SWEET SORGHUM BIOETHANOL PRODUCTION

RESEARCH, DEMONSTRATION, AND

OPERATIONAL ISSUES

By

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Safety First!

Onward and Upward…

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Title of Study: SWEET SORGHUM BIOETHANOL PRODUCTION: RESEARCH, DEMONSTRATION, AND OPERATIONAL ISSUES

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Abstract: Sweet sorghum or *Sorghum bicolor (L) moench* is sugar rich, grows with minimal inputs, and is suitable for cultivation in dry regions making it a desirable energy crop for Oklahoma. In-field fermentation of sweet sorghum reduces storage and transportation hurdles of the biofuel supply chain. For the overall success of the on-farm approach, the ethanol dewatering step is crucial. The Alcohol Separation Unit (ASU) at Oklahoma State University is a research and demonstration prototype unit, the first of its kind to study bioethanol production from fermented sweet sorghum liquid feedstock. A two-column distillation set up for concentrating ethanol from 250 gal/h liquid fermented sweet sorghum feed, with associated heat exchangers, pumps, and storage units was designed and constructed for on-farm ethanol separation. The ASU was commissioned in Fall 2013. Product ethanol (190 proof) was produced in October 2013. Ethanol purities of 193.6 proof at 74^{o}F with rectifier column conditions of 0.8 psig and 171^{o}F were established during processing. Overall economics of the pilot plant indicate fixed capital costs at 403,000 USD. Major unit operation equipment (columns, heat exchangers, storage tanks) makes approximately 50% of the costs. Major energy usage at the pilot plant for ethanol production is steam, which accounts for 75% of total annual operating costs of 68,000 USD.

Experiments to quantify sweet sorghum fermented juice fouling were undertaken to understand potential operational issues. Total solid loading of fermented juice was determined to be approximately 3 wt%, significantly lower compared to corn-based process streams. Heat transfer resistance (R_f) of fermented sweet sorghum was found to be approximately 9.25 x 10^{-5} m^2 ^{o}C/W and fouling intensity is equivalent to that of tap water. Fermented sweet sorghum R_f values were found to be one tenth of corn and maize ethanol thin stillage fouling resistance. The induction period for fermented sweet sorghum fouling was more than 5 hours. With a significantly lower fouling resistance value and longer induction times for fermented sweet sorghum, fouling in equipment and the frequency of cleaning in processes is expected to be lower. Using sweet sorghum as a bioethanol crop is then advantageous over starch-based feedstocks.

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CHAPTER 1

INTRODUCTION

1.1 Background and Motivation

In a changing energy landscape, diversification, use of alternative or sustainable forms of fuel, and move to efficient processes is no longer a choice for governments, organizations, businesses, or individuals. In United States renewable energy consumption including solar, wind, biofuels, hydrothermal, and geothermal energy was 8.620 quadrillion Btu in 2013 and ethanol accounted for 13% of this energy utilization [1]. Liquid biofuel (ethanol and biodiesel) production is estimated to average 1,009,000 bbl/day in 2014 according to EIA estimates, retaining an increasing trend. These numbers indicate a continued increasing presence of ethanol as an alternative liquid fuel.

The Renewable Fuel Standard (RFS) has facilitated this surge in renewable fuels – as part of the mandate approximately 10% of non-renewable fuel produced in the US needs to be from alternative sources [2]. Apart from total renewable fuel production targets, specific fuel sub-categories such as cellulosic, bio-based biodiesel, and advanced feedstocks have individual volume targets. As per RFS2, mandate for advanced feedstocks grows from 1 billion gallons in 2010 to 21 billion gallons of ethanol by 2050. The mandate for cellulosic ethanol grows from 100 million gallons in 2010 to 16 billion gallons in 2022 [3]. Cellulosic ethanol is produced from lignocellulosic material and covers switchgrass, miscanthus, wood chips [4]. Advanced feedstocks on the other hand include all non-cellulosic non-corn sugar and starch feedstocks [5].

Within these non-corn starch and sugar feedstocks, sweet sorghum stands out as a suitable option for bioethanol fuel manufacture.

Sweet sorghum or *Sorghum bicolor* (L) Moench is sugar rich, like sugarcane, with sugars concentrated in the stalk [6]. Sweet sorghum grows with minimal nutrient addition [7-9], requires limited quantities of water (12,000 m^3/ha which is a third of the water required for sugarcane) [10], and is suitable for cultivation in dry regions [11]. These characteristics make sweet sorghum well suited for Oklahoma. Sweet sorghum also provides additional carbohydrate sources – bagasse from stalks, leaves, and grains are all sources of additional energy and can produce between 25 to 75 tons/ha biomass [12-14].

Carbohydrates produced by sweet sorghum are six-carbon sugars that are directly fermentable to ethanol. These carbohydrates are present in the juices recovered when harvested sweet sorghum is pressed. The difficulty in using sweet sorghum for ethanol production is due to the fact that the freshly pressed juice is not readily storable. In chopped sweet sorghum glucose levels fell to 0 g/L, two days after harvest [15]. The juice must be fermented immediately to prevent conversion of the sugars to non-ethanol products by naturally occurring bacteria [16]. Co-locating fermentation and sweet sorghum harvesting provides a solution for minimizing loss of sugars. Concern about transporting dilute watery sugar rich solutions, or ensilage of the harvested crop is also mitigated with an on-farm approach [17].

An on-farm approach to bioethanol production from sweet sorghum, as suggested by researchers at the School of Biosystems and Agricultural Engineering and discussed by Kundiyana et al. [18] using sweet sorghum at smaller scale decentralized units removes the storage and transportation hurdle by fermenting sweet sorghum in-field. Their study found even with minimal pH, temperature control and nutrient addition, up to 95% sugar to ethanol conversion efficiencies could be achieved. For the overall success of a small-scale on-farm unit though, in addition to the in-field fermentation step, the downstream separation step to remove ethanol from the dilute post-fermentation process stream is crucial. Without a robust, economical, and safe ethanol separation process the success of on-farm bioethanol production can be compromised.

Initial design efforts reconfirmed that distillation remains the preferred dewatering technology for concentrating fermentation product (6 – 10 vol % ethanol) to near fuel grade, azeotropic (95 vol %) quality [19]. In the course of this technical evaluation of on-farm distillation, a rigorous first-principles simulation with associated capital and operating cost models was developed. Results from these models were incorporated in a comprehensive economic model of the sweet sorghum ethanol process [20]. The model establishes the potential economic viability of this process and indicates that the capital and operating cost of the dewatering system are among several key variables with a major impact on profitability.

On-farm distillation trials were common in late 1970s in United States. The energy crisis of the time motivated research into use of ethanol as a fuel substitute. The U.S. Department of Energy (DOE) and the U.S. Department of Agricultural (USDA) sponsored research into ethanol production from renewable sources, specifically on a small scale [21-24], and demonstration units followed suite [25-29]. In 1983 White wrote in a DOE report [30] that problems plaguing small scale ethanol facilities were due to "poor technical advice and inadequately-proven plant designs." He also noted in this report that "the distillation columns and associated equipment represent a major fraction of plant capital costs and consume a large portion of plant energy."

On-farm ethanol work from 1970's and 1980's was never concluded. Research funding for alternative fuels dried up after the decline of oil prices in 1982. Current efforts can be seen as resumption of previous efforts, but now with 30 years of additional experience and technology updates. Distillation and heat transfer technology have advanced significantly in the past 30 years. Technological advances appear theoretically adequate to provide the required technology updates.

However, to justify investment by the agricultural community, there is a need to demonstrate and document the cost of actual ethanol separation on a farm-scale. As Wynman emphasizes [31], process operation “presents constant opportunities and incentives for improvement, debottlenecking and innovation that cannot be duplicated in the laboratory or through paper studies.”

Anticipated costs include fixed capital costs for equipment and installation in addition to costs incurred for operation and maintenance for an ethanol separation facility built around sweet sorghum feedstock. Strong linkage between operation and maintenance were expected, based on the fouling tendencies observed in corn-based processes. Distillation byproducts from corn ethanol plants are concentrated in multiple effect evaporators and recombined with unfermented solids to create Distiller’s Dried Gains with Solubles (DDGS) [32]. Maximum observed resistance to heat transfer or fouling resistance of thin stillage streams from corn dry grind processes range between 0.2 to 0.35 m^2 K/kW, and indicate a high level of deposition [33-35]. Deposits are mixed inorganic/organic and can require up to 20 hrs of chemical cleaning [36], and need a rigorous Cleaning in Place (CIP) system.

Sweet sorghum process fluids have so far been studied mainly from a pre-fermentation perspective, as feed for food-based products [37] and process-related studies, specifically related to fouling are not present in literature. Without a clear picture on the extent of fouling, cleaning operations plant schedules including shut down procedures can’t be set in place. All of these steps will eventually impact the profitability of an on-farm ethanol facility – fouling monitoring at the demonstration facility, coupled with experiments to quantify, and model sweet sorghum fermented juice fouling will provide new valuable information critical for future development of a bioethanol process based on sweet sorghum. Identification of the fouling characteristics of sweet sorghum-based fermentation broth is a critical outcome.

1.2 Objectives

This doctoral investigation sought to demonstrate feasibility of sweet sorghum bioethanol production on a farm-scale from a technical, economical, and maintenance perspective by constructing and operating the Alcohol Separation Unit (ASU), a farm-scale demonstration facility. This investigation illustrates farm-scale, sweet sorghum-based bioethanol operation and maintenance concerns, specifically equipment fouling, by characterizing and quantifying fouling tendency of the feedstock. Specific objectives were:

1. Document and evaluate the design and cost of farm-scale bioethanol dewatering using best-available distillation technology. Detailed records were maintained to document true operating costs and energy usage for feeds varying between 6-10 vol % ethanol with the ASU run in research mode (all equipment and instrumentation, best case scenario). This scenario serves as the benchmark for future research (minimal equipment and instrumentation). Safety and the environment were first priorities in ASU operation. Operating and maintenance guidelines were consistent with accepted industrial practices.
2. Characterize sweet sorghum fermented liquid for its fouling potential. This was done by quantifying solid content in the feed stream, examining deposits, and determining transport properties of fermented sweet sorghum.
3. Determining fouling resistance of fermented sweet sorghum liquid. Deposition experiments under controlled hydrodynamic conditions using a heated test section were used to quantify the fermented sweet sorghum resistance to heat transfer. Two independent sets of experiments were conducted - the first apparatus described in detail was constructed at OSU and the second set of experiments was conducted using an annular flow fouling set up used by Challa *et al*. [38].

1.3 Structure of Dissertation

The dissertation is divided into five chapters. The first chapter is the Introduction. The following chapter, "Alcohol Separation Unit: Research and Demonstration of on-Farm Sweet Sorghum Bioethanol Production at Oklahoma State University" was submitted as AIChE Annual Meeting, 2014: 285e. The purpose of the work was to document bioethanol production from sweet sorghum from a first-of-its-kind farm-scale pilot plant, capable of producing 250 gal/h feed liquid feed. The Alcohol Separation Unit serves as both a research and demonstration unit. The facility has two distillation columns with Sulzer Chemtech internals, GEA Rainey air-cooled heat exchanger, stainless steel piping and auxillary equipment. The facility has successfully produced its intended product, 190 proof ethanol. The rectifier conditions at product purity were 0.8 psig and 171^{o}F. Fixed capital expenditure, based on actual spending, for the pilot facility are expected to be 403,000 USD. Operating costs were evaluated to be at 68,000 USD/year. Safety considerations have also been discussed in detail in this paper.

Chapter 3 is titled "Fouling Potential of Bioethanol Process Feed – An Analysis of Fermented Sweet Sorghum Solid Content". The work was intended to characterize fermented sweet sorghum for its fouling potential by quantifying its solid content and establishing its transport properties. National Renewable Energy Laboratory (NREL) Laboratory Procedures (LAP) [39] was used to establish total solid and dissolved solid content in fermented sweet sorghum. Standard density and viscosity determining techniques were used to establish transport properties. The total solid content in fermented sweet sorghum was less than 3%.

Chapter 4, "Experimental Investigation of Transport Processes and Fouling in Biofuel Process Streams," was submitted as AIChE Annual Meeting, 2014: 738a. This investigation determines fouling of fermented sweet sorghum under controlled hydrodynamic conditions. The fouling resistance factor of fermented sweet sorghum was established to be approximately 9.25 x 10^{-5}

m^2 $^oC/W$ and observed fouling is equivalent to that of tap water. Fermented sweet sorghum and process water R_f values were found to be in the same order of magnitude, one tenth that of corn and maize ethanol thin stillage. This has been established using two independent experiments. The induction period for fermented sweet sorghum fouling was more than 5 hrs. These results suggests that fouling from fermented sweet sorghum at the biofuel processes can expected to be lower than for corn ethanol facilities. This provides significant advantage to biofuel production from fermented sweet sorghum in terms of CIP schedules and intensity.

Chapter 5, "Farm scale ethanol separation from sweet sorghum" concludes the work of this doctoral investigation. In manner of the Solar Energy Research Institute (SERI) report [21] on small-scale ethanol production, this chapter is an updated technical manual for on-farm ethanol separation from sweet sorghum. Major research findings are presented in a simplified manner and key non-traditional learning from first hand construction and operation experience is also presented – these might be useful in future farm scale projects.

CHAPTER 2

BIOETHANOL FROM SWEET SORGHUM: A DECENTRALIZED PRODUCTION APPROACH

2.1 Abstract

This work aims to present implementation of renewable fuel production from sweet sorghum on a farm-scale. High concentrations of sugar in stalks, simple downstream technology, and minimum upkeep of feedstock make sweet sorghum an ideal candidate for decentralized energy production. The Alcohol Separation Unit (ASU) at Oklahoma State University (OSU) serves as both a research and demonstration prototype unit, first of its kind to study bioethanol production from fermented sweet sorghum liquid feedstock. This pilot plant establishes a robust downstream separation strategy crucial for on-farm bioethanol production. The study investigates four key aspects of the process – equipment and instrumentation, safety, operation and maintenance, and finally economics. The Alcohol Separation Unit uses two distillation columns for concentrating ethanol, with associated heat exchangers, pumps, and storage units. This plant was commissioned in Fall 2013. Sweet sorghum feed containing 5 wt% ethanol have been successfully processed at the pilot plant to 190 proof ethanol product. Total mass balance closes at 92% and ethanol purities of 193.6 proof at 74^{o}F with rectifier column conditions of 0.8 psig and 171^{o}F were established during processing. Overall economics of the pilot plant (250 gal/h feed) indicate fixed capital costs at 403,000 USD. Major unit operation equipment (columns, heat exchangers, storage tanks) makes approximately 50% of the costs. Major energy usage at the pilot plant for ethanol production is steam, which accounts for 75% of total operating costs of 68,000 USD.

2.2 Introduction

Nearly all gasoline sold in the United States includes ethanol blended in various percentages starting at 10%. So far this volume has primarily been produced from corn-based feedstock - in 2012 bioethanol production in United States was 13,300 million gallons from these starch-based sources [40]. Non-corn cellulosic production volumes between 8 and 30 million gallons of cellulosic biofuels are expected only in 2014, based on current EPA estimates [2]. Despite a potential decrease in 2014 mandate for renewable fuels being considered in the United States, the total volume of biofuels produced will still be a staggering quantity and feedstock diversification will continue to be a heavily investigated research area.

Amongst alternative non-corn feedstock options sorghum crops are increasingly being considered a viable feedstock option for energy production. Sorghum crops require relatively lower inputs than their starch counterparts. With an estimated fertilizer input of 80:50:40 N:P:K kg/ha and water input of 8000 m^3 sweet sorghum is one of the lowest input crops [7, 14]. Grain, forage, and sweet sorghum all provide various options for fuel development albeit from different conversion routes. Sweet and forage sorghum are both suitable crops for cellulosic biomass conversion, although sweet sorghum has a significant quantity of juice in its stalks also suitable for direct liquid fermentation [41, 42]. Sweet sorghum in particular can be used in multiple ways – juice pressed from its stalks can be fermented, bagasse left in the field can be converted either via liquid or solid state cellulosic fermentation processes to fuel – this makes sweet sorghum extremely productive from an energy crop standpoint. Sweet sorghum has been estimated to produce 4000 liters per ha at half the cost of sugarcane ethanol. The advantages of sweet sorghum which provide incentive for its use as an energy feedstock have been demonstrated. In addition to its use as a fuel, sweet sorghum is also used as a precursor to food products and animal feed items [43].

For a sweet sorghum fermentation-based process, immediate utilization of sucrose upon expression or ensilage [44], pose a technical challenge. Upon storage or ensilage a significant portion of the sugars are lost. Additionally, transporting the pressed juice, or cut stalks to a central facility has been estimated to be expensive and large facilities for sweet sorghum have been found not to be cost effective [45]. As suggested by researchers at the School of Biosystems and Agricultural Engineering and discussed by Kundiyana et al [18] using sweet sorghum at smaller scale decentralized processes removes the storage and transportation hurdle by fermenting the sweet sorghum in-field. Their study found even with minimal pH, temperature control and minimal nutrient addition, up to 95% sugar to ethanol conversion efficiencies could be achieved. For the overall success of a small-scale on-farm unit though, in addition to the in-field fermentation step, the downstream separation step to separate ethanol from the dilute post-fermentation process stream is crucial. Without a robust, economical, and safe process the success of on-farm bioethanol production can be compromised.

The Alcohol Separation Unit at Oklahoma State University is a research and demonstration prototype unit, the first of its kind to study bioethanol production from fermented sweet sorghum liquid feedstock. This pilot plant has been built to investigate and recommend a robust downstream separation strategy crucial, as discussed, for on-farm bioethanol production. Research activity at the pilot plant will translate into recommendations for farmers in terms of construction, operation, maintenance, and economics. The study investigates four key aspects of the process – equipment and instrumentation, safety, operation and maintenance, and finally economics. This includes design, set-up, and operation of the pilot-plant in addition to analytical requirements for product characterization.

2.3 Materials and Methods

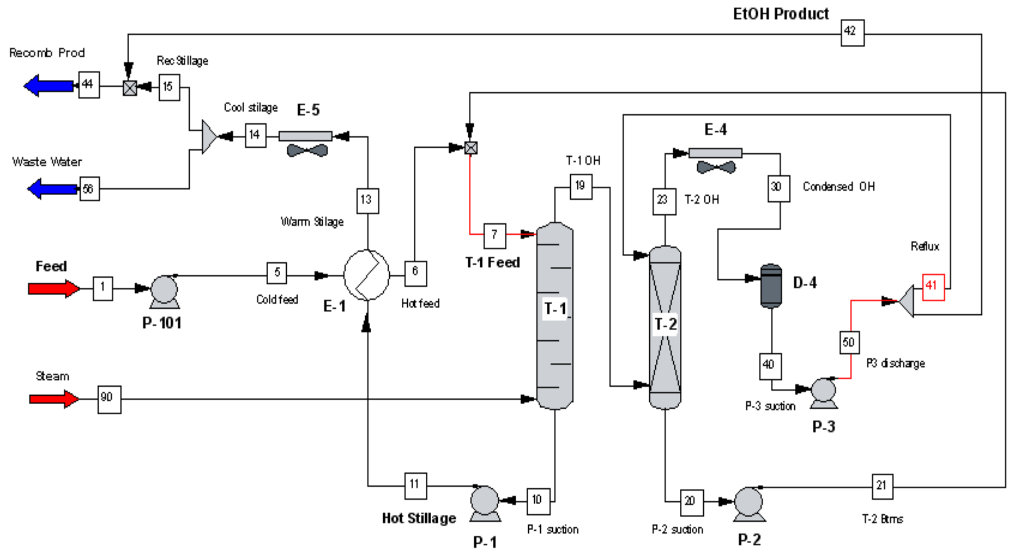
2.3.1 Alcohol Separation Unit: Design Criteria

Main considerations for the design of the Alcohol Separation at ASU are:

1. Feed rate of 250 gal/h, for continuous operation of a steady state process servicing a 500 acre farm. A 30 ton/acre sweet sorghum yield is expected, with juice yields of 4000-6000 gal/acre.
2. Ethanol concentration of 6-10 vol% in fermented liquid sweet sorghum can be processed.
3. Final ethanol product of 95 vol% or 190 proof ethanol at 15 gal/h is expected. Ethanol recoveries are at least 90%.
4. Process designed to operate at atmospheric pressure.
5. A two-column process with a beer column and rectifier is used.
6. Feed to the distillation column enters at 150^{o}F.
7. Steam is available onsite at maximum pressure of 150 psig.
8. Process has extensive data collection, control, and monitoring capability

2.3.2 Process Layout and Equipment Details

The Alcohol Separation Unit has a two-column distillation set up for concentrating ethanol, as seen in Figure 1, a common feature in ethanol separation units, both small and industrial scale. Details about and explanation of technology selection have been discussed in Mukherjee's work [19]. In theory, the column can be thought of as a single unit. From Figure 2.1 vapors from the beer column enter the bottom of the rectifier and contents from the bottom of the rectifier are added back into the beer column. However physical size/height restrictions limit the construction of the distillation unit as one column. This two-column configuration also allows use of packing in the rectifier, resulting in higher overall process efficiencies. In addition, solids are restricted to the beer column, where trays suitable for high solid loading process streams are used.



(a)

(b)

Figure 2.1: ASU from design to demonstration (a) Ethanol Simulation in ChemCAD (b) Alcohol Separation Unit: Storage and Process Area

Material of construction of all equipment, purchased or fabricated, is stainless steel 304 or 316. Low pH values of bio-based ethanol processes streams make them moderately corrosive and material resistant to acidic conditions, such as stainless steel is preferred [46]. The bioethanol distillation facility is equipped with a beer column (T-1), and a rectifier (T-2), as indicated in Figure 2.1. Associated equipment to ensure steady state function of the pilot plant include an air-cooled heat exchanger (E-4), a feed pre-heater (E-1), stillage cooler (E-5), storage tanks (Tk-101 and Tk-102) as well as pumps (P1, P2, P3, and P4) and a condensate receiver tank (D-4). Each custom built unit including the distillation columns and the air-cooled condenser were pressure tested before installation and are rated for 180 psig @ 350^{o}F.

Both beer column and rectifier are fabricated from 12 in. SA-312 TP304 pipe. Total height of the beer column is approximately 39 ft and the rectifier is 29 ft. Twenty three standard Sulzer cartridge SVG valve trays have been used in the beer column, each spaced at 15 inches The rectifier has a combination of four valve trays and 14 ft of M752Y packing (14 in HETP). In the beer column, temperature measurement ports have been provided at Tray 1, 5, 7, 12, and 22. On the rectifier, trays 1, 2, and 3 have a liquid withdrawal port as well as temperature measurement capability. The tray-packing combination in the rectifier is an industry standard [47, 48], and has been adopted for on-farm design – solid entrainment to the packing is avoided, by presence of the trays. Additionally, fusel alcohol removal is also facilitated from the rectifier by virtue of the trays.

A custom designed induced draft air-cooler from GEA Rainey is used as the condenser. A three row finned tube bundle with a 1-inch slope containing 10 fins per inch on tubes is used to facilitate condensation. The tube bundle itself is 3 ft x 4 ft in dimension. A ¾ HP motor is used to power the fan. Two heat exchangers are present on the feed return line – the stillage cooler, E-5, a BOL-725 Thermal transfer air cooler, and the feed pre-heater, E-1, a USSC 1036 4-pass shell and tube heater. The shell and tube heater (20 tubes per pass, 4 pass, 0.375 in OD x 0.02 in wall) and air cooler are arranged in series to ensure the stillage temperature is close to 120^{o}F, cool enough to store in polypropylene tanks, Tk-102 or Tk-101.

Either of two polypropylene tanks, each 5000 gallons in volume, can store sweet sorghum fermented juice feed. A spill protected storage area 40 ft x 24 ft x 1 ft high has been constructed around the tanks. Each tank has an Integral Flange Mounted Opening (IMFO) for easy cleaning and complete emptying of liquid content. A 24 in manway at the top of the tank for observation, a 2 in U-vent, and three 2 in nozzles are some of the features of the tank. Both tanks are stored separately on the south side of the building, as seen in Figure 1 (b). Both tanks are interconnected to facilitate transfer from one tank to the other, and provide mixing of contents. Four Goulds 1.5 HP, 1MF1S5B4 centrifugal pumps are used for the feed, stillage, rectifier bottoms, and condensate return streams.

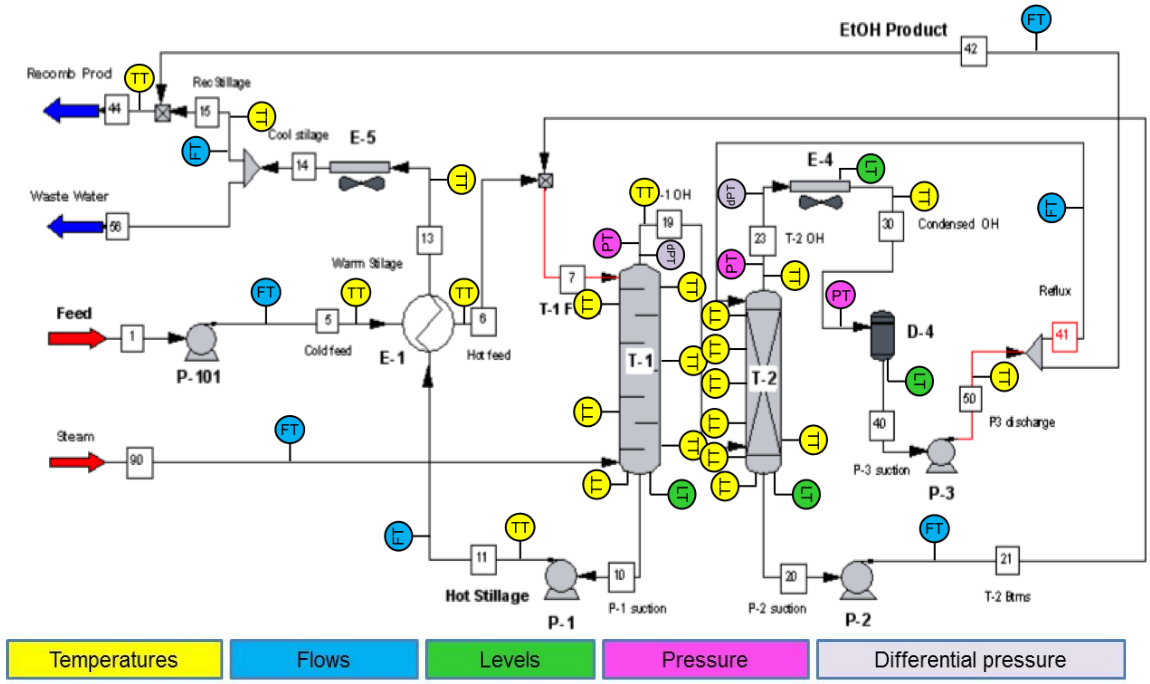
A Lattner 20 HP vertical boiler is used to provide steam to the distillation system. The steady state steam rate is 250 lb/h. Steam flow is increased gradually in increments of 50 lb/h. Steam system and steam piping blowdown is performed at start-up. The boiler is set at a maximum of 140 psi, with a 5 psi cutoff – in each cycle the pressure reaches 135 psi. The boiler trips at 140 psi. Steam is injected into the bottom of the beer column under Tray 1. The feed is introduced at the top of the beer column over Tray 23. The ethanol product stream is recombined with the stillage stream, and sent back to the tank area for storage. This is primarily due to Alcohol and Tobacco Tax and Trade Bureau (TTB) permitting requirements [49] for the facility, wherein a secured location is required for high purity ethanol storage. Therefore, ethanol product is recombined and stored in the tank area, which is secured by fence, and monitored by security cameras. In addition ethanol content and composition of the feed in Tk-101 and Tk-012 are monitored on a regular basis.

2.3.3. Instrumentation

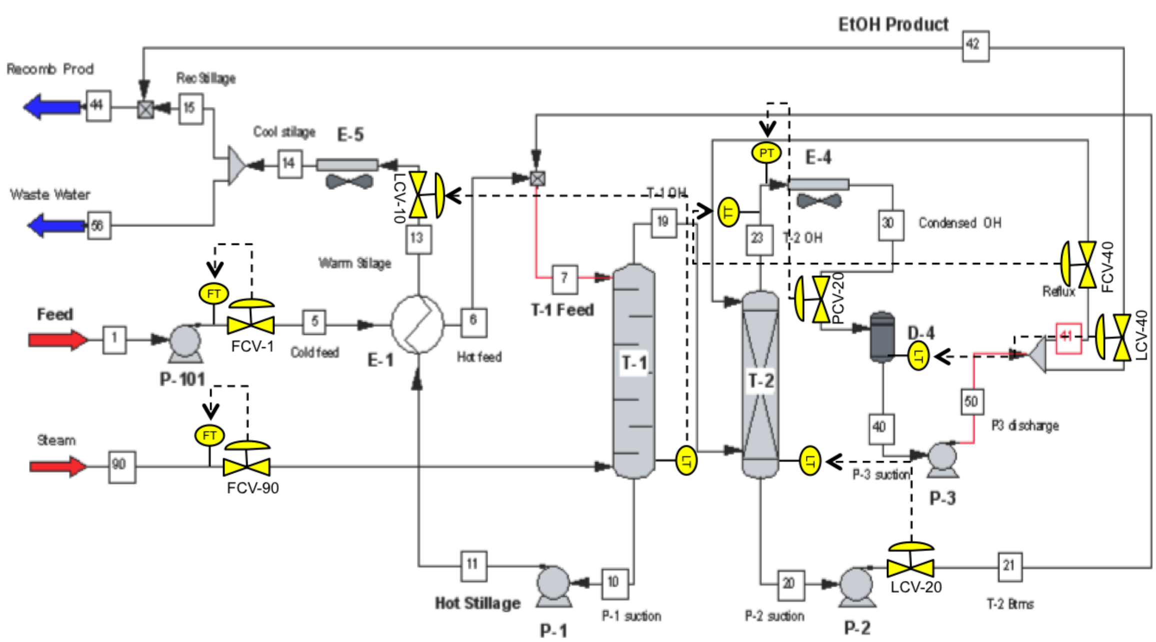
As listed in the design criteria, the process has an extensive data collection, control, and monitoring capability. Temperatures, flows, and pressures are recorded at multiple locations, and the process is fully automated to run during steady state operation. Data is collected as 4-20 mA signals from pressure or differential pressure transmitters, and flow meters. Two junction boxes are present at the base of T-1 and T-2 where all wiring terminates at the data collection device. Data from the junction boxes is transferred to the control room via Cat5e ethernet cables.

Six ports are available on T-1 with temperature measurements on Tray 1, 5, 12, 17, and 23. Similarly six ports are also available on T-2 with three on Tray 1, 2, and 3, and three at lower, mid, and upper points in along the packing, shown in Figure 2.2 (a). Process liquids and steam temperatures are also measured at each heat exchanger inlet and outlet, column inlet and outlet, as well as reflux drum and storage area tanks. Thermowells fabricated from 1/4 inch copper tubes were used along with T-type thermocouples at point of measurement. In-field pressure measurements are provided in addition to transmitted data signals for both temperatures and pressures. This helps evaluating whether the process is running according to protocol, monitoring and reacting quickly to process upsets, in addition to providing a layer of redundancy.

Steam pressure (PT-90), T-1 and T-2 column pressures (PT-10 and PT-20 respectively), reflux drum pressure (PT-40), as well as differential pressure measurements across the column (dPT-10 and dPT-20) are recorded using Rosemount 1151 type pressure/differential pressure transmitters. In-field pressure measurements include heat exchanger inlet and outlets, column top pressures, reflux drum pressure, and discharge pressure measurements at all pumps.



(a)



(b)

Figure 2.2: Instrumentation for process control and measurement at ASU (a) Instrumentation for measurement (b) Control strategy

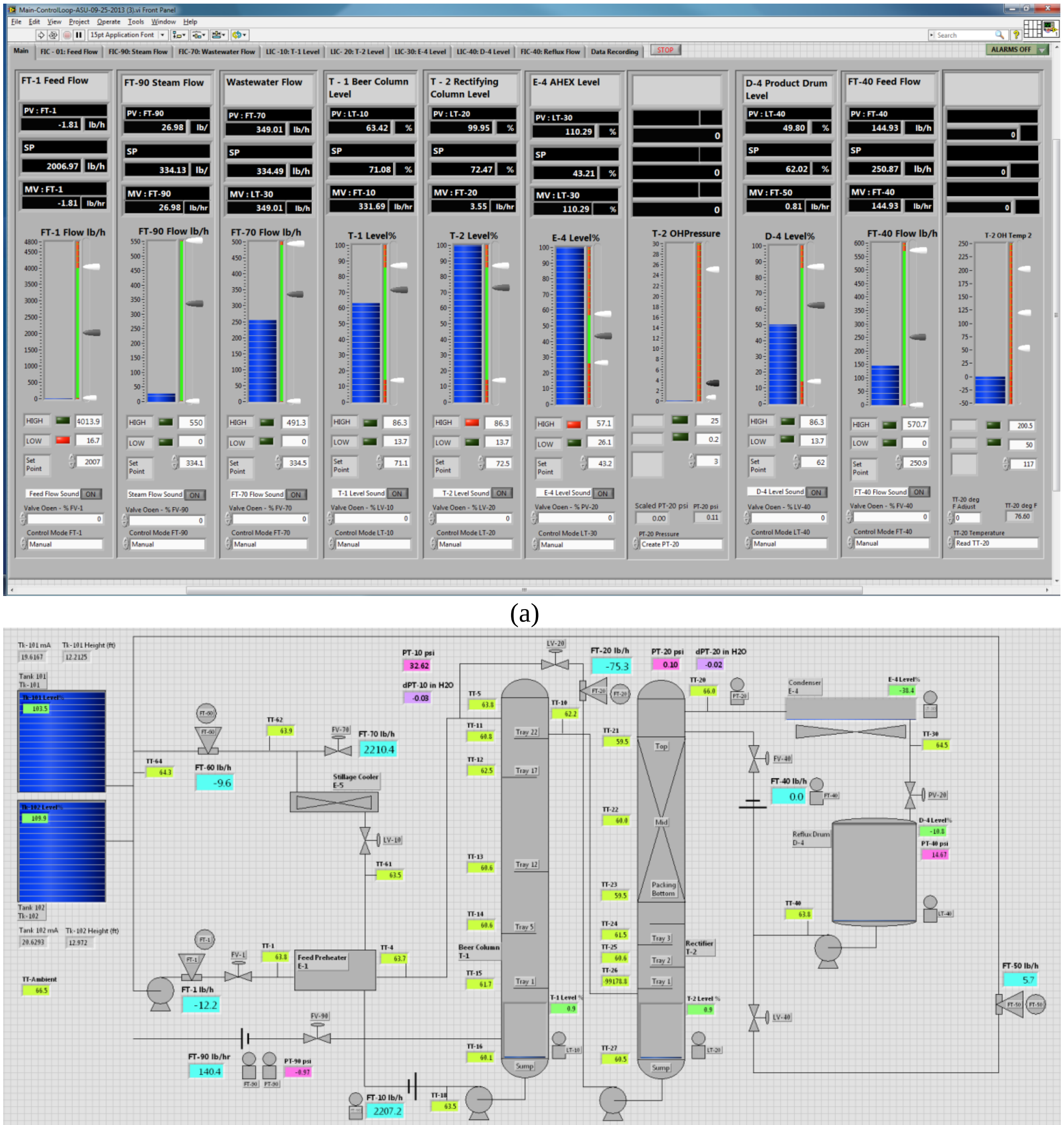
A total of seven flows are measured at ASU, which include feed, stillage, T-1 bottoms, T-2 bottoms, reflux, distillate, and steam, also indicated in Figure 2 (a). There are four highly accurate coriolis meters in use for feed (FT-1), stillage (FT-60), distillate (FT-50), and T-2 bottoms flow measurement. The other meters are orifice meters coupled with differential pressure transmitters and flow is calculated from the measured delta-P across the orifice. Process levels are measured with a wet-leg differential pressure approach. Silicone oil (specific gravity = 1.1) is used as the fill fluid on the low-pressure side of the transmitter. Sump levels on T-1 and T-2 as well as D-4 levels are measured using differential pressure. Tank Tk-101 and Tk-102 levels are measured using electric liquid level indicators. There is a dial for height display, and a 4-20mA current signal can be wired into the control program.

There are eight control loops at the pilot plant - control variables, manipulated variables, along with set point for each control loop are described in Table 2.1 and highlighted in Figure 2.2 (b). Overall, content coming into the system (steam and feed) is controlled in a flow control loop. Levels are maintained by controlling outflow from the columns. Pressure in E-4 and subsequently column pressure is maintained by keeping the liquid level in its tubes. Reflux flow into the column is set up as a flow control loop or a temperature cascade loop for T-2 top temperature.

Table 2.1: Control Loops at Alcohol Separation Unit

Control Loop	**Air-to-close/open**	**Stem**	**Cv**	**Tag**	**Service**
OSU-CV1	ATC	B-linear	2.00	PCV-20	Overhead Pressure/E-4 Level
OSU-CV2	ATC	F-linear	0.32	FCV-40	Reflux/T-2 Overhead Temperature
OSU-CV3	ATO	B-linear	2.00	FCV-1	Feed flow
OSU-CV4	ATO	B-linear	2.00	LCV-10	T-1 level
OSU-CV5	ATO	H-linear	0.13	LCV-20	T-2 level
OSU-CV6	ATO	B-linear	2.00	FCV-90	Steam flow
OSU-CV7	ATO	I-linear	0.08	LCV-40	D-4 level

Data acquisition from the process is via two National Instruments (NI) cDAQ 9188 chassis, and input modules. Ethernet based DAQ hardware provided a 50 percent cost savings on materials, which we would have otherwise spent on collecting measurement data.



(a)

(b)

Figure 2.3: User interface in control room (a) Main process control window for ASU (b) Critical process information on an easy to follow process flow diagram

A NI 9203 module is used for 4-20 mA current acquisition. This provided flexibility in process measurement as highly accurate coriolis flow sensors, level transmitters, and differential pressures were all coupled with the DAQ hardware.

