STANDARD OPERATING PROCEDURE
STAN MAYFIELD PILOT PLANT

TITLE: BIOMASS PRETREATMENT

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A. Scope

Biomass feedstock will be processed using steam and acid to soften and prepare the fibers for subsequent hydrolysis by enzymes in the Liquefaction step. A small portion of the biomass sugars derived from the hemicelluloses portion of the biomass will be released into the liquid phase using a screw press. This SOP pertains to the proper methods of pretreating biomass.

B. Safety and Training Requirements

The pretreatment process operates at high temperature (~190 °C/374 °F), high pressure (~ 200 PSI), and low pH (~2.0).

CAUTION: High temperatures (~190 °C/374 °F)

CAUTION: High pressure (~200 PSI)

CAUTION: Acid (phosphoric acid, 2.5 – 5.0% under high pressure)

During operations in the plant, the following safety gear will be utilized at all times:
- Safety Goggles or Face Shield
- Protective Gloves
- Hard Hat

C. Related Documents and SOPs

1. Experimental Plan
2. Metso Operation and Controls Manual P-280029
3. Phosphoric Acid System SOP-8110
4. Biomass Storage and Handling SOP-1215
5. CO2 Scrubber Operation SOP-7210
6. Bleach Scrubber Operation SOP-7211
7. Beer Well SOP-4000
8. Dry Weight Measurement by Moisture Balance SOP-0503
9. Steam Supply SOP-9305
10. Process Water System SOP-9505
11. Air System Operation SOP-9405

12. Seal Water Pump Manual - Metso Maintenance Manual, Chapter 22
13. Sample Cyclone SOP-2115
14. Flash Tank SOP-2106
15. Lime System SOP-8405
16. Flash Steam Condenser SOP-2106
17. Refrigeration System SOP-9210
18. Biomass Liquefaction SOP-2325
19. Waste Water System SOP-9530
20. Sampling SOP-0511
21. Dry weight measurement by Moisture Balance SOP-0503

D. Preparation/Materials/Equipment

1. Phosphoric Acid Solution (2.5 – 5.0%)
2. Process seal water
3. Seal water pump PC-1205
4. Biomass
5. Instrument Air
6. Steam (High Pressure (250 PSI) & Low Pressure (30 PSI))
7. Biomass Sampling Containers
8. Lime Slurry if needed
9. Biomass Dump Bin
10. Biomass sample buckets 3 x 5 gal

E. Detailed Procedure

1. **Initial valve position** settings are given in the Table below:

Pretreatment				
Line	**Line Number**	**Valve**	**Position**	**Check**
Seal Water to Hydrolyzer Pkg	RCW-9501-51-SS10	1202 V-03	~~Closed~~	
		1202 V-06	Closed	
	Drain	1202 V-04	Open	
		1202 V-05	Open	
		1202 V-36	~~Open~~	
	LP water to seals	1202 V-30	Closed	
		1202 V-31	Open	
		1202 V-32	Open	
		1202 V-33	Open	
	HP water to seals	1202 V-24	Closed	
		1202 V-25	Open	
		1202 V-26	Open	
		1202 V-27	Open	
		1202 V-28	Open	
		1202 V-29	Open	

Process Water to Hydrolyzer	RCW-9501-11-SS10	2101 V-01	Open	
		2101 V-02	Open	
	PSF	~~HIC-2102-03~~	Closed	
		2101 V-08	Closed	
	Dump Chamber	2101 V-17	Closed	
	Flash Tank	2101 V-18	Closed	
	Dump Chamber	~~HV-2101-24~~	Closed	
Phosphoric Acid to Hydrolyzer	PHOD-8102-15-SS97	2101 V-03	Closed	
		~~FIC2102-02~~	Closed	
High Press Steam to Hydrolyzer	SM-9302-22-CS73	~~PIC2101-05~~	Closed	
	T-pipe	2101 V-04	Open	
	CV-2102	2101 V-09	Open	
		2101 V-11	Open	
		2101 V-13	Open	
Flash Steam to Condensor	DGFS-2101-01-SS10	2101 V-06	~~Open~~	
		2101 V-15	~~Open~~	
~~Sample Valves~~	Cyclone	~~Valve 1~~	Closed	
	Cyclone	~~Valve 2~~	Closed	
	Flash Tank	~~Valve 1~~	Closed	
Lime Slurry to Screw Press	LMS-8403-05-SS10	FIC-2106-02	Closed	
		2102-V16	Closed	
Press Drain	HYDS-2102-02-SS94	2102 V-13	Closed	
	To CV-2106	2102 V-14	Open	
	To C5 Storage	2102 V-15	Closed	

