, A.J. Ramasamy, K., Thorson, M.R., "Catalytic Wet Air Oxidation of Hydrothermal Liquification Aqueous Stream." 2022 ,Submitted to: Water Research.
2. Cronin, D. J.; Subramaniam, S.; Brady, C.; Cooper, A.; Yang, Z.; Heyne, J.; Drennan, C.; Ramasamy, K. K.; Thorson, M. R., Sustainable Aviation Fuel from Hydrothermal Liquefaction of Wet Wastes. Energies 2022, 15 (4), 1306.
3. Snowden-Swan, L. J.; Li, S.; Jiang, Y.; Thorson, M. R.; Schmidt, A. J.; Seiple, T. E.; Billing, J. M.; Santosa, D. M.; Hart, T. R.; Fox, S. P. Wet Waste Hydrothermal Liquefaction and Biocrude Upgrading to Hydrocarbon Fuels: 2021 State of Technology; Pacific Northwest National Lab.(PNNL), Richland, WA (United States): 2022.
4. Santosa, D. M.; Wendt, L. M.; Wahlen, B. D.; Schmidt, A. J.; Billing, J.; Kutnyakov, I. V.; Hallen, R. T.; Thorson, M. R.; Oxford, T. L.; Anderson, D. B., Impact of storage and blending of algae and forest product residue on fuel blendstock production. Algal Research 2022, 62, 102622.
5. Choi, H.; Soland, N. E.; Moss, M. R.; Liu, J.; Prestangen, R. R.; Katahira, R.; Lee, S.-J.; Thorson, M. R.; Freeman, C. J.; Karp, E. M., The cell utilized partitioning model as a predictive tool for optimizing counter-current chromatography processes. Separation and Purification Technology 2022, 285, 120330.
6. Subramaniam, S.; Santosa, D. M.; Brady, C.; Swita, M.; Ramasamy, K. K.; Thorson, M. R., Extended Catalyst Lifetime Testing for HTL Biocrude Hydrotreating to Produce Fuel Blendstocks from Wet Wastes. ACS Sustainable Chemistry & Engineering 2021, 9 (38), 12825-12832.

Patents:

1. Thorson, M. R.; Snowden-Swan, L. J.; Schmidt, A. J.; Hart, T. R.; Billing, J. M.; Anderson, D. B.; Hallen, R. T., Hydrothermal liquefaction system. US Patent 11,279,882: 2022. - **Licensed**

Presentations:

1. TCBiomass, Denver, CO, 04/2022
2. WEF Process Innovations, 06/2022
3. HTL workshop, MetroVancouver, 06/2022
4. WEFTEC, 10/2022
5. AIChE, 11/2022

Commercialization Efforts:

1. Metro Vancouver is building a demonstration HTL plant based on PNNL's technology
2. Aloviam is scaling up the HTL process via an awarded FOA

2 – Progress and Outcomes
Task 5 – Aqueous Treatment

Objectives:

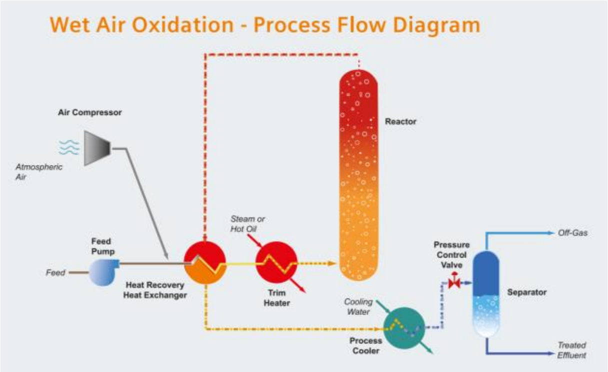
Advance mild oxidation to treat the HTL aqueous phase, and thereby enable sustainable recycling of this stream to the headworks of a water resource recovery facility (WRRF)

