Advancing Forest Biorefineries Towards Commercial Applications through Fractionation of Biomass Wastes

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Feedstock Technologies

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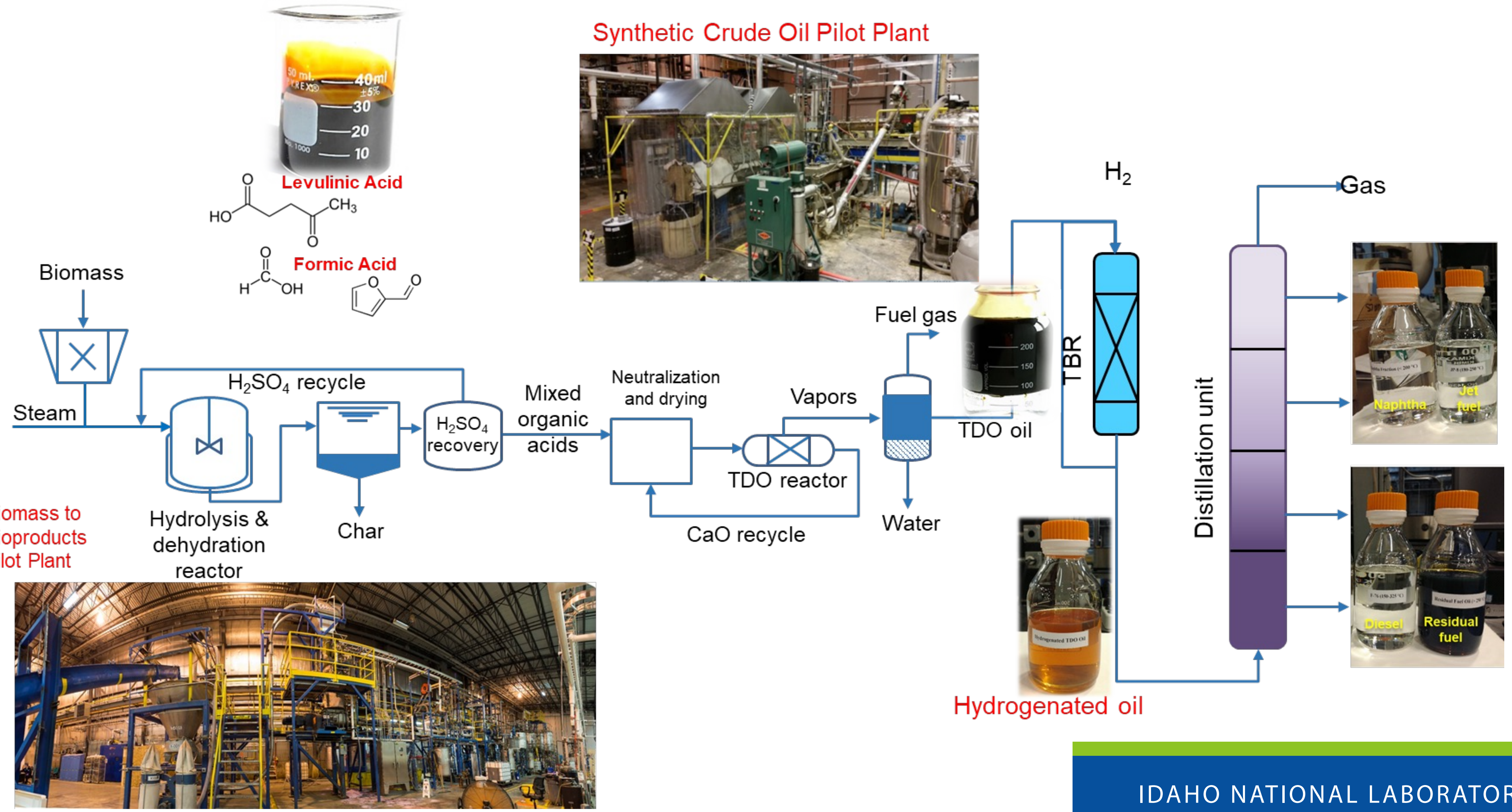
Project Overview

- This project supports the development of woody feedstocks for University of Maine’s Thermal Deoxygenation (TDO) pathway to produce renewable aviation fuel and chemicals.

- The goal of this project is to prepare woody biomass for conversion in TDO by *engineering particle attributes to improve flow reliability and separating detrimental components within the biomass using thermal fractionation*.

- This research will solve challenges in solids transport in compression screw feeders and progressive cavity pumps as well as investigate thermal fractionation to improve downstream quality by reducing char formation for improved yields.

