

**UNIVERSITY OF FLORIDA  
BIOREFINERY PILOT PLANT  
PROCESS DESCRIPTION**

The following is a description of the process that will be used in the University of Florida's Biorefinery Pilot Plant that will be located at the Buckeye Technologies Mill in Perry, Florida. The pilot plant will process approximately 3 tons per day dry of biomass feed stock, primarily bagasse, by using a dilute acid-enzyme process, utilizing phosphoric acid as the acid.

**Area 1200 – Feedstock Feeding**

Trucks will deliver feedstock to the plant. Bagasse feedstock will arrive at the plant already milled to approximately 1" lengths, washed and ready for processing. There will be no feedstock processing equipment included in the project. The feedstock will be stored in outdoor covered concrete bins located on ground level.

A "Bobcat" type front-end loader will transfer feedstock into the feed bin, which will be sized for two hours of feedstock storage. A shed roof will cover the feed bin. A level indicator with low alarm will alert the operator that the bin needs to be filled.

A live bottom in the feed bin will feed the bagasse feed conveyor that will lift the feedstock from ground level to the top of the hydrolyzer and discharge it into the pre-steam bin that will allow for steaming of the feedstock. A magnetic separator and a non-ferrous metal detector included with the bagasse feed conveyor will remove any metals that could be harmful to the process.

The temperature of the feedstock will control the flowrate of low-pressure steam into the pre-steam bin by means of a temperature control valve, and a flow transmitter will measure and totalize the steam flowrate.

The pre-steam bin will be equipped with a load cell that will maintain a constant level of feedstock in the bin by controlling the speed of the bagasse feed conveyor.

A live bottom in the pre-steam bin will feed the feedstock into the pre-steam bin transfer conveyor, which will feed the C5 hydrolyzer.

**AREA 2100 - HYDROLYZER**

The hydrolyzer partially converts the hemicellulose portion of the feedstock to soluble sugars, primarily xylose and oligomers, and the cellulosic fraction becomes more susceptible to further enzymatic hydrolysis. The use of dilute phosphoric acid, high temperatures and residence time accomplishes this conversion. Using high-pressure saturated steam generates the high temperatures.

Acetic acid, furfural, and hydroxymethyl furfural (HMF) are the by-products formed in hydrolysis, and process conditions minimize their formation.

**Hold Hydrolyzer description until vendor selection is made.**

**Following the hydrolysis step, the hydrolyzate is flash cooled, which removes a significant amount of water, a portion of the acetic acid, furfural and HMF. It is beneficial to remove these by-products, as they are fermentation inhibitors. The flash steam is condensed and sent to waste water tank. During normal operations the entire flashed hydrolyzate stream will by-pass the screw press and will go directly to the liquefaction tank. The flashed hydrolyzate is cooled and diluted with process water before entering the liquefaction tank.**

**When it is necessary to produce C5 rich material for use in propagators 1 and 2, the flashed hydrolyzate is fed to the screw press where a solid C6-rich stream is separated from the liquid C5- rich stream. The C5 rich stream goes to storage and the C6 rich stream is collected in a dumpster. Before going to the dumpster the C6 rich stream is cooled with process water and neutralized using slake lime.**

Liquid material from the screw press will flow by gravity into the C5 storage tank for further processing. The screw press transfer conveyor will transfer thickened material from the screw press to the liquefaction tank.

The C5 Storage Tank will store the C5 liquid from the screw press. The Process Water Pump will pump process water into the tank, and the C5 Storage Tank Agitator will continuously mix the tank contents. The C5 Pump will pump the tank contents to the C5 pH Adjustment Tank.

Aqueous Ammonia Metering Pump #2 will meter aqueous ammonia into the C5 pH Adjustment Tank to control the pH of the liquid. The Mix Tank Pump in the Propagation and Fermentation Area will pump nutrients such as trace metals and magnesium sulfate needed for propagation into the tank, and the Process Water Pump will deliver process water to the tank for dilution. The C5 pH Adjustment Tank Agitator will continuously mix the tank contents.