Thermocouple temperatures are acquired directly using NI 9213 module. Output signals to control valves are generated using the NI 9265 modules. The control strategy is implemented using Labview programming (Figure 2.3). Critical process information including levels, pressures, temperatures, and flows are displayed, complete with an alarm system for high or low level occurrences on the graphical user interface, as displayed in Figure 2.3 (a) and (b). Control valve tuning, level, flow, temperature control capabilities have also been built into the control system using PID algorithm provided in NI's Control and Fuzzy logic toolkit. Program development, initial testing of control loops, controller tuning, and initial commissioning activities were accomplished in three months of summer 2013. The intensity of all start-up and commissioning activities, were equivalent in intensity to a level present in any chemical process or plant.

2.3.4 Safety Considerations at the ASU

Before the design was finalized and construction activities on-site began, a detailed Process Hazard Analysis (PHA) was conducted. As with any chemical process, safety is the highest priority right from design to operation. This exercise not only helped identify safety concerns in ASU's design, but as a group various possible solutions were identified. Over-pressurization, firefighting, storage of alcohol, spills, and monitoring were acknowledged as concerns regarding the facility and safeguards were included into the design to address these concerns.

As required by the Alcohol and Tobacco Tax and Trade Bureau (TTB), any ethanol produced on site must be gauged and stored under lock and key. Therefore, to tackle this primary concern, ethanol content of feed tanks is measured on a regular basis - this provides an accurate estimation of ethanol content in the fermentation broth. The storage tanks are in a fenced area, and are monitored with security cameras. To eliminate the storage of high purity ethanol the process operates in redistillation mode where ethanol produced in the process is mixed back into stillage and pumped back to storage area. When not in operation, process liquids are expected only in the storage area. Spills in this area will be restricted with a dyke 1 ft tall, built around the storage tanks. The volume of the diked area is large enough to accommodate contents of the largest tank inside it, 5,000 gallons in our case, as recommended in guidelines set by the National Fire Protection Agency (NFPA) in NFPA 30 [50].

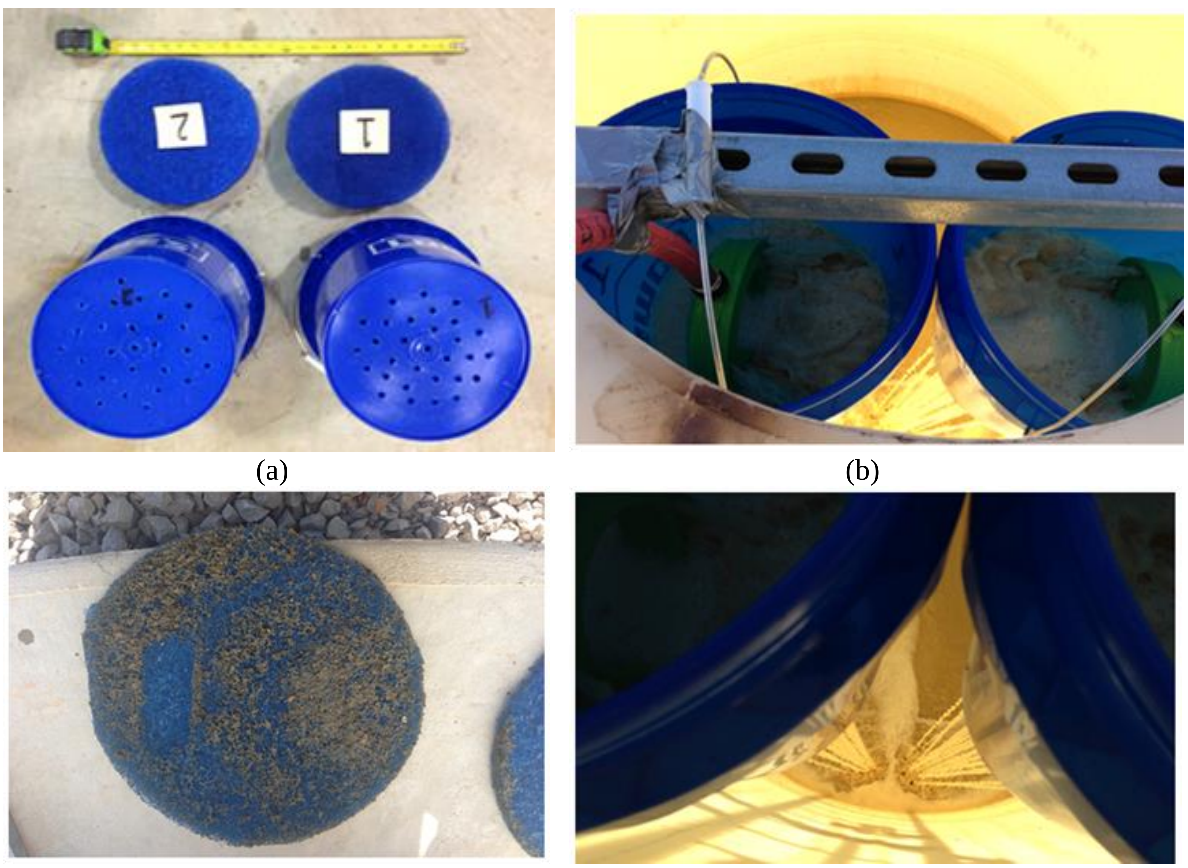
Detailed relief calculations with three over-pressurization scenarios were examined to address over-pressurization concerns - (a) fire at the base of columns, (b) runaway steam boiler, and (c) loss of electricity and failure of air-cooler. Two rupture disk assemblies, on the basis of these calculations, were selected to protect columns and associated equipment from over-pressurization. Fire fighting measures were analyzed using the NFPA 11 standard [51]. For process area spillage, AR-AFFF requirements included contents in the T-1 and T-2 sumps, reflux drum, and air cooler.

2.3.5 Steady State Operations and Commissioning:

Liquid sweet sorghum juice was procured from Delta Renewables Inc. – a total volume of 4890 gallons was transferred to the storage tanks Tk-101 via a 2 in inlet valve using the transfer pump on the delivery truck. The liquid was allowed to circulate in the tank using feed pump P-101 for half an hour before samples were taken for feed ethanol determination.

An initial filtration step was set up for removal of large sized leafy and solid particles. Mesh filters (constructed from lining holed buckets with air conditioner filters) were installed in Tk-102 manway (Figure 2.4) and liquid from Tk-101 was diverted through these filters, and into Tk-102, and back into Tk-101. Feed in Tk-101 was recirculated for at least half an hour prior to processing.

Steam was let into the system through the steam line into T-1 till target of 250 lb/h was reached. Vents on T-2 overhead line and D-4 was closed once both distillation columns were allowed to fill up with steam. A liquid level in the beer column, T-1, the rectifier, T-2, and condensate drum, D-4 respectively develops upon condensation of vapors in the system. Upon reaching the 20% level on D-4, the collected liquid was sent back to T-2, as reflux, at approximately 100 lb/h. Feed was added to the column in increments till a steady state value of 1700 lb/h was reached.



(a) (b)

(c) (d)

Figure 2.4: Filtration Setup Details at ASU (a)Filtration equipment (b) Set-up (c) Retentate (d) Filtrate

Headspace gases from the air cooler/condensate drum were diverted through a sparger assembly – carbon dioxide gases trapped in the fermented sweet sorghum juice still retained in the distilled product were removed by dissolving in water. Some ethanol is lost in this process, however over-pressurization of air cooler and condensate drum are avoided. A constant pressure of 0.8 psig was maintained in the reflux drum. As ethanol is concentrated in the column, temperature separation was observed along the columns. Levels in T-1 and T-2 were maintained between 10 to 15%. Maintaining low levels in columns assists in a faster shut down process as equipment is emptied out quickly. Product ethanol concentration was checked on-site with hydrometers. 193.6 proof ethanol concentration at 74^{o}F (translates to 190 proof at 60^{o}F) was recorded as product from ASU.

2.3.6 Analytical Sample Analysis

An Aminex HPX-87H column (Bio-Rad, Sunnyvale, CA, USA) at 60^{o}C was used for off-site HPLC analysis. Sample flowrate was 0.6 mL/min and a 0.01 N H_2SO_4 solution was used as eluent and refractive index detection (1100 series, Agilent) was used for quantification. All samples were examined in triplicate. Samples with high ethanol concentration (T-2 samples) were diluted to 1:100 and feed and stillage samples (from T-1) were diluted to 1:10. Ethanol standards were prepared from 200 proof laboratory grade ethyl alcohol (AL200-500). A calibration standard with fructose 20.002 g/L, glucose 19.870 g/L, sucrose 20.074 g/L, and acetic acid 19.78 g/L was prepared for non-ethanol constituent analysis. Dilutions of 1:1, 1:2, 1:4, 1:10, and 1:20 were used for calibration samples.

2.3.7 Economic Analysis

Capital and installation costs of constructing a farm scale unit were documented. These costs represent actual spending on equipment and installation. Installation costs include labor and contractual services (not including engineering salaries of the PI and graduate student), piping, hardware, fittings material cost, and construction equipment rental costs. Operating costs include operation boiler steam and electricity. Electricity costs also includes demand from all pumps (P1, P2, P3, P4, and P101 at 1.5 HP, E4 and E-5 fan motor at 2 and 1.5 HP respectively).

2.4 Results

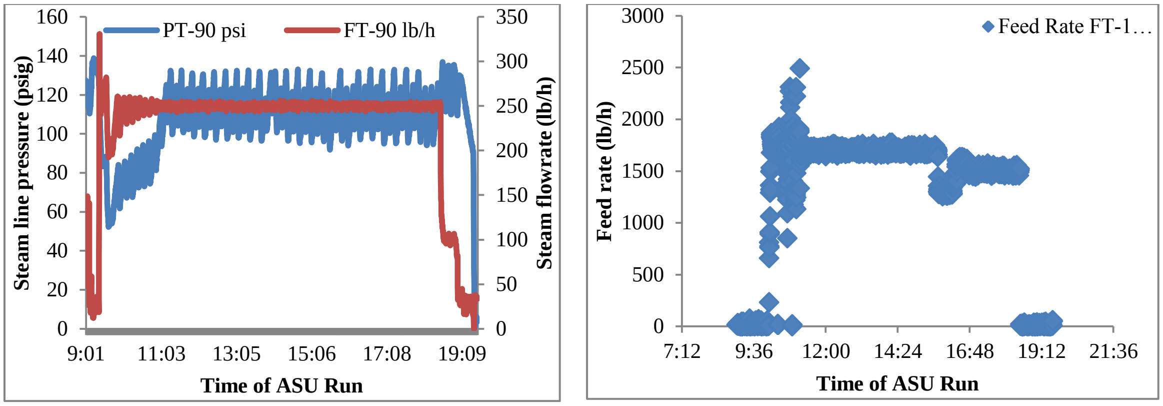
2.4.1 Process Operation

Feed and steam flows account for all mass entering the system. Stillage and product flows account for all mass exiting the process. Mass flows shown in Table 2.2 were recorded at azeotropic product formation - 190 proof at 60^{o}F, mentioned earlier. This mass balance closes at 92% for total material.

Table 2.2: Total Mass Balance over ASU

Total Flows (lb/h)	Mass In	1752	Mass Out	1610
Stream	Feed	Steam	Stillage	Product
Sensor	FT-1	FT-90	FT-60	FT-50
Flowrate (lb/h)	1500	252	1557	53

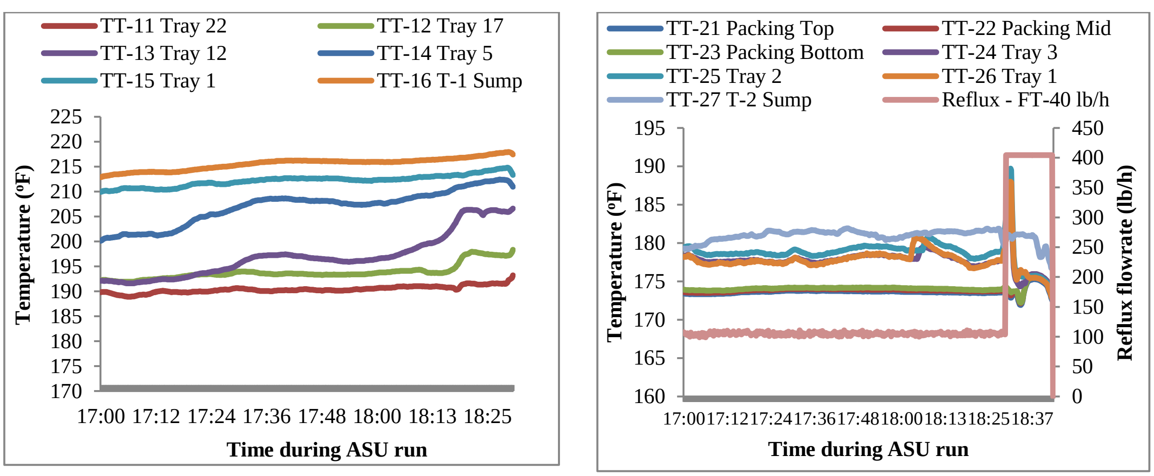
With the exception of FT-90 all sensors accounting for the flow are high accuracy Micromotion flow devices. An orifice meter is recommended and used for steam flowrate measurement along with calibrated differential pressure transmitter. The data recorded has some inherent scatter (for each flowrate), which is possibly contributing to the difference. Steam flowrate was maintained at 250 lb/h, at an average delivery pressure of 110 psig, as seen in Figure 2.5 (a).



(a) (b)

Figure 2.5: Flows entering the system (a) Steam (b) Feed. Also shown in 2.5 (a) is steam boiler pressure behavior

Typically boiler pressure swings between 100 and 130 psig, reflecting cold make up water addition cycles in the boiler, also seen in Figure 2.5 (a). The cut-off pressure for the boiler is 140 psig. We found that the boiler could maintain a maximum steam flowrate of 250 lb/h without tripping. Feed was introduced in 200 lb/h increments till 1700 lb/h was achieved, as seen in Figure 2.5 (b).



(a) (b)

Figure 2.6: Temperature profile of column (a) T-1, Beer column (b) T-2 Rectifier

As ethanol is concentrates in the column, temperature separation was observed along the columns as shown in Figures 2.6 (a) and (b). The bottom temperature of approximately 216^{o}F and a T-1 top temperature of 191^{o}F were observed at product specification. T-2 top and bottom temperatures were 171^{o}F and 181^{o}F.

2.4.2 Analytical Results

The initial ethanol concentration on delivery was determined to be 4.25 wt% using HPLC (Table 2.3). Apart from using hydrometer readings, which provide accuracy to 0.2 proof, HPLC protocols were also used to verify ethanol concentrations in various liquid samples taken throughout the pilot plant during operation.

Table 2.3: Fermented sweet sorghum feed composition on arrival

Name	**Sucrose**	**Glucose**	**Fructose**	**Glycerol**	**Acetic Acid**	**Ethanol**
wt%	1.30	0.18	1.64	0.47	0.48	4.25
Std dev	0.25	0.01	0.16	0.04	0.04	0.25

Table 2.4 compares results from hydrometer tests as well as HPLC results for ethanol samples drawn downstream of E-4 at three time intervals. Temperature corrected hydrometer results for ethanol volume concentrations agree with ethanol concentrations established within 0.5%.

Table 2.4: Ethanol product concentration: Hydrometer and HPLC comparison

Time (pm)	**Measured Proof (^{o}F)**	**Temperature at proof measured (^{o}F)**	**Corrected Proof at 60^{o}F**	**Hydrometer Results Vol%**	**HPLC Results Vol%**
3:23	190.0	77.7	185.7	92.9	93.03
3:36	191.0	78.1	186.8	93.4	93.03
6:13	193.6	74.0	190.0	95.0	95.64

Feed, stillage, T-1 and T-2 bottoms product, T-2 tray 3, and sparger samples were also collected and are tabulated in Table 2.5. Ethanol was not found in stillage stream, leaving the process. For feed ethanol at 4.5 vol% the ethanol content found on tray 3 on T-2 was 43.4 vol%. The ethanol content in rises in the liquid as we traverse the column height, as intended in distillation.

Table 2.5: Ethanol composition in various process streams in ASU

Process Stream	Feed	Stillage	Product	T-2 Tray3	T-2 Btms
vol%	4.49	0.00	96.50	43.35	28.43
Std Dev	0.05	0.00	0.03	3.06	

In order to fulfill TTB requirements of tracking ethanol concentrations experiments were conducted to establish the variation of ethanol content in the feed storage tanks over time. In addition to ethanol sugars (sucrose, fructose, glucose) and fermentation products, namely acetic acid, and glycerol were also measured in HPLC experiments. Average ethanol concentrations of feed was approximately 5wt% (Figure 2.7) and increased slightly from the time sweet sorghum shipment was received, 10th Oct 2013, to process run on Nov 4th 2013. This can be attributed to continued fermentation inside storage tanks after delivery. Unfermented sugars were still present in the fermented broth and accounted for 2.4 wt% of the solution.

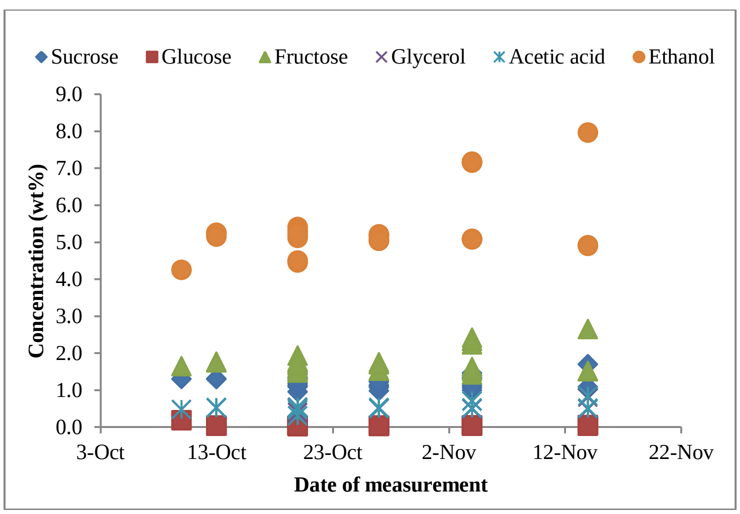


Figure 2.7: Feed characterization: Level of sugars, ethanol, and fermentation by products over time

Stillage and ethanol product are recombined and recycled back to the storage area. The contents are diluted with water equivalent of steam added into the process. Average ethanol concentration of Tk-102 was found to be 3.4wt%. Overall, stratification in tanks was not very significant.

GC experiments were conducted to estimate levels of dissolved CO_2 in sweet sorghum feed based on Liu et al's estimation method [52]. Additionally, CO_2 level in the waste stream (recombined product and stillage from process stored in Tk-102) was also measured to see if sparging the process streams had caused CO_2 reduction. Carbon dioxide levels in feed samples from Tk-101 were 0.062 g/L and from Tk-102 was 0.03 g/L. A 52% reduction in carbon dioxide levels is seen. We expect the level of CO_2 to decrease with time and with the progress in fermentation occurring in the feed.

2.4.3 Economics of Small Scale Ethanol Separation

Capital and installation costs of constructing a farm scale unit are tabulated in Table 2.6. These costs represent actual spending on equipment and installation spending. An initial total fixed capital cost of 251,000 USD was estimated in 2009. These values were based on industry quotes and internet pricing of readily available items.

Table 2.6: Fixed capital and installation costs of ASU and comparison with initial estimates

Costs	Initial Estimate	Actual Cost	%Difference
Instrumentation (Control and Measurement)	60,400	74,300	19
Column Body and Internals	49,500	95,590	48
Boiler	16,000	33,800	53
Heat Exchangers	2,700	41,000	93
Pumps	3,700	2,400	(54)
Storage Tanks	36,500	28,300	(29)
Equipment Cost	168,800	275,390	39
Installation Cost	82,700	127,600	35
Fixed Capital Costs	251,500	402,990	38

A 49% cost factor was used for installation costs. In reality, the total expenditure was 38% higher than initial estimates. Installation costs include labor and contractual services (67,400 USD not including engineering salaries of the PI and graduate student), piping, hardware and associated material cost (38,000 USD), and construction equipment rental costs (16,000 USD).

Main contributors to the operation of a steam stripping ethanol separation facility are from operation of the boiler, which account for 75% of operating costs, seen in Table 2.7. Total annual operating costs for a plant operating for 45 weeks is 68,300 USD. Electricity costs includes demand from all pumps (P1, P2, P3, P4, and P101 at 1.5 hP, E4 and E-5 fan motor at 2 and 1.5 hP respectively).

Table 2.7: Operational costs for ASU

Direct Operating Costs - excluding raw materials	Usage		Unit Cost		Annual Cost $/yr
Utilities	Utility use		$/unit		(45 weeks operation)
Electricity (distillate, bottoms, reflux and feed pumps, condenser, air cooler blowers)	42877	kW-hr	0.09	$ / kW-hr	3,859
Boiler fuel cost	678	SCFH	9.78	$/1000 SCFH	50,827
Boiler water cost	67	gph	2.99	$ / kgal	1,531
Total Utility Costs					56,217
Maintenance and repairs (3% of fixed capital)					12,090
Total Annual Operating Expenses ($/yr)					68,307

2.4.4 Safety Considerations

A total relief rate of 1,877 lb/h of an ethanol-water mixture is expected from the first scenario (fire at base of column) at a Maximum Allowable Working Pressure (MAWP) of 50 psig. Liquid inventory included sump liquids for both T-1 and T-2, all liquid in trays (23 trays with 2 inches of liquid) and packing (14 ft of packing with 3.5% liquid hold up and 4 trays), reflux drum level, and air cooler tubes liquid hold up. The pilot plant was thus equipped with two union type rupture disk assemblies, each with a rated capacity of 1251 lb/h air (relief rate is 5,329 lb/hr for ethanol-water mixture both calculated at 50 psig and 277 ^{o}F) providing adequate relief capacity in case of over-pressurization.

Solutions of ethanol with even 80% water are flammable [53] and burn with a near smokeless flame (depending on the concentration of the ethanol in the liquid). Ethanol fires containing more than 10% ethanol need an Alcohol Resistant (AR) Aqueous Film Forming Foam (AFFF) [54]. The real concerns for fire at the pilot plant are in the process area, where high purity ethanol, up to 95 vol% can be found. The storage area contains only dilute concentrations of ethanol (less than 10 vol%), not considered flammable.

Liquid accumulation from T-1 and T-2 sumps, reflux drum, and air cooler liquid in the process area (of dimensions 20 ft x 11 ft) would account for a liquid layer of 0.84 inches. For the storage area, flammable liquid content is 6.5 vol% (design ethanol content in sweet sorghum liquid) of the 10,000 gallons present in both tanks. Calculations showed the height of flammable liquid in spills in process and storage areas was lower than the 1 in. contained spill height. This indicates the pilot plant is an exceptional case for fire response since the spill and flammable substances volumes are low. In addition the City of Stillwater fire department is not equipped with AR-AFFF foam, and it was not economical to maintain an inventory for such small quantities of alcohol. According to the EERC it is “important to understand all you can safely do is contain the incident and let the fire run its course. Knowing when to let this happen is an important component of safety” [55]. After discussions with the Fire Marshall for OSU it was established the best response in case of fire was indeed to let the fire run its course.

2.5 Conclusion

The Alcohol Separation Unit successfully served as a research and demonstration unit for bioethanol separation (190 proof product) from fermented sweet sorghum. Detailed records documented true operating costs and energy usage for feeds containing 6-10 vol % ethanol. This operational pilot plant serves as a benchmark for the development of sweet sorghum bioethanol. Updated equipment were used to set up rigorous measurement and control strategies making ASU one-of-a-kind multi functional bioethanol production unit. Start-up, shut down, on-site and laboratory analytical procedures for ethanol product determination were established. This demonstrative research strengthens existing body of sweet sorghum research.

CHAPTER 3

FOULING POTENTIAL OF BIOETHANOL PROCESS FEED – AN ANALYSIS OF FERMENTED SWEET SORGHUM SOLID CONTENT

Abstract

Use of sweet sorghum as a bioenergy feedstock for ethanol production via liquid or solid-state fermentation is motivated by several factors – accessible sugars, low inputs, simple process set-up for bioethanol production, and a potential for complete crop utilization provide incentives for increasing interest in the crop. Areas of concern for any bioenergy feedstock are equipment fouling under process conditions. Characteristics of fouling deposits need research to arrive at an effective cleaning strategy and ensure economic and safe biofuel production. We focus on sweet sorghum fermented juice solid quantification, deposit examination, and transport property (density and viscosity) determination. The National Renewable Energy Laboratory procedure for total and dissolved solids in liquid biomass was employed to determine solid content in sweet sorghum fermented liquid. Standard density and viscosity measurement techniques were used for physical property determination of sweet sorghum fermented liquid. Total solid loading of fermented juice was determined to be less than 3 wt%, significantly lower compared to corn-based process streams. Solids were observed to contain porous, three dimensional, multi-component deposits (crystallization, particulate) from fermented sweet sorghum juice. Deposit morphology was seen to change with solid content - as a result deposit thermal properties and fouling nature of liquid are expected to be different in fermented vs. unfermented juice.

3.2 Introduction

With growing emphasis on cellulosic and advanced biofuel production, sorghum crops are gaining popularity as potential bio feedstocks. Within sorghum crops, sweet sorghum or Sorghum bicolor (L.) Moench is attractive due to the high sugar content in its stalks and its tolerance to dryer conditions [56]. All sweet sorghum crop components (grain, stalk, sugars, bagasse) can potentially be used to produce energy [57], bio based products, and/or animal feed [8]. Using direct extraction of sugars from the stalk in conjunction with in-field fermentation and an on-farm small-scale separation unit in place, shared by several farmers or run as a co-op, the economics of decentralized sweet sorghum ethanol become attractive [20]. Perhaps the most important aspect of a decentralized process is the advantage to the rural agricultural community: small-scale ethanol manufacturing units within agricultural communities will provide farmers with additional income, create jobs, and provide renewable fuels.

Amid growing interest in sweet sorghum biofuel production, one area of renewable energy research at Oklahoma State University (OSU) is a small-scale sweet sorghum based decentralized bioethanol production model. This demonstration prototype ethanol manufacturing facility has been built to highlight the feasibility of an on-farm sweet sorghum to ethanol process. Experiments conducted at the Alcohol Separation Unit (ASU), OSU's bioethanol research and demonstration facility will help establish the feasibility of a small-scale on-farm ethanol model, and bring to attention any technical issues with the process [19]. Some of these bioethanol production issues, which have been discussed in literature include – finding cost and energy efficient downstream production processes [58], fouling of equipment in downstream processes for ethanol removal from fermented streams [59], and as discussed by Kolmetz *et al*. finding and troubleshooting "inappropriate design, inappropriate operation, and damage to equipment". With increasing solid loading in process streams, compounded by an increasing interest in solid-state fermentation as well, the possibility of fouling is expected to proliferate with respect to biofuel production.

Sweet sorghum process fluids and its properties have so far been studied mainly from a pre-fermentation perspective, as feed for food based products [60] and process related studies, specifically related to fouling are infrequent in literature. Without a clear picture on the extent of fouling, cleaning operations plant schedules including shut down procedures can't be set in place. All of these steps will eventually impact the profitability of an on-farm ethanol facility. Sweet sorghum juice fouling under processing conditions, fouling behavior models, and characteristics of its deposits are some of the research areas that need to be addressed in order to arrive at an effective cleaning strategy and ensure economic and safe biofuel production. In order to understand the fouling process, the first step should establish what is entering the system (proteins, detritus, minerals, organics, etc). The focus of this article is sweet sorghum fermented juice solid characterization achieved by solid quantification, deposit examination, and transport properties (density and viscosity) determination.

3.3 Materials and Methods

3.3.1 Solid quantification

Three major foulant groups are expected to contribute to fouling on heat exchanger surfaces – suspended particulates, dissolved solids, and yeast biomass, pictorially depicted in Figure 3.1. In fermented sweet sorghum liquid total solids include (a) suspended solids - yeast used as fermentation agents as well as particulates, and (b) dissolved solids that are fermentation by products. In unfermented sweet sorghum liquid, dissolved solids are typically expected to be sugars present in the liquid pressed from the stalks.

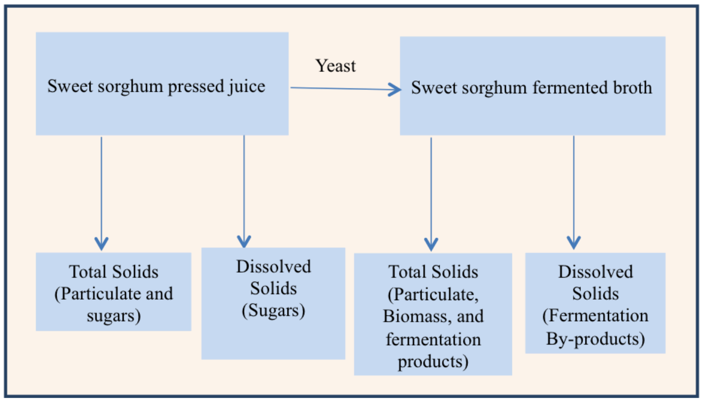


Figure 3.1: Solid quantification procedure of sweet sorghum liquids

Suspended solids in unfermented sweet sorghum broth are expected residues of the crushing process employed. Suspended solid levels for both fermented and unfermented broth were inferred from total solids and dissolved solid measurements using Equation 1.

$$\text{Suspended solids} = \text{Total solids} - \text{Dissolved solids} \qquad (1)$$

Sweet sorghum samples collected were from Topper variety harvested and fermented in October 2011 from Stillwater. Final solid quantification results are reported in weight %.

Gravimetric techniques are used to establish moisture loss and thus calculate solid content in sample. The National Renewable Energy Laboratory's (NREL) analysis methods was used to find solid content of the sweet sorghum feed slurry [39]. According to NREL's Laboratory Analytical Procedure (LAP) dissolved solids (present in the liquid slurry after filtering through a 0.2 μm pore), as well as particulate matter can be estimated gravimetrically. Samples were collected from a well-mixed container. Mixing was achieved using a Thermo scientific magnetic stirrer operated at 170 rpm. Liquids were allowed to mix for 10 minutes prior to sampling. Samples were collected in 50 mL centrifuge tubes from the container, drained into the aluminum plate, weighed and transferred to the oven. Samples used for dissolved solids were also withdrawn with a 50 ml tube. A 2 mL volume was used, centrifuged at 13 G for 10 minutes, prior to filtration through 0.2 μm syringe filter. Although NREL recommended volumes were 10 ml for liquor samples, and these recommendations were followed for total solid measurements, only 2 ml sample size was used for dissolved solid samples. This was in part because it was extremely difficult to filter manually through the syringe filter assembly. However, even with low sample volumes experimental errors were maintained low (less than 0.5%).

In addition to quantification steps to account for the liquid retained in the syringe, filter, and centrifuge tube each of these components were weighed before and after the liquid transfer. These were also dried along with the aluminum pans at 105^{o}C – difference between before and after drying weights indicate the solids trapped in transfer equipment (syringe, centrifuge tubes, filters). Dissolved constituents, mainly sugars (sucrose, fructose, glucose) in unfermented juice, and ethanol, acetic acid, and glycerol in fermented juice were determined using HPLC measurements described in Vijayakumar's work [61].

3.3.2 Determination of Physical Properties

In order to estimate Reynolds number for sweet sorghum juice accurately, transport properties, namely density and viscosity need to be determined.

Density measurement: An analytical balance (Model BP-301S) with a density measurement kit (Sartorius, YDk-01) was used to determine liquid sweet sorghum (unfermented and fermented broth) density using a plummet (made of AR glass, V=10 cm^3 volume, hanging on a constantan wire), provided with the kit.

Viscosity Measurement: A Brookfield RV-1 viscometer was used to measure the viscosity of the sweet sorghum fermented juice with Spindle #1. The temperature of the liquid was recorded at the time of the experiment. The spindle was allowed to rotate till a constant viscosity reading was observed. Samples were placed in a 600 mL glass beaker. Ten single point measurements (in cP) at each shear stress value were recorded to ensure statistical robustness. Three sample volumes for each type of sweet sorghum liquid were used.

3.3.3 Deposit Analysis:

The FEI Quanta 600 field-emission gun Environmental Scanning Electron Microscope was used to study the initial dried solid deposits from the solid quantification experiments. The SEM experiment was conducted at 12 kV. A gold/Au coating was applied to the deposit to ensure conductivity within the sample during the experiment – sputtering times of 30 s was used. A magnification range from 500-5,000x was used for image capture. A 5 mm x 5 mm section of the aluminum weighing pan, containing the dried layer of deposits from solid quantification experiments, was cut away and mounted onto the SEM sample stub using a double-sided adhesive pad.

3.4 Results and Discussion

3.4.1: Solid Quantification

Samples were collected from a well-mixed container. Samples used for dissolved solids measurement were centrifuged prior to filtration through 0.2 μm syringe filter. The results are shown in Table 3.1.

Table 3.1: Solid quantification of sweet sorghum juice – Fermented and Unfermented

Sample	**Total Solids, wt %**		**Dissolved Solids, wt %**		**Suspended Solids, wt%**
	Avg.	Standard Deviation	Avg.	Standard Deviation	
Fermented Sweet Sorghum	1.9705	0.0021	1.4261	0.0041	0.5444
Unfermented Sweet Sorghum	12.3525	0.0492	11.9301	0.0419	0.4224

In unfermented sweet sorghum juice the total solids are 12.3 wt%, and majority of these solids are present in dissolved form. The total solids in fermented sweet sorghum broth are less than 2 wt %. These results confirm that the total solids present in the liquid fermentation based sweet sorghum bioethanol process stream are low compared to corn based bioethanol process streams, which often have 20-30% solid content [62]. A lower solid content in process streams in bio-refineries will increase pump and heat exchanger efficiencies and ensure longer equipment life. In addition centrifugal pumps are capable of handling non-settling low solid content in liquid streams, and are easily available – this allows for off the shelf equipment and cost saving in terms of specialized pumping equipment or customization. Majority of the solids present in fermented sweet sorghum are present in dissolved form.

In order to corroborate the NREL procedure a mass balance study was initiated – a mass balance across each experimental container was carried out. This included the centrifuge tube, syringe, filter, and aluminum pan. Each of these items was weighed with liquid sample uptake, and after discharge, and post drying. For dissolved solid measurement, any solid content in the pans can safely be assumed < 0.2 microns in size (X), collected after filtration. The dissolved solids content in the pan (X_p) was experimentally determined in the solid quantification experiment. We also assume any X found in the filter and syringe, or centrifuge tubes are dissolved in the liquid retained. Since liquid retained in each stage of the separation process was measured, the dissolved solid content was calculated using X_p and liquid weights. The percentage total dissolved solids measured for all liquid volumes found in syringe (Xs), filter (Xf), and centrifuge tube (Xc) should match the percentage found in the pan, within experimental error. An example set of measurements is shown in Table 3.2.

Table 3.2: Mass balance of dissolved solids in experiment

Experimental Equipment	Liquid (Measured)	Liquid Retained (g)	Solid	Dissolved solid (g)	Dissolved Solid%
		L		DS	L x DS
Centrifuge	L_c	0.1320	X_c	0.00188	L_c x X_c
Syringe	L_s	0.0156	X_s	0.00022	L_s x X_s
Filter	L_f	0.2024	X_f	0.00289	L_f x X_f
Pan	L_p	1.4811	X_p	0.02108	Measured
Sample	L_t	1.8767	X_t	0.02607	$X_c+X_s+X_f+X_p$
Dissolved solid in liquid sample				1.38918	X_t/L_t x 100

It was found only 80% of the sample volume was used in the drying experiment (in the aluminum pan) – the rest was trapped in the centrifuge (7.9%), syringe (0.8%), and filter (11%). From rigorous mass balance experiments and calculations it was found despite the lower sample volume the filtered sweet sorghum liquid was indeed representative of the initial sample. Dissolved solid content calculated from mass balance experiment was compared to measured dissolved solid content in pan. All experimental observations were checked for statistical differences using ANOVA – as seen the p-value, 0.05 < F-critical indicating the results were not statistically different from each other. Results are tabulated in Table 3.3.

Table 3.3: Experimental Data and ANOVA results: Dissolved solids

Type	Data	Measured	Calculated	p-value	F-critical
Fermented	FDS1	1.4233	1.38918	0.126	18.512
	FDS2	1.4290	1.40947		
Unfermented	UDS1	11.9005	11.76272	0.768	18.512
	UDS2	11.9597	12.26904		

Initial doubts concerning partitioning of dissolved solid content due to liquid retention in equipment was thus dispelled. The need for rigorous experimentation and multiple redundancies arise from the fact that none of these liquids (unfermented or fermented sweet sorghum juice) have previously been tested with the NREL LAP for dissolved solids or total solids. A rigorous mass balance corroborates the procedure – we can be assured that the dissolved solids captured will be representative of that in the liquid feed and thus in fouling estimates or calculations these figures can be used without hesitation.

3.4.2 Composition

The feed composition was established using high-pressure liquid chromatography (HPLC) experiments following a previously established protocol for sweet sorghum fermented juice. A calibration standard with fructose 20.002 g/L, glucose 19.870 g/L, sucrose 20.074 g/L, acetic acid 19.78 g/L, and ethanol 20.02 g/L was prepared. Dilutions of 1, 2, 4, 10, and 20x were used for calibration. Sweet sorghum liquid samples were diluted 1:10 for measurement purposes. Results of the HPLC experiment are tabulated in Table 3.4.

Table 3.4: Feed Characterization – Composition of fermented sweet sorghum juice

	Sucrose	**Glucose**	**Fructose**	**Glycerol**	**Acetic Acid**	**Ethanol**
Sample	Average Concentration (wt%)					
Raw Juice	5.17	4.10	3.57	0.01	0.00	0.04
Fermented Juice	0.00	0.02	0.00	1.03	0.21	5.18
	Standard Deviation					
Raw Juice	1.33	1.04	0.92	0.01	0.00	0.03
Fermented Juice	0.00	0.02	0.00	0.13	0.06	0.25

The main constituents of the sweet sorghum fermented juice are ethanol, glycerol, and acetic acid. Main dissolved constituents of sweet sorghum unfermented juice are sugars (sucrose, glucose, fructose). The total average dissolved solid content in fermented sweet sorghum juice is 6.44 wt%, that includes ethanol.

During drying over the course of experimentation ethanol is expected to evaporate and the dissolved solid content expected in measurements will include glycerol, acetic acid, and unfermented sugars – these compounds account for 1.26 (± 0.17) wt% of the dissolved content in fermented samples which is similar, although slightly lower than the dissolved solid value measured in the filtration experiment. Similarly in unfermented juice, the dissolved solid content level determined by HPLC is 12.89 (± 3.43) wt%, and reflects relatively closely results from the filtration experiment. The HPLC results also corroborate the NREL method for solid content measurement – these results are significant because a simple inexpensive filtration based measurement can very accurately provide an overview of the solid loading of process liquids.