Thermal Deoxygenation Pathway Overview



Synthetic Crude Oil Pilot Plant

Levulinic Acid

Formic Acid

Biomass

Steam

H_2SO_4 recycle

Hydrolysis & dehydration reactor

Char

H_2SO_4 recovery

Mixed organic acids

Neutralization and drying

TDO reactor

CaO recycle

Vapors

Fuel gas

Water

TDO oil

H_2

TBR

Gas

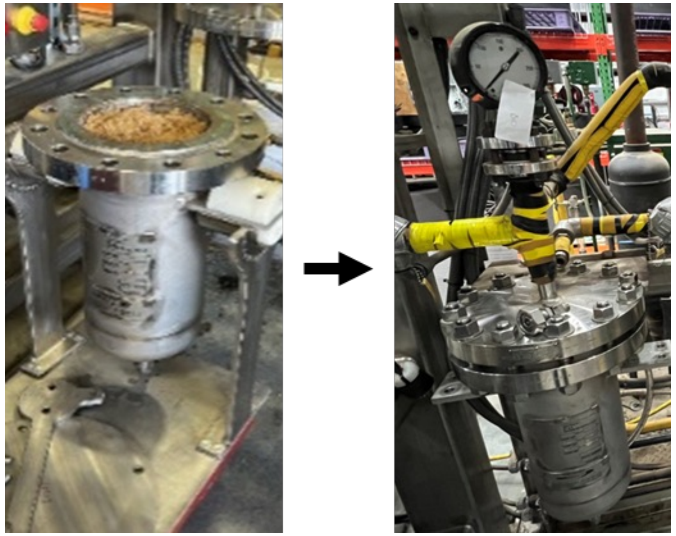
Distillation unit

Hydrogenated oil

Biomass to Bioproducts Pilot Plant

1 – Approach – Overview Biomass Feedstock National User Facility (BFNUF) Material Preprocessing and Conditioning

BFNUF processing of pulp as an “ideal feedstock” baseline before moving onto woody residues that have significantly different physical and chemical properties.



Batch scale thermal fractionation testing of 2 kg (dry basis) samples of wood chips has provided insight as to the CMA of woody feedstock prior to half-ton scale runs.

1 – Approach – Overview University of Maine Thermal Deoxygenation (TDO) Summary

Solids Inlet

→

Progressive Cavity Pump

→

Flash Drum Separations

→

Solid Carbon After Filters

The University of Maine has a pilot scale Thermal Deoxygenation system to turn cellulose into bio-oil and value-added chemical co-products. **This system is being used understand how feedstock physical and compositional changes in woody wastes impact material flowability against pressure gradients and the downstream product quality / yields after acid pretreatment**.

1 – Approach – Research Team and Roles

Idaho National Laboratory

- Feedstock engineering through size reduction, densification, and contaminant thermal fractionation in the Chemical Preprocessing System (CPS) to remove detrimental components.

Luke Williams

David Thompson

John Aston

Eliezer Reyes Molina

National Renewable Energy Laboratory

- Advisory role on biomass fractionation and lignin / hemicellulose removal from biomass using sulfide pretreatment

Xiaowen Chen

1 – Approach – Research Team and Roles

University of Maine

- Primary research institution for the “Thermal Deoxygenation” (TDO) process
- The Technology Research Center is lead by Amy Luce and has about 20 staff members.
- The bench scale screening work involves researchers from the US, Nigeria, Ghana, Sri Lanka, and India.
- This project sources feedstocks from rural areas in Maine.
- The DEI goal of this project is to expand research opportunities to the most qualified and diverse personnel possible.