The C5 pH Adjustment Pump will pump the tank contents through the C5 pH Adjustment Filter to the Propagators 1 and 2. A side stream from the pump discharge will continuously recycle into the C5 pH Adjustment Tank, and the pH transmitter used to control the feed rate of aqueous ammonia into the tank will be located in this recycle line.

**AREA 2300 – LIQUEFACTION**

In the Liquefaction Tank, the addition of an enzyme (cellulase) combined with high temperature and residence time will convert the cellulose in the feed stream to glucose. Higher temperature will be used in the liquefaction step than in the fermentation step to

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University of Florida  
Pilot Plant FEL 3  
FB&D Project No. Y7275

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take advantage of the increased activity of the enzyme and to reduce the time necessary for liquefaction/saccharification.

The Cellulase Metering Pump will feed cellulase into the tank. Aqueous Ammonia Metering Pump # 1 will feed aqueous ammonia into the tank to adjust the pH. The Liquefaction Tank Agitator will continuously mix the tank contents.

The Liquefaction Tank will be fully jacketed to allow for heating with low-pressure steam and cooling with chilled water. The tank will vent to the Beer Well.

The Liquefaction Tank Pump will transfer the tank contents through either Liquefaction Cooler #1 or #2 to the Hydrozylate pH Adjustment Tank. A level control valve in the pump discharge piping will control the level in the Liquefaction Tank.

Liquefaction Cooler # 1 will be a shell and tube heat exchanger, and Liquefaction Cooler # 2 will be a plate and frame heat exchanger. Chilled water will cool the liquid as it passes through the coolers.

The Mix Tank Pump will feed nutrients such as trace metals and magnesium sulfate into the Hydrozylate pH Adjustment Tank. Aqueous Ammonia Pump #3 will feed aqueous ammonia into the tank for pH adjustment. A pH transmitter in the discharge line from the tank will control the speed of Aqueous Ammonia Pump # 3.

The Hydrozylate pH Adjustment Tank will be fully jacketed to allow for cooling with chilled water. The tank will vent to the Beer Well.

The Hydrozylate pH Adjustment Tank Pump will transfer the contents of the tank Propagator 3 and to the Fermentors. A level control valve in the pump discharge line will control the level in the Hydrozylate pH Adjustment Tank.

The Liquefaction Tank, the Hydrozylate pH Adjustment Tank and the Liquefaction Coolers have provisions to be cleaned in place (CIP'd).

### **USE TEXT BELOW IN CHEMICAL STORAGE AREA WRITEUP.**

**The cellulase enzyme used in this step will be handled in drums or tote bins and charged using a metering pump. The hydrolyzate, which is now much less viscous due to the enzymatic reaction, is transferred through a cooler to the pH adjustment tank.**

### **AREA 3200 – PROPAGATION & FERMENTATION**

The Propagation and Fermentation area will include one mixing tank, three propagator trains, three fermentors and associated pumps and auxiliary equipment. The train will ferment both C5 and C6 materials individually or together.

The fermentation train consists of Mix Tank, Propagators 1A&B, 2A&B and 3A&B and Fermentors 1,2 & 3. The propagators will be jacketed vessels with agitation and sterile air sparging capabilities. The fermentors will be jacketed vessels with sterile air sparging and provisions for venting carbon dioxide.

Propagators 3A&B and Fermentors 1,2 & 3 will ferment the entire stream from the hydrolyzer, and Propagators 1A&B and 2A&B will ferment only C5 liquid. The propagators will transfer their contents to each other and to the fermentors by air pressure and/or gravity. The purpose of the Propagators will be to grow specially designed bacteria (microorganisms), and each propagator will be approximately ten times the volume of the previous one until the volume of the final propagators will be large enough to support the production fermentation.

**ADD LATER IN THIS SECTION:**

The mixing tank will be arranged for either bag addition at the tank platform, for ease of adding bags, or drum/tote addition from the drum unloading stations. A diaphragm pump will be used for transfers from the mixing tank. The propagators and fermentors will be jacketed vessels with agitation, carbon dioxide removal, sterile air sparging capabilities and will have facilities for CIP and SIP.