The quantification of solids and dissolved solids in bioethanol feed streams also provide a first step in establishing fouling potential of process streams in bio-refineries. Fouling potential of processes is closely related to the concentration of foulants in fluids – in Fickak et al's study of whey concentrate, fouling rates increased with higher protein concentrations. In their study heat transfer coefficient recorded for 2 wt% whey concentrate solution, 300 $Wm^{-2}K^{-1}$, was higher than that for 6 wt% solution, 200 $Wm^{-2}K^{-1}$, indicating intensity of fouling was higher in the latter case [63]. In Kazi *et al*.'s study $CaSO_4$ deposition on stainless steel was seen to increase with concentration of the salt in solution [64] – after 4000 minutes deposition from 3.0 g/L of $CaSO_4$ solution was 50 g/m^2 compared to 150 g/m^2 deposition of 3.6 g/L $CaSO_4$ solution. The concentration of solids in sweet sorghum broth is approximately 2 wt% and we might expect a significant level of fouling (compare 2 wt% concentration for whey solutions).

Unfermented liquid total solid and dissolved solid content was found to be 12.3 wt% and 11.9 wt% respectively, using the NREL LAP for solid content determination in liquid biomass process streams. Sugar content of the unfermented sweet sorghum juice (sucrose 5.2 wt%, fructose 3.5 wt%, and glucose 4.1 wt%) makes up majority of the solids in this process liquid. From a fouling perspective, in fermentation processes, specifically in-field equipment, deposition of dissolved solid in compact layers can be expected.

3.4.3 Density

A Sartorius liquid density measurement kit with an analytical balance was used to determine the density of the fermented sweet sorghum liquid and the results can be found in Table 3.5.

Table 3.5: Feed Characterization: Density

Sample	**Average Density (g/cm^3)**	**Standard Deviation (g/cm^3)**
Raw Juice	1.0589	0.0002
Fermented Juice	0.9978	0.0005

The density of fermented sweet sorghum juice was 0.9978 g/cm^3. This is expected as ethanol has a lower density than water and constitutes a little more than 5 wt % of the solution. Water-like densities indicate that liquid displacement equipment need not be exceptionally large or specific and off the shelf equipment suitable for water can be used.

3.4.4 Viscosity

A Brookfield viscometer, RV-1 with Spindle #1 attachment was used to measure viscosity. The RPM was set at 20 and a total of 10 readings were taken for each sample. Three different replicate samples were used for viscosity measurement. The viscosity was recorded at an average temperature of 73.4°F for fermented samples, and 52.47°F for unfermented samples. Results for experiments are shown in Figure 3.2.

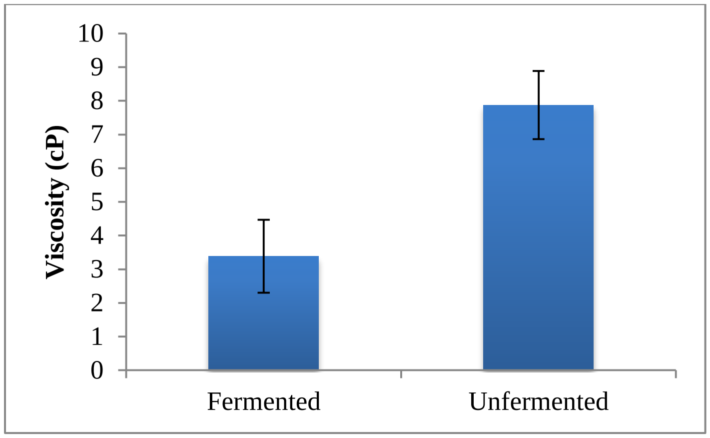
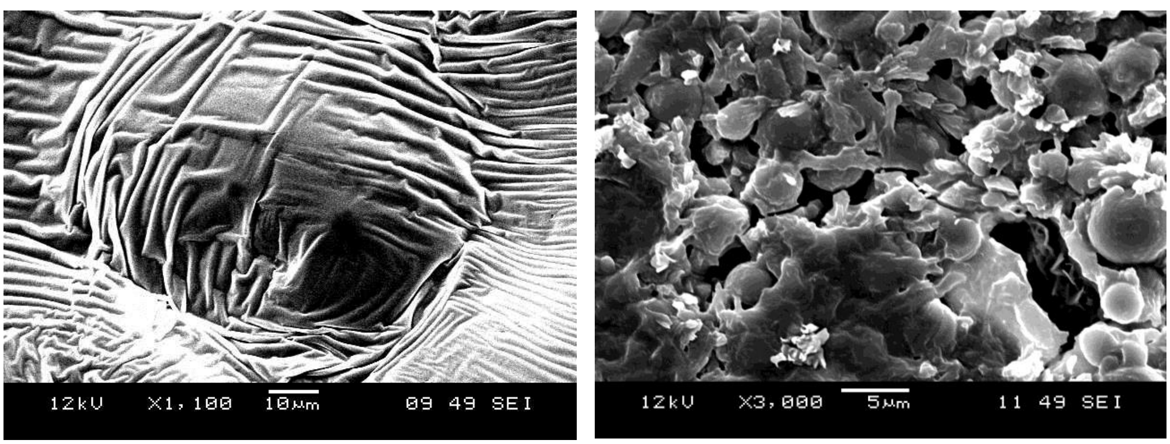


Figure 3.2: Viscosity of sweet sorghum – fermented and unfermented broth

The values for sweet sorghum raw juice viscosity have been reported to be 3 to 8 cP, and results for sweet sorghum juice tested here falls in this range [65]. Viscosity values for fermented sweet sorghum were approximately 3.387 cP. The fermented sweet sorghum liquid viscosity measurement is especially important, as literature instances for these measurements are rare. Measured values will help estimate, with additional accuracy, Reynolds number for piping and fluid transfer equipment – for example, in heat exchanger tubes where fouling is expected to be severe, the calculated Reynolds number using sweet sorghum physical properties (ρ = 997.8 kg/m^3 and μ = 3.387 cP) and tube dimensions (0.375 inch outside diameter, 0.02 inch wall, 2.38 ft tube length, 20 tubes per pass) at 4.2 gal/min flowrate will be 556, compared to 1629 using physical properties used for water (ρ = 997.8 kg/m^3 and μ = 3.387 cP).

3.4.5 Deposit Examination

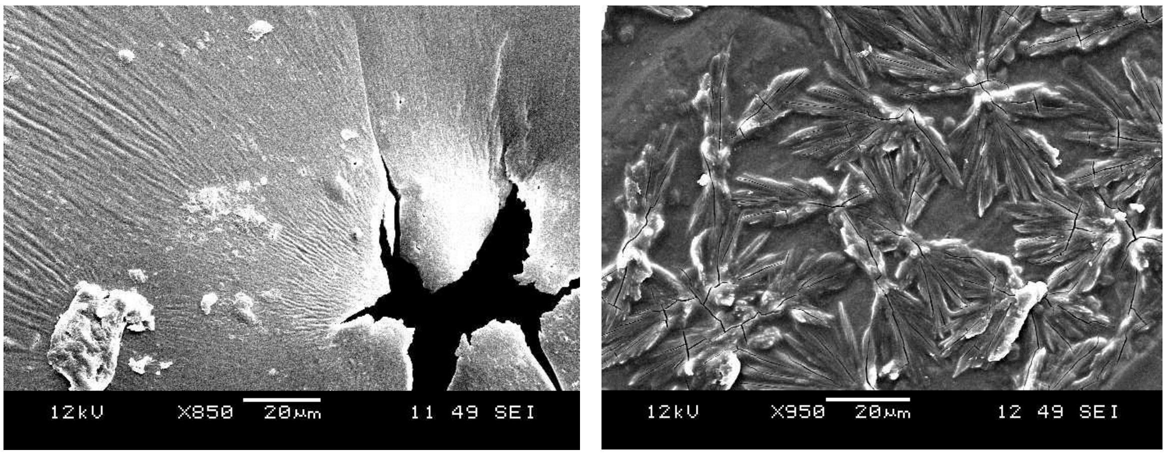
The deposits obtained in solid quantification experiments were examined using the SEM – this was primarily to observe the structure of deposits, the size of particulate matter, and to try and differentiate between different morphological details of deposits. Deposits used for SEM experiments were collected from solid quantification experiments, conducted at 105^{o}C. Unfermented juice deposits are seen to be compact with interconnected surface, as seen in Figure 3.3 (a). From initial observations it can be seen there is a prevalence of yeast cells between 2 and 5 μm in fermented sweet sorghum samples (before filtration) as seen in Figure 3.3 (b) – these results were similar in other findings where yeast cells deposited on sweet sorghum bagasse or other substrates were observed to be approximately 1 to 5 μm in size [66-69]. In Figure 3.3 (b), micrograph for fermented sweet sorghum deposits, yeast cell matrix is also seen interspersed with crystalline residues. Particulate and crystallization fouling often result in an interdependent connected 3-D structure [70].



(a) (b)

Figure 3.3: SEM Photos - Samples used are in total solid experiment: (a) Unfermented (b) Fermented

Particulates often provide nucleation site for the growth of crystal structures, and upon progression these structures trap a growing number of particles. Influence of multiple foulants and process conditions on deposit morphology will only be evident after dynamic deposition experiments.



(a) (b)

Figure 3.4: SEM Photos: Samples used are in dissolved solid experiment: (a) Unfermented (b) Fermented

Compact deposit layers have been observed in sugar process evaporator residues found along evaporator walls [71, 72]. The porosity of deposits change with distance from the heated surface on which fouling takes place, as well as deposit composition. Under conditions of solid quantification experiments (105^{o}C) sweet sorghum juice deposits are seen to have interconnected vein-like surface assemblies. These deposits show similar morphological features across the deposit as the residue heated the aluminum pans have been heated uniformly over the course of the experiment. Unfermented sweet sorghum juice will not be used in the bioethanol separation processes at ASU, but deposit morphology information can be useful for upstream fermentation processes, which use raw juice. Compact deposits are expected to have higher thermal resistance and will thus impact performance of heat exchange equipment with greater severity.

Figure 3.4 (a) also shows a compact deposit layer, in filtered liquid from unfermented juice samples, with presence of crystalline deposits on the smooth layer. Dissolved solid deposits on the other hand contain elongated crystalline structures, as seen in fermented dissolved solid samples, Figures 3.4 (b). Crystalline deposits found in sugar-related processes, particularly in evaporation equipment, cause severe fouling issues and have been found to contain calcium oxalates, which originate primarily from plant sources – the presence of sugar, silica and acids complicate the deposition process and result in crystalline deposits of varying morphology depending on composition [73]. Elemental composition analysis of fermented sweet sorghum deposits using X-ray diffraction (EDX) experiments coupled with SEM will be conducted in future to understand the nature of deposits and role of mineral impurities in fouling in sweet sorghum fouling processes. The exact nature of deposits will depend on processing temperatures, and flowrates, which will be tested using hydrodynamic experiments, in a separate publication.

3.5 Conclusions

NREL LAP for total and dissolved solids in liquid biomass was tested and found to accurately determine solid content in sweet sorghum fermented liquid. Standard density and viscosity measurement techniques can also be used for physical property determination of sweet sorghum fermented liquid. Total solid loading of fermented juice was determined to less than 3 wt%, significantly lower compared to corn-based process streams. Deposits are observed to be porous, three-dimensional with multiple components (crystallization, particulate deposition). Deposit morphology was seen to change with solid content - as a result deposit thermal properties and fouling nature of liquid are expected to be different in fermented vs. unfermented juice.

CHAPTER 4

EXPERIMENTAL INVESTIGATION OF TRANSPORT PROCESSES AND FOULING IN BIOFUEL PROCESS STREAMS

4.1 Abstract

The increasing popularity of sweet sorghum as a bioenergy feedstock for ethanol production via liquid or solid-state fermentation is motivated by easily accessible sugars, low inputs for crop growth, simple process set-up for bioethanol production, and a potential for complete crop utilization. Total solids content in sweet sorghum fermented liquid is approximately 3wt%. The movement of these solids through the system can have a significant downstream impact in terms of fouling particularly on heat exchange equipment. Experiments to quantify sweet sorghum fermented juice fouling were thus undertaken – the primary objective was determining fouling resistance of fermented sweet sorghum liquid. Resistance to heat transfer value of fermented sweet sorghum was found to be approximately 9.38 x 10^{-5} m^2 $^oC/W$ and fouling intensity is equivalent to that of tap water. Fermented sweet sorghum and water's R_f values were found to be in the same order of magnitude, one tenth of corn and maize ethanol thin stillage fouling resistance. This has been established using two independent experiments. The induction period for fermented sweet sorghum fouling was more than 5 hours. We expect low levels of fouling at bioethanol facilities will use fermented sweet sorghum for ethanol production.

4.2 Introduction

The increasing popularity of sweet sorghum as a bioenergy feedstock for ethanol production via liquid or solid-state fermentation is motivated by easily accessible sugars, low inputs for crop growth, simple process set-up for bioethanol production, and a potential for complete crop utilization. Pressed sweet sorghum juice contains between 16-18% fermentable sugars [74] which is directly fermented into 6 to 10 vol% ethanol beer solution using *Saccharomyces cerevisiae* [75]. The subsequent ethanol-water separation step, tailored towards small scale operation, is under investigation at Oklahoma State University (OSU) - experiments conducted at the OSU research and demonstration facility will establish the feasibility of a small-scale on-farm ethanol model, and bring to attention technical issues with the process [76].

The Alcohol Separation Unit (Figure A1.1) has two 38+ ft tall distillation columns central to the ethanol separation step. The beer column is equipped with 23 MVG type fixed valve trays with 15 inch tray spacing; the rectifier has a combination of trays and packing. Four MVG trays are present in at the bottom, and 13 feet of M752Y packing is present in the rectifier. The process has two heat exchangers – a 304 stainless steel shell and tube feed preheater, with 3/8 in (0.0095 m) OD tubes and stillage air-cooled condenser.

Feed is introduced into the top of the beer column, and 190 proof product is condensed and collected from the top of the rectifier. Stillage exits the process as bottoms product from the beer column. Sweet sorghum fermented juice, used as feedstock, and stillage from the process are both potentially fouling liquids. Total solids content in sweet sorghum fermented liquid is approximately 3 wt%. The absence of a biomass/cell removal unit operation at the OSU bioethanol pilot plant implies that the biomass containing fermented broth will be circulated through the process. Due to the relatively low initial solids content (when compared to corn based feedstocks which have a solid loading of approximately 16 wt%) of the sweet sorghum fermented broth, it can be pumped through pipes without extensive solids removal [77, 78]. Even though this is hydraulically feasible, the movement of biomass through the system is likely to have a downstream impact in terms of fouling specifically on the heat exchange equipment.

Limited information is available about fouling in heat exchangers from biological material and bio-based process streams. Fouling is expected in heat exchangers at the bioethanol separation facility, which is critical in terms of operation and on-spec production, and thus fouling occurring from sweet sorghum fermented broth on heated surfaces will be the focus of this study. The use of Tubular Exchangers Manufacturers Association (TEMA) resistance to heat transfer due to fouling (or fouling resistance) in heat exchanger design still remains the standard method to account for fouling. Standard prescribed R_f values are restricted to water and hydrocarbon process fluids [79]. Instances of over-design of heat exchangers remain common, at times 65% higher surface area [80], due to inclusion of high fouling resistances (R_f) in the overall heat transfer coefficient. An accurate estimation of sweet sorghum fouling resistance will provide new information critical for future development of heat exchange equipment and bioethanol processes that use sweet sorghum.

Experiments to quantify sweet sorghum fermented juice fouling were thus undertaken – the primary objective was determining fouling resistance of fermented sweet sorghum liquid. This was achieved by studying deposition under controlled hydrodynamic conditions using a heated test section. Two independent sets of experiments were conducted – the first apparatus described in detail was constructed at OSU and the second set of experiments were conducted using an annular fouling set-up used by Rausch et al [81]. The fouling resistance was estimated from surface and bulk temperature measurements. Fluid under investigation was sweet sorghum fermented broth. Test section surface was also analyzed after deposition to determine thickness of fouling layer, visual characteristics, and morphology of deposits.

4.3 Materials and Method

Fouling experiments were conducted in two separate apparatus - both experiments had electrical resistance heating for heat generation.

4.3.1 Apparatus

OSU Apparatus: In the apparatus constructed at Oklahoma State University an electrically heated stainless steel tube section was used to simulate the 3/8 in tube side flow in the shell and tube heat exchanger, E-1. Any deposition is expected to occur on the inside surfaces of the tube. A stainless steel skid assembly, equipped with a ½ HP Liquiflow geared pump for continuous recirculation of liquid, was central to the experiment. A 16 in x 24 in (0.4 m x 0.6 m) OD stainless steel tank was also included on the skid, which was used as the feed reservoir. Sweet sorghum fermented liquid flowrate inside the tube was 15.43 lb/min (359.6 kg/h) and velocity was 5.67 ft/s (1.72 m/s). A 304 stainless steel tube, 7 ft x 0.375 in OD x 0.02 in thick (2.13 m x 0.009525 m x 0.000508 m) section provided the test section.

Exterior surface temperature measurements (TT-1, TT-2, TT-3, TT-4) were taken at four locations as shown in Figure 1. TT-1 was furthest from the entrance, at 6 ft (1.82 m) and others were attached at 1 ft (0.3 m) intervals. The closest thermocouple to the entrance, TT-4, was 3 ft (0.91 m) from the entrance. Exterior surface temperature measurements were made in fully developed flow - the entrance region was 0.75 ft (0.22 m). K type thermocouples were secured to the tube with ¾ inch (0.019 m) electrically insulating 3M-vinyl professional grade electrical tape. Foam rubber tubing insulation for 0.375 inch OD x 0.5 inch (0.0095 x 0.0127 m) thick was wrapped around the test section. Inlet and outlet temperature measurements (TT-5 outlet and TT-6 inlet) were recorded using K-type thermocouples in Omega 0.25 inch x 6 inch (0.00635 x 0.1524 m) stainless steel thermowells.

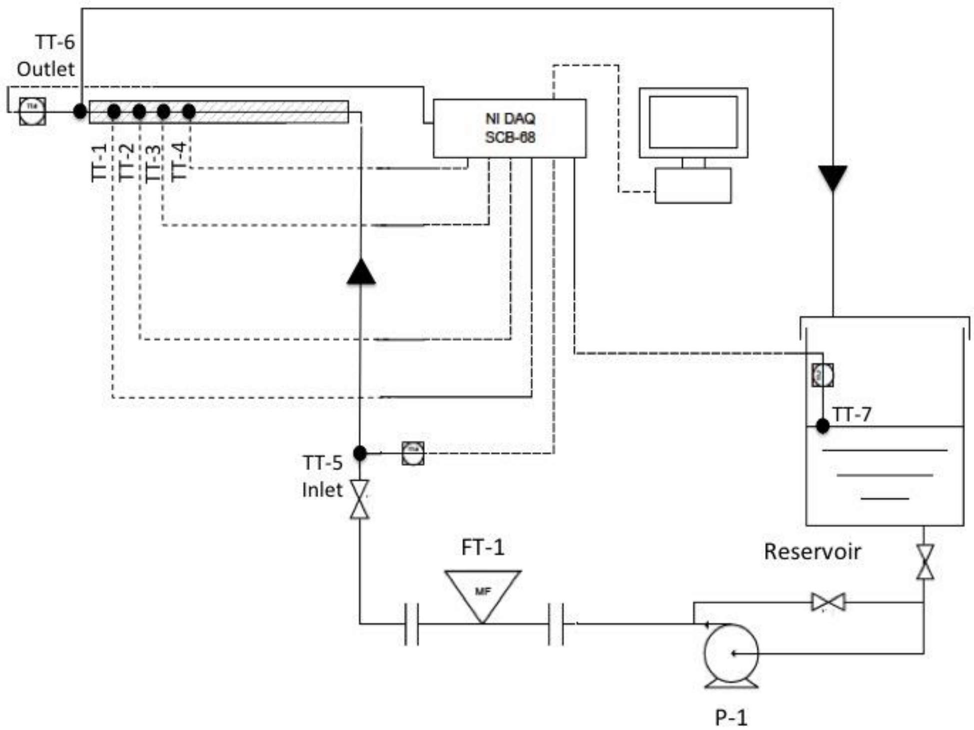


Figure 4.1: OSU fouling loop process flow diagram

Temperature of sweet sorghum liquid in tank was measured as well (TT7-Reservoir). All thermocouples were calibrated against a NIST traceable K-type handheld thermocouple. Calibration was carried out using a water bath - temperature of water varied from 0oC and 40oC. Table 1 shows the correction factors for all thermocouples. TT-1, TT-3, TT-Reservoir factors appear to be higher. This could be due to uneven heating/cooling of the water bath. Corrections to measured temperatures were made using Equation 1. Here, C is the correction factor listed in Table 4.1.

$$T_{correct} = T_{read} + C \qquad (1)$$

All temperatures were recorded using a National Instruments SCB-68 data acquisition unit connected to a computer. The DAQ unit provides cold junction compensation.

Table 4.1: Thermocouple details in experiment

Thermocouples	**TT1**	**TT2**	**TT3**	**TT4**	**TT5-Inlet**	**TT6-Outlet**	**TT7-Reservoir**
Distance from entrance (ft)	6	5	4	3		7	
Correction Factor (^oC)	1.2	0.7	1.3	1.2	0.4	0.8	1.1

A Lincolweld SA-250 electric welder was used to generate a voltage drop across the test section, required for electrical resistance heating.

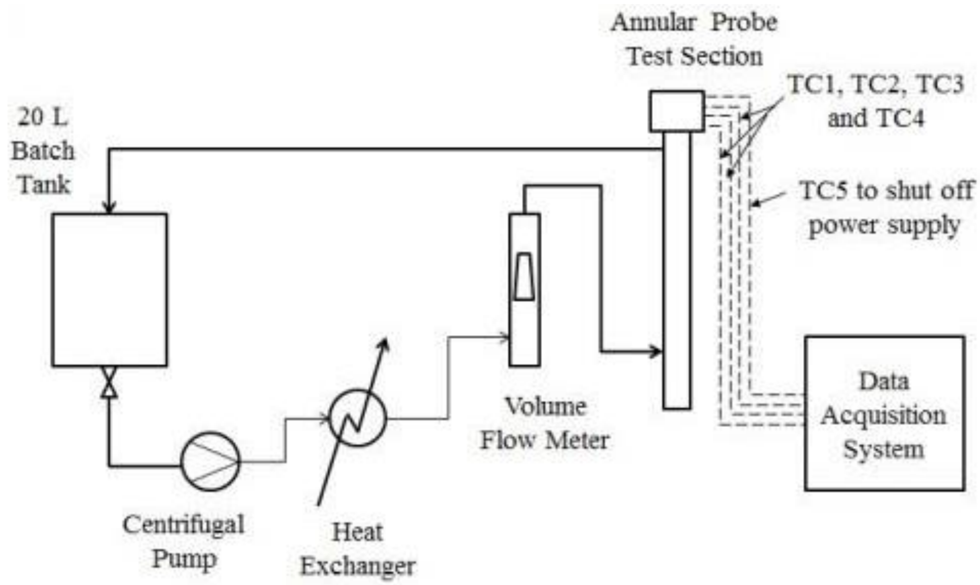
In addition to temperature measurements, current flowing in the system as well as the voltage drop across the tube section was measured using a HHM90 Omega multimeter. The current generated by was measured across a 500 milli-ohm shunt. A clamp on Extech 382060 power meter was also used to measure power applied to the test section. Temperatures were recorded every 60 s. Each fouling test run was carried out for 5 hrs. Accuracy and relevance of the experimental set up was established by conducting experiments with water. All conditions remained same and are tabulated in Table 4.2.

Table 4.2: Experiment conditions: Water and Sweet sorghum

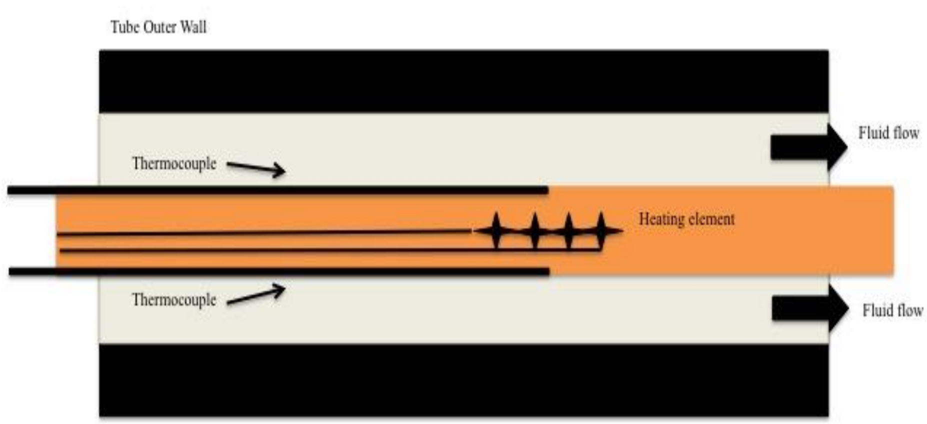
Experiment Conditions	Measurement	Units
Time	5	hrs
Flowrate	359.6	kg/h
Voltage across section	6.25	V
Current in tube section	124	A
Tube outside diameter	0.375	in
Heated test section length	1.93	m

Fermented sweet sorghum liquid used in the fouling experiments was used in the Alcohol Separation Unit in 2014. Five 15 L samples were withdrawn from the 5000 gallon storage tank on site. In order to ensure mixing the liquid was allowed to recirculate for 3 hours prior to sample withdrawal. Three samples were tested with Rausch *et al.*'s apparatus and the rest were tested with the OSU apparatus.

Rausch *et al.*'s apparatus: A 316 stainless steel annular fouling probe was used as the main test section (Watlow FIREROD 1025, Figure 2) – a 2000W/208 V heat source was housed in the center, with fluid flow in the annulus.



(a)



(b)

Figure 4.2: Rausch *et al*. (a) Experimental set up and (b) Annular fouling apparatus:

Total tube length was 0.61m and the diameter was 0.0126 m. Total heat transfer area available was 0.0034 m^2. The heated section was 0.286 m from thermocouple lead end and provided a 0.1016 m heating zone. Four thermocouples were located close to the inner wall of the test section of which three were used for wall temperature measurement. The fourth thermocouple was used to monitor wall surface temperature - a cut-off maximum limit of 200^{o}C was in place, beyond which the power supply to the experiment was cut off. A 20 L batch tank was used to hold liquid and a liquid batch size of 7 L was used in all experiments. Reynolds number was 460 (±10) and the power supply to the unit was constant at 410 (±10) W to maintain an initial probe surface temperature of 120^{o}C. The bulk temperature of the sweet sorghum liquid was maintained at 75^{o}C. Further details of the experimental set up can be found in Challa *et al*. [82].

4.3.2 Calculations

OSU Calculation: Fouling resistance was calculated from test section wall temperature T_{wall} and fluid bulk temperatures T_{bulk}. The local heat transfer coefficient, h (kW/m^2 ^{o}C), was experimentally determined from temperature measurements using Equation 2. Q is the power supplied to the test section in watts; A is the surface sectional area available for heat transfer in m^2.

$$h = \frac{Q}{A(T_{wall} - T_{bulk})} \tag{2}$$

Bulk liquid temperature was estimated using Equation 3 from inlet and outlet fluid temperatures. Heat generation along the length of the tube is assumed to be uniform. Under uniform heating conditions the fluid bulk temperature inside tube [83] can be approximated with a straight line. Fully developed flow conditions are assumed and the temperature increases linearly along the length of the tube from T_{inlet} to T_{outlet} as per Equation 3.

$$T_{bulk} = T_{inlet} + (T_{outlet} - T_{inlet})\, x/L \qquad (3)$$

T_{inlet} is recorded using TT5 and T_{outlet} is measured using TT6. Surface temperature measurements on the heated section include TT1, TT2, TT3, and TT4. These record the outside surface/wall temperature. The inside wall temperature was calculated using Equation 4. Conditions in this experiment were analogous to heat conduction in a cylindrical pipe with internal heat generation [84].

$$T_{wall} = -Sr_1^2/4k + a * \ln r_1 + b \qquad (4)$$

Power in the system, Q was determined using Equation 5, calculated from current and voltage measurements. Here, V (volts) is the voltage across the test section and I (ampere) is the current flowing in the system.

$$Q = V\, x\, I \qquad (5)$$

In order to estimate hydrodynamic and heat transfer parameters (Reynolds number, and Nusselt number) accurately physical parameters, namely density and viscosity were determined experimentally. An analytical balance (Model BP-301S) with a density measurement kit (Sartorius, YDk-01) for liquid densities was used for density measurement. The sweet sorghum fermented liquid sample was heated to 75^oC and the glass plummet was suspended in the liquid. The density was recorded as the liquid cooled, to 30^oC. The density data was regressed against temperature to generate a response curve (R^2 = 0.97), described by Equation 6 (kg/m^3).

$$\rho = 1{,}024.017 - (1.465)T + (0.037)T^2 - (2.807 \times 10^{-4})T^3 \quad (6)$$

Sweet sorghum rheological properties were determined using a SALS 60 mm parallel plate rheometer. A stainless steel 997436 plate with 500 μm plate distance was used for the experiment. The temperature was varied from 25°C to 60°C. The viscosity (Pa.s) was regressed with temperature ($R^2 = 0.97$) and is given in Equation 7

$$\mu = 0.00307 - (6.800 \times 10^{-5})T + (4.4 \times 10^{-7})T^2 \quad (7)$$

Due to the absence of physical property data for fermented sweet sorghum liquid equation 8 was used for specific heat (J/g °C) – this equation is valid over temperature range 0°C to 80°C [85].

$$C_p = 4.2085 - (3.022 \times 10^{-3})T + (7.833 \times 10^{-5})T^2 - (4.90 \times 10^{-7})T^3 \quad (8)$$

Temperature dependent fluid thermal conductivity values were also obtained from correlations used to describe water. Over the temperature range of 1 to 97°C Equation 9 was used to estimate thermal conductivity (W/m K) values [86]. $T^* = T/298.15$ and k^* is thermal conductivity of water at 298.15 K and 0.1 MPa, 0.6065 W/m K.

$$\frac{k}{k^*} = -1.48445 + (4.12292)T^* - (1.63866){T^*}^2 \quad (9)$$

The Sieder Tate equation was used to compare predicted heat transfer coefficient and experimentally determined heat transfer coefficients for water and fermented sweet sorghum. Fouling resistance was calculated using Equation 10

$$R_f = \frac{1}{U_t} - \frac{1}{U_0} \tag{10}$$

Rausch *et al*. Calculation: The overall heat transfer resistance (W/m^2 ^{o}C) was calculated from average of three values, representing three thermocouple locations. Equation 2 was used to calculate the local heat transfer coefficient at each thermocouple location. The outside wall surface temperature, T_w was calculated using Equation 11. Here T_w is the temperature measured at the inside wall of the fouling probe and *x* is the radial distance of the thermocouple from the surface. Also, *k* is the thermal conductivity of the metal.

$$T_s = T_w - \left(\frac{Q}{A}\right)\left(\frac{x}{k}\right) \tag{11}$$

The ratio (*x/k*) was determined experimentally using techniques described in Arora *et al*. [87]. The ratio, *x/k* was, from T1 to T4 **-** 0.075, 0.109, 0.097, and 0.097 for this experiment.

4.3.3 Deposit Collection and Examination

A Joel 500 Scanning Electron Microscope was used to study the morphology of the deposits, and characterize the layer formed. The SEM experiment was conducted at 16 kV. A gold/Au coating was applied to the deposit to ensure conductivity within the sample during the experiment – sputtering times of 30 s was used. A magnification range from 500x was used for image capture. A section of the tube (0.5 in x 0.5 in) was used to study deposits.

4.4 Results and Discussion

Total solids content in the fermented sweet sorghum liquid was 3 wt%. The concentration of ethanol in the broth was 4.5 vol%. Total sugar (glucose, sucrose, and fructose) levels were less than 2 g/L. Glycerol and acetic acid levels in fermented sweet sorghum broth were 0.7 g/L and 1.2 g/L, respectively.

4.4.1 OSU Fouling Experiments

From experiments conducted with water, neither fouling on tube surface or increase in fouling resistance was observed. Average heat transfer coefficient values for two tests with water gave reproducible results (Figure 4.3 a). Average fouling resistance for tap water was determined to be 1.0 x 10^{-4} m^2 $^oC/W$ comparable to fouling resistance factors reported in literature [88]. It was thus concluded the set-up would fulfill its intended purpose. For fermented sweet sorghum liquids typical values for fluid properties estimated at bulk fluid temperature of 47^{o}C (Table 4.3).

Table 4.3: Fermented liquid sweet sorghum properties and other conditions

Density	Viscosity	Inside Wall Temp	Fluid Temp	Fluid Velocity	Reynolds Number	Specific Heat	Thermal Conductivity
kg/m^3	Pa.s	^{o}C	^{o}C	m/s		J/kg K	W/m^2 K
1069.58	1.13 x 10^{-3}	49.3	47.4	1.64	13,284	4177	0.636

The average rise in fluid temperature from the inlet to the outlet was 3.0^{o}C. Temperature of the reservoir fluid increased from 25^{o}C to 60^{o}C over the course of the experiment. Average heat losses in the experimental set up were found to be 12%, from Figure 4.3 (b). The average local heat transfer coefficients calculated using Equation 2 was 6,453 W/m^2 ^{o}C for fermented sweet sorghum liquid. These observed values were close to that of water, as seen from Figure 4.3 (c).

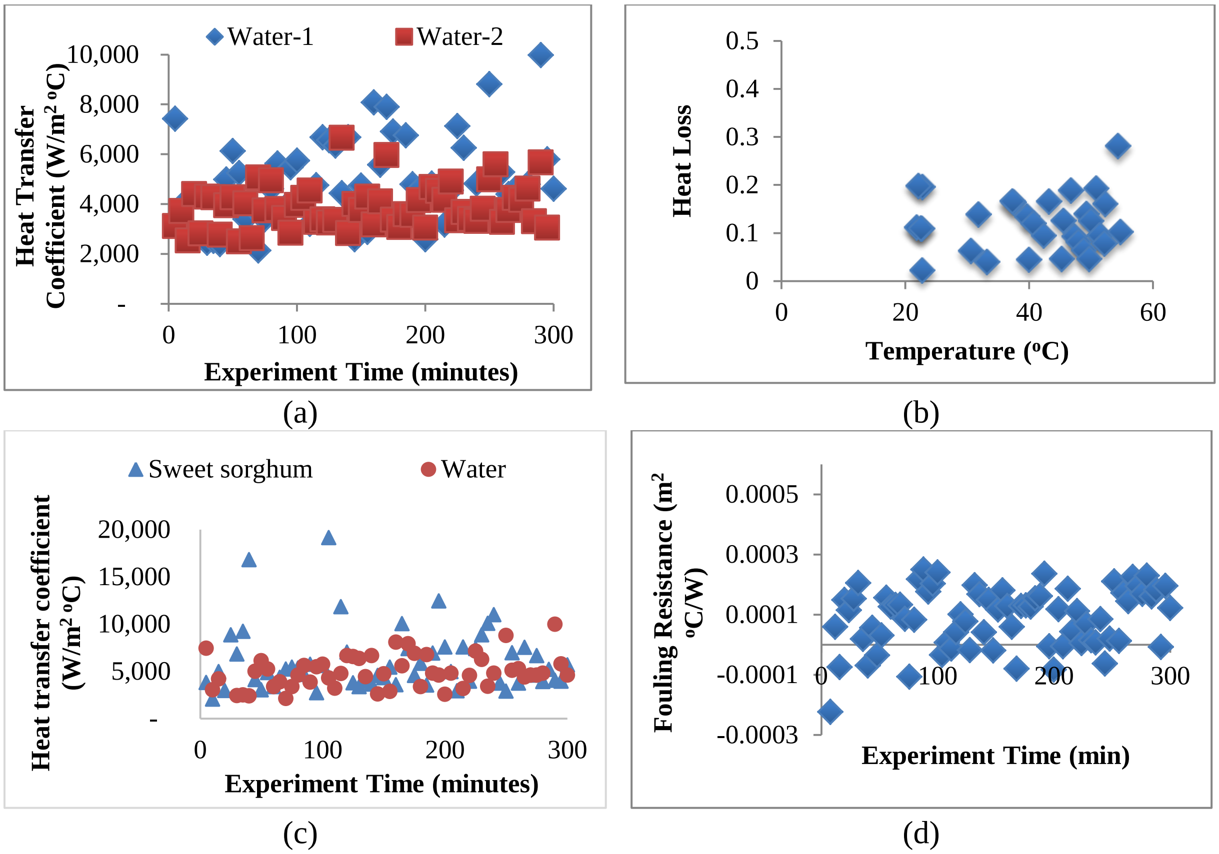
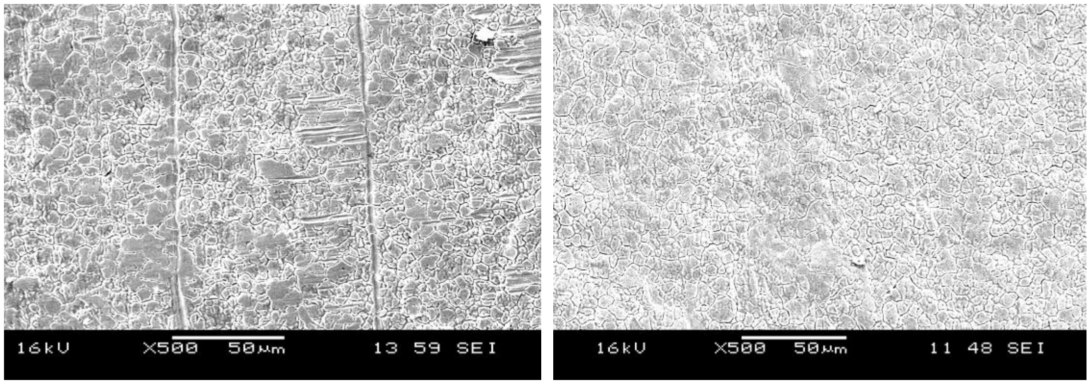


Figure 4.3: Experimental Results (a) Water tests for reproducibility and accuracy of apparatus (b) Heat loss from experimental set up (c) Heat transfer coefficient for water and sweet sorghum (d) Fouling resistance calculated for fermented liquid sweet sorghum

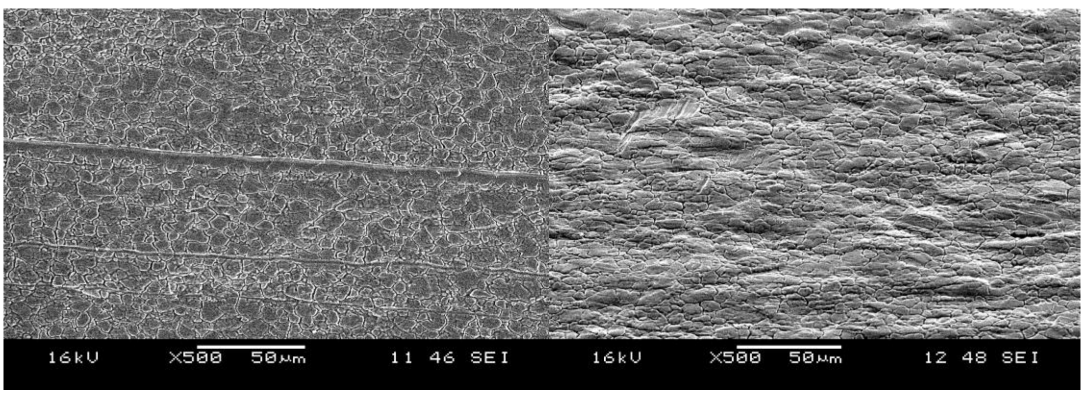
Fouling resistance was not seen to increase with time – it remained near constant after five hours of experimentation, as seen from Figure 4.3 (d). Maximum fouling resistance value is the value of fouling resistance recorded after five hours, and was 9.38 x 10^{-5} $m^2\,^oC/W$. This value was in the same range as water - fouling resistance of tap water was 1.5 x 10^{-4} $m^2\,^oC/W$ at 1.5 m/s after 300 hours established by Sung *et al.* [88].