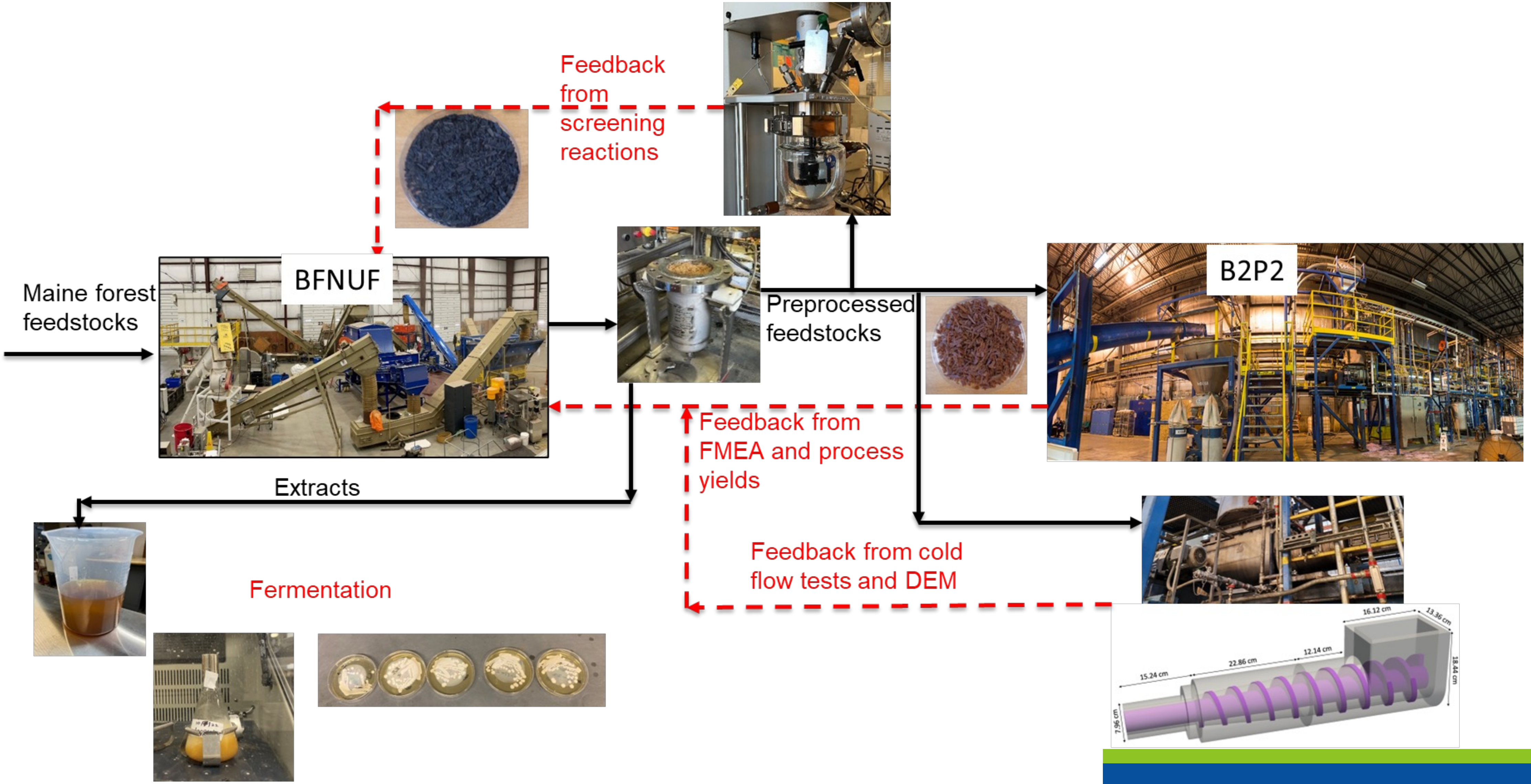
Graduate Researchers and Faculty

Technology Research Center Professionals

1 – Approach – Experimental Plan

- A half ton run of preprocessed Brownstock Pulp (Unbleached Kraft) was used to establish a “baseline” for the best possible conversion of woody material in the TDO process.
- Hemicelluose and extractives thermal fractionation was performed at the kg scale in the BFNUF’s Chemical Preprocessing System (CPS). *Variables investigated included particle size, temperature, initial moisture content.*
- Fractionated material was tested for acid hydrolysis yields in UMaine’s bench scale system
- Cold flow in compression screw feeders and progressive cavity pumps is occurring in INL and UMaine pilot plants. *Cold flow and batch tests will help dictate the conditions for UMaine’s first pilot scale run with thermally fractionated clean softwood material.*
- Depending on the results from early experiments later tests will advance to softwood forest residues, clean hardwood chips, and hardwood forest residues.
- Failure Modes and Effects Analysis (FMEA) and Discrete Element Models are being developed to improve process learning as the research progresses along with technoeconomic analysis (TEA)

1 – Approach – Experimental Plan Visualization



Feedback from screening reactions

Maine forest feedstocks

BFNUF

Preprocessed feedstocks

B2P2

Feedback from FMEA and process yields

Extracts

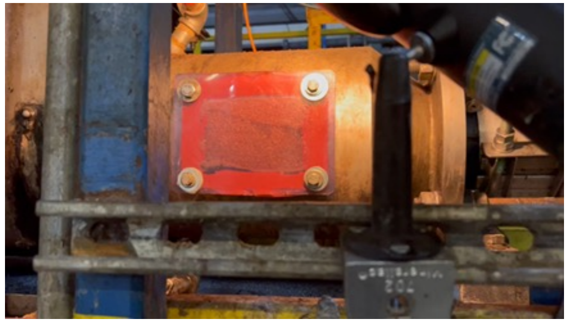
Feedback from cold flow tests and DEM

Fermentation

1 – Approach – Challenges with the Technical Approach

The main challenges for improving woody biomass in TDO are:

1. Particle formulation (size, aspect ratio, compressibility, wettability) to improve process throughput (increased solids loading) and reliability in the progressive cavity pump at UMaine's Biomass to Bioproducts Pilot Plant (B2P2)



A window into the entrance of the progressive cavity pump in the B2P2 allows visualization of flow properties.

2. Selective fractionation of detrimental components in the woody wastes being utilized as feedstocks. There is a balance between hemicellulose and extractive removal without losing carbon associated with C_6 sugars and mitigating sticky char formation and filter plugging downstream in the B2P2 due to resin-type material formation

Thermal fractionation of detrimental components needs to be evaluated holistically with downstream results

1 – Approach – Go / No-Go

The Go/No-Go incorporates metrics for both process throughput (and by extension reliability as evaluated with FMEA) as well as product yields as compared to an ideal “baseline”. **In the case of successful CMA identification, the project will move to new woody waste materials.** If throughput and/or yields are not close enough to the baseline, then CMA’s will be reevaluated.