2. Services.

The following services are required to start the pretreatment operations:

i. Electrical power for drive motors, controls, etc.
   1. Press Skid I/O Panel ON.
   2. Disconnects for:
      a. CV-1204 on top deck
      b. BN-1202 on top deck
      c. CV-1202 on top deck
      d. ME-2101 on second deck
      e. PC-1205 on second deck
      f. CV-2101 on second deck
      g. FE-2102 on second deck
      h. CV-2102 on second deck
      i. CV-1201 behind Feed Bin
      j. CV-1203 behind Feed Bin
      k. BN-1201 behind Feed Bin

ii. Low pressure steam (30 PSI min) according to Steam Supply SOP-9305.
iii. High pressure steam (~200 PSI or as determined in experimental plan) according to Steam Supply SOP-9305.
iv. Process water according to Process Water System SOP-9505.
v. Seal water for low pressure seal applications including Flash Tank Agitator (AG-2102) and Screw Press Feed Screw (FE-2102).
    1. Open valve ~~9501-V-30~~ and verify that the pressure is 50 – 60 PSI on PI-9502-09 and PI-9502-08.
        a. Assure valves 9502-V-38 and V-39 are open to allow pressure to the pressure gauges.
        b. Should the pressure difference between PI-9502-09 PSI and PI-9502-08 be more than 10 PSI, replace the filter element according to Process Water System SOP-9505.
    2. Close valve 1202-V-36 in front of high pressure seal water pump (PC-1205).
    3. Open valve 1202-V-03 to provide water to the high pressure seal water pump.
    4. Open valves 1202-V-04 and V-05 to open the seal water drain lines.
    5. Open valve 1202-V-06 to provide water to the low pressure seal water header.
    6. Open valve 1202-V-31 to provide seal water to the Flash Tank Agitator (AG-2102).
    7. Open valve 1202-V-32 to provide seal water to the Screw Press Feed Screw DE (CV-2105).
    8. Open valve 1202-V-33 to provide seal water to the Screw Press Feed Screw NDE (CV-2105).
vi. High Pressure Seal Water for the High Shear Mixing Conveyor, Digester, and Digester Discharger mechanical seals.
    1. Open valve 1202-V-34 in line downstream from Hydrolyzer HP Seal Water Pump (PC-1205).
    2. ~~Open valve 1202-V-37~~ to supply water to pressure gauge PI-1205-01.
    3. Verify that spare valve 1202-V-24 is CLOSED.
    4. Open the following valves to seals:
        a. Valve 1202-V-25 for the High Shear Mixing oConveyor (CV-2102) DE seal.
        b. Valve 1202-V-26 for the High Shear Mixing Conveyor (CV-2102) NDE seal.
        c. Valve 1202-V-27 for the C5 Hydrolyzer (ME-2101) seal.

d. Valve 1202-V-28 for the Hydrolyzer Discharger (CV-1201) DE seal.
e. Valve 1202-V-29 for the Hydrolyzer Discharger (CV-1201) NDE seal.