SEM examination of tube sections were done from three sections – 1) surface of tube before deposition (2) top surface of tube after fouling experiment (3) bottom surface of tube after experiment. Average roughness factor (Ra) of 30 nm have been reported for stainless steel [89] and topology examinations via SEM experiments show heterogeneous surfaces also with cracks, as seen in Figure 4.4 (a).



(a) (b)

Figure 4.4: SEM Micrograph for stainless steel 304 surface before deposition (a) Top and (b)



(c) (d)

Figure 4.4: Bottom. Stainless steel surface after deposition (c) Top and (b) Bottom.

Initial tube surface is devoid of any deposits (Figure 4 a). We did not observe any significant deposition of sweet sorghum solid content on the stainless steel surface, as seen from Figure 4 (b), (c) and (d).

4.4.2 Rausch *et al.* Experiments

Fouling resistance measured using the annular fouling probe did not show increase during the course of the experiment, as seen in Figure 4.5 (a), indicating induction periods longer than 5 hours. The maximum average fouling resistance value of sweet sorghum fermented broth was 3.5

x 10^{-6} m^2 $^oC/W$. The probe surface was visually observed to be clear of any deposition, as seen in Figure 5 (b).

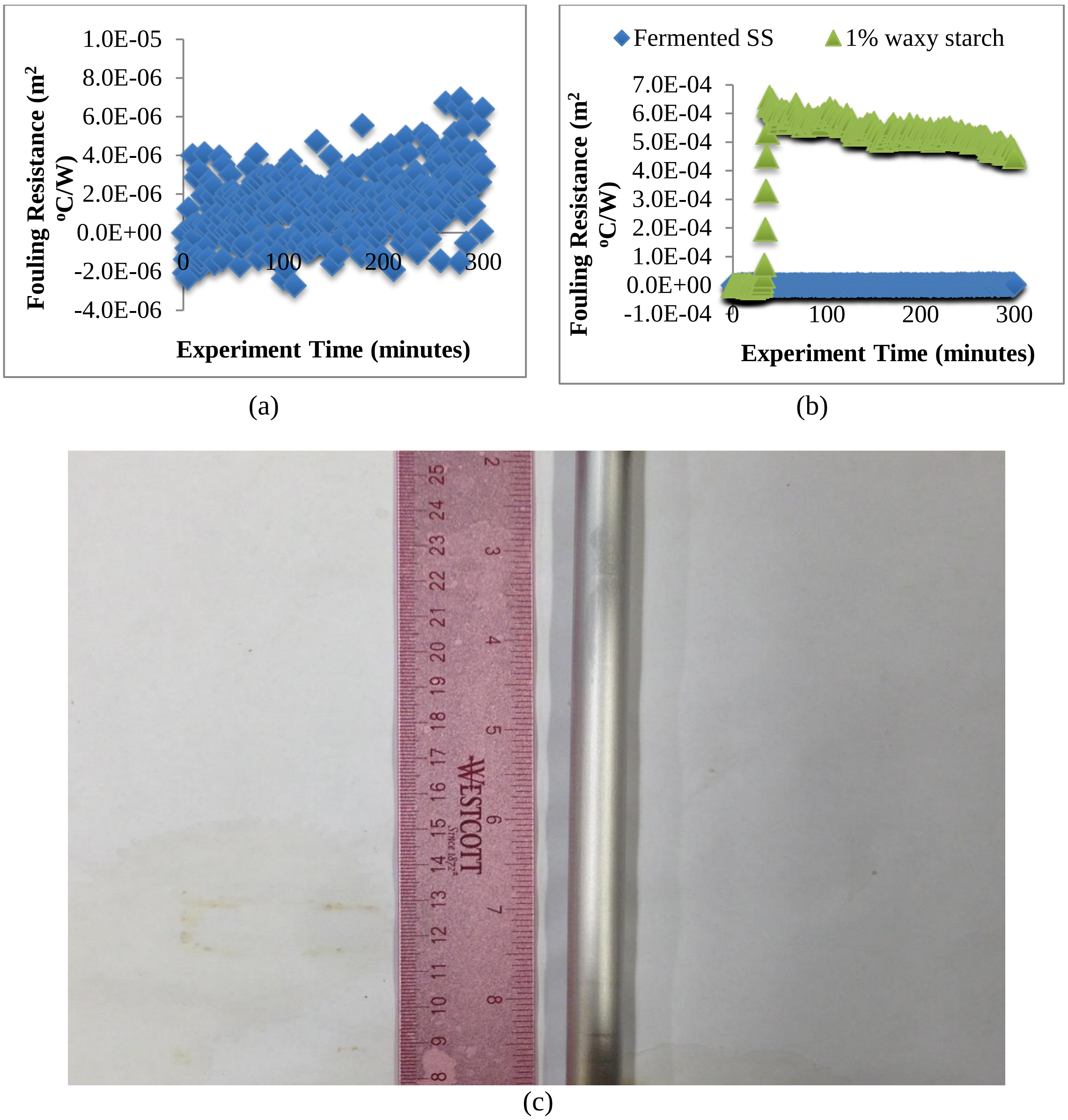


Figure 4.5: Rausch *et al*. Results: (a) Fermented sweet sorghum (b) Compared to 1% waxy starch (c) Fouling probe after experiment – no apparent deposition

Fouling experiments done in both OSU and Rausch *et al.*'s experimental set-ups establish that fermented sweet sorghum liquid provides a low fouling alternative bio feed environment. Overall, fouling problems are pervasive in process industries; however the manifestation tends to differ industry-wise – for example, in petroleum refineries heat exchangers are cleaned once or twice a year [90]. Heat exchangers serving milk processing operations, on the other hand, are cleaned daily to remove deposits.

In ethanol manufacturing operations from molasses, solid removal was found to be useful in reducing fouling in downstream distillation columns [91]. A two-step clarification operation used to remove sludge (containing suspended solids, inorganic) provided three fold improvement in the cleaning cycle times, increasing the time between cleanings from 3 to 4 months to 9 to 12 months.

In starch-based ethanol processes, fouling is an issue in distiller's dry grains with solubles (DDGS) related streams and equipment. Pipe plugging and heat exchanger efficiency reduction have been reported in DDGS based pilot plant operations [92], but the majority of the fouling problems are present in multi-effect evaporators used to concentrate corn or maize stillage streams into DDGS in dry grind ethanol plants. Fouling resistance of thin stillage from corn ethanol dry ethanol processes is approximately 3.5×10^{-4} m^2 $^oC/W$ [34, 93], an order of magnitude higher than fermented sweet sorghum liquid. Fouling induction period for thin stillage was very short, as low as 2.5 min at low Reynolds numbers, and increase in fouling resistance values was immediate for maize thin stillage [94]. Compared to this, fermented sweet sorghum induction times were more than 5 hr (Figure 4.5 (b)). Actual induction times will be ascertained after longer (150 hr) experiments are conducted.

With 12-21% directly fermentable sugars, and no starch to convert, sweet sorghum is a good choice as ethanol feedstock [95]. With sugar conversion efficiencies of more than 90% [18], leftover sugars in the fermented broth are minimal with levels in the current investigation at less than 1.2 g/L. The absence of starch in fermented sweet sorghum broth could be the reason behind such low fouling rates seen from the liquid, when Challa *et al.*'s work is considered [82]. In preliminary experiments with DDGS constituents, glucose and corn syrup solids did not show any fouling, but long chain polymeric starch fouled significantly [38, 81]. Starch fouling was immediate and R_f values in the order of 4 x 10^{-4} $m^{2o}C/W$ were recorded (Figure 4.5 (b)).

With a significantly lower fouling resistance value and longer induction times for fermented sweet sorghum, fouling in equipment and the frequency in cleaning in processes is expected to be lower. Using sweet sorghum as a bioethanol crop is then advantageous. Pilot plant experiments at the Alcohol Separation Unit will be able to confirm the effect of fouling on industrial sized heat exchangers.

4.5 Conclusion

Resistance to heat transfer of fermented sweet sorghum is equivalent to that of tap water. The two liquid fouling resistances were found to be in the same order of magnitude, only about one-tenth of the resistance found in corn and maize ethanol thin stillage. This has been established using two independent experiments. The induction period for fermented sweet sorghum fouling was more than 5 hr. We expect low levels of fouling at bioethanol facilities which use fermented sweet sorghum for ethanol production.

4.6 Acknowledgements

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4.7 Symbols

a	$(T_{inside-wall} - T_{outside-wall}) + \frac{(\frac{S}{4k})\times(r_2{}^2 - r_1{}^2)}{ln\frac{r_2}{r_1}}$	
A	Surface area of test section	m^2
b	$T_{inside-wall} + \frac{Sr_1{}^2}{4k} - a\ lnr_1$	
C	Correction factor	^oC
C_p	Specific heat of water	J/g ^oC
h	Local heat transfer coefficient	$W/m^2\ ^oC$
I	Current in test section	ampere
k	Thermal conductivity	W/m ^oC
k^*	Thermal conductivity of water at 298.15K and 0.1 MPa	W/m K
L	Length of test section	m
Q	Power supplied to the test section	W
r_1	Inside radius	m
r_2	Outside radius	m
R_f	Fouling resistance	$m^2\ ^oC/W$
S	Heat generated per unit volume in test section	W/m^3
$T_{correct}$	Corrected measured temperature	^oC
T_{read}	Measured temperature	^oC
T_{wall}	Inside wall surface temperature	^oC
T_{bulk}	Fluid temperature	^oC
T_{inlet}	Fluid temperature measured at inlet	^oC
T_{outlet}	Fluid temperature measured at outlet	^oC
T^*	T/298.15	
U_0	Overall heat transfer coefficient at time t=0	$W/m^2\ ^oC$
U_t	Overall heat transfer coefficient at time t=t	$W/m^2\ ^oC$

V	Voltage across test section	volts
x	Distance from entrance of test section	m
ρ	Density of water	kg/m^3
μ	Viscosity of water	Pa.s

Rausch *et al.*

T_s	Outer surface temperature of heated rod	^oC
T_w	Inner wall temperature measured by thermocouples	^oC
Q	Power supplied to the heater	V
A	Surface area of the heated section of probe	m^2
x/R	Calibration constant specific to probe	

CHAPTER 5

FARM SCALE ETHANOL SEPARATION FROM SWEET SORGHUM

5.1. Introduction

This chapter is a techno-informational guide to assist in building and operating an on-farm distillation unit. This on-farm unit is fit to handle ethanol separation from fermented liquid feedstock with low solid content. Technology for small-scale ethanol production has been demonstrated at Oklahoma State University using sweet sorghum fermented juice as feedstock. The design was robust, and successful operation and ethanol production was demonstrated in 2013. We have shared information on different forums via publications – but simple explanations, timelines, key learning, simple fixes, and troubleshooting experience need special attention. We have tried documenting all of the mentioned information in this handbook. The purpose is to provide an overview of how to prepare for and implement distillation operations on a small scale.

Sweet sorghum provides a low input, drought tolerant, and easily implementable option for bioethanol production. Once harvested though, carbohydrates in sweet sorghum have a short window before being consumed by in-field contaminants. Storage and transportation are both either unviable or uneconomical [96, 97]. One way to circumvent the degradation problem is to ferment the carbohydrates immediately, in-field. The on-farm concept for production of ethanol from sweet sorghum germinates here. An on-farm facility for separating and dewatering ethanol can be conceptualized as a small- scale unit, operated by the farmer, which uses a decentralized model for ethanol production [98].

Sorganol™, which is ethanol produced using sweet sorghum as feedstock, is central to the concept of on-farm ethanol dewatering and is described in detail in Fig 5.1 The process involves harvesting and pressing the juice out of sweet sorghum stalks on farm, fermenting the sugar juice followed by the ethanol separation step to produce fuel grade ethanol on farm.

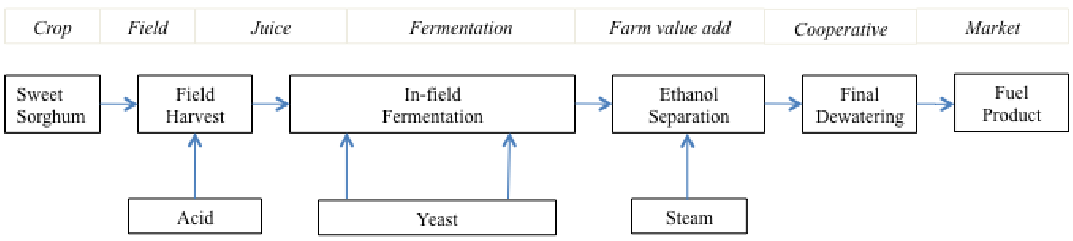


Fig 5.1: The sweet sorghum bioethanol process

Economic modeling work at the Department of Agricultural Economics as well as other investigations into small-scale ethanol production suggest smaller units for azeotropic ethanol production, followed by a central cooperative (Figure 1) servicing satellite on-farm units is an economically attractive and technologically feasible solution [20, 99, 100]. Activity in early 1980s around small-scale ethanol production and renewable energy in general was ripe, after the oil embargo of the late 1970s. Several demonstration units were constructed all over United States and several government organizations including the United States Department of Agriculture (USDA) and Department of Energy (DOE) had funded these projects for on-farm ethanol production. The Solar Energy Research Institute (SERI) released a technical manual for farm to fuel production in 1982 [21], which provided practical information for farmers interested in pursuing farm scale ethanol production. Although the information provided was valuable for farmers looking to invest in ethanol production, technical issues with the separation step were never resolved.

In a DOE report [30], the author, White noted that on-farm facility technical issues were attributable to "poor technical advice and inadequately-proven plant designs." Attention to fuel ethanol production was declining by mid 1980s due to a significant decline in oil prices and the resulting loss of state gasohol exemptions. The work started in the late 1970's and early 1980's for on-farm distillation was never concluded. Initial results revealed the need for improvements to reduce costs but the required follow-up was never initiated due to the rapid decline in oil prices in 1982. The work that was pursued at Oklahoma State University can be viewed as a resumption of earlier effort with the benefit of 30+ years of additional experience and technology evolution. The Alcohol Separation Unit in Figure 5.2 is a robust separation process compared to its predecessors from the 1980s.

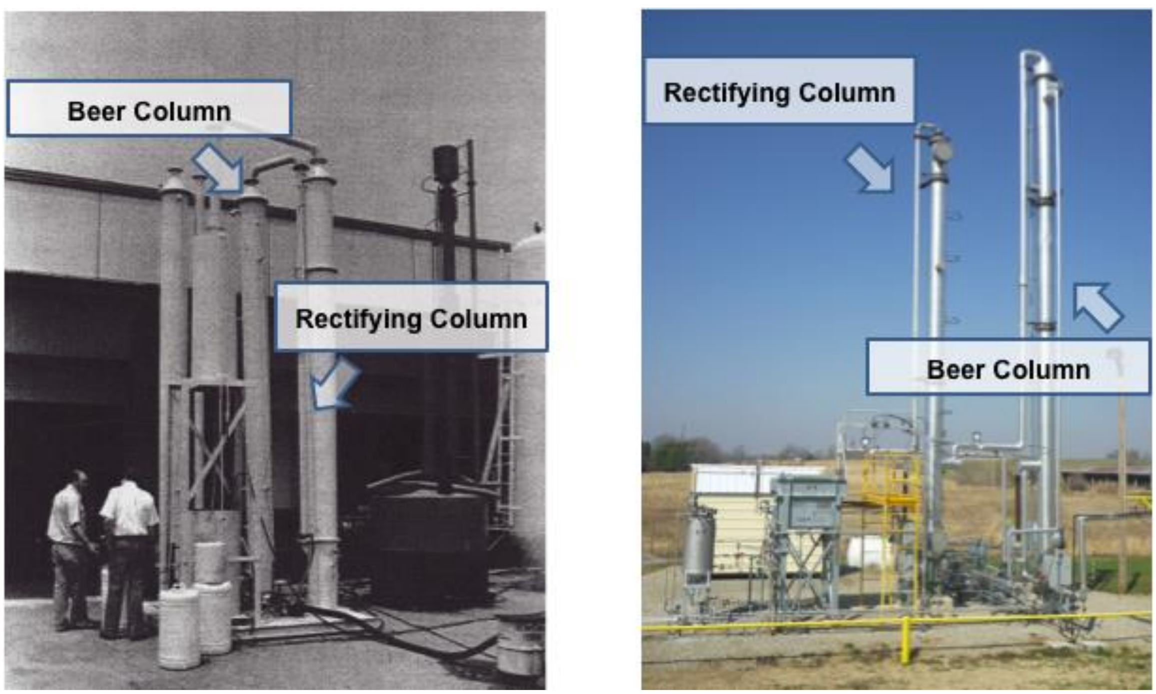


Figure 5.2: Alcohol Separation Unit compared to on-farm work from 1980s

Preliminary studies at the Department of Biosystems and Agricultural Engineering at Oklahoma State University shows that successful ethanol fermentation is possible from sweet sorghum with minimum process, pH, or temperature control. A dilute ethanol solution, with 6-10 vol% ethanol is obtained. This dilute ethanol solution is concentrated to 99.9 vol% to reach fuel grade specifications. The Alcohol Separation Unit at Oklahoma State University was constructed to demonstrate the initial ethanol water separation step (from 6 vol% to 95 vol%). The incoming sweet sorghum liquid was already fermented on reception. Also final dewatering was not part of this construction and only the distillation design results are shared here. Framework for workflow, plant design criteria, as well as materials, instruments and equipment selection are discussed in this document. A detailed start up and shut down process is also present in this document.

5.2. Plant Design Criteria

5.2.1 Distillation Technology

The step from low to fuel grade ethanol specifications often takes two distinct processes: the first process, commonly distillation, concentrates the ethanol to a 95 vol% solution and a final dewatering step is needed to reach fuel grade specifications. Ethanol-water solutions form a constant-boiling ‘azeotropic’ mixture, which do not separate after 95 vol% upon boiling. Distillation, which uses difference in boiling point as a physical property basis for separation, thus falls short in delivering ethanol concentrated above 95 vol%. Dewatering steps typically use membrane separation, or molecular sieve adsorption to physically remove water from the concentrated ethanol water mixture to produce fuel grade ethanol product. Such hybrid or combination technologies have been tested for bioethanol concentration, but all dewatering facilities still have a partial distillation step [101]. This is to ensure a concentrated (80+ wt% ethanol) and clean (free from solids) stream going into the dewatering unit.

Ethanol separation process schemes involve two columns – the beer column and the rectifier. The columns themselves are separated due to height restrictions. A thin self-supported column more than 40+ ft can pose safety concerns. Having separated beer and rectifier columns allows for solids to concentrate in the beer column, which are typically equipped with column internals appropriate for dirty or fouling liquids. Column technology development over the 30+ years from initial trials of farm scale that have been included into the distillation unit are:

(a) Column Internals: cartridge valve trays for high solid content liquids and fouling service and high efficiency M752Y structured packing for the rectifier
(b) Column design features: combination of trays and packing for improved column efficiency and to facilitate fusel draw removal.
(c) Air cooled heat exchanger as a condenser for reduction in water consumption
(d) Stainless steel piping and equipment for longevity and minimal cleaning requirement

These features were included as design details in the process were established. The first step in workflow of the pilot plant was determining the design basis of the pilot plant. This also established hydraulic, piping design, and all engineering decisions involved in detailed design.

5.2.2 Design Basis

The conditions that were considered during initial design phase:

1. Continuous operation of a steady state process servicing a 500 acre farm. A 30 ton/acre sweet sorghum yield is expected, and juice yields of 4000-6000 gal/acre. A 250 gal/h feed rate for the process is appropriate (plant operates for 300 days).
2. Ethanol concentration of 6-10 vol% in fermented liquid sweet sorghum can be expected. Design was based on ethanol concentration of 6.5 vol%.

3. Azeotropic ethanol product from distillation step expected. Final ethanol product of 95 vol% or 190 proof ethanol was the target. Ethanol recoveries were at least 90%. A product rate of approximately 15 gal/h was calculated, from initial design simulation.
4. Process designed to operate at atmospheric pressure. This minimizes operating and initial capital expenditure in addition to providing a safe operating environment.
5. A two-column process with a beer column and rectifier was used.
6. Feed to the distillation column entered at 150^{o}F. Entering feed was heated from the hot stillage bottoms stream leaving the column. This increased the efficiency of the operation.
7. Steam is available onsite at maximum pressure of 150 psig. The vertical tubeless 15 HP boiler present on site was used for steam injection into the beer column. A steady state steam rate of 333 lb/h was initially assumed.
8. Process had extensive data collection, control, and monitoring capability

Control strategy for distillation column controls was selected to reflect updated practical process control and includes (a) material balance (b) product quality and (c) satisfaction of process constraints as discussed by Luyben *et al*. [102]. For material balance all inventories (this includes all liquid levels and column pressure) were controlled. Composition control (for product quality) was achieved by controlling/maintaining top temperature. Steam control was provided to ensure safe operation. Steam flowrate control also ensured the process was always operating within its design constraints.

5.3 Framework for Setting Up Pilot Plant

5.3.1 Design Basis and Equipment Selection

As seen in Figure 3, the first step in establishing a successful process is establishing a working design basis. A CHEMCAD or ASPEN simulation to establish steady state operating conditions for the process is required (Figure 5.3).

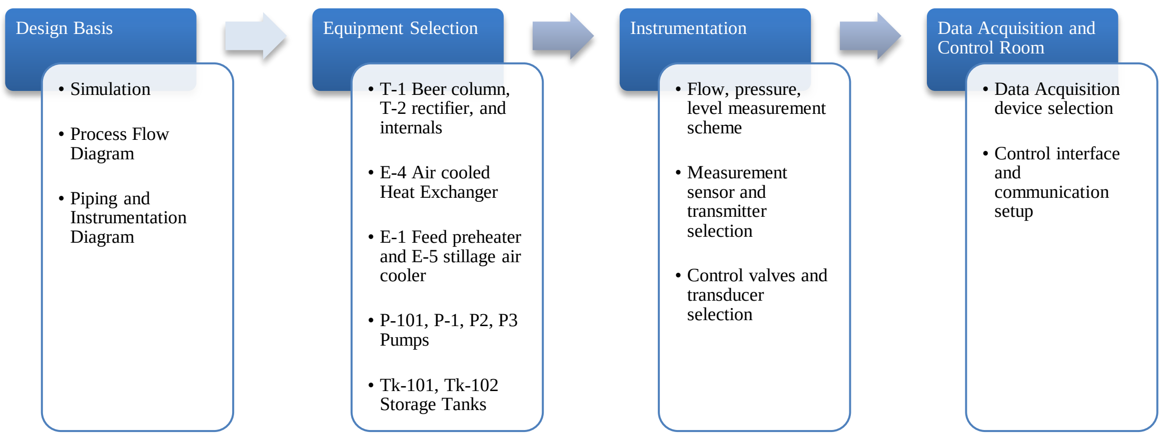


Figure 5.3: Framework for Detailed Design

For a chemical process all subsequent steps will be based around this design. Appendix A1 contains the CHEMCAD simulation output for the Alcohol Separation Unit. A process flowsheet, detailing pressures, flows in all process lines is part of Appendix A2. A Piping and Instrumentation Diagram (P&ID) outlines piping connections, line sizes, control strategy, and instrumentation (in-field and transmitted). The P&ID was/is always a work in progress and was changed according to any piping modifications made on site. A P&ID for the Alcohol Separation Unit and hydraulic summary of piping are available in Appendix A3.

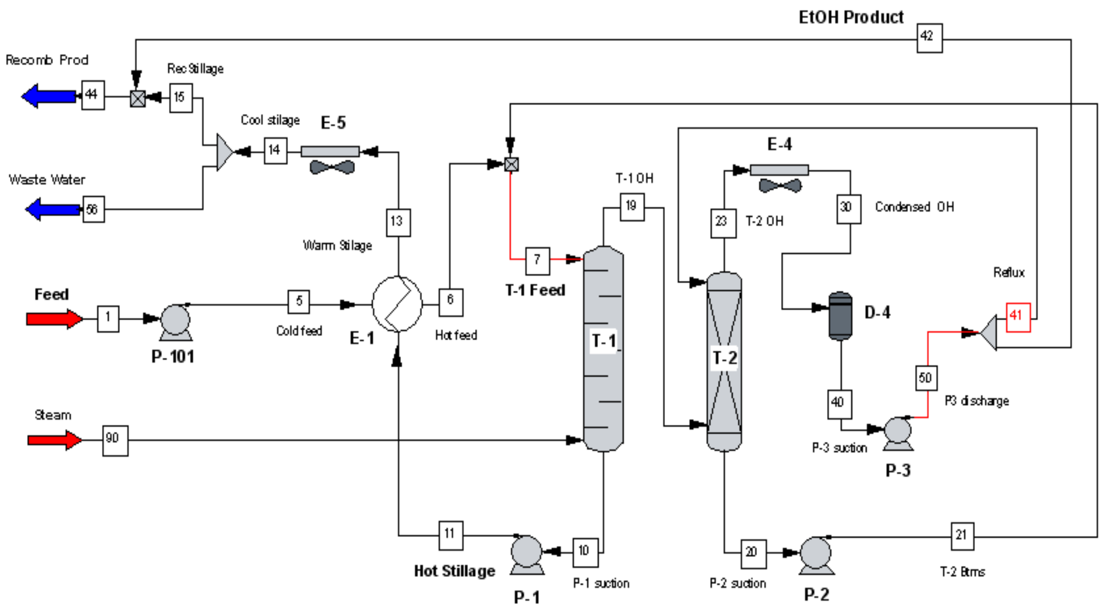


Figure 5.4: Alcohol Separation Unit Process Flow Diagram

The design basis forms the foundation for equipment selection procedure of the pilot plant. We found information exchange while reaching out to suppliers for quotes, and meeting manufacturers or fabricators for orders primarily revolved around information in the primary design basis. A Request for Quotes (RFQ) for the feed heater E-1 is in Appendix A5 and shows how physical property values from simulated process streams in CHEMCAD, as well as heat curves were used while ordering equipment.

Equipment selection affected nature and location of piping, as well as the type of fittings used in the pilot plant. For example, in the distillation columns, the location and size of the feed entry, steam inlet, and vapor outlets nozzles, as well as temperature measurement ports were decided early on. The piping connections then required appropriate reducers to match process piping. Steam piping connection to beer column needed a 2 in x 1 ½ in eccentric reducer connection at T-1. This was true for instrumentation as well. Decisions made on which type of sensors, transmitters, and control valves also impact the P&ID. For example, a ½ in control valve in a 1 in line implies placing 1 in x ½ in (and vice versa) reducing connections on either end of the control valve. A list of selected equipment is provided in Appendix A4.

The final piece of the puzzle is the data acquisition equipment – this aspect of the process has probably a profound impact on the success of the operation. Data acquisition devices need to work across platforms. Fortunately 4-20 mA signals are standard across platforms and were used for input output signals from and to transmitters. It is also important to also keep in close consideration power (DC vs. AC power or 110V vs. 220V) availability on location when selecting equipment. Equipment can be susceptible to weather damage and if placed outdoors, weather protection is a must – all transmitters purchased were field-ready and outdoor friendly. All data acquisition devices were placed in weatherproof junction boxes.

5.3.2 Plant Layout and Drawings

Once the P&ID is established and equipment selection is complete a series of drawings are required to proceed to on-site work (Figure 5.5). A three dimensional rendering of the plant is required. Google SketchUp, free software from Google, was used to produce the 3-D rendition of the pilot plant. AutoCAD or Solid Works are alternative software packages, which can also be used for the 3-D layout. The location of equipment was guided by process safety guidelines – a minimum distance of 50ft from the nearest wall of a building is required for an outdoor process area. Detailed equipment design contains dimensions of the unit as well as orientation and locations of inlet and outlet fittings.

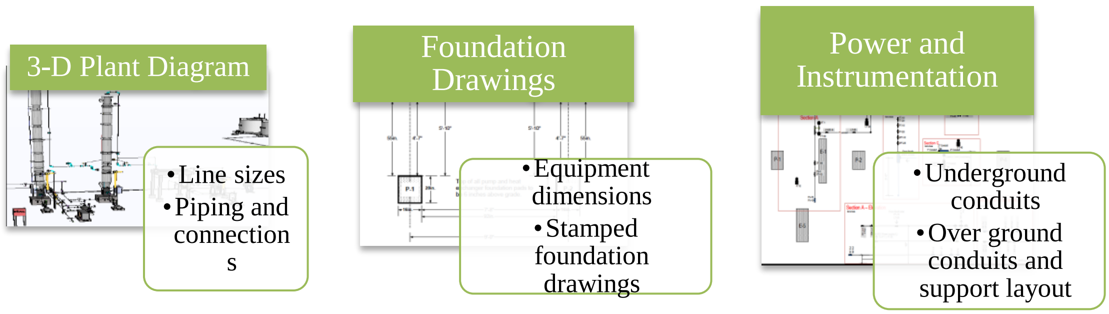


Figure 5.5: Drawings needed at the Pilot Plant

The locations of individual equipment were decided with inlet outlet locations in mind. For example the pumps were located west of the columns directly across the bottom outlet nozzles on both columns. Views of the storage area and process areas are provided in Appendix B1.

Certified foundation drawings for equipment were required before delivery to facilitate equipment installation. Engineering firms specializing in structural and foundation work were recruited for this purpose. ASU drawings were completed by Tulsa based Payne Huber Engineering. Details for all engineering design contractors, as well as companies we purchased equipment for can be found in Appendix B2. A foundation plan drawing with concrete slab specifics was made after certified foundation diagrams were delivered. This drawing was passed on to the engineering, works, or earth moving contractors for start of on-site preparation. Foundation plan drawings for ASU are included in Appendix B3.

Electrical drawings to complete conduit placement and confirm instrument location were also required. Appendix B4 shows the electrical and instrumentation mechanical layout – all instrument (sensor and transmitter) locations were identified and aboveground conduit were located in tandem. T or L connections were placed close to sensors and transmitters to minimize flex conduit length (Figure 5.6 a). Once the foundation blocks were laid, underground channels were dug out and rigid conduits were laid connecting power sources with equipment. Support structures were constructed with strut and all transmitters were bolted to the individual supports (Figure 5.6). A complete list of instruments with power supply and connection specifications is available in Appendix B4.

(a)

(b)

Figure 5.6: Sensor and transmitter installation (a) Micromotion and 1151DP as level transmitter installation (b) Control valve air and sensor connections to rigid conduit

The use of differential pressure transmitters as level transmitters and flow meters (with orifice plates) are described in B4 as well.

For sensor transmitter pairs (CMF025 and RFT9739) wiring included was:

1. Sensor to transmitter wiring (specified by the manufacturer). For the Micromotion equipment a 9-wire cable was specified and used between the sensor and transmitter. The cable is color coded for identifiable connections.
2. Transmitter power supply wiring. Depending on the type (AC or DC) power supply used, the wiring assignments will change. For DC power supply, a battery was installed in the junction box and two wire cables were extended to the field mount transmitter via rigid conduit.
3. A two wire cable was connected from transmitter output for a 4-20mA signal.

For loop-powered transmitters (1151DP) only one two-wire cable connection was needed. Differences in the two loops and wiring diagrams are explained in Appendix B5. All in-field wiring terminates in an in-field weatherproof junction box. The back panel connections were designed based on National Instruments DAQ device power supply and connection requirements. Wiring connections, termination strip location, and connection to the Data Acquisition (DAQ) device is explained in Appendix B6.

5.3.3 Fabrication and On-Site work

Column construction, erection, and all subsequent events follow the path in Figure 5.7. Local fabrication shop, Total Fabricators was enlisted for column fabrication. Both columns were constructed from sections of Schedule 40 12 in 304 stainless steel pipe sections. Stainless steel ¾ in couplings were welded at 6 specified locations along the beer column and 7 locations along the rectifier. Support lugs for lifting columns were installed. Beer column cartridge trays were installed at the fabrication shop before the column was transported to the pilot plant site. Each cartridge was rotated 180° for alignment. Before column erection, each column was pressure tested (180 psig with compressed air) and leak checked (soap-water test on joints) on site. Pictures from internals and column installation are in Appendix B7.

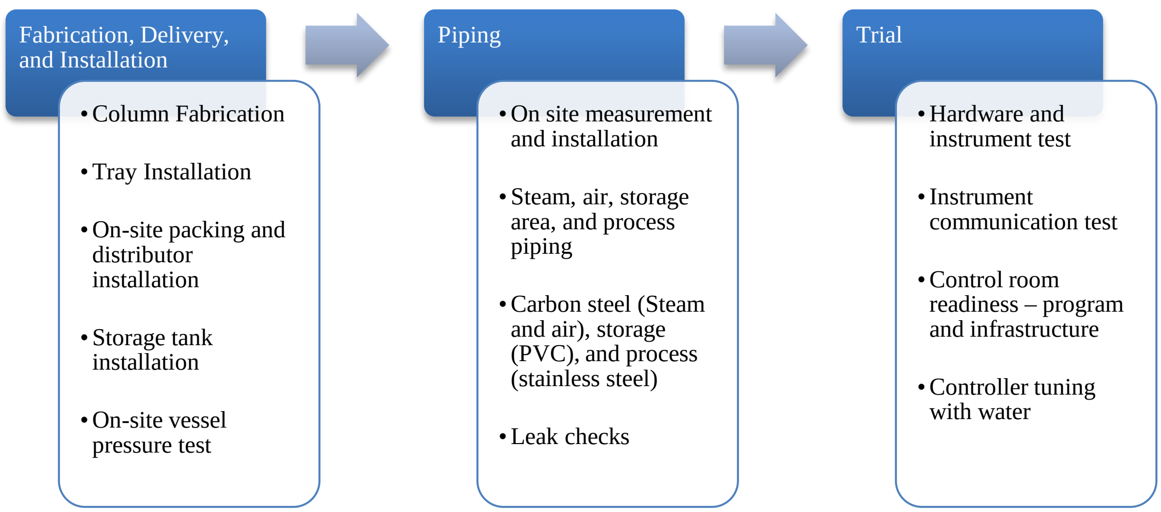


Figure 5.7: Onsite work list to trial

Belger Cartage Services Inc., were hired to erect both columns. A 22-ton all terrain crane was used for the project. Total beer column weight including internals was 3200 lbs and rectifier column weight was 2300 lbs. Tasks covered with a crane and boom lift rental were:

1. Structured packing installation in rectifier
2. Re-torqueing and tightening of bolts on both beer column and rectifier columns
3. Installation of thermowells, thermocouples, rigid and flex conduits, and support assembly from temperature measurement points to bottom of the column

4. Installation of rectifier top portion and top flange
5. Installation of vapor line connecting beer column top and rectifier bottom
6. Installation of vapor line connecting rectifier top to condenser

The 12 bolt assembly on both column flanges were re-torqued and tightened according to sequence in Appendix B8. Structured packing sections were lowered into the column – each section was rotated 90^{o}. The structured packing liquid distributor was leveled first at grade and then once placed on the packing inside the rectifier (Figure 5.8). This is considered a crucial installation step – without level placement of the distributor, liquid in the column is susceptible to flowing in channels, with minimum vapor-liquid contact and mixing. This compromises the separation efficiency of the column. Once the distributor was placed, leveled, and centered the top flange of the rectifier was put in place and bolted.

(a) (b) (c)

(d)

Figure 5.8: Packing and distributor installation. Clockwise from left: (a) Flange installation using lifts (b) Packing support (c) Distributor leveling on column/packing (d) Structured packing installed in column

Boiler Repair Co. executed all piping fabrication and installation work at the Alcohol Separation Unit. The work included steam piping (threaded) and 304 stainless steel piping (threaded and TIG welded). Three dimensional isometric drawings provided by our research team were used as guides for fitting placement and perspective, but final measurement and assembly was done on site. All materials were purchased and provided by our research team. After several experiments with threading glues (including Rectorseal Tru-Blu and Loctite 592 PST thread sealant), nickel based anti-seize compound was found to be effective in sealing threaded (brush top cans) 304 stainless steel connections.

A layer of the threading sealant brushed on to the threads and a Teflon tape was wrapped around the pipe threads before connecting male and female ends of the piping/fittings. All connections and piping in the feed tank area was Schedule 40 PVC and was assembled by the ASU research team. Once installed all piping was checked for leaks. A 150 psig portable compressor was used to pressurize the piping – soap water solution was then sprayed on each connection. Connections which had bubble formations were marked and either re-threaded or welded.