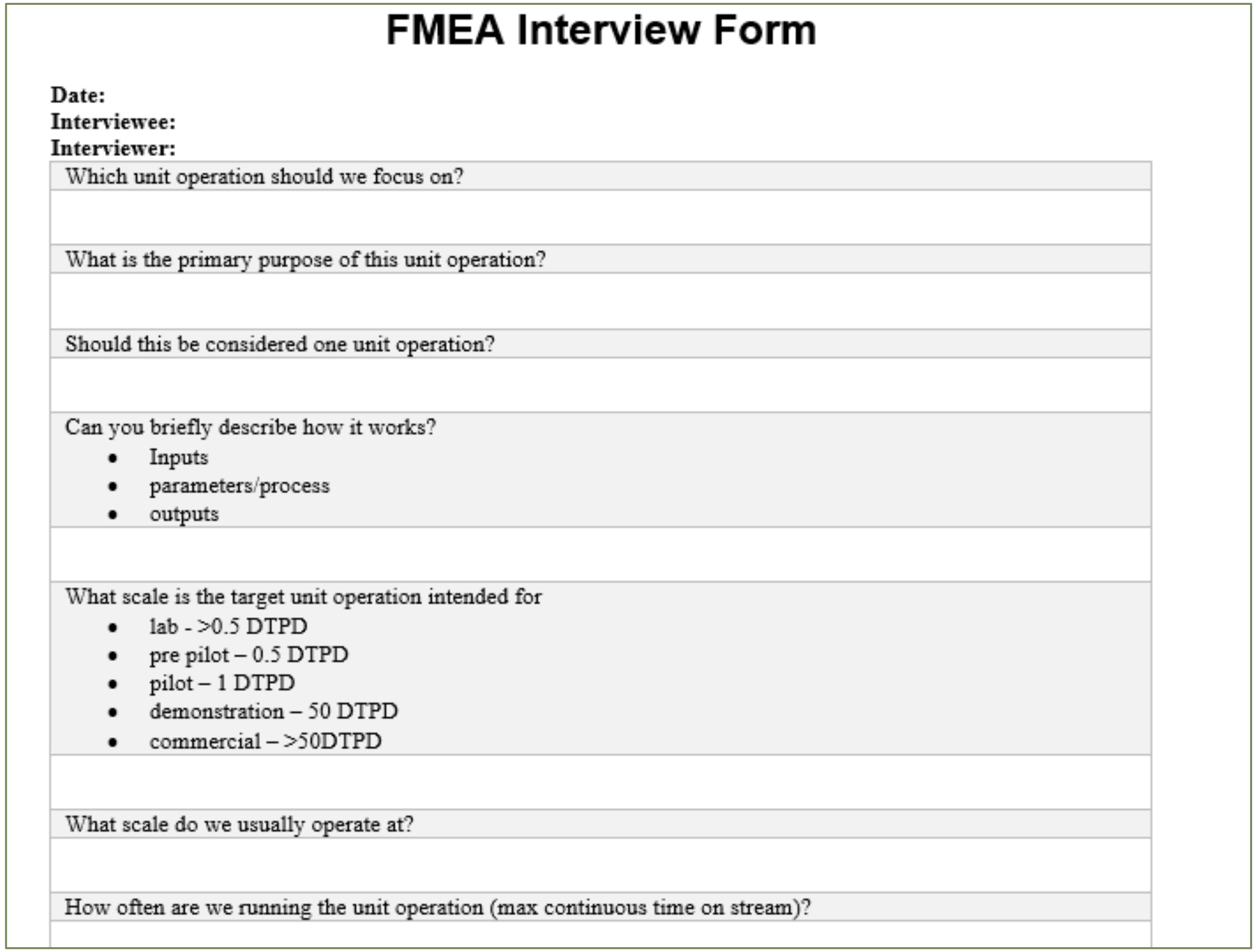
Go: If initial preprocessing achieves 50% of the throughput and/or cellulose conversion of pulp for clean softwood, and the hemicellulose fractionation is enough to reduce C_5 sugar content to an acceptable level for downstream processing after AHDH, then begin research studies with forest residues.

No-Go: If a throughput of 50% is not achieved then continue adjusting the identified CMA’s with tests at the pilot scale to better understand the transfer function of the CMAs between the screening and pilot experiments.

1 – Approach – Risks and Mitigation Strategies

Risk	Description / Mitigation
Feedstock variability.	Wide compositional variability of forest feedstock. Increase preprocessing steps to reduce the variability.
Pump failure / wet solids handling difficulties	Failure of progressive cavity pump while handling slurries. Re-engineer the feedstock for better performance.
Student staff acquisition delays	Delay in commencing graduate research until summer or fall 2022. Conduct as much research with existing staff as possible.
Information accessibility restrictions for proprietary equipment.	Proprietary information on the design of the progressive cavity pump makes modeling impossible. Switch modeling task (i.e., a piece of equipment other than the progressive cavity pump) or focus modeling efforts on the FMEA only.

2 – Progress and Outcomes (Quarter 1) – Facility Visits and Historical Analysis



FMEA Interview Form

Date:
Interviewee:
Interviewer:

Which unit operation should we focus on?
What is the primary purpose of this unit operation?
Should this be considered one unit operation?
Can you briefly describe how it works? • Inputs • parameters/process • outputs
What scale is the target unit operation intended for • lab - >0.5 DTPD • pre pilot – 0.5 DTPD • pilot – 1 DTPD • demonstration – 50 DTPD • commercial – >50DTPD
What scale do we usually operate at?
How often are we running the unit operation (max continuous time on stream)?