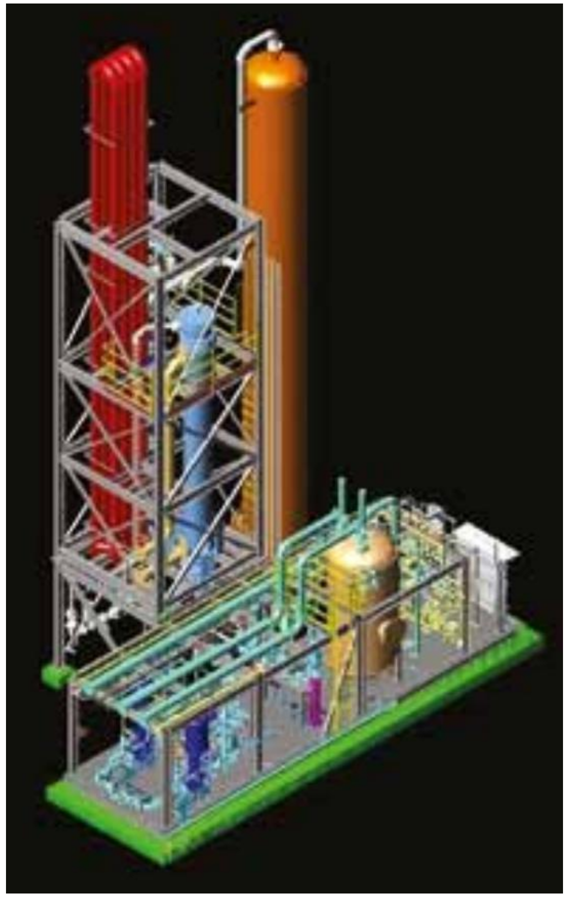
Motivation:

- HTL Aqueous phase if full of recalcitrant compounds limiting recycle to WRRFs
- Commercial process with multiple licensers with modular designs
- Widely used in refining, petrochemicals, and wastewater treatment

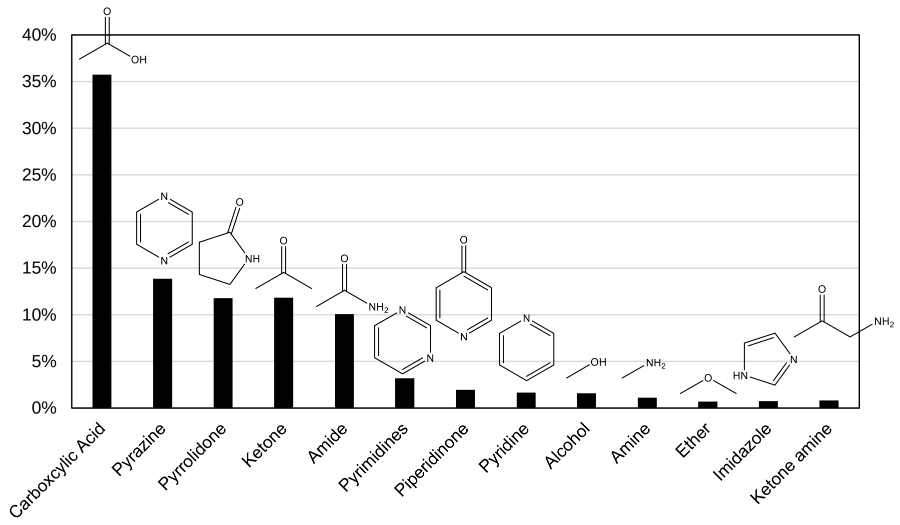
Milestones:

- FY23Q2 (6/2023): Reduce the residual solids volume >50%. Track the residual organic nitrogen with 90% mass balance. Partner with WRRF to assess the recyclability of this stream at their WRRF.
- FY23Q4 (9/2025): Develop a process for aqueous and solids treatment via wet air oxidation (>2000 hr of continuous operation) with sufficient treatment to meet solids disposal requirements and aqueous recycle at a WRRF