Instrument connections were set up in three parts (Figure 5.9):

1. Wiring connections from transmitter to junction box terminals
2. Wiring connection from terminals to hardware acquisition device
3. Ethernet connection from data acquisition device to computers

Terminal strips, the NI cDAQ9188 chassis, DIN rail mount for the supply were attached to the panel first. Labels describing each termination (transmitter tag and description, LT-10 for example) were pasted appropriately (Figure 10a). Labels are crucial to identify bad wiring in order to isolate and replace. Junction boxes knockouts were located and holes made with a punch and die set – two 1 in knockouts, and one ½ in knockout were drilled in the north junction box. The south junction box had one 3 in knockout and two ½ in knockouts.

16 BWG 2 wire Belden cable was pulled with a 125 ft electrical fish tape from each transmitter connection to the junction box. Each wire was tagged and labeled on both ends. Again, this is essential when identifying and isolating malfunctioning wiring. Connections from the terminal strips to the DAQ device were made with 22-gauge single conductor wire (Figure 10b). Panel connections (North Junction box panel 4-20 mA input and output, thermocouple signal connections, DAQ device mounting, and power supply) have been described in detail in Appendix B8.

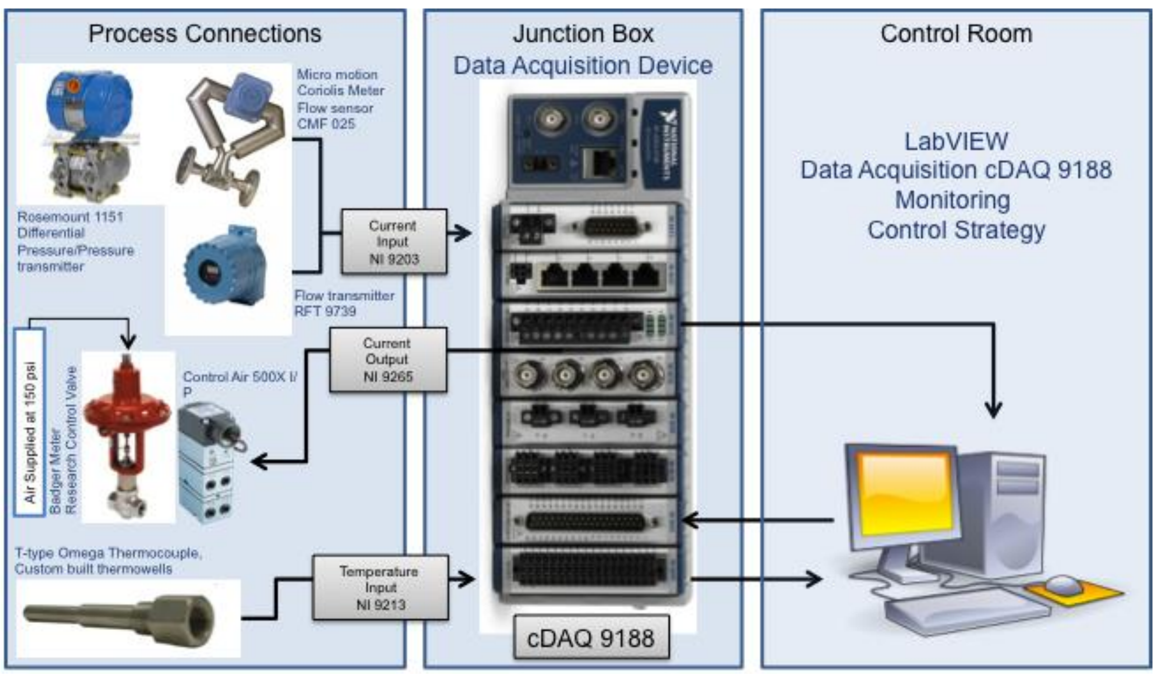
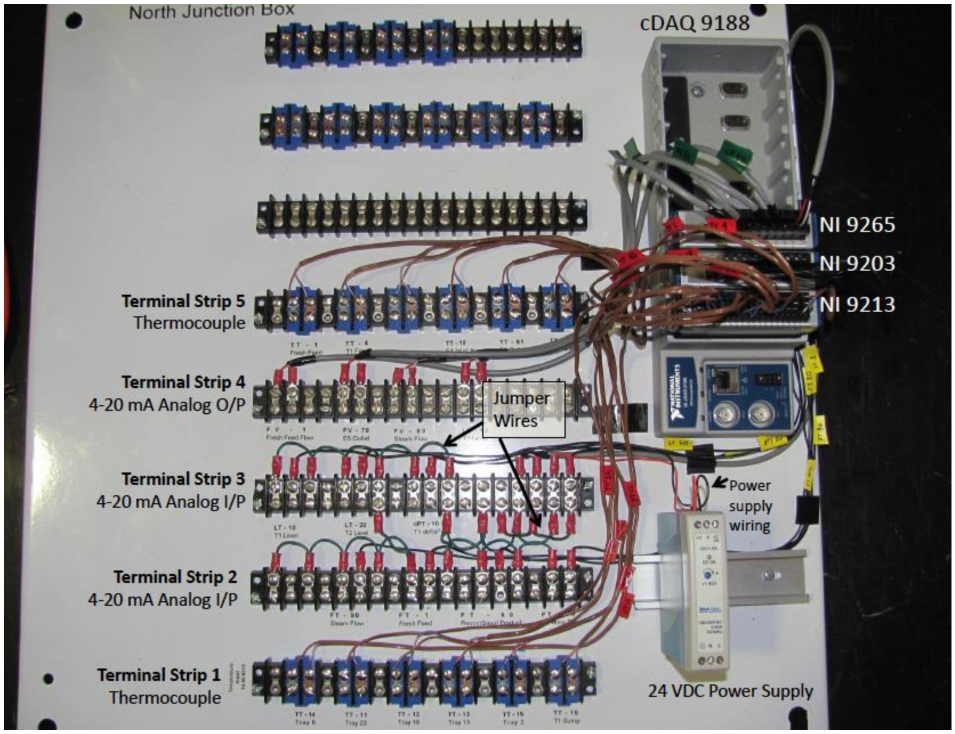
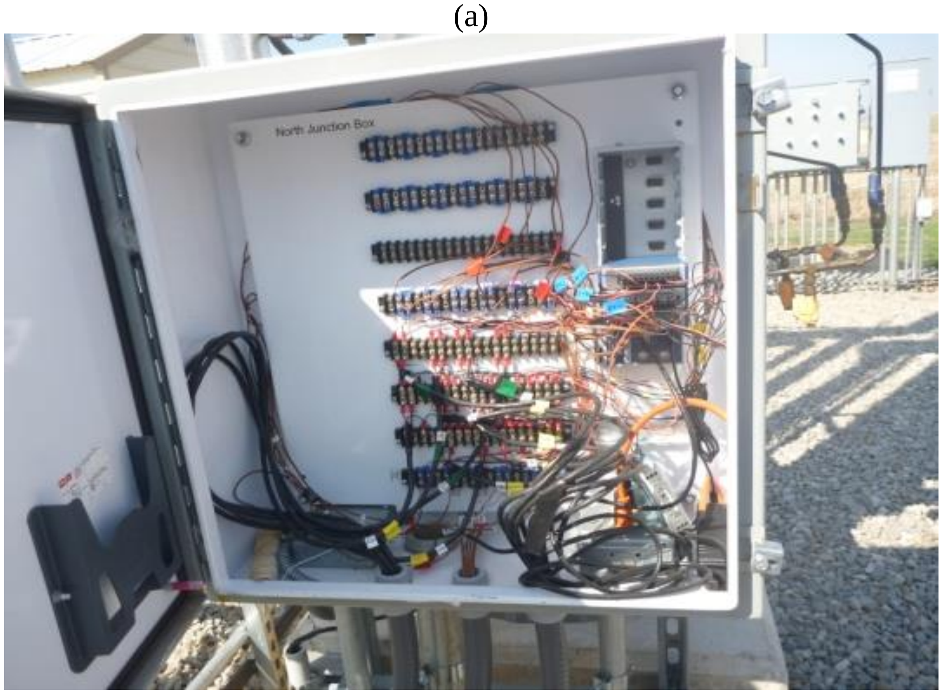


Figure 5.9: Input output signals to and from the control room to the pilot plant

Once wiring was completed each transmitter was tested for a 4 mA zero value signal. This was achieved using NI Measurement and Automation System (MAX) software interface. A graphical or tabulated readout of the signal strength is available through the software. Each channel on all modules can be tested individually. The test and setup procedure is explained in Appendix B9.



(a)



(b)

Figure 5.10: Junction box (a) Labeling and wiring back panel (b) Connection inside box on-site

The control room process interface was programed using Labview 2011. The interface has the following features (Appendix B10):

1. Real time updates of critical process information including levels, pressures, temperatures, and flows
2. An alarm system for high or low level occurrences
3. Easy to follow graphical user interface, built on the LabVIEW platform, as seen in Appendix B10.
4. Process control strategy, control valve tuning, level, flow, temperature, and cascade control capabilities have also been built into the control system using PID algorithm provided in NI's PID and Fuzzy logic toolkit.

This makes the operation of the Alcohol Separation Unit completely automated. Before the Alcohol Separation Unit was commissioned all transmitters were field tested and calibrated.

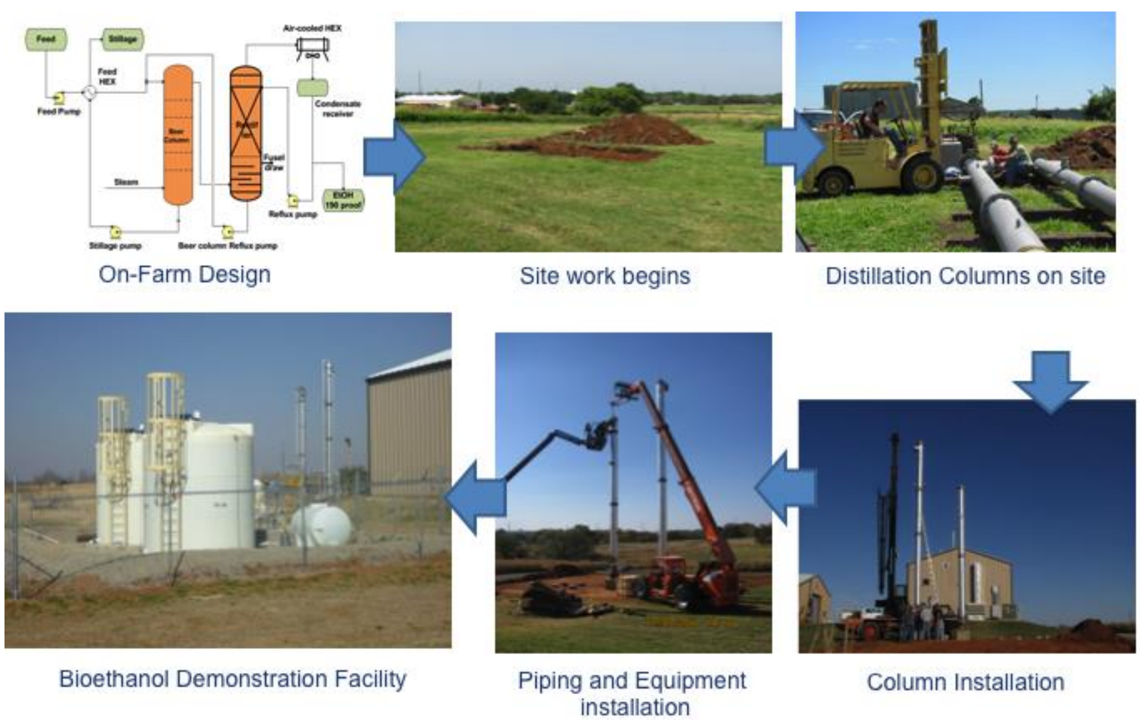


Figure 5.11: From design to demonstration: Highlights

All 4-20 mA transducers were calibrated for an air pressure signal range of 3-15 psig range corresponding to 0-100% opening on the control valve. All level transmitters were calibrated for a 0-100% level (100% corresponding to a full liquid height). Flow transmitters were calibrated for individual flow ranges. For example the feed and stillage coriolis meter have a calibrated flow range of 0-4800 lb/h, whereas the steam meter has a flow range of 0-500 lb/h. All calibration information can be found in Appendix B11. Overall the timeline from design to construction and commissioning ready unit spanned three years (Figure 5.11).

5.3.4. Commissioning Activities

All control loops were individually tested with water as test fluid in each line. The controller tuning parameters were adjusted to reflect the best estimates needed for quick or timely response, and remaining at set point. Tuning parameters are listed in Appendix B12. For commissioning, steam was let into the columns – flow rate, pressure, and temperatures were checked along the beer column, the rectifier, as well as the condenser. Level build up with steam condensation in both columns were tracked with LT-10 and LT-20. Flows were checked on T-1 bottoms flow FT-10, rectifier bottoms flow FT-20, and feed flow meter FT-1 by diverting water from the columns. A water level in E-4 was also tracked by LT-30 control loop. Steam condensation in D-4 also built a water level the reflux drum. The level controller in D-4 was checked with the slowly building liquid level. Once adequate level was found in the reflux drum, some liquid was diverted to check distillate flow in FT-50 and reflux flow in FT-40. Commissioning went as per plan and all instrumentation and communication channels responded as per design. Start up and shut down procedures were established (Appendix B13).

5.4. Safety:

During design, construction, start up, shut down, and operation of the pilot plant safety was always the first priority and cannot be overstressed. A detailed Hazard and Operability (HAZOP) study via a Preliminary Hazard Analysis (PHA) checklist was conducted during the detailed design of the pilot plant. Major outcomes of the event were:

1. Firefighting measures needed at the pilot plant – after careful consideration Ethanol Emergency Response Coalition recommendation of letting the fire run its course was adopted.
2. Spill and containment measures needed at the pilot plant – A 1 ft high berm surrounds the storage area. The volume of the storage area is large enough to contain spill contents of the largest tank.
3. Over pressurization prevention strategies – rupture disk assemblies are installed in two locations. One on the overhead vapor line on T-1 and another on the condensate receiver D-4.

In addition, safe operating guidelines exist which are enforced during operation of the pilot plant (Table 5.1).

5.5 Expenses

Major expenses that can be expected for pilot plant are for equipment (47% of expenditure), piping and hardware materials (12%), and contractual services (22%). Table 5.2 lists all expenditures incurred during the project life from 2010 to 2013 for the pilot plant. These figures do not include any labor costs. The graduate student and Principal Investigator salary were not considered.

Table 5.1: Safety precautions employed at the Alcohol Separation Unit

Hazard	Precautions
Boiler operation	Blowdown boiler before start up Check maximum boiler pressure. Should be compatible with manufacturers recommendation Addition of water treatment chemicals and follow up on maintenance as per boiler manual instructions
Burns and scalding from hot surfaces	Insulation on all pipes with temperatures higher than 150^{o}F Protective gear to be worn at all times on site – gloves, full length shirt, goggles, and hard hat
Handling sweet sorghum liquid	Protective gear to be worn hen hadling liquids on site – gloves, full length shirt, goggles, and hard hat
Handling ethanol product	Protective gear to be worn when sampling on site – gloves, full length shirt, goggles, and hard hat
Fires	No ignition sources while plant is in operation

Table 5.2: All expenditure on materials and services for ASU from 2010 to 2013

Year	2010	2011	2012	2013
Columns		8,322.90		
Internals		16,677.01		
Storage tanks		25,000.00		
Reflux drum		3,250.00		
Column fabrication		71,578.00		
Condenser E4		34,900.00		
Air Cooler E5			2,983.75	
Shell and Tube E1		3,162.75		
Pumps			2,838.92	
	-	162,890.66	5,822.67	-
Equipment				**168,713.33**
Rupture disk assembly			1,147.55	
304 SS Pipe and fittings	870.06	429.77	14,109.57	2,064.68
PVC Pipe			26.86	46.88
PVC Fittings			100.56	68.56
Tools and supplies		174.24	3,837.30	1,111.63
Bolts, screws, nuts, fasteners		574.87	1,344.68	478.78
Foundation, earth moving, grvael supplies		2,764.78	1,457.33	121.39
Valves and fittings		837.86	2,221.23	83.03
Antisieze		45.55		
Hardware			809.08	25.38
Scaffold			475.00	
Insulation		1,154.84	1,499.35	
	870.06	5,981.91	27,028.51	4,000.33
Piping, Hardware, and Materials				**37,880.81**
Micromotion transmitters			105.00	5,941.21
Rosemount, Wellmark level transmitters			1,520.45	325.74
NI Hardware			4,076.59	206.45
LabView			739.70	
4-20 mA Wiring		195.95		
Control Valve trims, strainers, I/P			5,625.26	841.00
Conduit			668.03	602.48
Electrical, power supply			299.21	9.97
Thermocouples		163.00		
Thermocouple wires			1,048.06	
Orifice meter-Steam	454.77		917.41	
Indicators - P, T	338.06		599.57	
Instrument fittings		285.71		
Tubing		32.40	523.35	
Panels and Electrical		281.78	1,426.75	694.45
Computer, cables, switches			496.94	547.71
	792.83	958.84	18,046.32	9,169.01
Instrumentation				**29,000.00**

Year	2010	2011	2012	2013
Boiler chemicals				813.08
Filter assembly				74.72
Pump, scrubber				291.29
	-	-	-	1,179.09
Maintenance Materials				**1,179.09**
Wallace Engineering	484.90			
Payne Hueber		10,000.00		
Biosystems and Ag		1,310.25		
Kinnunen		754.44		
Belger Cartage Services		1,140.00		
PM Insulation		2,000.00	4,706.69	
Carrier ditching and excavation		2,700.00		
Physical plant			17,244.40	
Boiler Supply and Co. - Piping			26,960.00	
	484.90	17,904.69	48,911.09	-
Contractual				**67,300.68**
Kinnunen		505.27	555.23	
Biosystems and Ag		1,501.00	3,463.45	
Allied Steel Crane			1,917.60	
RSC Equipment Boom Lift			3,623.92	
Simon's Towing			540.00	
Pioneer rental			3,556.82	
Shipping equipment		325.34		
	-	2,331.61	13,657.02	-
Rental				**15,988.63**
Analytical			1,065.08	327.36
Sigma/Fusel study - Standards	325.71			
Stillwater steel - Gases ?		26.00		
McMaster		180.26		
BioRad- deashing/refill		310.50		
Lab supplies - syringes etc		11.20		
Equipment use rent		798.00	88.50	
	325.71	1,325.96	1,153.58	327.36
Lab Supplies/Analysis				**3,132.61**
Safety Cameras			1,411.89	
Welding			493.19	
Stationary etc			122.78	104.93
	-	-	2,027.86	104.93
Miscellaneous				**2,132.79**

CHAPTER 6

SUMMARY AND CONCLUSIONS

This doctoral investigation sought to establish technical feasibility of bioethanol separation from fermented sweet sorghum. The overall objective was to demonstrate bioethanol production from sweet sorghum fermented juice on a farm scale production unit. Technical validity, troubleshooting experience, and economics of the process were understood in due course. Operation and maintenance concerns, specifically fouling behavior of fermented sweet sorghum in bioethanol production conditions, which were unknown previously were also studied. Building and operating the Alcohol Separation Unit at Oklahoma State University validated technical feasibility of the on-farm ethanol design. Fermented sweet sorghum characterization studies established solid content in the feed as well as transport properties of the liquid. Dynamic fouling experiments were conducted to quantify resistance to heat transfer for fermented sweet sorghum. Overall, production of 190 proof ethanol product from fermented sweet sorghum on a farm scale facility is possible without major equipment fouling concerns.

In Chapter 2, data from ASU operation are presented in addition to analytical results and economic analysis. Ethanol product purities of 95 vol% (at 60^{o}F) can be obtained from 3.5 vol% fermented sweet sorghum feed. HPLC techniques were also successfully used for ethanol product purity determination in addition to hydrometer readings. Steam consumption accounts for majority of the operational costs (75%). The facility itself contained two distillation columns. Steam was used as an energy source.

Updated instrumentation, modern data acquisition hardware, and easily implementable testing software LabVIEW ensured accurate measurements and quick deployment. Major operational issues encountered were removal of solid and carbon dioxide content from the fermented liquid. A filtration set up to remove large particulate matter was installed in the storage area, before operation. A sparger assembly was incorporated into the pilot plant to continuously remove the gas and prevent accumulation in the condensate receiver.

Fermented sweet sorghum liquid was characterized for solid content using NREL LAP, as described in Chapter 3. The total solid content was found to be less than 3 wt%. The solid loading is significantly lower than corn based process streams such as DDGS (solid content 16-20%). Deposition experiments were conducted with fermented sweet sorghum liquid in a heated test section. Results were discussed in Chapter 4. The resistance to heat transfer for fermented sweet sorghum was found to be equivalent to tap water. The fouling resistance was one tenth of reported corn or maize fouling resistance values. This was established using a heated stainless tube experiment, with deposition inside the tube and validated by a second experiment conducted on an annular fouling probe set up, deposition in annulus. Both experiments confirmed deposit formation on stainless steel tube surface was negligible. The absence of long chain starch compounds in fermented sweet sorghum juice was seen as the primary reason for low fouling resistance values. This provides a significant advantage for sugar based sweet sorghum bioethanol processes.

Finally, experience gathered at the pilot plant was discussed in Chapter 5. Setting up the farm scale plant involved paying attention to specifics and several layers of details were required for successful implementation – from design to demonstration. Technical information essential for decision making were discussed. These include but were not limited to the framework required to initiate construction, drawing requirements, instrumentation connections, contractual work on site, rental equipment, programming, safety on site, and detailed expenditures. This section was included to ensure technology transfer of successful engineering practices to future sweet sorghum bioethanol applications.

During the course of this doctoral work construction of ASU was completed, the farm scale unit was commissioned, operating procedures were developed for safe operation of the unit, procedures for feed and product sample analysis were established, and fouling from fermented sweet sorghum liquid was quantified. In addition sweet sorghum bioethanol separation economic and operational costs were established. With further research farm scale scenario of sweet sorghum bioethanol production will be established. Providing operating procedures for operation in 'farm mode' is a short-term goal in place for ASU activities. In addition, fouling studies at the pilot scale will corroborate laboratory findings indicating low fouling from fermented sweet sorghum juice. Ultimately, continued experiments at the facility will establish economic viability and assist in commercializing the sweet sorghum bioethanol process.

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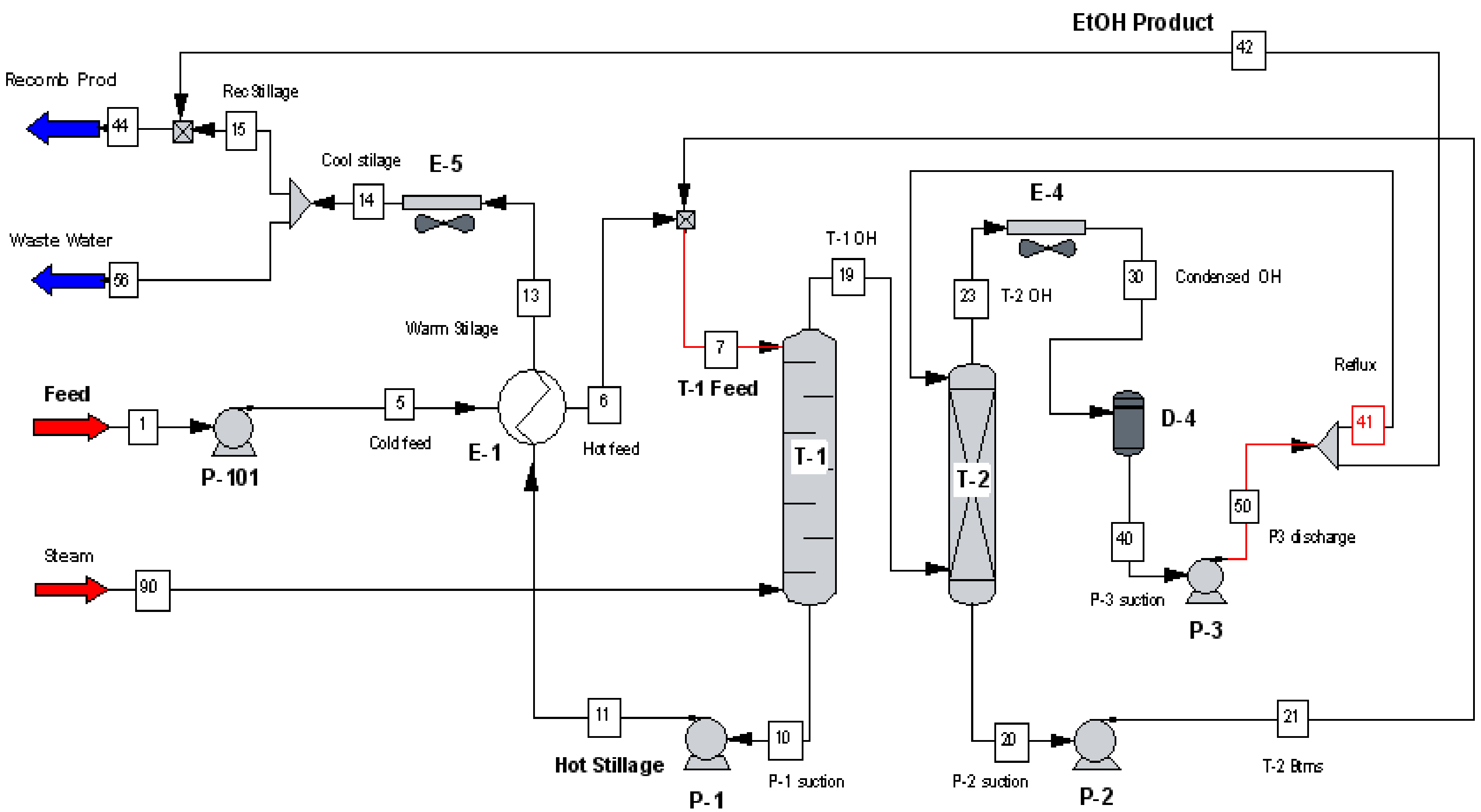
APPENDICES

Appendix A1

CHEMCAD Simulation Diagram



Appendix A2

Process Simulation Flow Rates

CHEMCAD 6.4.1
Simulation: EtOH full simulation - cons Date: 01/03/2012 Time: 13:12:12
FLOW SUMMARIES:

Stream No.	1	5	6	7
Stream Name	Feed	Cold feed	Hot feed	T-1 Feed
Temp F	80.0000*	80.1091	158.4917	162.2633
Pres psia	14.7000*	50.0000	50.0000	45.0000
Enth MMBtu/h	-13.524	-13.524	-13.367	-14.394
Vapor mole frac.	0.00000	0.00000	0.00000	0.00000
Total lbmol/h	109.9449	109.9449	109.9449	118.4889
Total lb/h	2062.2114	2062.2114	2062.2114	2224.6201
Total std L ft3/hr	33.4202	33.4202	33.4202	36.0767
Total std V scfh	41721.74	41721.74	41721.74	44964.00
Flow rates in lbmol/h				
Ethanol	2.3441	2.3441	2.3441	2.6370
Water	107.3574	107.3574	107.3574	115.6020
Lactic Acid	0.0685	0.0685	0.0685	0.0685
Glycerol	0.1003	0.1003	0.1003	0.1003
Acetic Acid	0.0696	0.0696	0.0696	0.0760
Succinic Acid	0.0050	0.0050	0.0050	0.0050

Stream No.	10	11	13	14
Stream Name	P-1 suction	Hot Stillage	Warm Stilage	Cool stilage
Temp F	227.1614	227.2394	158.1913	130.0000
Pres psia	19.7000	45.0000	45.0000	45.0000
Enth MMBtu/h	-15.124	-15.124	-15.282	-15.346
Vapor mole frac.	0.00000	0.00000	0.00000	0.00000
Total lbmol/h	125.6347	125.6347	125.6347	125.6347
Total lb/h	2279.6533	2279.6533	2279.6533	2279.6533
Total std L ft3/hr	36.4645	36.4645	36.4645	36.4645
Total std V scfh	47675.66	47675.66	47675.66	47675.66
Flow rates in lbmol/h				
Ethanol	0.0197	0.0197	0.0197	0.0197
Water	125.3716	125.3716	125.3716	125.3716
Lactic Acid	0.0685	0.0685	0.0685	0.0685
Glycerol	0.1003	0.1003	0.1003	0.1003
Acetic Acid	0.0696	0.0696	0.0696	0.0696
Succinic Acid	0.0050	0.0050	0.0050	0.0050

CHEMCAD 6.4.1

Simulation: EtOH full simulation - cons Date: 01/03/2012 Time: 13:12:12

FLOW SUMMARIES:

Stream No.	15	19	20	21
Stream Name	RecStillage	T-1 OH	P-2 suction	T-2 Btms
Temp F	130.0000	211.3092	210.0539	210.1393
Pres psia	45.0000	18.3000	18.3000	45.0000
Enth MMBtu/h	-13.082	-1.1578	-1.0279	-1.0278
Vapor mole frac.	0.00000	1.0000	0.00000	0.00000
Total lbmol/h	107.1010	11.3445	8.5440	8.5440
Total lb/h	1943.3590	278.0693	162.4086	162.4086
Total std L ft3/hr	31.0853	4.9480	2.6565	2.6565
Total std V scfh	40642.55	4304.99	3242.26	3242.26
Flow rates in lbmol/h				
Ethanol	0.0168	2.6174	0.2929	0.2929
Water	106.8767	8.7207	8.2446	8.2446
Lactic Acid	0.0584	0.0000	0.0000	0.0000
Glycerol	0.0855	0.0000	0.0000	0.0000
Acetic Acid	0.0593	0.0064	0.0064	0.0064
Succinic Acid	0.0043	0.0000	0.0000	0.0000

Stream No.	23	30	40	41
Stream Name	T-2 OH	Condensed OH	P-3 suction	Reflux
Temp F	182.4012	140.3000	140.3000	140.4523
Pres psia	18.0000	17.9000	17.9000	45.0000
Enth MMBtu/h	-1.1249	-1.3260	-1.3260	-0.99493
Vapor mole frac.	1.0000	0.00000	0.00000	0.00000
Total lbmol/h	11.2187	11.2187	11.2187	8.4182
Total lb/h	463.3379	463.3379	463.3379	347.6772
Total std L ft3/hr	9.1798	9.1798	9.1798	6.8883
Total std V scfh	4257.27	4257.27	4257.27	3194.54
Flow rates in lbmol/h				
Ethanol	9.3118	9.3118	9.3118	6.9873
Water	1.9070	1.9070	1.9070	1.4309
Lactic Acid	0.0000	0.0000	0.0000	0.0000
Glycerol	0.0000	0.0000	0.0000	0.0000
Acetic Acid	0.0000	0.0000	0.0000	0.0000
Succinic Acid	0.0000	0.0000	0.0000	0.0000

CHEMCAD 6.4.1

Simulation: EtOH full simulation - cons Date: 01/03/2012 Time: 13:12:12
FLOW SUMMARIES:

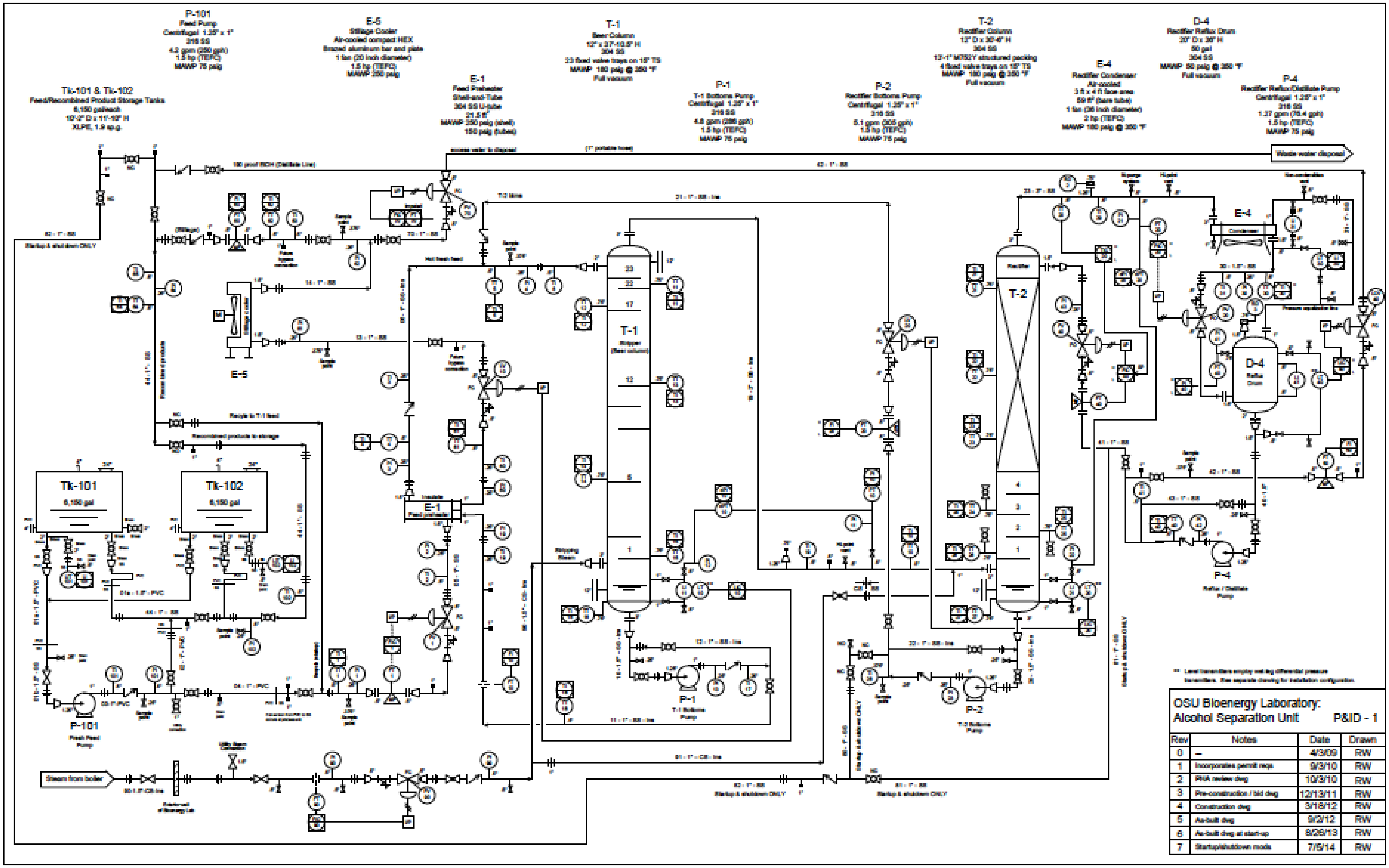
Stream No.	42	44	50	56
Stream Name	EtOH Product	Recomb Prod	P3 discharge	Waste Water
Temp F	140.4523	130.4108	140.4523	130.0000
Pres psia	45.0000	45.0000	45.0000	45.0000
Enth MMBtu/h	-0.33095	-13.413	-1.3259	-2.2638
Vapor mole frac.	0.00000	0.00000	0.00000	0.00000
Total lbmol/h	2.8002	109.9013	11.2187	18.5336
Total lb/h	115.6515	2059.0103	463.3379	336.2945
Total std L ft3/hr	2.2913	33.3766	9.1798	5.3792
Total std V scfh	1062.63	41705.19	4257.27	7033.11
Flow rates in lbmol/h				
Ethanol	2.3243	2.3411	9.3118	0.0029
Water	0.4760	107.3527	1.9070	18.4948
Lactic Acid	0.0000	0.0584	0.0000	0.0101
Glycerol	0.0000	0.0855	0.0000	0.0148
Acetic Acid	0.0000	0.0593	0.0000	0.0103
Succinic Acid	0.0000	0.0043	0.0000	0.0007

Stream No.	90
Stream Name	Steam
Temp F	358.5585
Pres psia	150.0000*
Enth MMBtu/h	-1.8877
Vapor mole frac.	1.0000*
Total lbmol/h	18.4901
Total lb/h	333.1000
Total std L ft3/hr	5.3358
Total std V scfh	7016.62
Flow rates in lbmol/h	
Ethanol	0.0000
Water	18.4901
Lactic Acid	0.0000
Glycerol	0.0000
Acetic Acid	0.0000
Succinic Acid	0.0000

Appendix A3

Alcohol Separation Unit P&ID

Next page


P-101
Feed Pump
E-5
Stillage Cooler
T-1
Beer Column
T-2
Rectifier Column
D-4
Rectifier Reflux Drum
Tk-101 & Tk-102
Feed/Recombined Product Storage Tanks
E-1
Feed Preheater
Shell-and-Tube
P-1
T-1 Bottoms Pump
P-2
Rectifier Bottoms Pump
E-4
Rectifier Condenser
Air-cooled
P-4
Rectifier Reflux/Distillate Pump
Waste water disposal
Tk-101
Tk-102
E-5
T-1
T-2
D-4
E-4
E-1
P-101
P-1
P-2
P-4
Steam from boiler
OSU Bioenergy Laboratory:
Alcohol Separation Unit
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7/5/14
RW

Appendix A3

Alcohol Separation Unit Hydraulic Summary

Table A3: Hydraulic Summary of Alcohol Separation Unit

		Nominal	Pipe	Flow Rate			
Line #	Service	Pipe Size inches	MOC	Volumetric gpm	gph	acfh	Mass lbm/hr
1	P-101 suction	1.5	PVC	4.19	251.1	33.57	2,062.2
2a	**P-101 recycle** (line 3 to line 44)	1	PVC	**10.81**	648.6	86.71	5,326.0
2b	P-101 recycle (unit return line to tank)	1	304 SS	10.81	648.6	86.71	5,326.0
3a	**P-101 discharge w/ recycle**	1	PVC	**15.00**	899.7	120.28	7,388.2
3b	P-101 discharge w/o recycle	1	PVC	4.19	251.1	33.57	2,062.2
4	Cold feed from storage to processing unit	1	PVC	4.19	251.1	33.57	2,062.2
5	Cold feed to unit & E-1	1	304 SS	4.19	251.1	33.57	2,062.2
6a	Hot feed to T-1 (from E-1 to T-2 btms tie-in)	1	304 SS	4.28	256.6	34.31	2,062.2
6b	Hot feed + T-2 btms to T-1	1	304 SS	4.62	277.4	37.09	2,224.6
10a	T-1 btms to P-1	1.5	304 SS	4.78	286.7	38.33	2,279.6
10b	P-1 suction (T-1 btms + recycle)	1.5	304 SS	15.00	900.1	120.32	7,156.0
11a	**P-1 discharge w/ recycle**	1	304 SS	**15.00**	900.1	120.32	7,156.0
11b	Hot stillage to E-1	1	304 SS	4.78	286.7	38.33	2,279.6
12	**P-1 recycle**	1	304 SS	**10.22**	613.3	81.99	4,876.4
13	Warm stillage from E-1 to E-5	1	304 SS	4.65	279.1	37.31	2,279.6
14a	Cool stillage + waste water	1	304 SS	4.61	276.8	37.00	2,279.6
14b	Cool stillage to be recombined	1	304 SS	3.93	235.9	31.54	1,943.3
-56	Waste water to septic system	1	Hose	0.68	40.8	5.46	336.3

Table A3: Hydraulic Summary of Alcohol Separation Unit Contd.