- **Objective:** Use Failure Modes and Effects Analysis (FMEA) to:
  - Semi-quantitatively evaluate impacts to risk and risk reduction based on project system design and material
  - Identify primary system pinch points unit operations and the associated CMAs, CPPs, and CQAs to support virtual process assessment tool

2 – Progress and Outcomes (Quarter 1) – Facility Visits and Historical Analysis

- Char build-up primary failure (conversion system)
- Extractives, flow properties, and particle size are the primary CMAs

Failure	**Impacts**	**CQAs**	**VERITY**	**Causes**	**CMAs**	**CPPs**	**OCCURRENCE**	**Detection methods**	**DETECTION**	**RPN**
Pump Plug				s settling based on and particle size butions lids content Agitation style Dirty feed tube Consistent %solids	Moisture Particle size Particle size distribution	Hydrolyze flow configuration System pressure Pump Speed	1 (3% solids) 3 (5% solids)	Software for pressure tracking and system shut off Visual and aural observation by trained operator	1	**8 (3% solids)** **24 (5%solids)**
Char Build-up in reactor	Downstream system plugs Product loss (30%) Reduction in residence time Reduction in reactor agitator efficiency Reactor temperature increase	Yield (Proc, Eco)	8	Poor operation of level control valve to remove char Flow properties of char compared to non-char slurry Material of construction of reactor Overcooking Cascading failure from pump failures	Extractives Flow properties (char) Particle size	Residence time Flow rate Heating	10	Level control detectors Software Manual detection	3	**240**

2 – Progress and Outcomes (Quarter 2) – Initial Pulp Baseline - INL

First Pass Pulp
“Crumbles”
(Discarded)

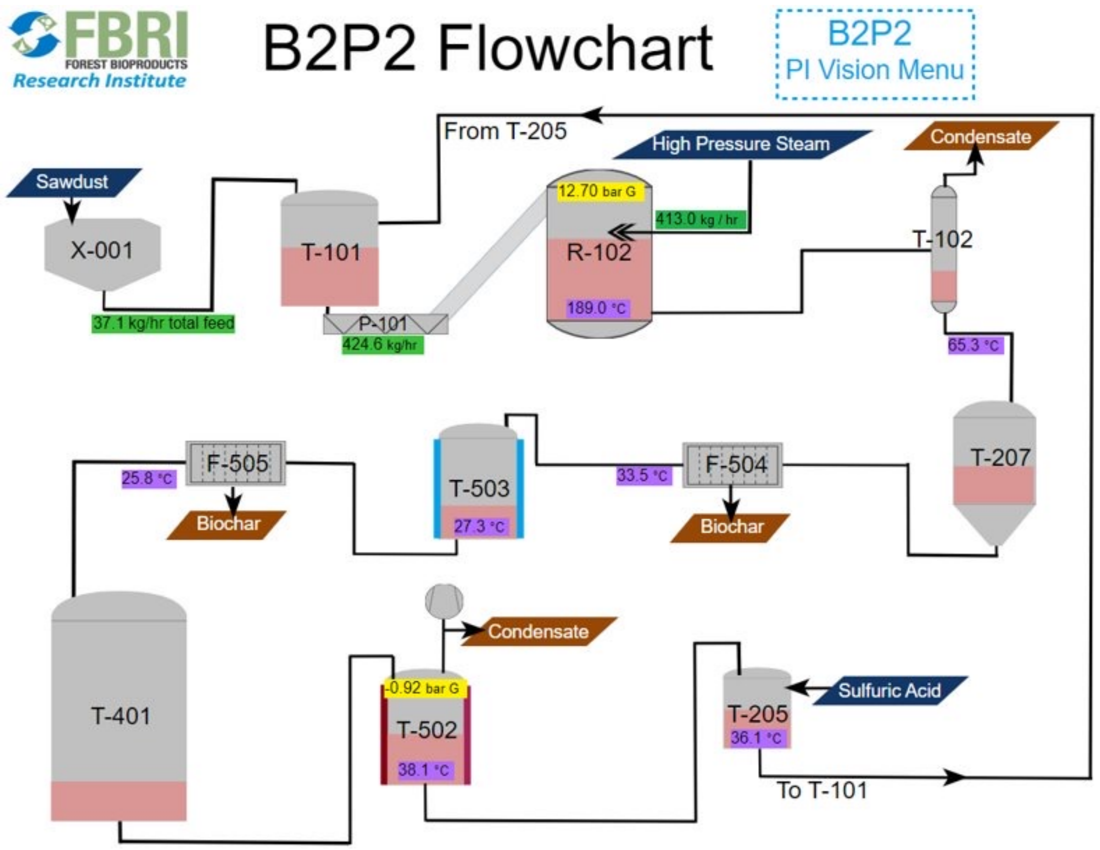
Knife Mill with
2” Screen

Crumbler On
Spec Material

Rejected
Crumbler Fines

Initial processing with the Crumbler and a mixture of knife milling and crumbling lead to a lot of strips of pulp and eventually plugged equipment. The **pulp processing greatly expands the properties tested in particle flow through these systems since it’s physical and chemical properties are vastly different than raw biomass**.