vii. Phosphoric acid solution according to Phosphoric Acid System SOP-8110.
viii. Instrument air supply at 70 PSI according to SOP-9405.
1. ~~Open valves 9401-V-10 and 9401-V-14~~ to supply air to the Hydrolyzer Package.
2. Verify that the following spare valves are CLOSED.
a. 2" valves 1202-V-14 and V-23
b. 3/8" valves 1202 ~~V-08~~, V-12,V-13, ~~V-17, V-21~~, V-22
3. Open valves 1202-V-01 and 1202-V-02 to supply air to the two Hydrolyzer Package air headers.
4. Open the following supply valves in the Hydrolyzer Package:
a. 1202-V-07 to the pneumatic actuator valve FV-1202-07
b. 1202-V-09 to the pneumatic actuator valve PV-2101-05
c. 1202-V-10 to the sample valve of the C5 Hydrolyzer (ME-2101).
d. 1202-V-11 to the sample valve of the Hydrolyzer Discharger (CV-2101).
e. 1202-V-15 to the pneumatic actuator valve HV-2101-12.
f. 1202-V-16 to the pneumatic actuator valve HV-2101-13.
g. 1202-V-18 to the pneumatic actuator valve HV-2106-03.
h. 1202-V-19 to the pneumatic actuator valve HV-2106-04.
i. 1202-V-20 to the Screw Press (ME-2105).

ix. Flash Steam Condenser is ready for Blow Tank vent gas according to Flash Steam Condenser SOP-2106 (including CO2 Scrubber SOP-7210, Bleach Scrubber SOP-7211, Beer Well SOP-4000, and Waste Water SOP-9530)
x. Lime System is ready for neutralization of solids according to Lime System SOP-8405, if needed.

3. Biomass Delivery System

i. Begin filling the Feed Bin (BN-1201) with biomass according to Biomass Storage and Handling SOP-1215 so that the low level alarm (LAL-1201-02) is satisfied.
ii. On the HMI, start the Bagasse Feed Conveyor #2 (CV-1204), ensuring that the level in the Bagasse Presteam Bin (BN-1202) is not at the High High Level Alarm (LIC-1202-03).
1. The high high level alarm is interlocked with the Feed Conveyor CV-1201
iii. On the HMI, start the Bagasse Feed Conveyor #1 (CV-1201).
iv. On the HMI, start the Bagasse Feed Bin Collecting Conveyor (CV-1203).

v. On the HMI, start the Bagasse Feed Bin live bottom screws (BN-1201), ensuring:
   1. the Presteam Bin (BN-1202) level is not at High Alarm (LIC-1202-03),
   2. the Metal Dumper (X-1201) is closed (by visual inspection), and
   3. the Metal Detector controller XA-1201-07) is in automatic (green indicator on Metal Detector panel).

vi. Fill the Presteam Bin (BN-1202) to the normal operating level (between low and high level alarms). The High Level Alarm LIC-1202-03 will shut down the delivery system when the Presteam Bin (BN-1202) is full.

vii. Add steam to Presteam Bin (BN-1202).
   1. Verify that the manually operated access gate on the Presteam Bin (BN-1202) is CLOSED.

      CAUTION: Gate must be closed and locked to prevent exposure to steam

   2. Open valves ~~9302-V-15~~ and 1201-V-02 to supply steam to the unit.
   3. Open valve ~~1201-V-01~~ to direct steam to the pressure gauge PI-1202-06.
   4. On the HMI, slowly begin to add steam to the Presteam Bin (BN-1202) via FIC-1202-07 in manual mode by ramping the valve open.
   5. When the temperature reaches 180 °F on TIC-1202-08, switch to remote/auto on the HMI with a set-point of approximately 195 °F.
      a. The flow control valve FV-1202-07 will regulate the flow of steam to control temperature in the Presteam Bin (BN-1202).

4. Calibration of biomass feed system

i. Once the system has been fully charged with biomass according to Section E.3 above, the rate of biomass feed to the system must be calibrated.

ii. On the HMI, set the Steam Bin Transfer Conveyor (CV-1202) to REVERSE.

iii. Place a 5 gal bucket below the conveyor opening.

iv. Remove the cover from the conveyor opening.

v. On the HMI, start the biomass feed system at the final set-point on the master controller SIC-1202-08.

vi. Collect a full bucket of biomass and discard.

vii. Place a second bucket under the stream, record the time required to fill the bucket, and remove the bucket from the stream.

viii. Place an empty bucket under the stream.

ix. Stop the biomass feed using SIC-1202-08.

x. Determine feed rate in dry weight per minute according to Sampling SOP-0511 and Dry Weight Determination using Moisture Balance SOP-0503.