		Nominal	**Pipe**	**Flow Rate**			
Line #	Service	Pipe Size	MOC	Volumetric			Mass
		inches		gpm	gph	acfh	lbm/hr
19	T-1 OH	3	304 SS			4,406	278.1
20a	T-2 btms to P-2	1.5	304 SS	0.35	20.9	2.79	162.4
20b	P-2 suction (T-2 btms + recycle)	1.5	304 SS	15.00	899.9	120.30	6,998.1
21a	**P-2 discharge w/ recycle**	1	304 SS	**15.00**	899.9	120.30	6,998.1
21b	T-2 btms to T-1	1	304 SS	0.35	20.9	2.79	162.4
22	**P-2 recycle**	1	304 SS	**14.65**	879.0	117.51	6,835.7
23	T-2 OH	3	304 SS			4,196	463.3
30	Condenser liquid to D-4	1.5	304 SS	1.21	72.4	9.68	463.3
31	D-4/E-4 pressure equalizationl ine	1	304 SS		0.0	0.00	0.0
40a	Reflux + distillate liq from D-4 to P-4	1.5	304 SS	1.21	72.4	9.68	463.3
40b	P-4 suction (reflux + distillate + recycle)	1.5	304 SS	15.00	900.0	120.31	5,755.5
41	T-2 reflux	1	304 SS	0.91	54.4	7.27	347.7
42a	**P-4 discharge w/ recycle**	1	304 SS	**15.00**	900.0	120.31	5,755.5
42b	T-2 reflux + distillate (no recycle)	1	304 SS	1.21	72.5	9.69	463.3
42c	Distillate to be recombined	1	304 SS	0.30	18.1	2.42	115.7
43	**P-4 recycle**	1	304 SS	**13.79**	827.5	110.63	5,292.1
44	**Recombined product to storage**	1	304 SS	**4.23**	253.9	33.94	2,059.0
45	Recycle jumper from recomb prod to feed	1.5	304 SS				
Utilities							
90	Steam	1.5	CS				333.1
95	Utility air	1	CS				

Table A3 Continued

				Fluid Properties			Fluid Composition			Line
Line #	Service	Density	sp.g.	Viscosity	Viscosity	T	EtOH	Water	Velocity	Length
		lbm/ft^3	wtr=1	CC6, cp	x1.5 cP	°F	std liq volume basis		ft/s	ft
1	P-101 suction	61.43	0.9849	0.9008	1.3512	80.0			0.66	12
2a	**P-101 recycle** (line 3 to line 44)	61.43	0.9849	0.9000	1.3500	80.1			4.01	4
2b	P-101 recycle (unit return line to tank)	61.43	0.9849	0.9000	1.3500	80.1			4.01	11
3a	**P-101 discharge w/ recycle**	61.43	0.9849	0.9000	1.3500	80.1	6.50 vol%	92.7 vol%	5.57	2
3b	P-101 discharge w/o recycle	61.43	0.9849	0.9000	1.3500	80.1			1.55	6
4	Cold feed from storage to processing unit	61.43	0.9849	0.9000	1.3500	80.1			1.55	180
5	Cold feed to unit & E-1	61.43	0.9849	0.9000	1.3500	80.1			1.55	43
6a	Hot feed to T-1 (from E-1 to T-2 btms tie-in)	60.11	0.9638	0.4137	0.6206	158.5	6.50 vol%	92.7 vol%	1.59	7
6b	Hot feed + T-2 btms to T-1	59.98	0.9617	0.4013	0.6020	162.3	6.77 vol%	92.5 vol%	1.72	45
10a	T-1 btms to P-1	59.48	0.9536	0.2576	0.3864	227.2			0.75	6
10b	P-1 suction (T-1 btms + recycle)	59.47	0.9536	0.2576	0.3864	227.2			2.36	2
11a	**P-1 discharge w/ recycle**	59.47	0.9536	0.2576	0.3864	227.2	500 vppm	99+ vol%	5.57	2
11b	Hot stillage to E-1	59.47	0.9536	0.2576	0.3864	227.2			1.77	12
12	**P-1 recycle**	59.47	0.9536	0.2576	0.3864	227.2			3.79	
13	Warm stillage from E-1 to E-5	61.09	0.9795	0.4126	0.6189	158.2			1.73	8
14a	Cool stillage + waste water	61.62	0.9879	0.5270	0.7905	130.0	500 vppm	99+ vol%	1.71	2
14b	Cool stillage to be recombined	61.62	0.9879	0.5270	0.7905	130.0			1.46	41
-56	Waste water to septic system	61.62	0.9879	0.5270	0.7905	130.0				

Table A3 Continued

		Fluid Properties					Fluid Composition			Line	line loss
Line #	Service	Density lbm/ft^3	sp.g. wtr=1	Viscosity CC6, cp	Viscosity x1.5 cP	T °F	EtOH std liq volume basis	Water	Velocity ft/s	Length ft	ex CV psi
19	T-1 OH	0.0631		0.0120		211.3	49.0 vol%	50.9 vol%	23.84		
20a	T-2 btms to P-2	58.18	0.9328	0.2854		210.1			0.05	6	0.0
20b	P-2 suction (T-2 btms + recycle)	58.17	0.9327	0.2854		210.1			2.36	2	0.1
21a	**P-2 discharge w/ recycle**	58.17	0.9327	0.2854		210.1	10.2 vol%	89.6 vol%	5.57	4	1.1
21b	T-2 btms to T-1	58.17	0.9327	0.2854		210.1			0.35	7	0.0
22	**P-2 recycle**	58.17	0.9327	0.2854					5.44		
23	T-2 OH	0.1104		0.0108		182.4	94.0 vol%	6.0 vol%	22.71		
30	Condenser liquid to D-4	47.84	0.7671	0.5627		140.3	94.0 vol%	6.0 vol%	0.19	8	0.0
31	D-4/E-4 pressure equalizationl ine		0.0000						--		
40a	Reflux + distillate liq from D-4 to P-4	47.84	0.7671	0.5627		140.3			0.19	8	0.0
40b	P-4 suction (reflux + distillate + recycle)	47.84	0.7670	0.5624		140.5			2.36	2	0.1
41	T-2 reflux	47.84	0.7670	0.5624		140.5			0.34	48	0.0
42a	**P-4 discharge w/ recycle**	47.84	0.7670	0.5624		140.5	94.0 vol%	6.0 vol%	5.57	2	0.5
42b	T-2 reflux + distillate (no recycle)	47.84	0.7670	0.5624		140.5			0.45	2	0.0
42c	Distillate to be recombined	47.84	0.7670	0.5624		140.5			0.11	18	0.0
43	**P-4 recycle**	47.84	0.7670	0.5624		140.5			5.12		
44	**Recombined product to storage**	60.66	0.9726	0.5271	0.7907	130.4	6.50 vol%	92.8 vol%	1.57	192	1.3
45	Recycle jumper from recomb prod to feed		0.0000								
Utilities											
90	Steam	0.3244		0.01563							

Appendix A4

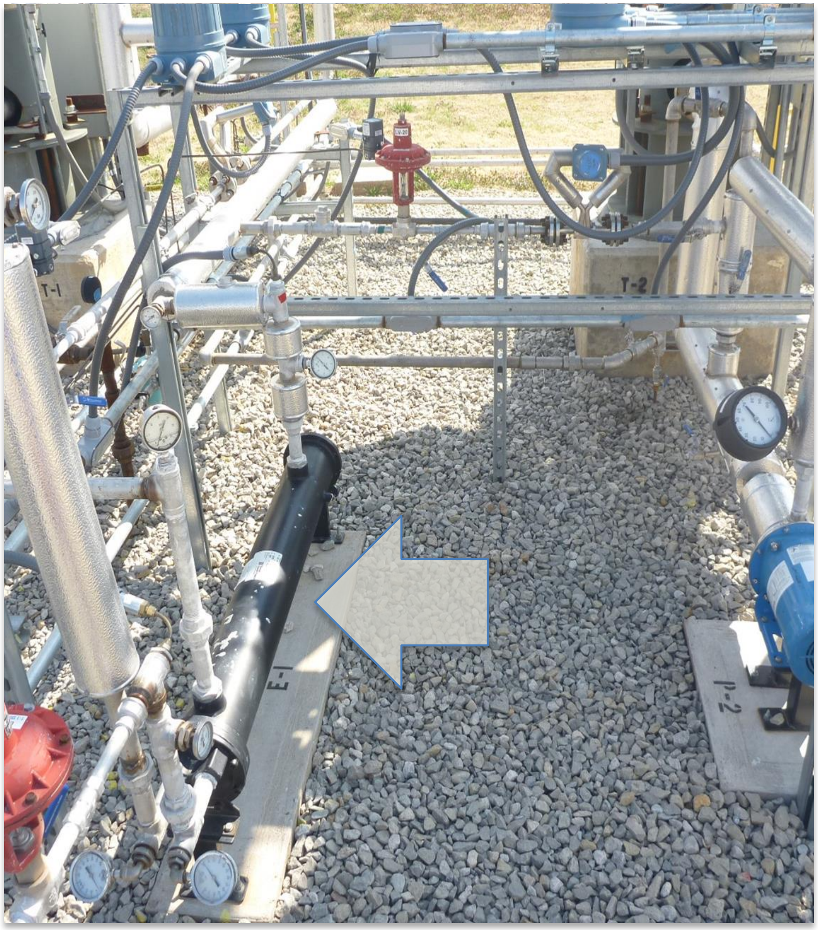
Equipment List

Equipment	Tag	Description
Storage Tank	Tk-101 and Tk-102	5000 gallons Integral Flange Mounted Opening (IMFO) for easy cleaning and complete emptying of liquid content 24 inch manway One 2 inch U-vent Three 2 inch nozzles
Beer Column	T-1	12 inch SA-312 TP304 pipe. Total height 39 ft Twenty three standard Sulzer cartridge SVG valve trays 15 in tray spacing Temperature measurement ports at Tray 1, 5, 7, 12, and 22 (3/4 in couplings)
Rectifier	T-2	12 inch SA-312 TP304 pipe Total Height 29 ft Combination of four valve trays and 14 ft of M752Y packing (14 inch HETP) Trays 1, 2, and 3 have a liquid withdrawal port + thermowells (3/4 in couplings)
Feed Preheater	E-1	USSC 1036 4-pass shell and tube heater. 20 tubes per pass, 4 pass 0.375 in OD x 0.02 in wall
Air cooled Condenser	E-4	Custom designed induced draft air-cooler from GEA Rainey Three row finned tube bundle with a 1-inch slope 10 fins per inch on tubes Tube bundle dimensions: 3 ft x 4 ft in dimension ¾ HP fan motor
Stillage Cooler	E-5	BOL-725 Thermal Transfer Air cooler

Equipment	Tag	Description
T-1 Bottoms pump	P-1	1.5 HP, 1MF1S5B4 centrifugal pump
T-2 Bottoms pump	P-2	1.5 HP, 1MF1S5B4 centrifugal pump
Distillate/Reflux pump	P-3	1.5 HP, 1MF1S5B4 centrifugal pump
Feed pump	P-101	1.5 HP, 1MF1S5B4 centrifugal pump
Condensate Reciever	D-4	Cylinder - SA-312 TP 304 Weld Pipe Head – SA 240 304 Design pressure180 psi 3 equally spaced legs at 120o 1.5 in WN flange connections for inlet and outlet
Boiler	Boiler	HE-15 Lattner 15 HP vertical boiler ASME vessel 150 psi Atmospheric natural gas boiler 680,000 BTUH LV16 Lattner boiler feed water system

Appendix A4 Continued

Stillage cooler



Feed preheater

Storage tanks

Distillation columns

Air cooled condenser

Reflux drum

Appendix A5

Request for Quotation: Feed Preheater Inquiry

Note: The requested quote is for purchase rather than budget purposes.

Provide separate quotes on FOB manufacturer's location and delivered basis (OSU Bioenergy Laboratory, 208 N. Mar Vista St., Oklahoma State University, Stillwater, OK 74078)

Feed Pre-heater, E-101

Quantity	1
Service	a) Tube side liquid: Feed (6 – 10 vol% ethanol and water), enters at 60^{o}F and heated to 150^{o}F b) Shell side liquid: Discharge from beer column bottoms (0.05 vol% ethanol and water), enters at 221.86^{o}F and cooled to at least 150^{o}F
Duty	–180,800 Btu/hr
Process stream □P	5 psi (max)
MOC	All surfaces exposed to the process fluid shall be 304 stainless steel.

		Shell Side Inlet	Tube Side Inlet
Pressure, psia		17.810	14.700
Temp, °F		221.860	60.000
Rate, lbm/hr	**Total**	2280.361	2062.211
	Ethanol	0.705	107.990
	Water	2259.480	1934.043
Volumetric flow rate, ft^3/hr		36.480	33.468
Density, lbm/ft^3		59.620	61.617
Avg. molecular weight		18.140	18.757
Cp, Btu/lbm-°F		18.259	18.290
Compressibility, Z		9.98E-04	1.05E-03
Viscosity, cP		0.266	1.162
Thermal conductivity, Btu/hr-ft-°F		0.389	0.316
Surface tension, dyn/cm		57.364	68.746

Heat curve attached.

Ambient air temperature,	maximum	100 °F
	minimum	–20 °F
Site elevation		890 ft above sea level
Electrical area classification		Class 1, Div. 2, Group D
Design/Test Pressure		Shell side: 300 psig (min), Tube side: 150 psig (min)
Fluid fouling factor (min)		0.001 hr-ft-°F/Btu

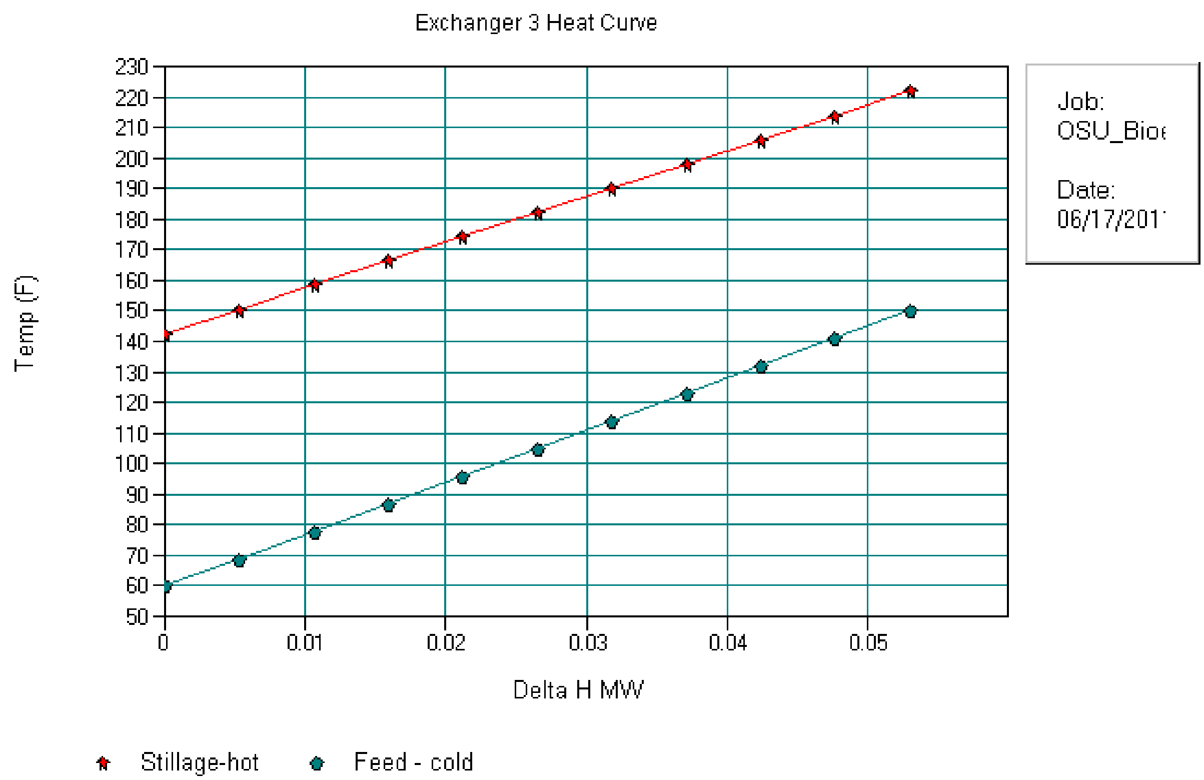
Single pass tube bundle arrangement.

Welded tubes are acceptable.

Additional Specifications

1. Fabrication of pressure vessels shall be in accordance with ASME Section VIII, Div. 1, latest edition.
2. Inspection and testing of pressure vessels shall be in accordance with ASME Section VIII, Div. 1, latest edition.
3. Dimensional tolerances for pressure vessels shall be as designated in the ASME code.
4. Prior to the final inspection and hydrostatic testing, the inside and outside of the new pressure vessel shall be cleaned and free of all foreign material.
5. The gaskets, joint rings and bolting used on all flanged connections during the hydrostatic pressure test of the new pressure vessel shall be of identical material and dimensions as those specified for service.
6. The water used for hydrostatic testing of the new pressure vessel shall have a chloride content of no greater than 50 ppm.
7. The Manufacturer’s quality control program shall be based on the ASME code.
8. It shall be the responsibility of the Manufacturer to ensure that the standards and practices of the Manufacturer’s quality control program are maintained by all of the Manufacturer’s subvendors and subcontractors.
9. The Manufacturer shall be responsible for suitably packaging, adequately supporting and securing all vessel components to prevent them from damage or loss prior to shipment. All packages shall include designated lift points or lifting lugs. All boxes, crates, bags, containers and skids shall be durably marked with the contents and the receiver’s address.
10. A protective, moisture-resistant coating shall be applied to all flanges and other machined surfaces prior to shipment.
11. Welding procedure specifications shall conform to the requirements of the ASME code.

Heat Curve



Data used in the chart: (ChemCAD 6.3.0 output)

Stream 4: Stillage - hot

NP	Temp F	Pres psia	Del H MW	Vapor lb/h	Liquid lb/h	Vap mole frac.	Vap mass frac.
1	221.9	17.8	0.0530	0	2280	0.0000	0.0000
2	214.0	17.8	0.0477	0	2280	0.0000	0.0000
3	206.1	17.8	0.0424	0	2280	0.0000	0.0000
4	198.2	17.8	0.0371	0	2280	0.0000	0.0000
5	190.3	17.8	0.0318	0	2280	0.0000	0.0000
6	182.3	17.8	0.0265	0	2280	0.0000	0.0000
7	174.4	17.8	0.0212	0	2280	0.0000	0.0000
8	166.4	17.8	0.0159	0	2280	0.0000	0.0000
9	158.5	17.8	0.0106	0	2280	0.0000	0.0000
10	150.5	17.8	0.00530	0	2280	0.0000	0.0000
11	142.5	17.8	0.000	0	2280	0.0000	0.0000

Stream 1: Feed - cold

NP	Temp F	Pres psia	Del H MW	Vapor lb/h	Liquid lb/h	Vap mole frac.	Vap mass frac.
1	60.0	14.7	0.000	0	2062	0.0000	0.0000
2	69.0	14.7	0.00530	0	2062	0.0000	0.0000
3	78.0	14.7	0.0106	0	2062	0.0000	0.0000
4	87.0	14.7	0.0159	0	2062	0.0000	0.0000
5	96.0	14.7	0.0212	0	2062	0.0000	0.0000
6	105.1	14.7	0.0265	0	2062	0.0000	0.0000
7	114.1	14.7	0.0318	0	2062	0.0000	0.0000
8	123.1	14.7	0.0371	0	2062	0.0000	0.0000
9	132.1	14.7	0.0424	0	2062	0.0000	0.0000
10	141.0	14.7	0.0477	0	2062	0.0000	0.0000
11	150.0	14.7	0.0530	0	2062	0.0000	0.0000

Appendix B1

3-D Rendition of the Alcohol Separation Unit

Next Page

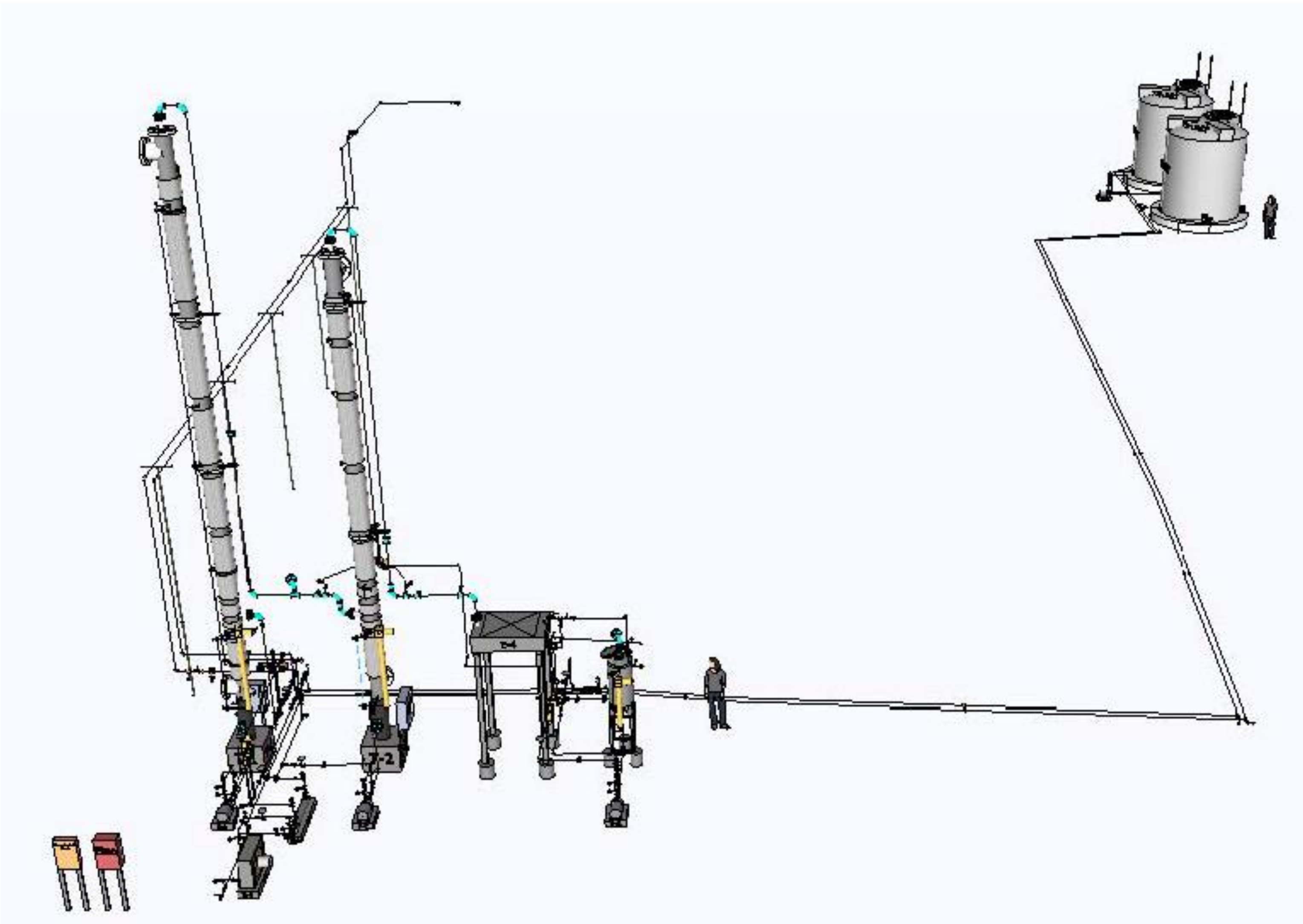


Figure B1: Process and Storage Area: View from

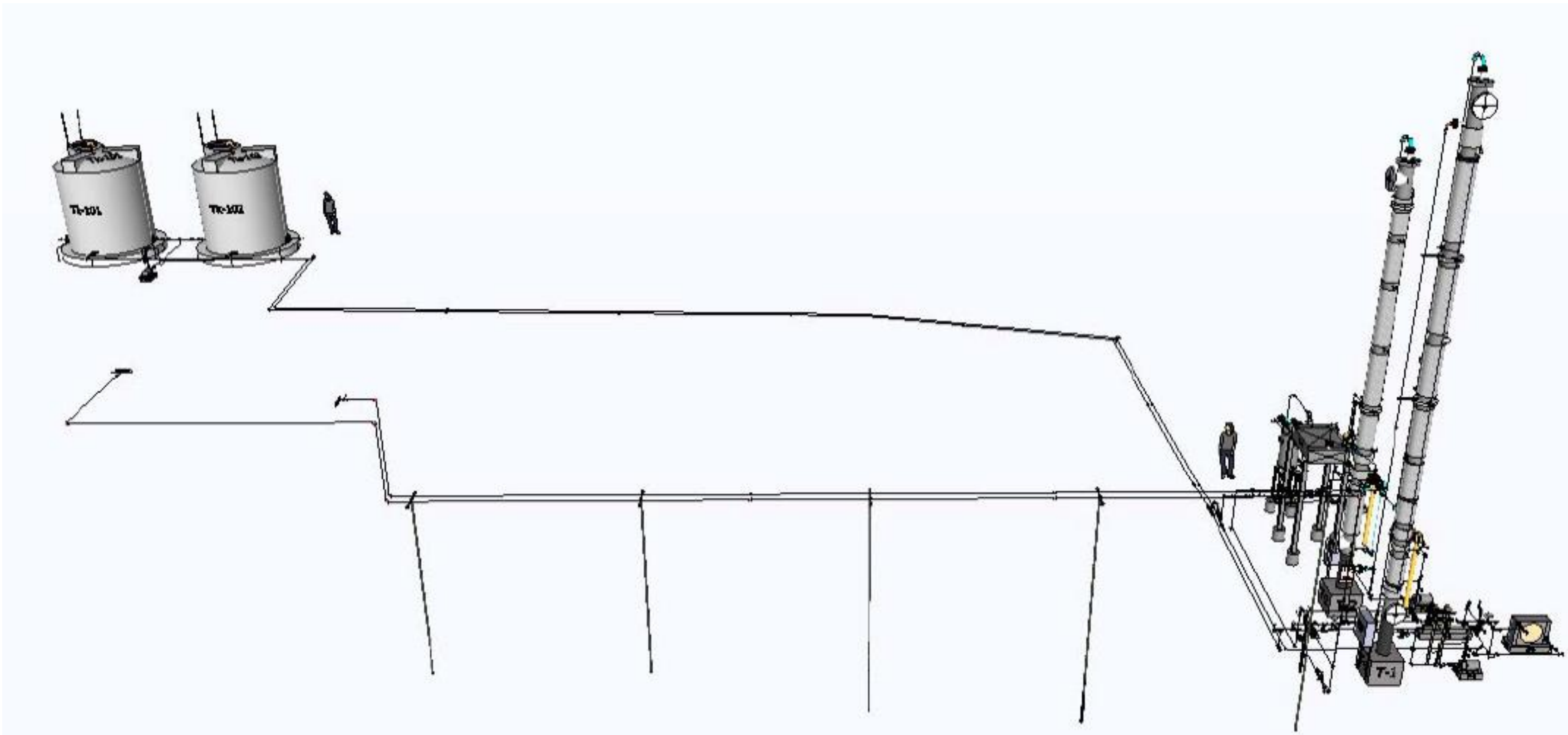
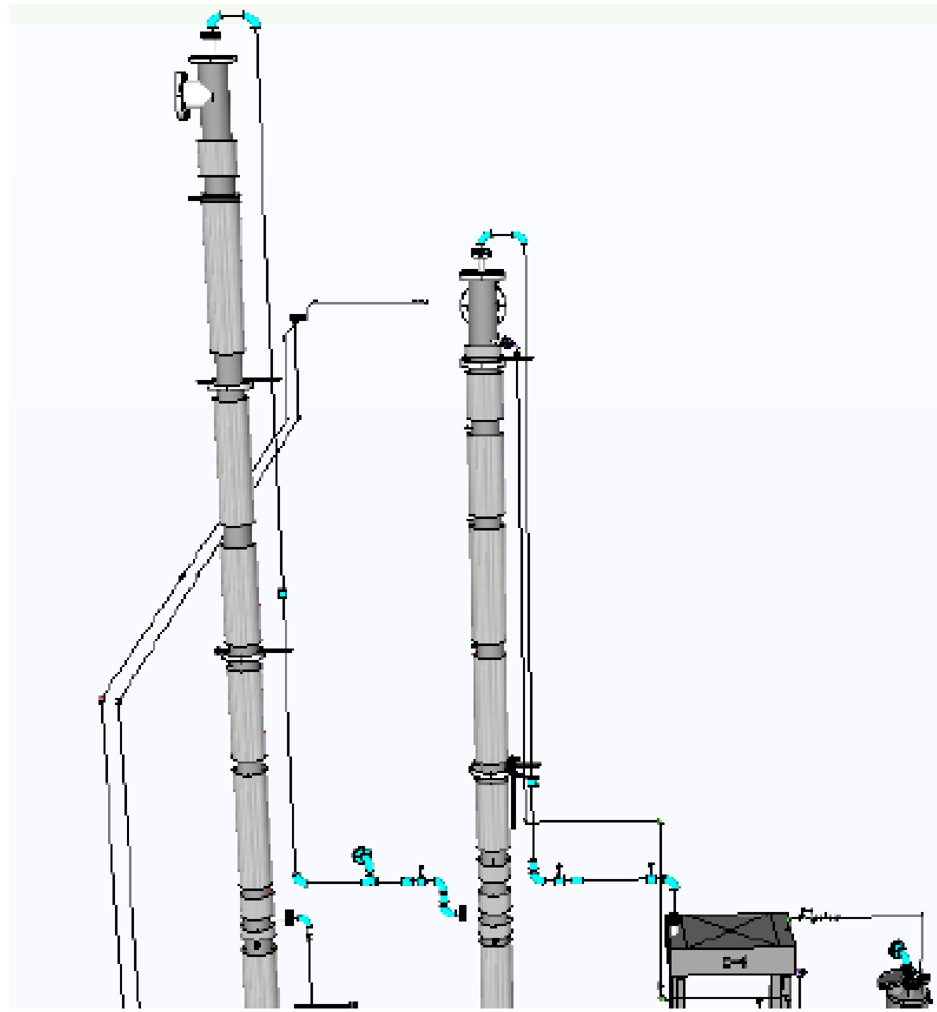