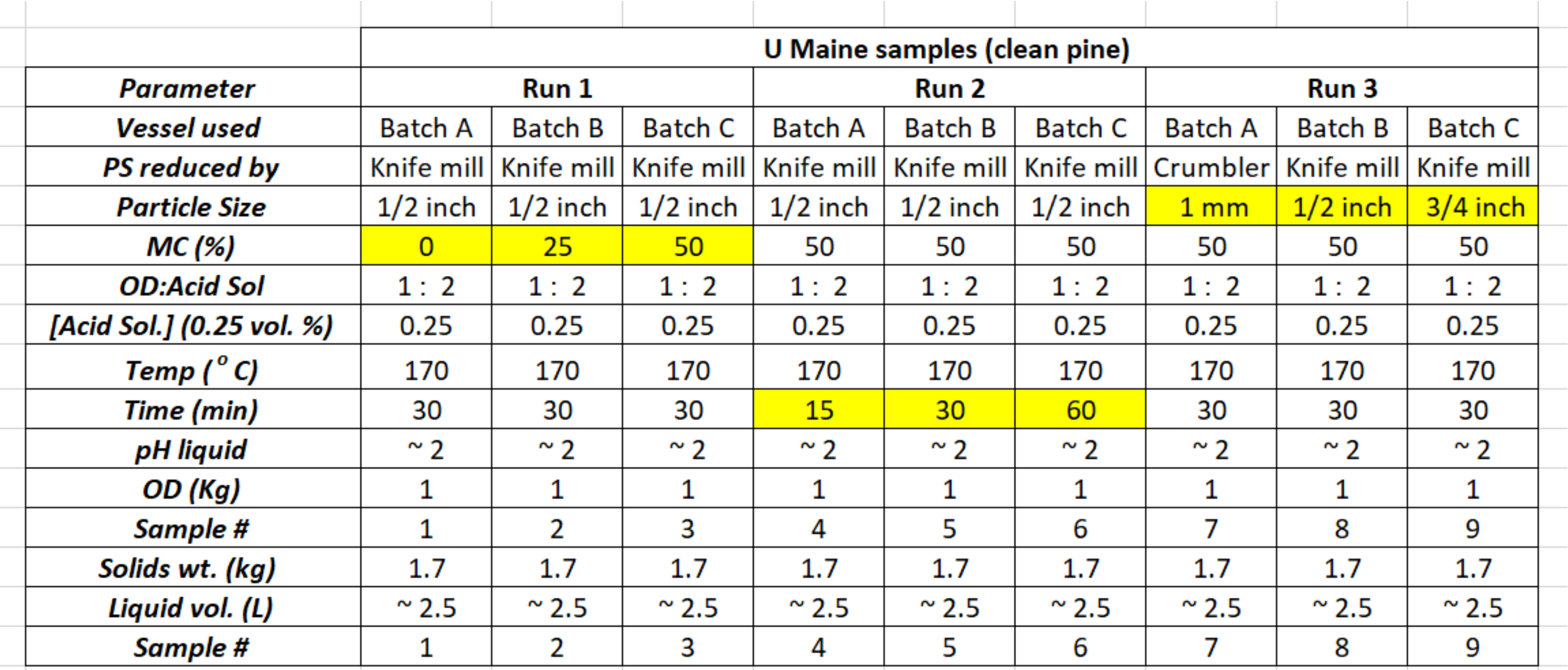
2 – Progress and Outcomes (Quarter 2) – Initial Pulp Baseline - UMaine



UMaine processed pulp at 5 wt% consistency for 19 hours without any flow aid additives. They performed compositional analysis of the pulp samples prior to processing it through their acid pretreatment step. **Process throughputs, as well as yields of levulinic acid, formic acid, and char were measured for pulp and will form the baseline for the rest of the project**.

2 – Progress and Outcomes (Quarter 3) – Batch Scale Screening Tests - INL

Variables investigated included particle size, residence time, initial moisture content, and temperature (not shown). *Solids conditioned with 0.25% H_2SO_4 in steam at 170 °C, and the resultant extract, have been sent to UMaine for screening and co-product development* through biological upgrading.



	U Maine samples (clean pine)								
Parameter	**Run 1**			**Run 2**			**Run 3**		
Vessel used	Batch A	Batch B	Batch C	Batch A	Batch B	Batch C	Batch A	Batch B	Batch C
PS reduced by	Knife mill	Knife mill	Knife mill	Knife mill	Knife mill	Knife mill	Crumbler	Knife mill	Knife mill
Particle Size	1/2 inch	1/2 inch	1/2 inch	1/2 inch	1/2 inch	1/2 inch	1 mm	1/2 inch	3/4 inch
MC (%)	0	25	50	50	50	50	50	50	50
OD:Acid Sol	1 : 2	1 : 2	1 : 2	1 : 2	1 : 2	1 : 2	1 : 2	1 : 2	1 : 2
[Acid Sol.] (0.25 vol. %)	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
Temp (° C)	170	170	170	170	170	170	170	170	170
Time (min)	30	30	30	15	30	60	30	30	30
pH liquid	~ 2	~ 2	~ 2	~ 2	~ 2	~ 2	~ 2	~ 2	~ 2
OD (Kg)	1	1	1	1	1	1	1	1	1
Sample #	1	2	3	4	5	6	7	8	9
Solids wt. (kg)	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
Liquid vol. (L)	~ 2.5	~ 2.5	~ 2.5	~ 2.5	~ 2.5	~ 2.5	~ 2.5	~ 2.5	~ 2.5
Sample #	1	2	3	4	5	6	7	8	9