5. Hydrolyzer Start Up

i. On the HMI, select "Start Reverse" for the Screw Press Transfer Conveyor (CV-2106) to C6 Storage Bin with valve HV-2101-13 in the open position.

ii. On the HMI, start Screw Press (ME-2105)

1. Ensure that the C6 Storage Bin is in place.
2. Ensure that instrument air is supplied at 60 PSI at the local pressure gauge for the dynamic cone of the screw core.
3. Set cone pressure to 5 PSI.
4. Verify that the spare valve 2102-V-13 is closed.
5. Close valve 2102-V-15 to the C5 Storage Tank.
6. Close valve 2102-V-?? to drain.
7. Step here to close the drain that is not shown in PID (valves not numbered yet, I believe it is two valves).
8. Open valve 2102-V-14 to send hydrolysate to the Screw Press Transfer Conveyor (CV-2106).
9. On the HMI, set the Transfer Conveyor (CV-2106) to REVERSE to send material to the C6 Dump Bin.

iii. Start the Screw Press Feed Screw (CV-2105) by:

1. Ensuring that the low pressure seal water is at 60 PSI on the DE and NDE mechanical seals as indicated by PSL-2105-08, FISL-2105-07, PSL-2105-11, and FISL-2105-10.
2. On HMI, start Screw Press.

iv. Start the CS Flash Tank Agitator (AG-2102) by:

1. Ensuring the low pressure seal water is at 60 PSI on the mechanical seal as indicated by PSL-2102-22 and FISL-2102-21.
2. Enable the Dump Chamber by opening the top valve (HV-2101-12) and closing the bottom valve (HV-2101-13) as shown by limit switches HIC-2101-12 and HIC-2101-13.
3. On the HMI, start the Flash Tank Agitator.

v. On the HMI, start the HP Seal Water Pump (PC-1205).

vi. On the HMI, start the Hydrolyzer Discharger (CV-2101).

1. Ensure that the High Pressure seal water is at 30 PSI higher than the digester operating pressure (determined in Experimental Plan) on the DE and NDE mechanical seals as indicated by PSL-2101-19, FSL- 2101-18, PSL-2101-22, and FISL-2101-21.

vii. On the HMI, start the C5 Hydrolyzer (ME-2101).
    1. Ensure that the High Pressure seal water on the DE mechanical seal as indicated by PSL-2101-16 and FISL-2101-15.

viii. Start the High Shear Mixing Conveyor (CV-2102) by:
    1. Ensuring High Pressure seal water is 30 PSI higher than the digester operating pressure determined the Experimental Plan on the DE and NDE mechanical seals as indicated by PSL-2102-15, FISL-2102-14, PSL-2102-18, and FISL-2102-17.
    2. On the HMI, start the Mixing Conveyor.

ix. On the HMI, start the C5 Plug Screw Feeder (FE-2102).
    1. Ensure that the Blow Back Damper air is on and set at 60 PSI on PSL-~~1202-06~~
    2. Once the Plug Screw Feeder is running, the solenoid valve (HV-2102-03) will open to supply water to the Plug Screw Feeder hood.

x. On the HMI, start the Bagasse Presteam Bin Collection Conveyor (CV-1202).

xi. On the HMI, start the Bagasse Presteam Bin (BN-1202) live bottom screws (HS-1202-08) to begin the transfer of biomass into the system.
    1. Set the initial speed to 50% rate.
    2. Once the live bin screws are running, the following controls are set to AUTO:
        a. the Bagasse Presteam Bin level (LIC-1202-03),
        b. the steam to the digester and High Speed Mixer (PIC-2101-05),
        c. the Presteam Bin temperature (TIC-1202-05), and
        d. the flow rate of the phosphoric acid solution to the hydrolyzer (FIC-2102-02).

xii. As bagasse begins to leave the Presteam Bin and enter the hydrolyzer, set the pressure (on the HMI) to 80% of the final operating pressure set-point (typically 150 – 180 PSI) using PIC-2101-05

xiii. When bagasse begins to enter the hydrolyzer, set (on the HMI) the master controller on the Presteam Bin (SIC-1202-08) at approximately 80% of the production set-point. This will set the bagasse withdrawal rate from the Presteam Bin, the speed of the C5 Plug Screw Feeder, and control the amount of phosphoric acid solution used for pretreatment.

xiv. As biomass begins to exit the hydrolyzer,
    1. On the HMI, set the production rate to the final set-point on SIC-1202-08.
    2. On the HMI, set the hydrolyzer pressure controller (PIC-2101-05) to AUTO with the typical set-point of 167 PSI for a temperature of 374 °F (190 °C). The set-point will be determined by the process requirements.

xv. Vent non-condensable gases
1. Open vent valve 2101-V-15 for 1 to 2 seconds.
2. Close vent valve 2101-V-15.