Figure B1.2: Process and storage area: View from North

(a) Process Top View from West



(b) Process high piping view from west

Figure B1.3: Process Views
Appendix B1 Continued

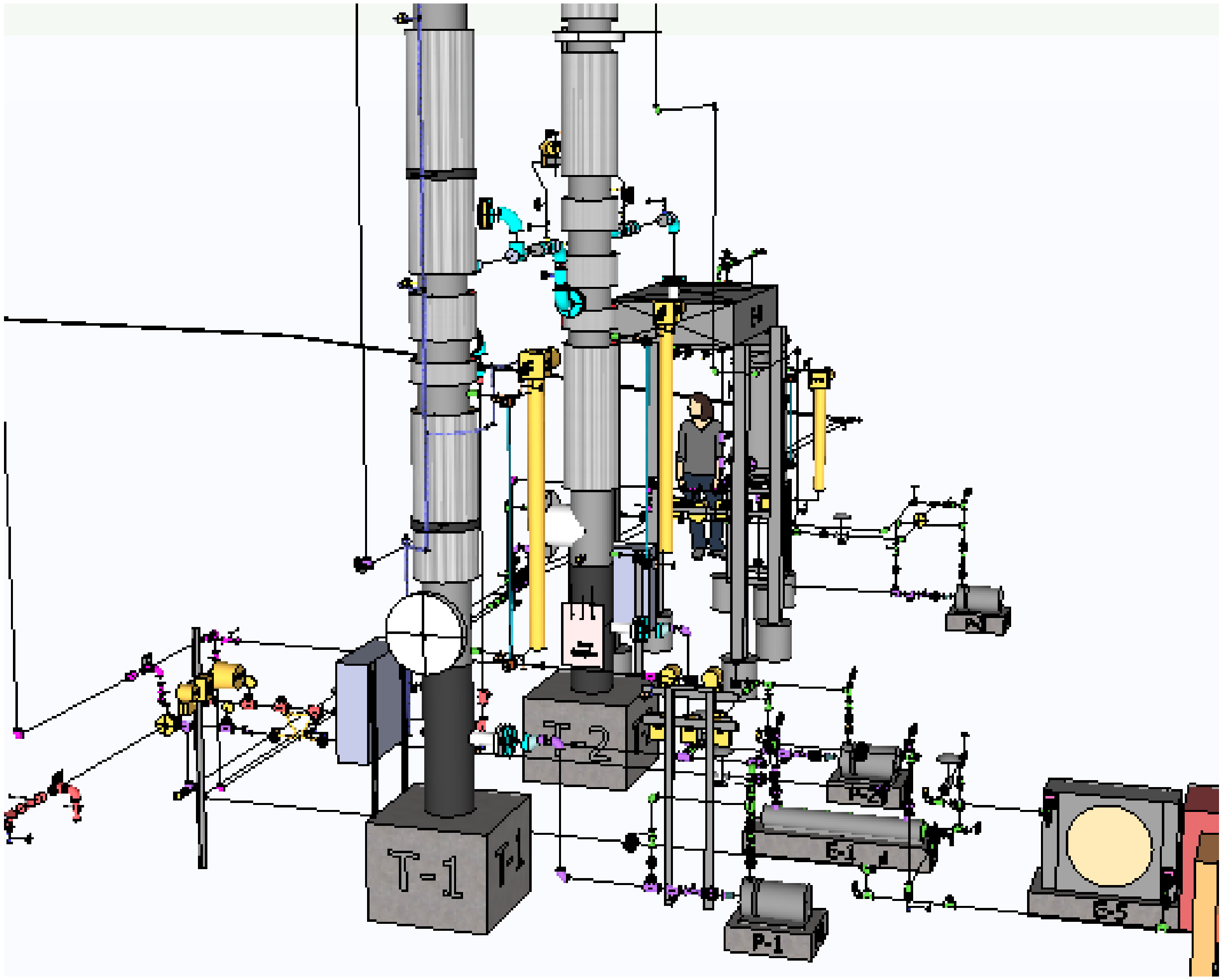


Figure B1.4: Process view from North

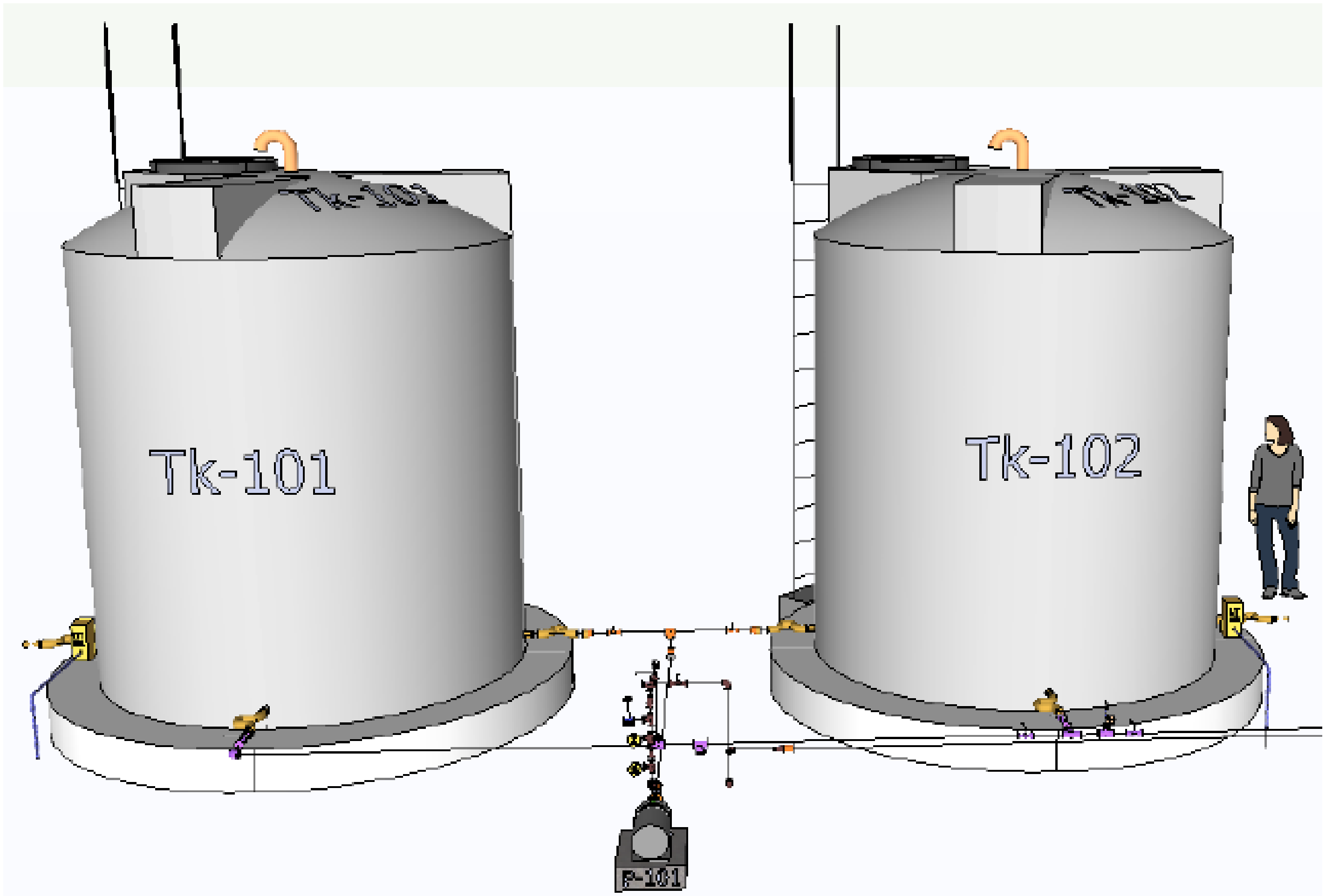


Figure B1.4: Storage area view from North

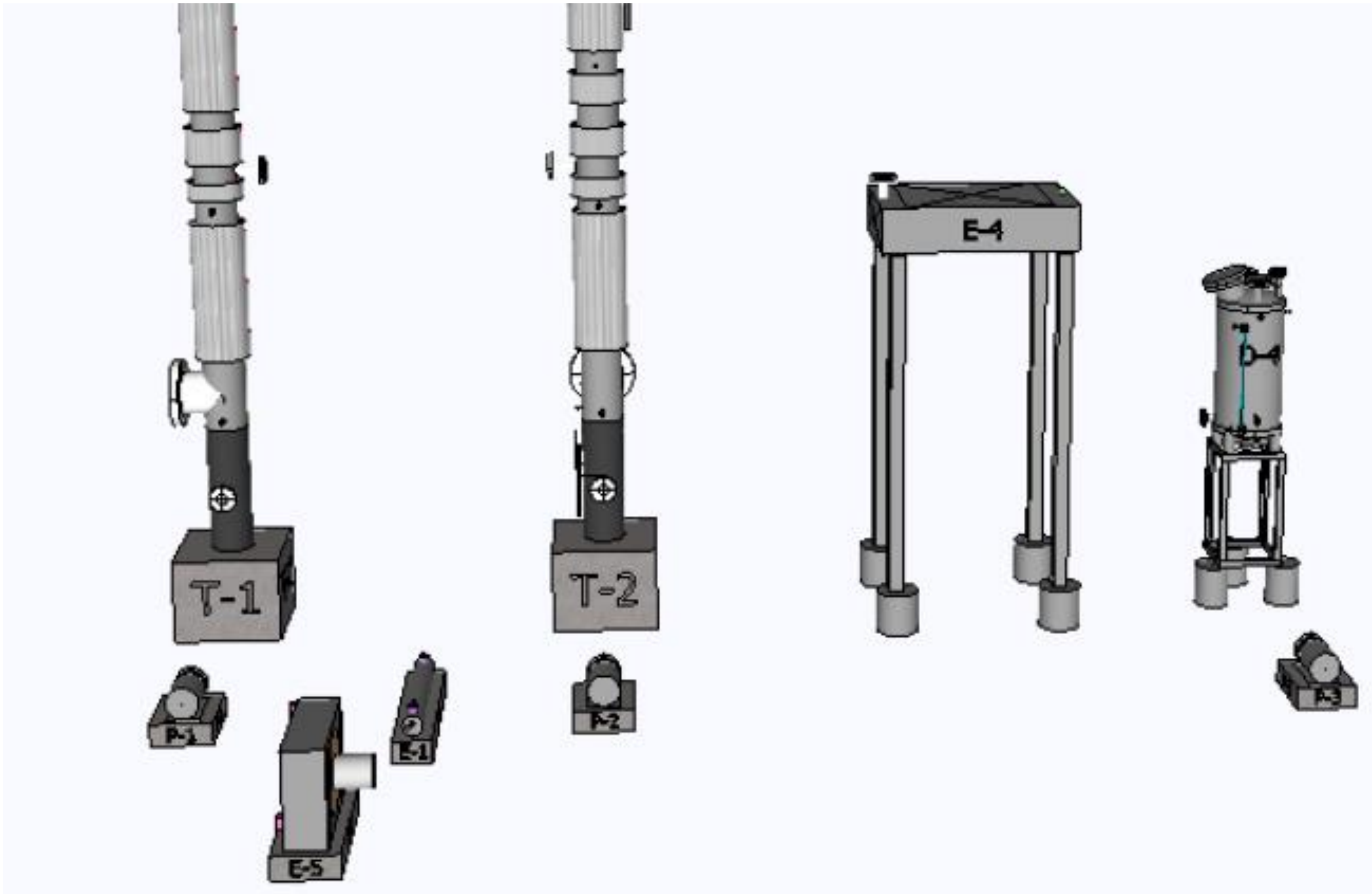


Figure B1.5: Major equipment – Process area only. View from West

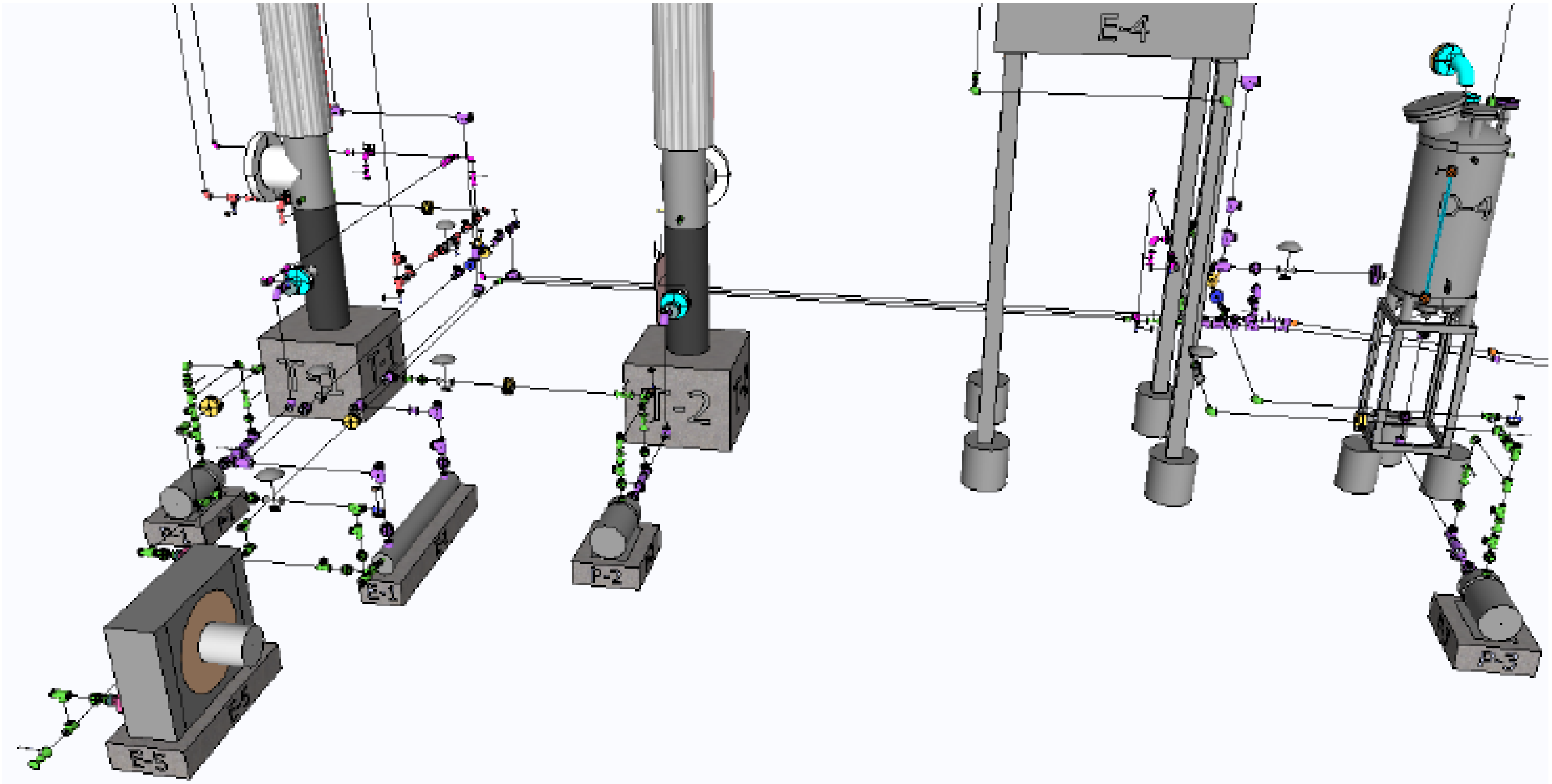


Figure B1.6: Low piping process area: View from West

Appendix B2: List of Suppliers

Storage Tanks
Arrowhead Plastic Engineering Inc.
2909 S Hoyt Ave.
Muncie, IN 47302
Phone: 765-286-0533
Fax: 765-286-1681

Heat Exchanger
Southwest Thermal Technology Inc.
3251 Corte Malpaso, Suite 507
Camarillo, CA 93012
Phone: (805)-484-2992
Fax: (805)-484-0049

Rupture Disk Assembly
Fike C/O Myers-Aubrey Co,
PO Box 470370
Tulsa, OK 74147
Phone : 918-622-3500
Fax : 918-628-0349
Website: www.myersaubrey.com

Air Cooled Condenser
GEA Rainey
5202 West Channel Road
Catoosa, OK 74015
Phone: (918)-266-3060
Fax: (918)-266-2464

Column Design: Shell and Internals
Sulzer Chemtech USA, Inc.
1 Sulzer Way
Tulsa, OK 74107
Phone :+1 918 446 6672
Fax: +1 918 446 5321

Piping Materials
Stillwater Steel and Supply
5320 East 6th Ave
Stillwater, OK 74074
Phone: 405-377-5550
Fax: 405-377-0377

Foundation and Support Drawings
Payne-Huber Engineering
7060 S. Yale, Suite 600
Tulsa, OK 74136
Phone: (918) 492-0975

Fechner Pump and Supply
1404 N Little Ave
Cushing, OK 74023
Phone: (918)-225-7867
Fax: (918)-225-7871

Column Fabrication
Total Energy Fabrication Corp.
2415 W Doolin Ave, Blackwell, OK 74631
100 W Airport Rd.
Stillwater, OK 74075
Phone: (580) 363-1500
Fax: (580) 363-1501 Fax
www.totalenergyfab.com

Wilson Supply
3401 Metcalf Drive
Oklahoma City, OK 73149
Phone: (405)-677-3382
Fax: (405)-670-1947

Insulation Materials
M&M Insulation Inc.
1625 S. Missouri Ave
Oklahoma City, OK, 73129
Phone: (405) 672-3373
Fax: (405) 672-3390
Email: corporate@mminsulation.com

Compression Fittings
Precision Fitting and Gauge Co.
1214 South Joplin
Tulsa, OK 74112
Phone: (918)-834-5011
Fax: (918)-835-6465

Hardware and Electrical Materials
Locke Supply Co.
901 E. 6th St
Stillwater, OK 74074
Phone: (405)-624-3900
Fax: (405)-377-8519
053@lockesupply.com

Lowes
1616 N Perkins Rd
Stillwater, OK 74075
Phone: (405)-377-3000
Fax: (405)-377-4206

Huzicker Brothers Inc.
707 E 3rd Ave
Stillwater, OK 74074
Phone: (405)-372-0542
Fax: (405)-372-5816

Fastenal Inc.
329 E Harned Ave
Stillwater, OK 74075
Phone: (405)-624-1010
Fax: (405)-624-1016

Internet Retailors
www.grainger.com
www.mcmastercarr.com
www.ebay.com

Specialty Fittings
Swagelok
9421 E 54th St
Tulsa, OK 74145
Phone: (918)-258-8661
Fax: (918)-258-1262

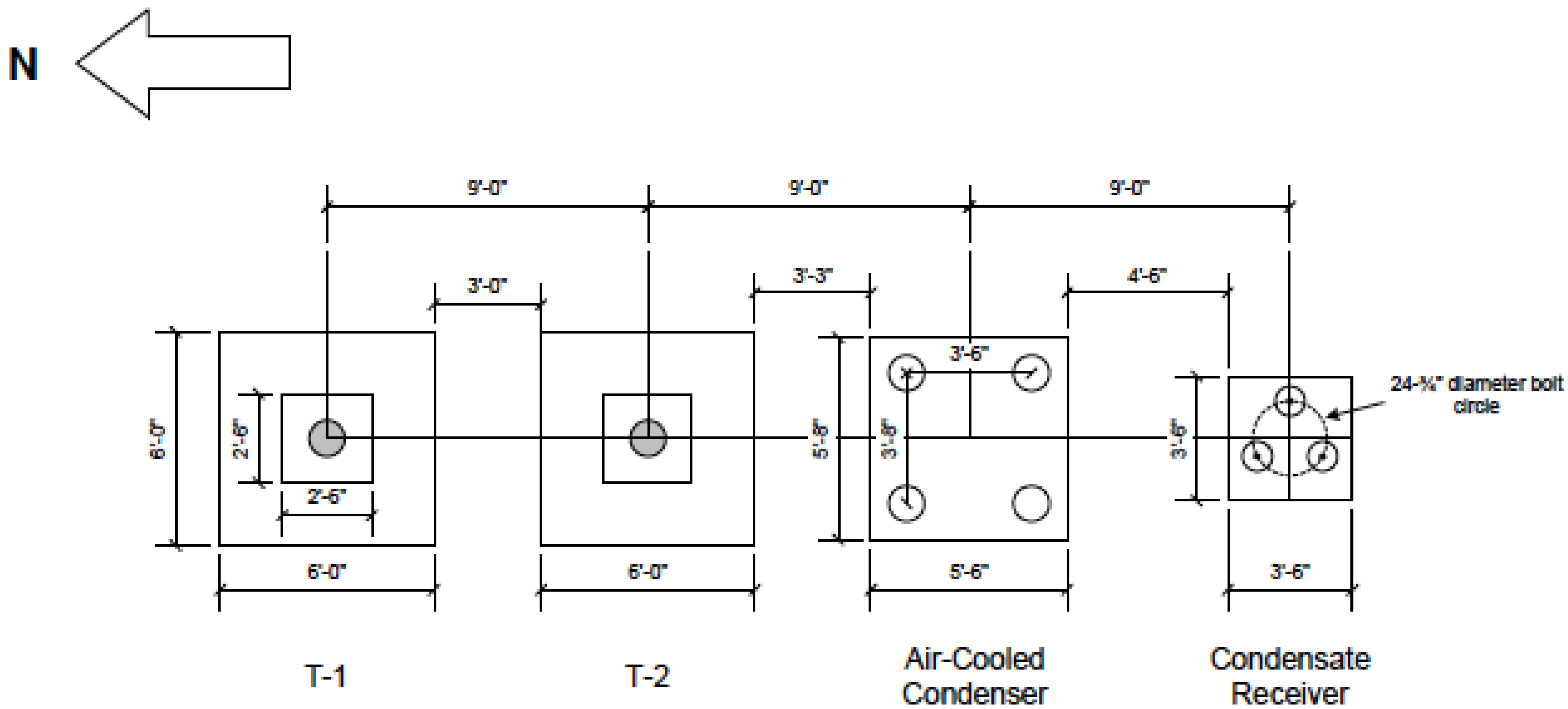
Sensor and Transmitter (Support)
Micromotion
7070 Winchester Circle
Boulder, CO 80301
Phone: (800)-522-6277
Fax: (800)-735-2585

Data Acquisition Hardware
Graphical Design Software
National Instruments
11500 N Mopac Expressway
Austin, TX 78759
Phone: (888)-280-7645

Appendix B3

Foundation Plan

Bioethanol Dewatering Unit
Foundations Layout – Final
5-Sep-2011



Appendix B4

Electrical Connection Layout

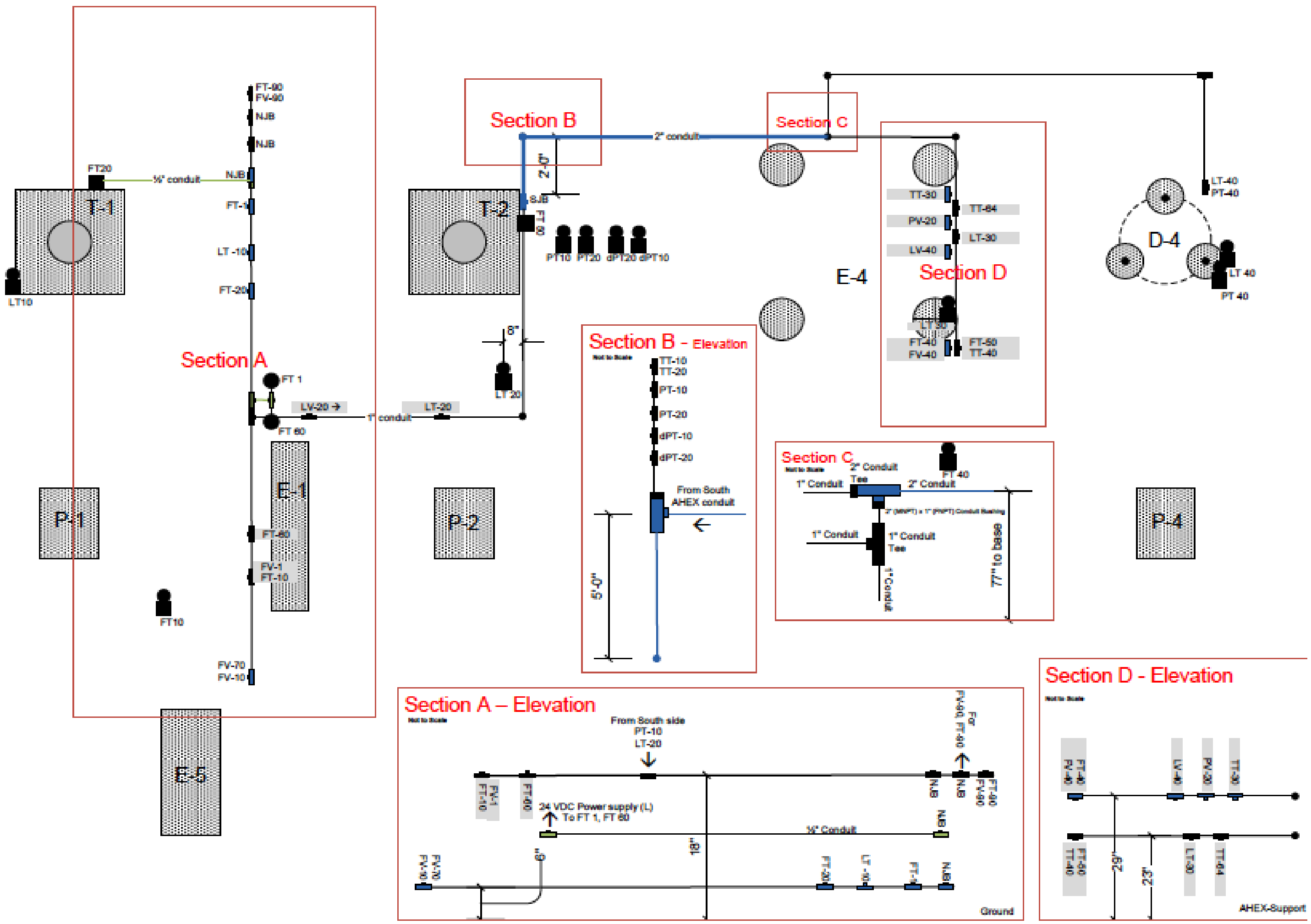


Figure B4: Electrical conduit layout at ASU

OSU BIOETHANOL PILOT PLANT: JUNCTION BOX WIRING DIAGRAM

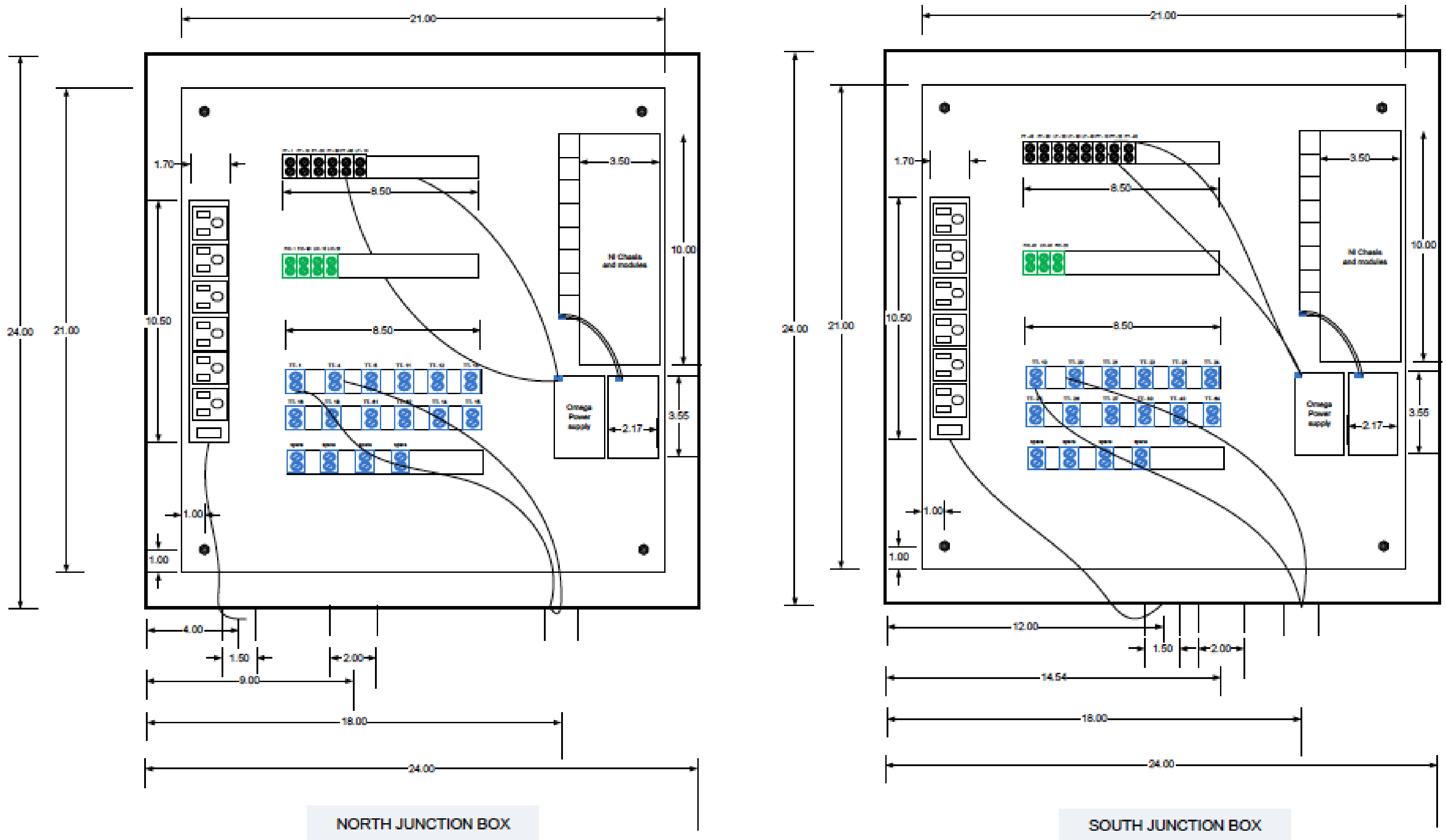
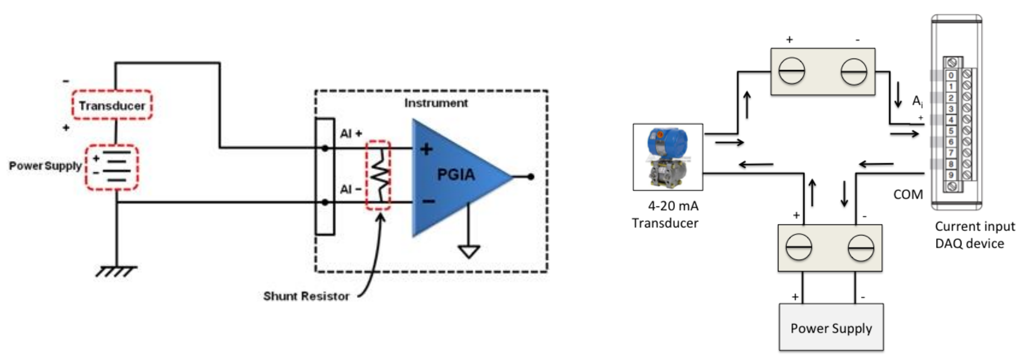


Figure B4.2: Junction box knockout connections and layout

A DC power supply is needed in order to complete the 4-20 mA signal loop. An Omega 20W, 1A, 24VDC power supply was mounted on a DIN rail (Figure B5.1). The number of transmitters that can be loop powered by a single power supply was calculated using Equation B5.1

$$Transmitter_{total} = \frac{Power\ Supply\ Amp}{Max\ Amps\ output\ for\ transmitter} \qquad \text{Eq. B5.1}$$

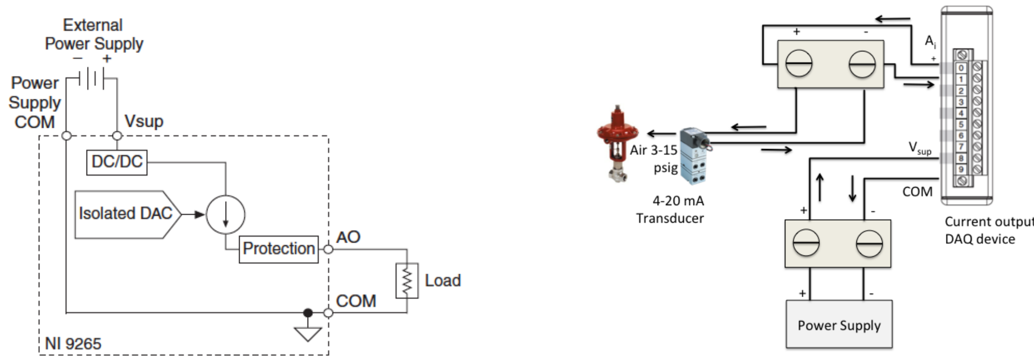
The maximum ampere rating on the 1151 DP loop powered transmitters were 28mA. The power supply rating was 1 A (=1000 mA). The number of transmitters that can be powered using this power supply is 35. Two power supplies were installed, one in each junction, due to limitations of space and connectivity (North and South junction boxes are separate). To connect multiple transmitters (7 transmitters in the north junction box) in a single powered loop, the power supply voltage was connected via jumper wires (Figure B5.2) across the terminal strip (from let to right in Figure B5.1). Also the (-)/COM terminal on the NI 9203 is connected to the one of the distributed V- from the power supply, on the terminal strip. This completes the 4-20 mA loop for all 1151DP style transmitters. All modules are grounded via the cDAQ chassis.



(a) 4-20 mA Circuit Diagram (b) 4-20 mA Current loop set up

Figure B5.2: 4-20 mA current loop

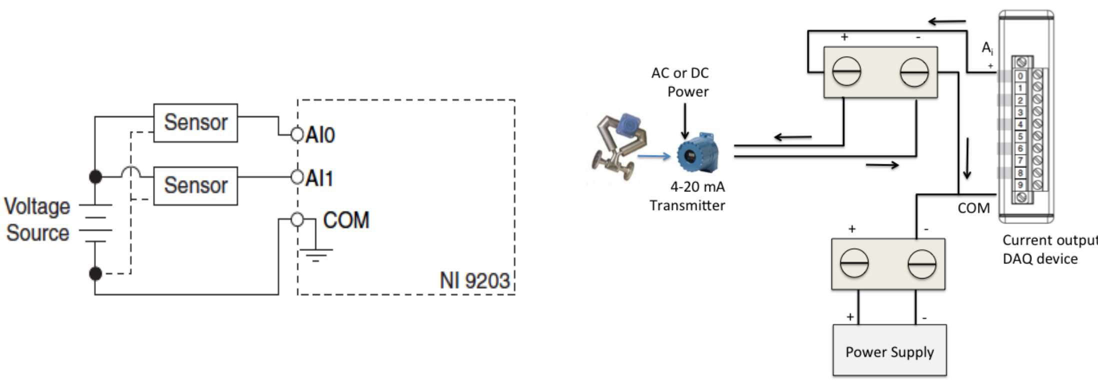
The NI 9265 was powered using jumper wires as well (spreading out V+ and V- from a single power source). The distributed power supply V+ and V- on terminal strips were used for NI9265 power input at V_{sup} (+) and COM terminals (Figure B5.3).



(a) 4-20 mA Output Current Diagram (b) 4-20 mA Output loop set up

Figure B5.3: 4-20 mA current output connections

Micromotion Field Mount RFT9739 transmitters were powered with individual power sources (AC or DC). The 4-20 mA signal diagram was different (Figure B5.4). The negative lead of all current signals connected to the COM port and the positive lead individually connected to A0 to A7. The negative connection from the 24VDC power supply connected to COM also.



(a) 4-20 mA Input Current Diagram (b) 4-20 mA Input loop set up

Figure B5.3: 4-20 mA current input connections for Micromotion Transmitters

Appendix B6

Wiring Connections

The National Instruments data acquisition device cDAQ 9188 was mounted on the right and three modules (NI 9213, 9203, and 9265) were mounted in the first three slots. Two extra thermocouple strips and one extra 4-20 mA terminal strip were provided for future connections. A DIN rail section was mounted to the panel to mount the 24 VDC power supply.

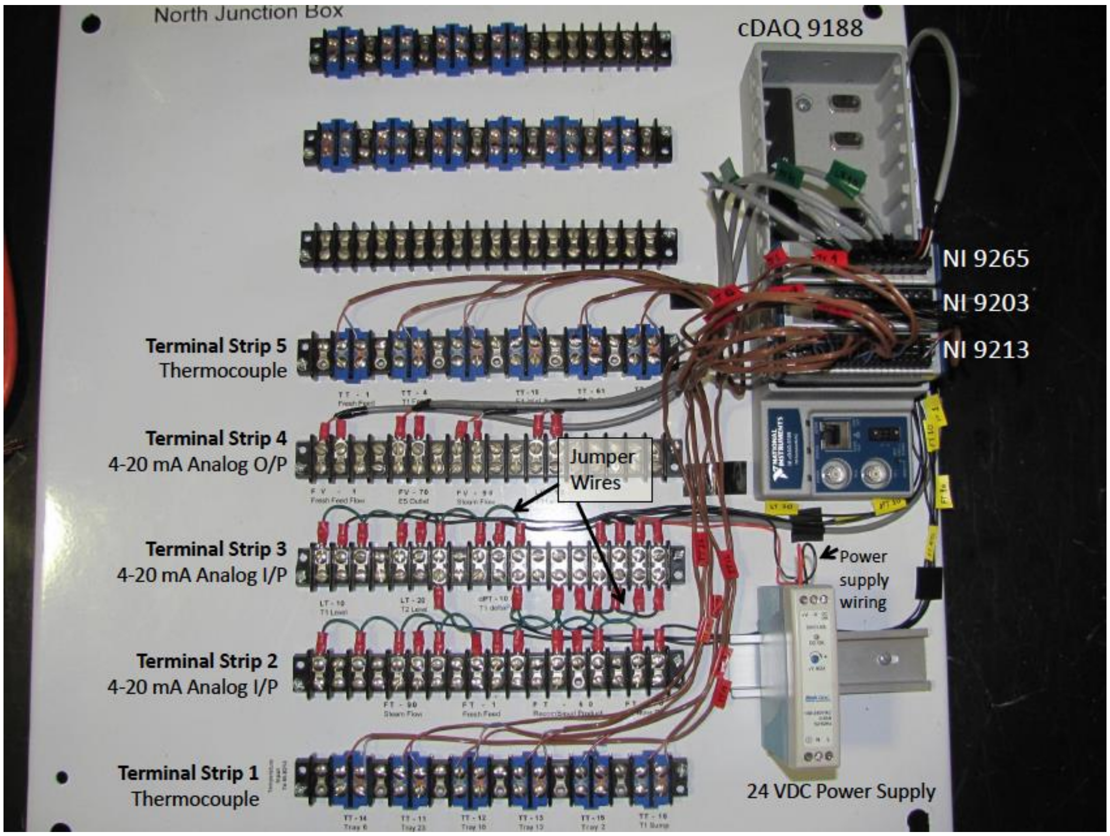


Figure B5.1: Wiring connections for North junction panel

Signal wires from transmitters and thermocouple wires were pulled into the junction box. Once in the junction box each wire pair was connected to its labeled location on the terminal strips mounted on the panel. Terminal strip functions include (Figure B5.1)

1. 12 thermocouple wires (Terminal strips 1 and 5) to NI 9213
2. 7 Transmitter wires (Terminal strips 2 and 3) 4-20 mA, analog input to NI 9203
3. 4 I-to-P Transducer connections (Terminal 4) 4-20 mA, analog output from NI 9265

Jumper wires were used to provide common V+ connections on the terminal strip. Loop powered signal wires (4-20 mA from Rosemount transmitters) terminate at terminal strip, at labeled location. Similarly, thermocouple wires pulled into the junction box and terminate on Terminal Strip 1 or 5, where indicated. All connection locations were assigned sequentially numerically.

Appendix B7

Column Internals Installation

(a) Cartridge trays for beer column

(b) Tray installation in beer column

(c) Column Base

(d) Rectifier Top Flange

(e) Liquid distributor for rectifier and packing support plate

(f) Column at Total Fabrication Inc.