2 – Progress and Outcomes (Quarter 3) – Batch Scale Screening Tests - UMaine

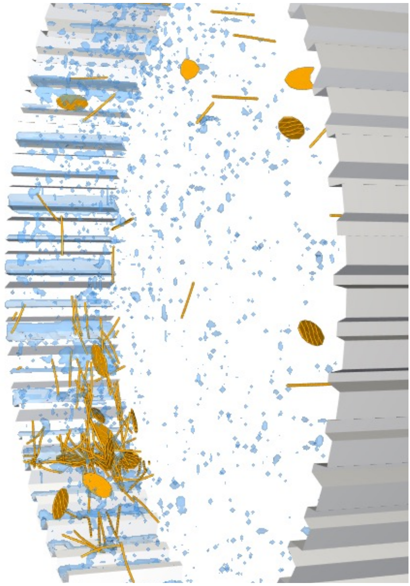
Interestingly, the initial moisture content had the greatest impact on reduced yields after acid pretreatment. Dry particles had undergone pore collapse that limited the effectiveness of the acid pretreatment but *particle size varying by over a factor of ten did not impact results*.

Sample	Glucan	Xylan	FA yield based on Glucan (g/g)	LA yield based on Glucan (g/g)	Furfural yield based on xylan (g/g)
0 (untreated)	39.5%	3.8%	0.15	0.26	0.29
1	37.6%	1.8%	0.13	0.37	0
2	43.8%	2.4%	0.12	0.30	0
3	43.3%	2.0%	0.14	0.38	0
4	45.6%	1.7%	0.12	0.33	0
5	44.6%	1.3%	0.12	0.34	0
6	44.7%	1.1%	0.13	0.33	0
7	43.7%	0.9%	0.15	0.37	0
8	45.4%	1.1%	0.15	0.36	0
9	43.7%	1.2%	0.15	0.38	0

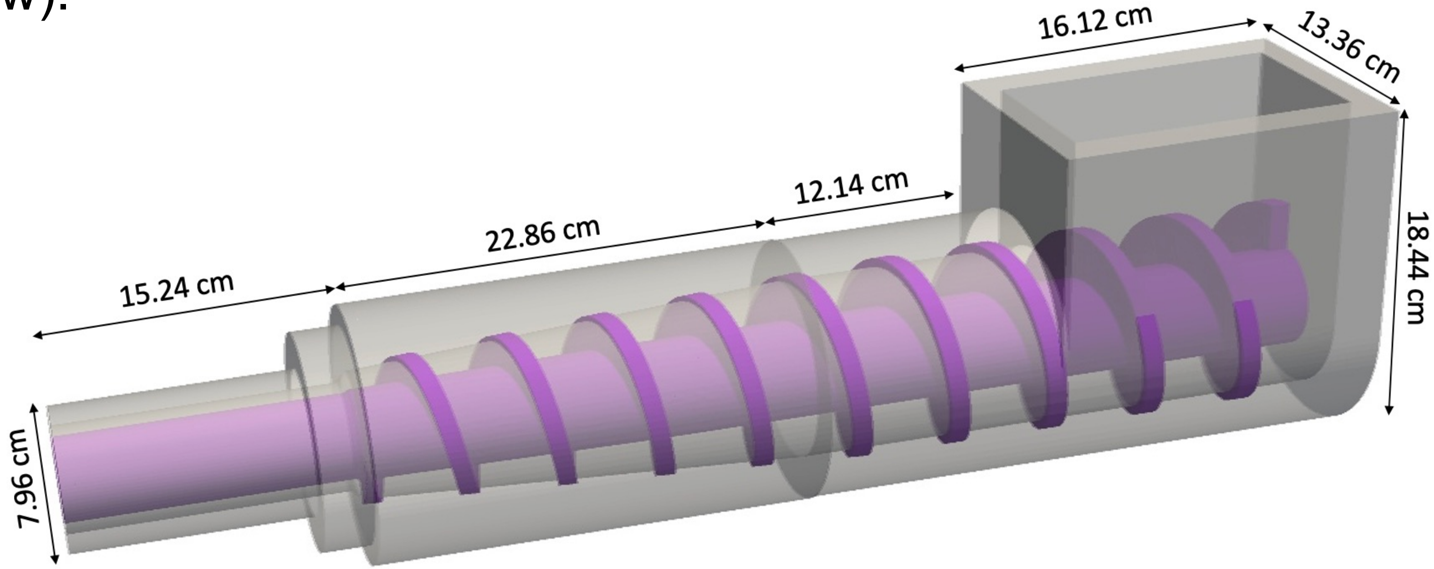
2 – Progress and Outcomes (Quarter 4) – Pilot Run of Thermally Fractionated Biomass - INL

Cold flow tests of three different particle sizes (1/2” knife mill, ¼” knife mill, and 1mm “Crumbles”) in the compression screw feeder that is part of the CPS have been started. These different particle sizes will also be run with at least two different moisture contents to study the impact of both attributes on mass throughput and energy requirements.

Discrete element modeling (DEM) of the CPS is also in its initial phases (digitally rendered feeder pictured below).