6. The Hydrolyzer Operation.

i. Normal operating parameters:

1. The production rate of the Digester unit is set by the speed of the Presteam Bin live bottom discharge screws via master controller SIC-1202-08. This also controls,
   a. the phosphoric acid feed rate (FIC 2102-02), and
   b. the speed of the C5 Plug Screw Feeder (SIC 2102-04).
2. The pretreatment temperature is set by the hydrolyzer pressure controller PIC-2101-05.
   a. If the temperature and pressure deviate from standard saturated steam tables, vent the hydrolyzer by opening valve 2101-V-15 for 1 to 2 seconds to vent non-condensable gases.
3. The pretreatment time is controlled on the HMI by the speed of the digester screw rotation (SIC-2101-10), which will be approximately 5 - 10 minutes.
4. Run the unit for at least 30 minutes of steady state operation before taking the first sample of C5 Hydroysate.
5. Sample the C5 Hydrolysate according to C5 Hydrolysate Storage SOP-2135 to verify if it meets the initial quality specifications.
6. When the C5 Hydrolysate meets the required quality specifications,
   a. make sure that HV-2101-12 is in the open position, and
   b. select “Start Forward” on the HMI for the Screw Press Transfer Conveyor (CV-2106) in order to start the transfer of material to the Liquefaction Tank (VS-2301).
7. Begin the Liquefaction process according to the Biomass Liquefaction SOP-2325.

ii. Re-Start from Power Failure

1. Following an electrical power failure, it is assumed that all utility services are also lost and that all valves have failed in their designed failed safe position.
2. Operators should inspect all process equipment immediately and ensure the all manual isolation valves are set to the initial valve setting Table in Section E.1

3. Once the power is re-established:
    a. Confirm that all utilities are re-established as shown in Section E.2: air, water, and electrical power.
    b. When the control system is active, make sure that all the control valves are closed and in a safe position according to the initial valve settings table in Section E.1.
    c. Shut down any pumps or conveying equipment that may have started when the power was re-established until the process is fully understood. Keep any tank agitators running.
    d. For the Biomass Delivery System, follow the normal start-up procedure in Section E.5 from this point.
    e. For the Pretreatment :
        i. Re-start the Blow Tank agitator (AG-2102).
        ii. Re-start the system following the procedure outlined in E.5

7. Shutdown Procedures

i. Biomass Delivery System

1. On the HMI, stop the Bagasse Feed Bin live bottom screws (SIC-1201-06).
    a. Biomass can be left in the bin.
    b. The bin should be emptied after one week by:
        i. Open Metal Detector Dumper (X-1201)
        ii. Start Feed Bin Collecting Conveyor (CV-1203).
        iii. Start Feed Feed Bin live bottom screws (BN-1201)
        iv. Run until Feed Bin is empty
2. On the HMI, stop the Bagasse Feed Bin Collecting Conveyor (CV-1203).
3. On the HMI, stop the Bagasse Feed Conveyor #1 (CV-1201) when the conveyor is empty by visual inspection.
4. On the HMI, stop the Bagasse Feed Conveyor #2 (CV-1204) when the conveyor is empty by visual inspection.