The Mix Tank Pump will feed the initial seed inoculum and nutrients into Propagators 1A&B. Base B Metering Pumps will feed a basic chemical into the propagators for pH control, and the C5 pH Adjustment Pump will deliver C5 filtrate to them.

The seed inoculum grown in the Propagators 1A&B will be used as the seed inoculum for the Propagators 2A&B. Air pressure will transfer the contents of Propagators 1A&B to Propagators 2A&B. Pumps will deliver nutrients, base, and C5 filtrate to the propagators, and the batch will be held for its propagation cycle.

The second propagation batch will be used as the seed inoculum for Propagators 3A&B. In this case, flashed hydrolyzate will be added with the base and nutrients instead of the C5 liquid. At the end of this propagation cycle, the batch will be large enough to support fermentation.

The propagators will all be straight batch propagators, meaning that all ingredients will be added together and the "clock" starts when the tank is full. Base addition will be available for any pH adjustments.

Like Propagators 3A&B the fermentors will use flashed hydrolyzate, however the fermentors will be "fed batched". Fed batching to the fermentors includes the initial charging of a heel by "pitching" the last propagator into the fermentor and then gradually adding the hydrolyzate during the first twenty-four hours of fermentation. After filling the fermentors are held for additional time to complete the fermentation process. When the fermentation cycle is complete, the resulting ethanol broth (beer) will be transferred to the beer well.

Base addition is available to the fermentors for any pH adjustments. The fermentation step generates carbon dioxide and heat. Heat is normally removed by cooling water in the jacketed however, each fermentor will have the capability of connecting its recirculation loop to an external plate and frame heat exchanger for cooling the fermentation broth during the fermentation process. Carbon dioxide is vented along with any other gases to the beer well.

### **Beer Well & CO<sub>2</sub> Scrubber**

The beer well is a collection tank for the batch fermentors and helps make the transition from a batch process (fermentation) to the continuous distillation process. It also acts as temporary containment for vapor and liquid from the propagators and fermentors vents, which contain mostly carbon dioxide and some ethanol but could potentially carryover the microorganism that cannot be released to the atmosphere.

After leaving the beer well the beer is heated in the beer preheater before going to the beer stripper. Any microorganisms in the beer can be killed at this step if the beer is going straight to the decanter and bypassing the stripper. The beer heater uses stripper bottoms as the heating medium but if this is unavailable steam can also be used.

Carbon dioxide vented from the beer well is first scrubbed in a water scrubber , which recovers most of the ethanol. The scrubbing liquid is pumped to the beer well. The "cleaned" carbon dioxide is then vented to a second scrubber, which uses a bleach solution as the scrubbing liquid. These scrubbers function as the vapor containment system for the microorganism. The spent bleach solution is sent to waste treatment and the clean carbon dioxide to the atmosphere.

The beer well can be CIP'd as needed.

### **Stripper & Lignin Separation**

The distillation and dehydration process equipment will be used to separate the ethanol from the fermentation beer producing 200 proof ethanol. Distillation is accomplished using a two-column system. The first column is the stripper or beer column in which the dissolved carbon dioxide, most of the water and solids (mostly insoluble lignin) are separated from the ethanol. The stripper column bottoms before being fed to the lignin separation step are used to preheat the beer before it is fed to the stripper column. Steam is used to drive the stripping column. As mentioned above, the stripper column also functions as the microorganism kill step.

Insoluble lignin and phosphates are removed from the stripping column bottoms stream by a combination of decanting. The first decant is completed using a decanting centrifuge which removes the lignin cake which is collected and either land filled or sold for its fuel value. The filtrate from the decanting centrifuge is treated in the phosphate conversion tank with lime to form calcium phosphate, which is then removed by settling in the nutrient recovery tank. The calcium phosphate settles to the bottom of the nutrient

recovery tank, is recovered and will be used as a fertilizer. The top layer from the nutrient recovery tank is further treated to remove phosphates on an as needed basis. It is finally sent to waste water tank.