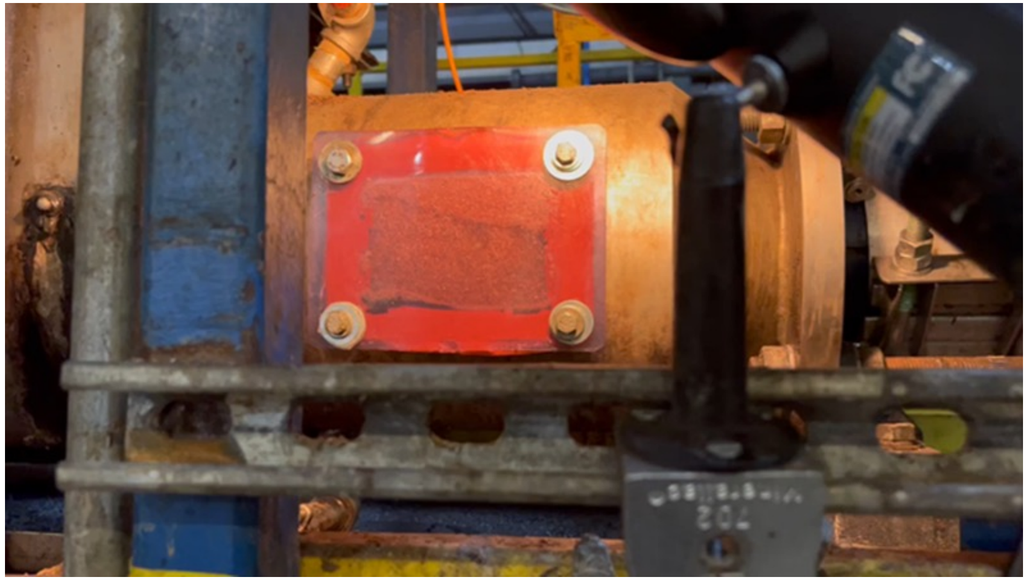


Application of advanced DEM model (from FCIC) with resolved particles (and optionally resolved water content)



2 – Progress and Outcomes (Quarter 4) – Pilot Run of Thermally Fractionated Biomass - UMaine

Cold flow test particles are identical to those tested in the CPS at INL but the UMaine team is testing them in a progressive cavity pump. These two independent tests will help gain knowledge about how identical particles behave in two different types of wet solids conveyance systems.



Pine sawdust has been run at UMaine with a solids loading up to 7% before needing addition of a flow aid additive.

3 – Impact –

1. Failure Modes and Effects Analysis combining historical information from the University of Maine B2P2 with the recent pulp baseline has provided a semi-quantitative approach for evaluating the effectiveness of particle formulation and thermal fractionation. *This has been a successful adaption of an accepted industry technique to a DOE project.*

2. A side-by-side comparison of two different types of solids transport systems (a compression screw feeder and a progressive cavity pump) *provides impactful data on equipment selection for any biorefinery processing lignocellulosic feedstock*. The Quality by Design (QbD) approach taken to evaluating flow characteristics in these systems will lead to more reliable pilot scale operability and *increase the chances of scale up success*.

3. Thermal fractionation of biomass to remove components that mitigate the formation of sticky chars and tars will also provide materials for other co-product pathways. This preprocessing step could be *applicable to any process that utilizes acid pretreatment.*

Summary

1. Historical information from the University of Maine, and a “baseline” run with kraft pulp, was used to inform a baseline Failure Modes and Effects Analysis and experimental standard.
2. Initial thermal fractionation tests showed the importance of not letting the woody feedstock dry too much in storage and that particle size has little importance on the effectiveness of a strong acid pretreatment step.
3. Cold flow tests have illustrated the difficulties of processing larger (>1/2” knife milled) particle sizes through compression screw feeders and progressive cavity pumps.

Quad Chart Overview

Timeline

- *Start Date: 04/01/2022*
- *End Date: 03/31/2024*

	FY22 Costed	Total Award
DOE Funding	$291,809	$3,000,000
Project Cost Share *	None	

TRL at Project Start: 4
TRL at Project End: 5

Project Goal

This project will turn two of the four woody biomass waste streams investigated at the bench and pilot scales into feedstocks with defined critical material and processing attributes for the AHDH/TDO pathway while meeting BETO's MFSP and GHG emissions targets.

End of Project Milestone

INL will thermally condition a half ton of hardwood feedstock for pilot scale testing at UMaine. Material and process attributes such as particle size particle, aspect ratio, and composition will be evaluated for effects on process throughput and yield in the AHDH preprocessing for TDO. Results will be compared with the pulp baseline.

Funding Mechanism

Annual Operating Procedure

Project Partners

- **University of Maine**
- **National Renewable Energy Laboratory**

*Only fill out if applicable.

Additional Slides

Responses to Previous Reviewers' Comments

This project has not been peer reviewed before.

Publications, Patents, Presentations, Awards, and Commercialization

1. Lessons Learned from De-Risking the Production of Fuel Intermediates from Forest Residues, AIChE November 2022, Phoenix, AZ
2. Low Temperature Hydrothermal Liquefaction of a Mixture of Lignin and Hemicellulose Derived Product, AIChE November 2022, Phoenix, AZ
3. Lessons Learned from De-Risking the Production of Fuel Intermediates from Forest Residues (Manuscript in preparation)
4. Effects of chemical preprocessing conditions on the wood derived organic acid yields and the char types (Manuscript in preparation)