The Biomass Delivery System is now shutdown.

ii. Hydrolyzer

1. On the HMI, stop the Bagasse Presteam Bin live bottom screws (SIC-1202-08).
    a. Empty the bin if outage is to be more than 48 hours.

   b. Shut the steam supply to the bin by switching FIC-1202-07 to MANUAL and closing valve 1201-V-02.
2. On the HMI, stop the Bagasse Presteam Bin Collection Conveyor (CV-1202) after the conveyor is empty.
3. On the HMI, stop the Plug Screw Feeder (FE-2102) and raise the Blow Back Damper pressure to maximum (?? PSI – to be determined at commissioning).
4. On the HMI, set the reversing screw to REVERSE to dump to the C6 Storage Bin using HV-2101-13.
5. On the HMI, stop the acid feed by setting the acid flow controller (FIC-2102-02) to MANUAL and ramping the flow rate to zero in 10% steps.
6. Empty the High Shear Mixing Conveyor (CV-2102) and turn it off.
7. Empty the C5 Hydrolyzer (ME-2101) and turn it off.
8. Empty the Hydrolyzer Discharger (CV-2101) and turn it off.
9. Run the Dump Chamber through the auto dump cycle, ensuring all pretreated biomass is through the system.
   a. Verify that the Hydrolyzer pressure is less than 50 PSI on PI-2101-07.
   b. Keep the Flash tank agitator (AG-2102) ON.
   c. Close the top Dump Valve (HV-2101-12).
   d. Open the bottom Dump Valve (HV-2101-13).
   e. On the HMI, set the Screw Press Transfer Conveyor (CV-2106) to REVERSE in order to send the rinse to the C6 Storage Bin.
   f. On the HMI, set the Recycle Water Valve controller (HS-2101-11) to AUTO.
   g. Open valves 1201-V-02, V-18, and V-17 to flush the Dump Chamber and Flash Tank.
10. On the HMI, turn OFF the steam supply to the hydrolyzer by closing PV-2101-05.
11. When the digester pressure falls to 0 PSI,
    a. retract the Blow Back Damper,
    b. run the Plug Screw Feeder empty, and
    c. stop the Plug Screw Feeder.
12. When the Dump Chamber and Flash Tank are empty,
    a. CLOSE valves 1201-V-02, V-17, V-18, and
    b. on the HMI, turn off the Flash Tank Agitator (AG-2102).
13. Empty the Screw Press Feed Screw (CV-2105).
14. Empty the Screw Press (ME-2105).

15. Empty the Screw Press Transfer Screw (CV-2106).
16. When the system is empty of material, turn off all equipment.

The Pretreatment and Washing Processes are now shutdown.

8. Emergency Stop of the Pretreatment Process

i. The pretreatment process contains an acid mixture under pressure and high temperature that can be dangerous. Therefore, it is necessary to have a procedure that will rapidly stop the process in an orderly fashion, depressurize the system, and cool off the material if required.

ii. The following steps should be followed if an immediate stop is required:

1. On the HMI, ramp the Master Controller SIC- 1202-08 down to zero rate. This will stop the flow of bagasse into the pretreatment system and cut the flow of phosphoric acid into the digester.
2. On the HMI, CLOSE the steam valve PV-2101-05 to stop the supply of steam to the pretreatment system.
3. Continue to run the High Shear Mixing Conveyor (CV-2102), Digester screw (ME-2101) and Digester Discharger (CV-2101).
4. Continue to operate the Dump Chamber normally until the digester pressure is below 50 PSI.
5. Activate the recycle water valve HV-2101-24 to flush and cool the Dump Chamber. Ensure that valve 2101-V-17 is open.
6. Monitor the hydrolyzer and Flash Tank pressure and temperature via PI-2101-07, TI-2101-08, PI-2102-11, and TI-2102-12 until safe conditions are reached (less than 5 PSI and less than 100 °F).
7. Flush and cool the Flash Tank by opening valve 2101-V-18
8. Continue to operate the equipment downstream from the Flash tank to Liquefaction to purge the system of any off-quality material.

Once the digester is depressurized and empty, repairs can be made.

TITLE: BIOMASS PRETREATMENT

Pretreatment Data Log

Batch Number:_____ **Biomass type:**_____ **Biomass Lot #:**_____

Date	Time of Day	Elapsed Time (h)	Temp (deg F) TI-2101-08	Pressure (PSI) PI-2101-07	Biomass Feed (lb/h)	Acid Feed (lb/h)	Acid Conc. (%)	Rxn Time (min)	Screw Press Pressure (PSI)	Cyclone Sample	Comment

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