Breakdown of HTL aqueous compounds by GCMS peak area



2 – Progress and Outcomes

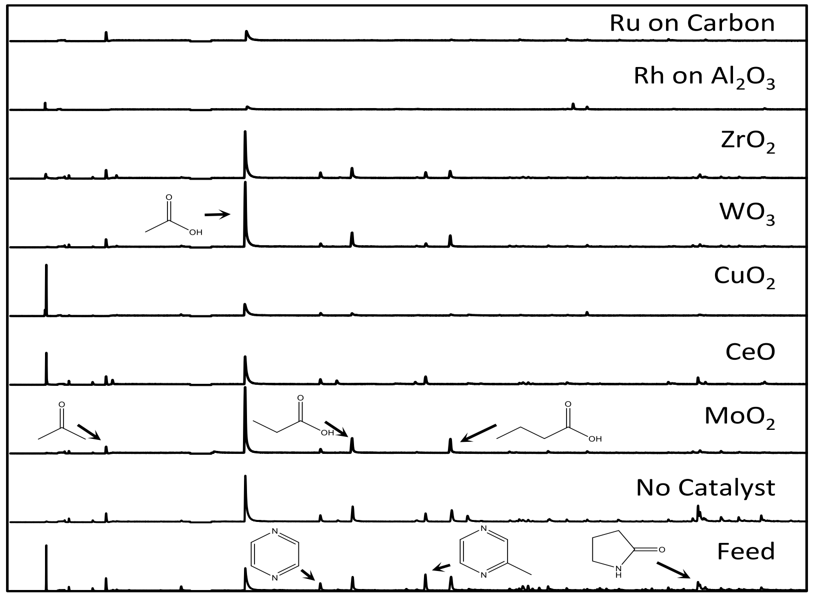
Task 5 – Aqueous Treatment

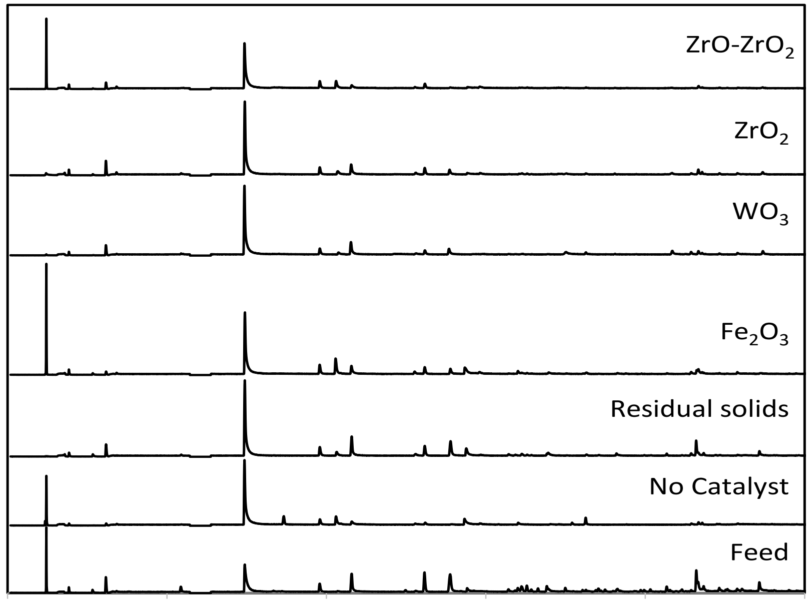
Key accomplishments:

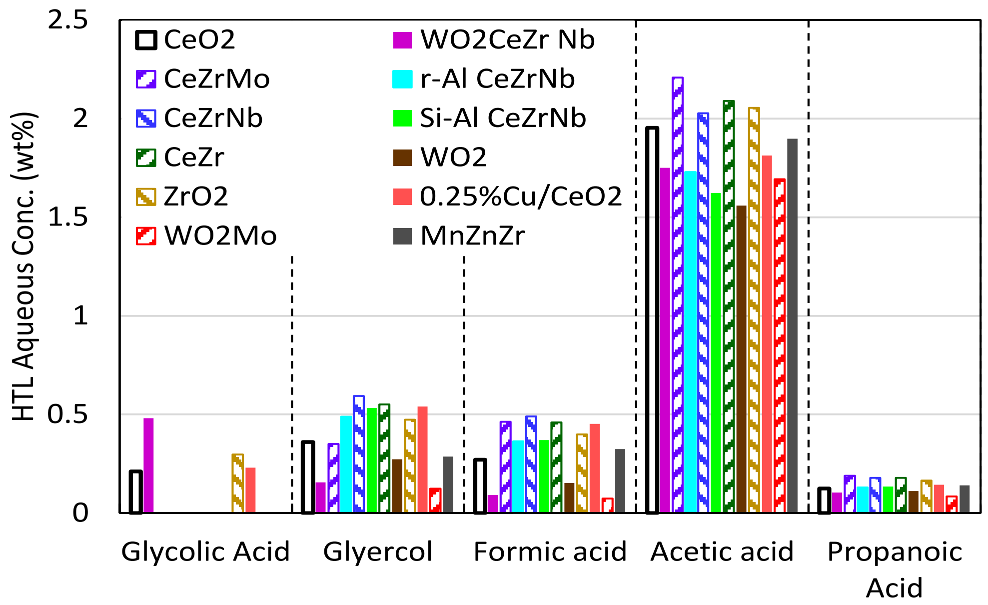
- Identified numerous catalysts effective for mild oxidation of the HTL aqueous stream
  - Residual solids from HTL proves to be a particularly effective catalyst
- Demonstrated the ability to oxidizes the nitrogen organics to carboxylic acids (>2x increase in acids)

Next Steps:

- Transfer batch process to a continuous process (CSTR) to understand deactivation
- Determine the optimal temperature (</=300°C), pressure, residence time, HTL residual solids addition (and recycle ratio) and heat integration
- Develop utilization strategy for carboxylic acids
- Determine the solids dewatering strategy following oxidation
- Develop strategy for heat integration in HTL







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Thank you