### **Rectification & Dehydration**

The stripper column overheads are fed to the rectification column, which removes water leaving an ethanol concentration at or near the azeotropic mixture at the column overheads. The overheads from the rectifying column are split, part is condensed and returns as reflux, the remaining is sent to the molecular sieves for further dehydration to 200 proof ethanol. The bottom of the rectifying column functions as a stripper to remove any ethanol in the bottoms stream. The rectifying column bottoms are then sent to the recycle or process water tank.

All the water from the nearly azeotropic mixture from the rectifying column overheads is removed by vapor phase molecular sieve adsorption. Regeneration of the adsorption vessel beds requires that an ethanol water mixture be recovered and recycled back to the rectifying column.

Steam is used to drive the rectifying column and to superheat the feed to the molecular sieves.

### **Ethanol Storage & Loadout**

200 proof ethanol is condensed and is transferred from the molecular sieves to the product shift tank. The 200 proof ethanol is denatured by adding gasoline as the denaturant. Denatured ethanol is loaded into trucks from the product shift tank.

### **Chemical Handling**

Most process chemicals used in the process will be handled in totes. Two drum or tote unloading stations will be provided, one for acidic material and one for basic materials. These stations will include a diaphragm pump, weigh scales and spill containment. Chemicals handled will include phosphoric acid, various enzymes and nutrients, aqueous ammonia, a second base and caustic. Nutrients can be passed through a sterilizing loop before being charged to a propagator or fermentor.

Aqueous ammonia is used as a base and as a nutrient. It will be handled from totes. Provisions have been made to handle a second base (possibly KOH) in a separate tote unloading system.

Slaked lime will be used in the process to neutralize the excess phosphoric acid. Bagged quicklime will be dumped into a slaker tank for mixing and producing slaked lime. The slaked lime transfer pump will be a centrifugal pump. A platform will be provided over the slaker tank for ease of dumping lime.

Concentrated phosphoric acid from totes is diluted using an inline mixer. The phosphoric acid will be diluted with process water. Dilute phosphoric acid will be transferred using a metering pump.

### **Utilities**

Utilities requirements for the plant include, steam, chilled water, cooling water, plant and instrument air, process water, and clean-in-place (CIP) system.

Steam to the pilot plant will be provided from the Buckeye main plant. The steam supply will be high pressure, which will be reduced to the required pressures for the individual users. Steam is utilized in the following areas: 1) hydrolyzer, 2) pre-steamer, 3) stripper column, 4) rectifier column, 5) mole sieve superheater, 6) CIP system, 7) C5 and nutrient sterilizers and 8) propagators and fermentation for sterilization (SIP).

A packaged chilled water system will be used to cool process water to replace a separate cooling tower. Cooling water is utilized in the following areas: 1) C6 cooler after liquefaction, 2) scrubber cooler, 3) fermentor external cooler, 4) propagator and fermentor jackets, 5) mole sieve regen condenser, 6) reflux condenser, 7) mole sieve ethanol condenser, 8) C5 cooler, and 9) C5 and nutrient sterilizers. Chilled water is used on the flash steam condenser.

Plant and instrument air system will include a sterile air system including filters for the propagators and fermentors.

Wherever possible process water will be recycled for reuse in the process. A process water tank will be provided including a city water line for make up. The process water pump will be a centrifugal pump. The process water system includes a shell & tube cooler and a sterilizing UV.

The CIP system will have two tanks, one is the dilute caustic tank and the other is a rinse tank. 50% caustic will be received in totes and diluted in the dilute caustic tank. The CIP supply pumps will be centrifugal pumps, and the return pumps will be air diaphragm pumps. CIP, as well as SIP (Steam in Place), will be utilized in the propagators, and fermentors after every batch and periodically in other tanks and vessels

Fire water will be provided by the Buckeye system and used in the sprinkler systems in the distillation area.

### **Waste Streams**

A wastewater tank will be provided for storing liquid wastes that will be sent to Buckeye's wastewater treatment facility utilizing a wastewater pump. Solids wastes streams will be collected in movable dumpsters.