Appendix B7 Continued

Column Delivery, Insulation, and Erection on site

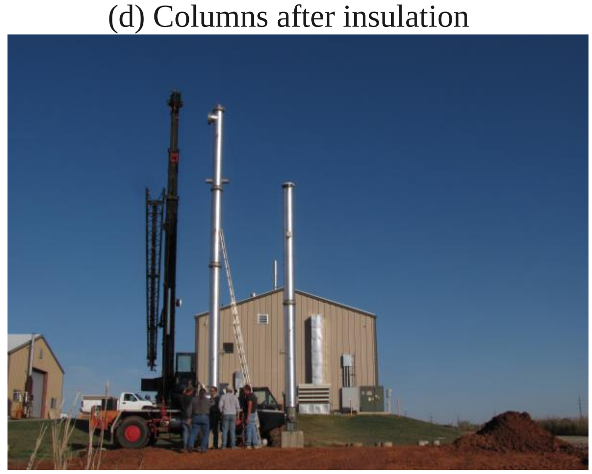
(a) Column delivery to ASU site

(b) Column transfer

(c) Columns assembled and delivered

(d) Columns after insulation

(e) Lifting trunnions in use



(f) Columns in place

Appendix B8

Torque Procedure Diagram

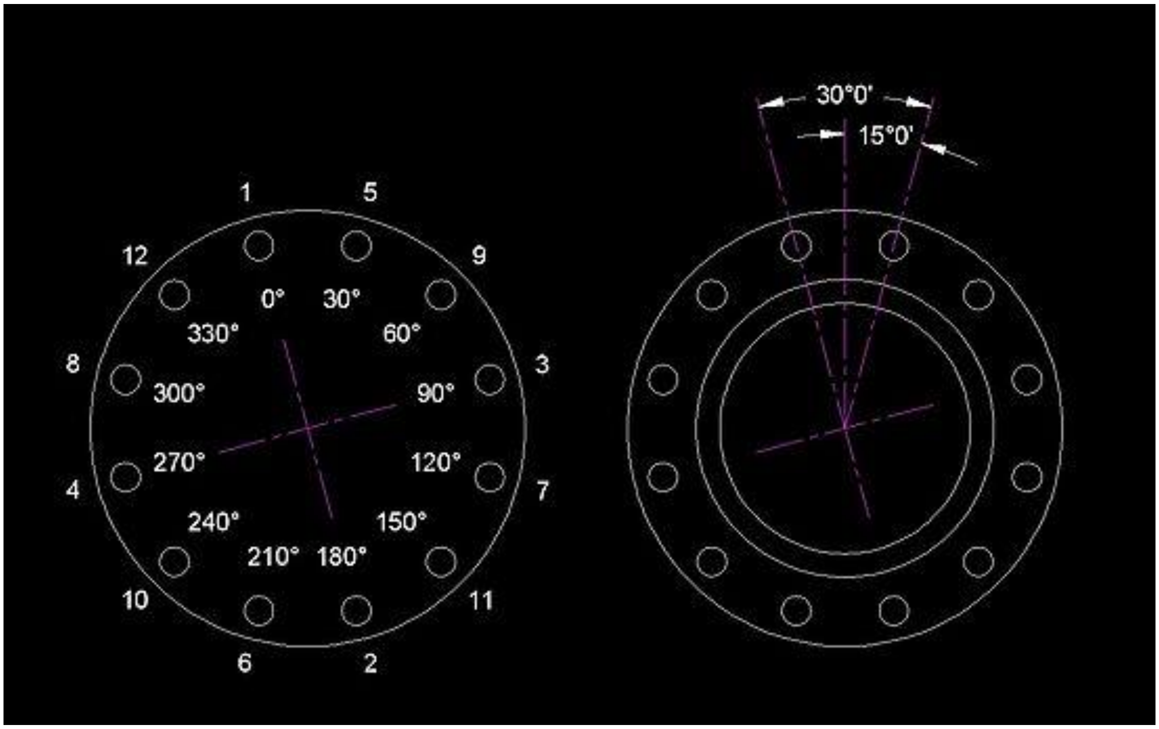


Figure B8.1: Torque Procedure for 12 bolt flange (connecting and end cap flanges on both columns)

Appendix B9

National Instruments Module Wiring Key

North Junction Box		
Key to NI Parts:		
Temperature Input		**9213**
cDAQ9188-16BD8BDMod1	NC	TT
cDAQ9188-16BD8BDMod1	ai0	11
cDAQ9188-16BD8BDMod1	ai1	12
cDAQ9188-16BD8BDMod1	ai2	13
cDAQ9188-16BD8BDMod1	ai3	15
cDAQ9188-16BD8BDMod1	ai4	16
cDAQ9188-16BD8BDMod1	ai5	62
cDAQ9188-16BD8BDMod1	ai6	61
cDAQ9188-16BD8BDMod1	ai7	18
cDAQ9188-16BD8BDMod1	ai8	5
cDAQ9188-16BD8BDMod1	ai9	4
cDAQ9188-16BD8BDMod1	ai10	1
cDAQ9188-16BD8BDMod1	ai11	
cDAQ9188-16BD8BDMod1	ai12	14
cDAQ9188-16BD8BDMod1	ai13	
cDAQ9188-16BD8BDMod1	ai14	
cDAQ9188-16BD8BDMod1	ai15	ambient
Analog Input, Current		**9203**
cDAQ9188-16BD8BDMod2	ai0	LT - 10
cDAQ9188-16BD8BDMod2	ai1	PT-90
cDAQ9188-16BD8BDMod2	ai2	dPT - 10
cDAQ9188-16BD8BDMod2	ai3	FT - 10
cDAQ9188-16BD8BDMod2	ai4	FT - 60
cDAQ9188-16BD8BDMod2	ai5	FT - 1
cDAQ9188-16BD8BDMod2	ai6	FT - 90
cDAQ9188-16BD8BDMod2	ai7	
Analog Output, Current		**9265**
cDAQ9188-16BD8BDMod3	ai0	FV - 1
cDAQ9188-16BD8BDMod3	ai1	FV - 70
cDAQ9188-16BD8BDMod3	ai2	FV - 90
cDAQ9188-16BD8BDMod3	ai3	LV - 10

Appendix B9 Continued

National Instruments Module Wiring Key

South Junction Box		
Key to NI Parts:		
Temperature Input		**9213**
cDAQ9188-16BD882Mod1	NC	TT
cDAQ9188-16BD882Mod1	ai0	20
cDAQ9188-16BD882Mod1	ai1	21
cDAQ9188-16BD882Mod1	ai2	22
cDAQ9188-16BD882Mod1	ai3	23
cDAQ9188-16BD882Mod1	ai4	24
cDAQ9188-16BD882Mod1	ai5	25
cDAQ9188-16BD882Mod1	ai6	26
cDAQ9188-16BD882Mod1	ai7	27
cDAQ9188-16BD882Mod1	ai8	30
cDAQ9188-16BD882Mod1	ai9	40
cDAQ9188-16BD882Mod1	ai10	64
cDAQ9188-16BD882Mod1	ai11	10
Analog Input, Current		**9203**
cDAQ9188-16BD882Mod2	ai0	PT-10
cDAQ9188-16BD882Mod2	ai1	dPT-20
cDAQ9188-16BD882Mod2	ai2	FT-40
cDAQ9188-16BD882Mod2	ai3	LT-20
cDAQ9188-16BD882Mod2	ai4	PT-40
cDAQ9188-16BD882Mod2	ai5	LT-30
cDAQ9188-16BD882Mod2	ai6	LT-40
cDAQ9188-16BD882Mod2	ai7	PT-20
Analog Output, Current		**9265**
cDAQ9188-16BD882Mod3	ai0	LV-20
cDAQ9188-16BD882Mod3	ai1	PV-20
cDAQ9188-16BD882Mod3	ai2	FV-40
cDAQ9188-16BD882Mod3	ai3	LV-40
Analog Input, Current		
cDAQ9188-16BD882Mod4	ai0	Tk-102
cDAQ9188-16BD882Mod4	ai1	Tk-101
cDAQ9188-16BD882Mod4	ai2	FT-50
cDAQ9188-16BD882Mod4	ai3	FT-20
cDAQ9188-16BD882Mod4	ai4	
cDAQ9188-16BD882Mod4	ai5	
cDAQ9188-16BD882Mod4	ai6	
cDAQ9188-16BD882Mod4	ai7	

Appendix B9 Continued

Instrument Tags

Measurement	Equipment	Tag	Service	Instrument
Temperature	T-1 Beer Column	TT-15, 14, 13, 12, 11	T-1 Trays - 1, 5, 12, 17, 23	Thermocouple, TT-type Cu-Co
		TT-16	T-1 Sump	Thermocouple, TT-type Cu-Co
	T-2 Rectifier	TT-26, 25, 24	T-2 Trays - 1, 2, 3	Thermocouple, TT-type Cu-Co
		TT-23, 22, 21	T-2 Packing - Bottom, mid, top	Thermocouple, TT-type Cu-Co
		TT-27	T-2 Sump	Thermocouple, TT-type Cu-Co
	E-1 Feed Preheater	TT-1	Feed inlet	Thermocouple, TT-type Cu-Co
		TT-4	Feed outlet	Thermocouple, TT-type Cu-Co
		TT-18	Stillage inlet	Thermocouple, TT-type Cu-Co
		TT-61	Stillage outlet/inlet to E-5	Thermocouple, TT-type Cu-Co
	E-5 Stillage Cooler	TT-65	Stillage outlet	Thermocouple, TT-type Cu-Co
	E-4 Condenser	TT-20	Product inlet	Thermocouple, TT-type Cu-Co
		TT-30	Product outlet	Thermocouple, TT-type Cu-Co
	Process	TT-40	Reflux	Thermocouple, TT-type Cu-Co
		TT-10	T-1 overhead	Thermocouple, TT-type Cu-Co
		TT-64	Recombined product	Thermocouple, TT-type Cu-Co
		TT-65	Ambient	Thermocouple, TT-type Cu-Co

Appendix B9 Continued

Instrument Tags

Measurement	Equipment	Tag	Service	Instrument
Flow	Feed	FT-1	Feed flow	Micromotion CMF050/RFT9739 transmitter
	Steam	FT-90	Steam	Orifice/Rosemount 1151DP transmitter
	T-1 bottoms stream	FT-10	Stillage	Orifice/Rosemount 1151 DP transmitter
	Product	FT-50	Distillate	Micromotion CMF010/RFT9739 transmitter
	Reflux	FT-40	Reflux	Orifice/Rosemount 1151 DP transmitter
	Stillage	FT-60	Stillage	Micromotion CMF025/RFT9739 transmitter
	T-2 bottoms stream	FT-20	T-2 bottoms stream	Micromotion CMF025/RFT9739 transmitter
Level	T-1 Beer column	LT-10	T-1 beer column level	1151 DP transmitter
	T-2 Rectifier	LT-20	T-2 Rectifier level	1151 DP transmitter
	D-4 Reflux drum	LT-40	Reflux drum level	1151 DP transmitter
	E-4 Condenser	LT-30	Condenser level	1151 DP transmitter
	Tk-101	Tk-101	Tk-101 level	
	Tk-102	Tk-102	Tk-102 level	
Pressure	T-1 Beer column	PT-10	T-1 beer column top pressure	1151 GP transmitter
	T-2 Rectifier	PT-20	T-2 rectifier top pressure	1151 GP transmitter
	T-1 Beer Column	dPT-10	T-1 beer column differential pressure	1151 DP transmitter
	T-2 Rectifier	dPT-20	T-2 rectifier differential pressure	1151 DP transmitter
	D-4 Pressure	PT-40	D-4 reflux drum pressure	1151 GP transmitter

Appendix B9 Continued

Measurement and Automation (MAX) Operating Instructions

Section 1: Operating Instructions: Hardware Connectivity

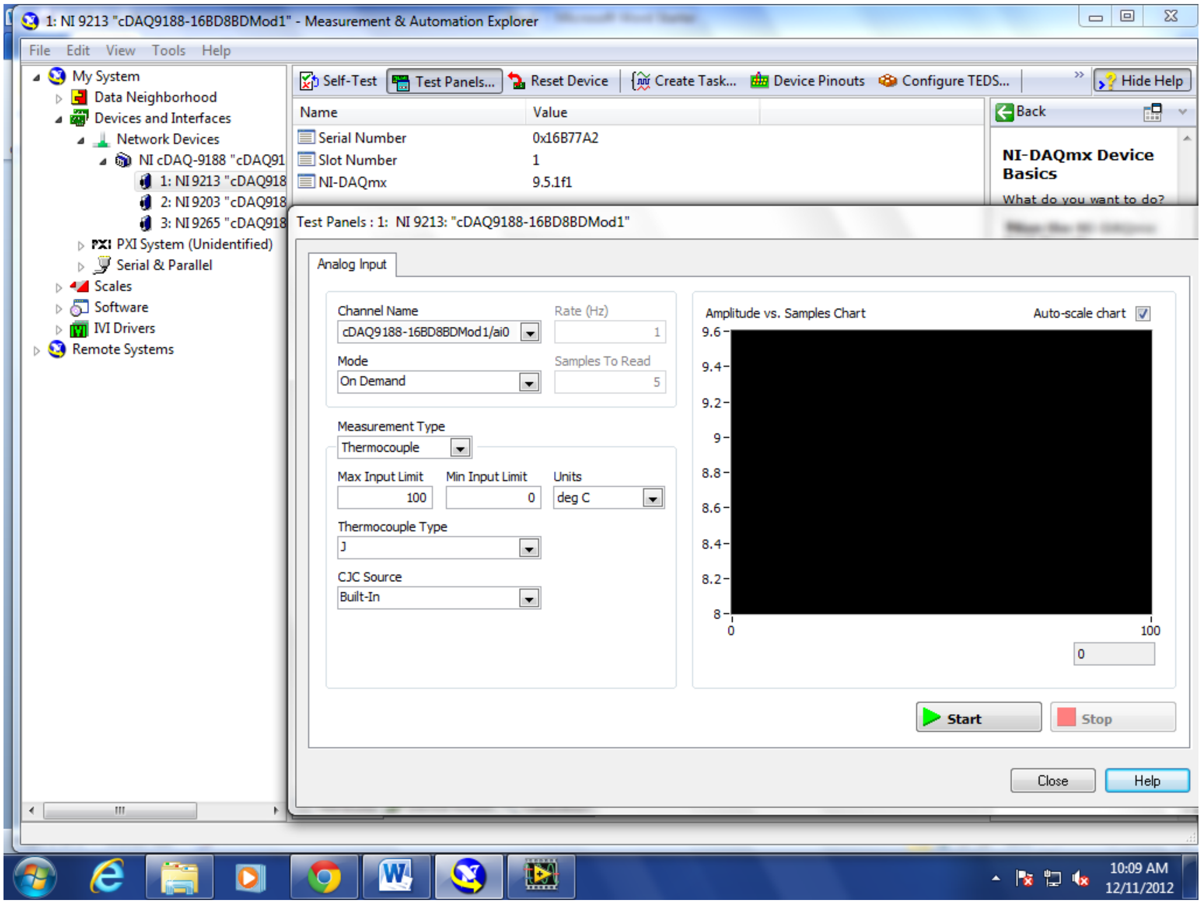
1. Unplug NI 9188 chassis and Omega 24VDC power supply inside the Junction Box (N and/or S) from AC power source/plug when not in use.
2. Also unhook CAT5e Ethernet cable from Belkin switch (inside the bioenergy lab) when not in use.
3. In order to start the measurement process:
    a. Connect Omega power supply and NI chassis to AC power socket.
    b. Connect Ethernet cable into Belkin switch.
    c. Make sure network cable is also inserted into the Belkin switch. The network cable is marked so with a tag. Also make sure the computer is connected to the Belkin switch using a short Ethernet cable.
    d. Once the connections have been made, and the PC started (ASU account, password asuBIOenergy@0!3) open MAX – Measurement Automation and Control from desktop (NI program to test connections to the chassis and modules.
    e. In the left had panel, under: My Systems → Devices and Interfaces → Network Devices → cDAQ9188-16BD8BD (N junction box chassis). Click on the chassis icon.
    f. Click on self-test icon on icon ribbon on top-center of the dialog box. After a Self-test dialog box you should get the message: Driver successfully communicated with cDAQ9188-16BD8BD
    g. Click on the arrow next to the chassis icon to reveal individual modules – NI 9213, NI 9203, NI 9265.

Section 2: Operating Instructions: Testing Hardware in MAX

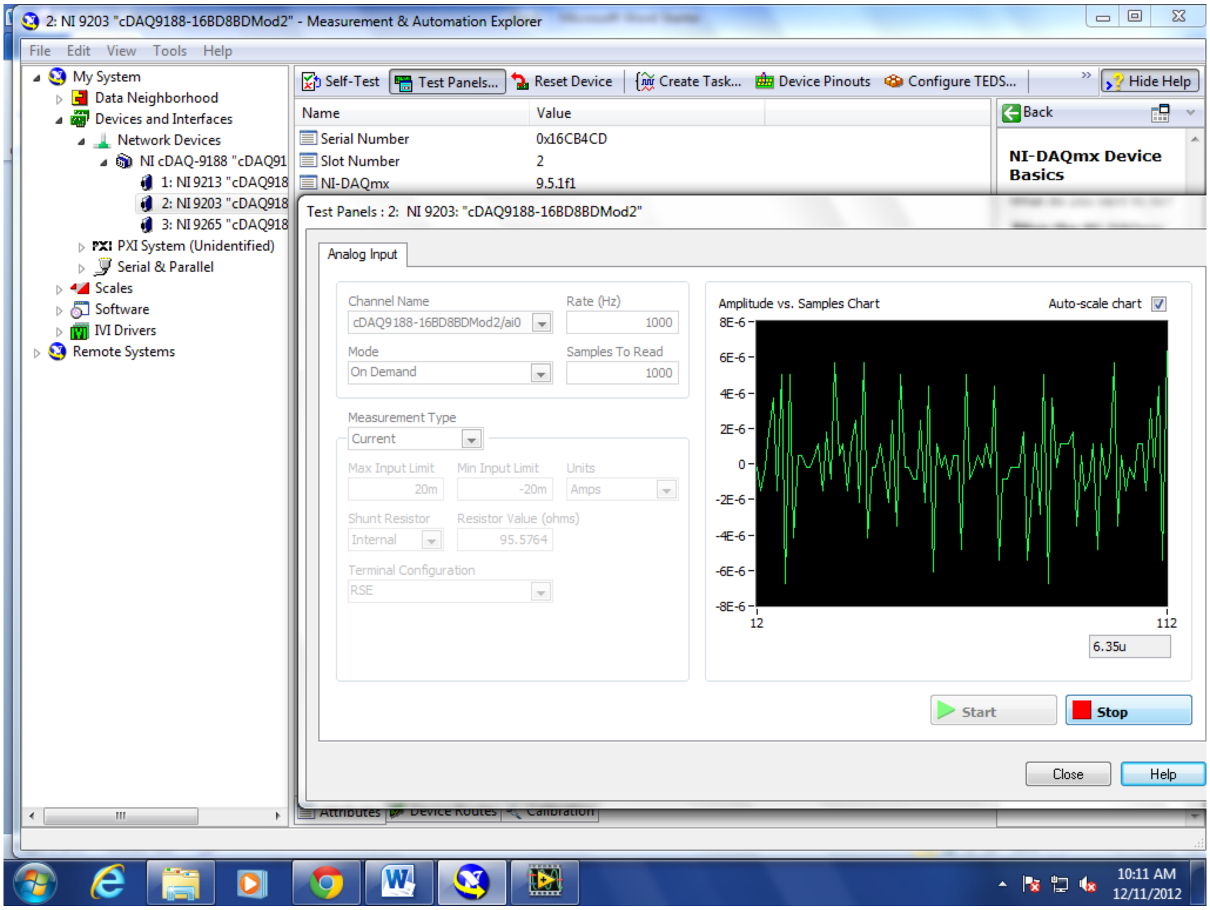
1. In order to test measurements on NI 9213, click on module icon, open Test-Panel (present on icon ribbon, top center of dialog box) and make following selections:
    a. Channel name: select channel of choice.
    b. Mode: Continuous
    c. Rate: 1 Hz, as is
    d. Samples to read: 5, as is
    e. Thermocouple: T
    f. Max Input: 150
    g. Min Input: 0
    h. Units: F

i. CJC source: Built-in as is

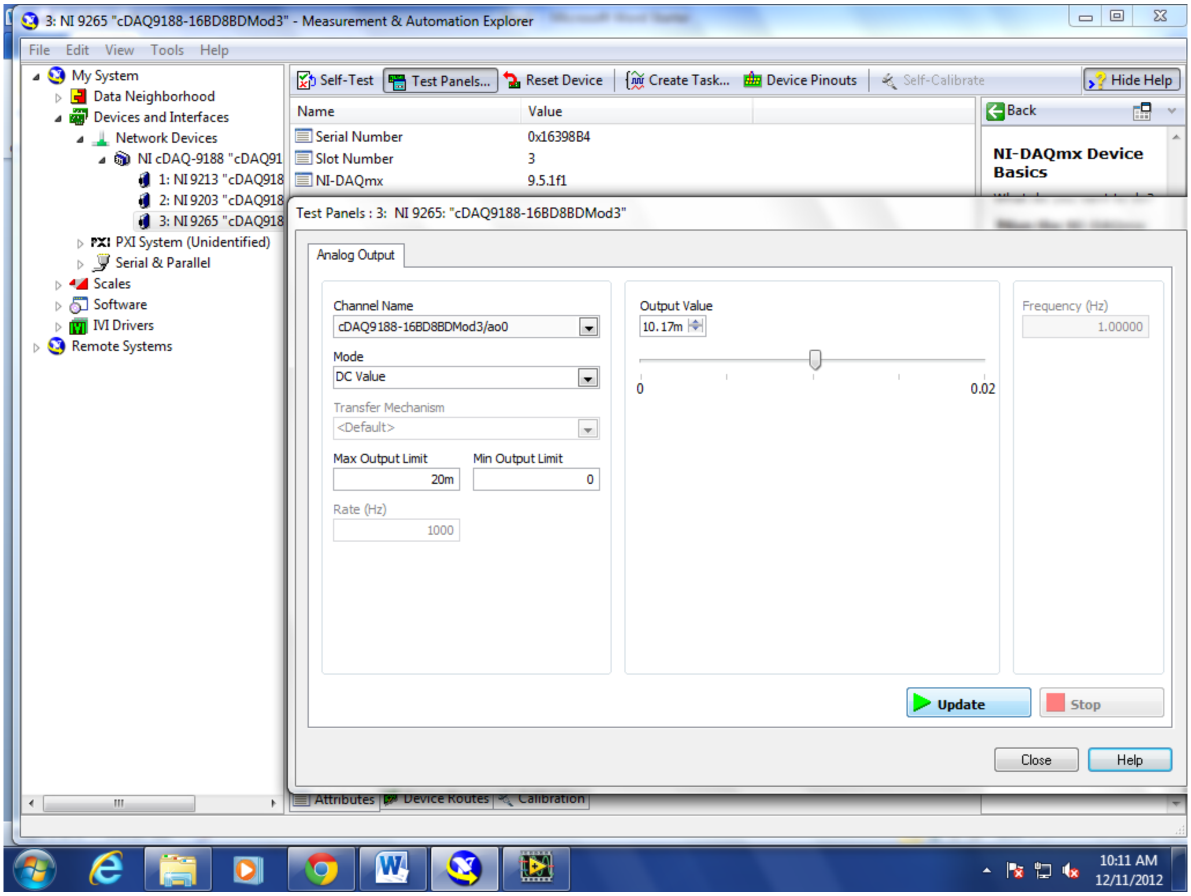
j. Click on Start to view data on chart. You can select/de-select Auto scale



2. In order to test measurements on NI 9203, click on module icon, open Test-Panel (present on icon ribbon, top center of dialog box) and make following selections:
    a. Channel name: select channel of choice.
    b. Mode: On-demand
    c. Rate: 1000 Hz, as is, (not an option in case of On-demand measurements)
    d. Samples to read: 1000, as is for continuous mode only
    e. Measurement type: Current, as is
    f. Max Input: 20mA, as is
    g. Min Input: 0
    h. Shunt resistor: internal, as is
    i. Resistor value: 95.57, as is
    j. Terminal connection: RSE, as is
    k. Click on Start to view data on chart. You can select/de-select Auto scale

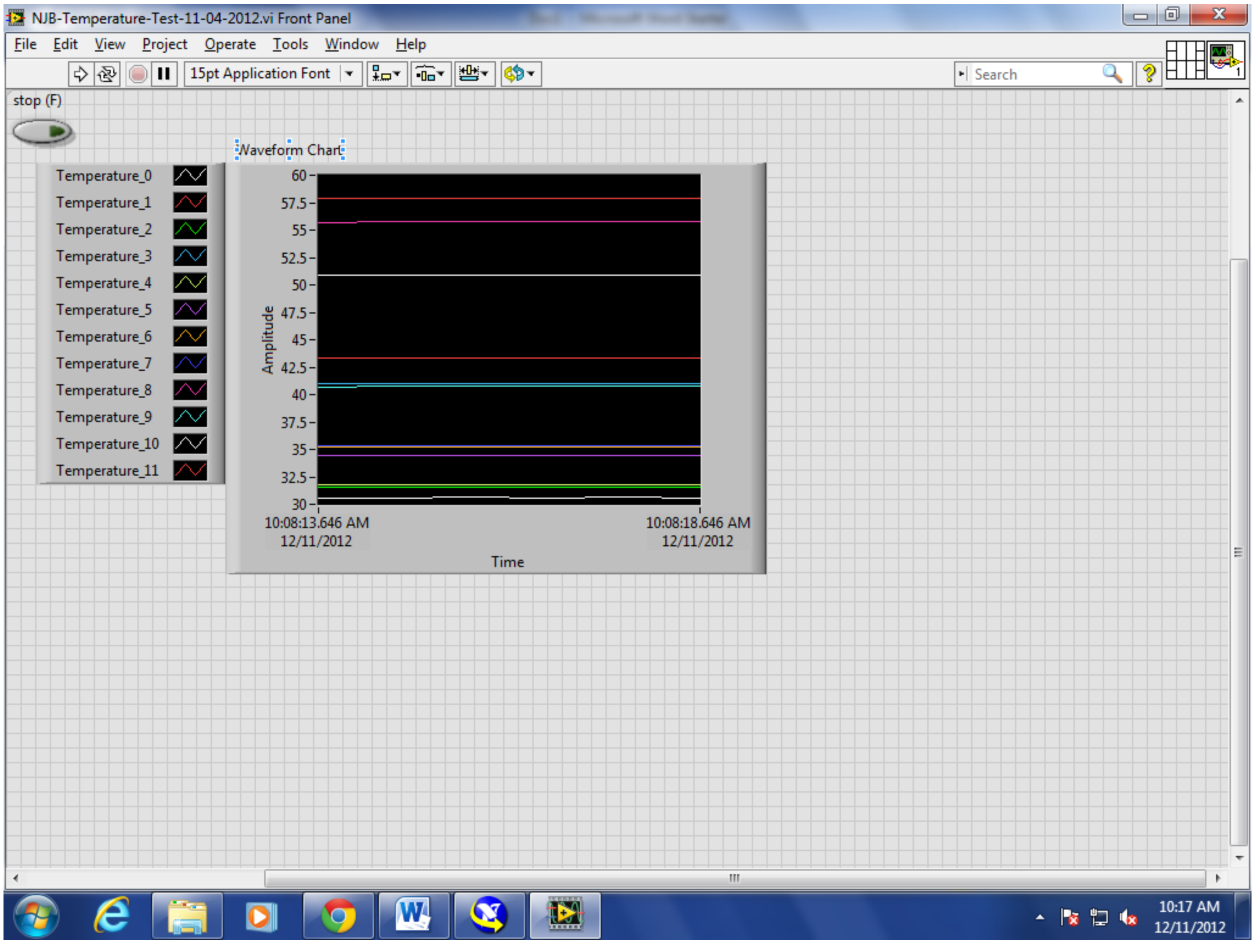


3. In order to test measurements on NI 9265, click on module icon, open Test-Panel (present on icon ribbon, top center of dialog box) and make following selections:
    a. Channel name: select channel of choice
    b. Mode: DC value
    c. Max output limit: 20m
    d. Min output limit: 0
    e. Adjust output value using slider.
    f. Press Update to apply.



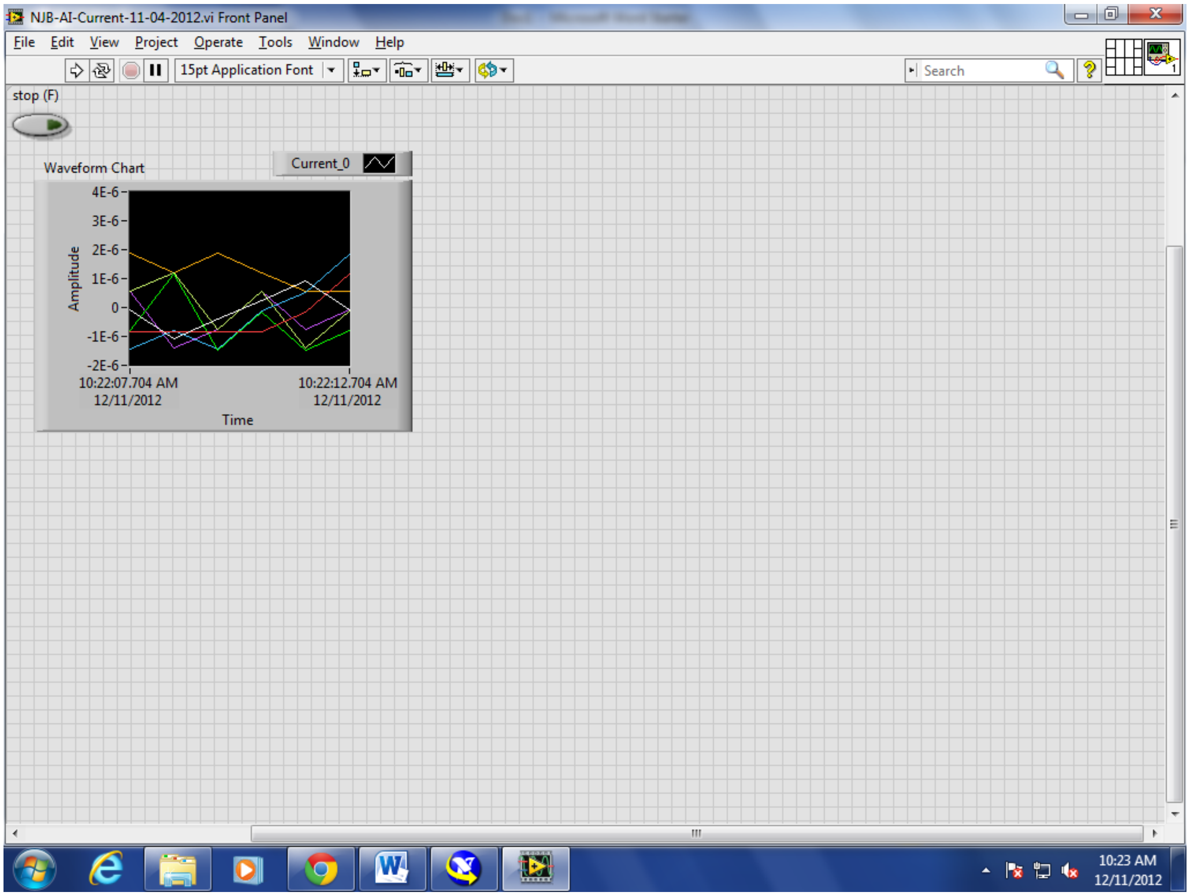
Operating Instructions: Temperature Measurements in North Junction Box Using LabView

1. A specific program for this purpose to read and display data from north junction box temperature connections has been made. Look under: Libraries → Documents → ASU-NJB-Test → NJB-Temperature-Test-11-04-2012
2. Once the front panel is open, you will see an arrow on the top left side of icon ribbon (top portion of front panel). Click to run program.
3. Measurements for each port will be displayed on the graph.
4. In order to stop recording, press “stop(F)” button, on left hand corner of front panel or stop sign (red hexagon) on icon ribbon.



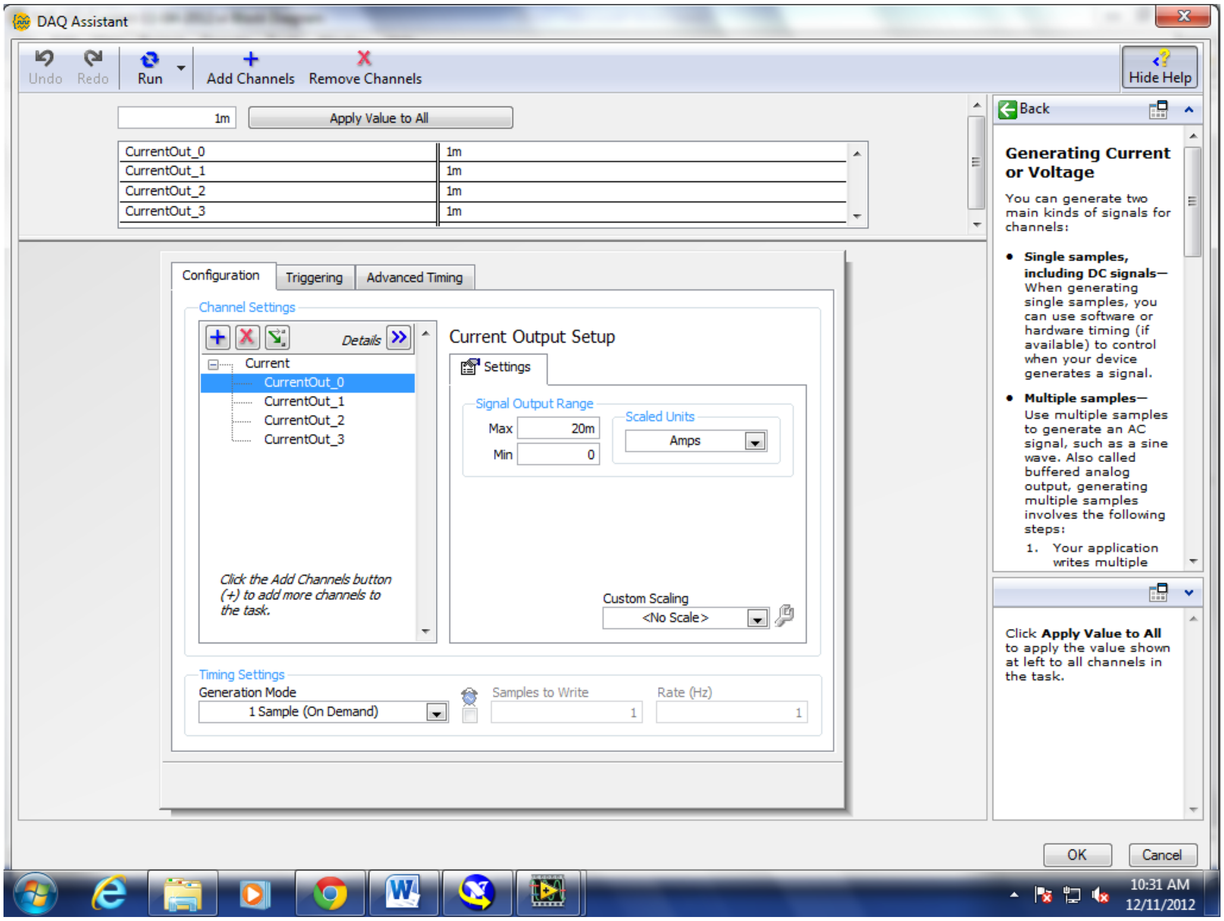
Operating Instructions Analog Input/Current Measurements in North Junction Box Using LabView

1. A specific program for this purpose to read and display data from north junction box temperature connections has been made. Look under: Libraries → Documents → ASU-NJB-Test → NJB-AI-Current-11-04-2012
2. Once the front panel is open, you will see an arrow on the top left side of icon ribbon (top portion of front panel). Click to run program.
3. Measurements for each port will be displayed on the graph.
4. In order to stop recording, press “stop(F)” button, on left hand corner of front panel or stop sign (red hexagon) on icon ribbon.



Operating Instructions: Analog Output to North Junction Box Using LabView

1. A specific program for this purpose to read and display data from north junction box temperature connections has been made. Look under: Libraries → Documents → ASU-NJB-Test → NJB-AO-Current-11-04-2012
2. Once the front panel is open, you will see an arrow on the top left side of icon ribbon (top portion of front panel). Click to run program.
3. The program has been set-up to send a 1.1mA signal to all four ports in NI 9265 module. This value can be changed from the block diagram using the following steps:
    a. On the front panel choose: Window→ Show Block Diagram
    b. Double click on DAQ Assistant
    c. In the dialog box, there will be space to type/enter values next to button "Apply Value to All": Enter any value from 0 to 0.02 A, and apply to all.



4. In order to stop recording, press "stop(F)" button, on left hand corner of front panel or stop sign (red hexagon) on icon ribbon.
5. In order to test communication, use the Omegaette Multi-meter. For mA measurements. Instructions from Omega HHM90 series instruction manual.

Appendix B10

User Interface

At any given point of time when in operation three screens are operational - Main with process control panel, process monitoring panel, and camera input from process. Please see Figure B10.1.

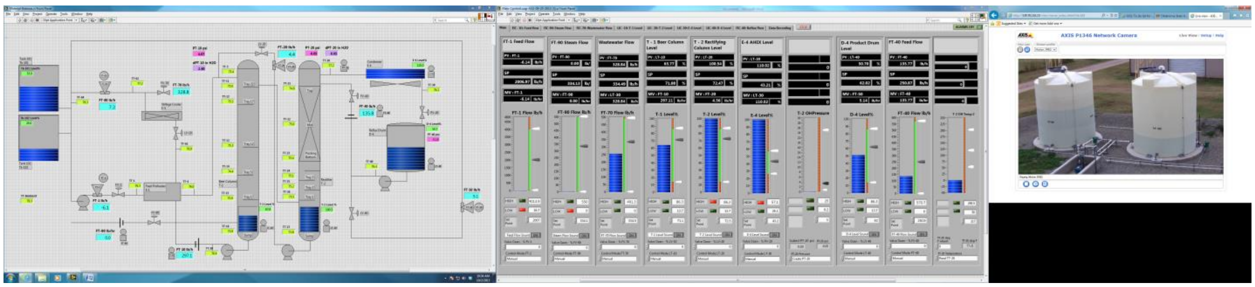


Figure B10.1: Operational screens at ASU control room

Process Monitoring

Labview 2011 program used to create control panel for ASU process. Front panel consists of displays for each control loop (Figure B10.2). These include:

1. FIC-01 - Feed flow
2. FIC-90-Steam flow
3. FIC-70-Wastewater flow
4. LIC-10-Beer column T-1 Level
5. LIC-20- Rectifier T-2 Level
6. LIC-30-Air cooler E-4 level
7. PIC-20-Rectifier T-2 Pressure
8. LIC-40 - Reflux drum D-4 level
9. FIC-40 - Reflux flow
10. TIC-40 - Rectifier T-2 product temperature

A slightly more realistic representation for process variable monitoring was adopted (Figure B10.3). All process variables with measurement locations can be found on the process diagram. Levels measurements are translated into a scale which can be easily read off from vessel/equipment. All measurements are color coded - flows are light blue blocks, pressures are pink, temperatures are yellow, and differential pressures are purple.

Temperatures, flows, pressures etc. are also recorded on a strip chart. Any process upsets or process trends can be followed (Figure B10.3).

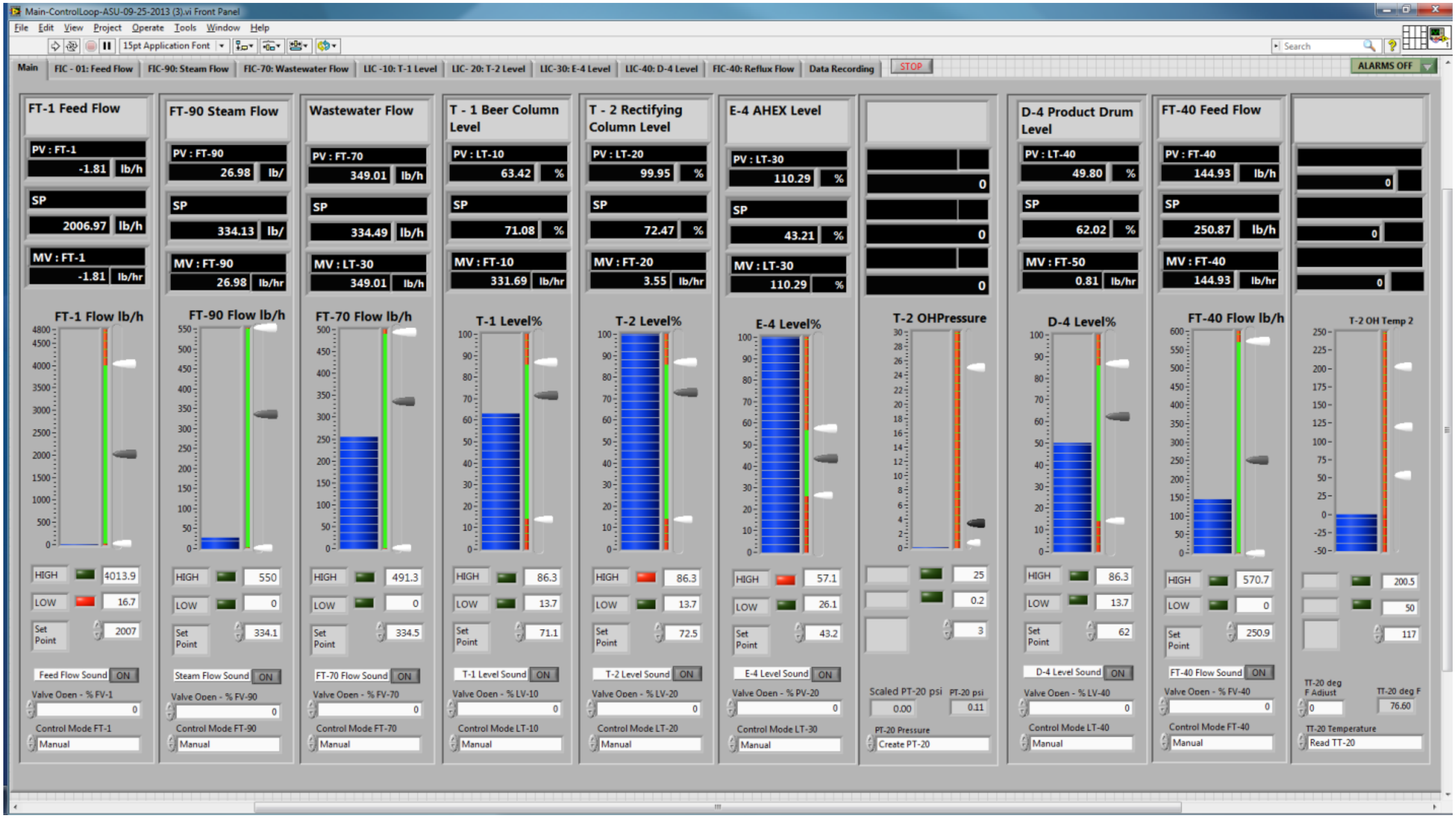


Figure B10.2: Front panel tiles for ASU process control - Manual to Automatic, process variable and set points, high and low level alarms.

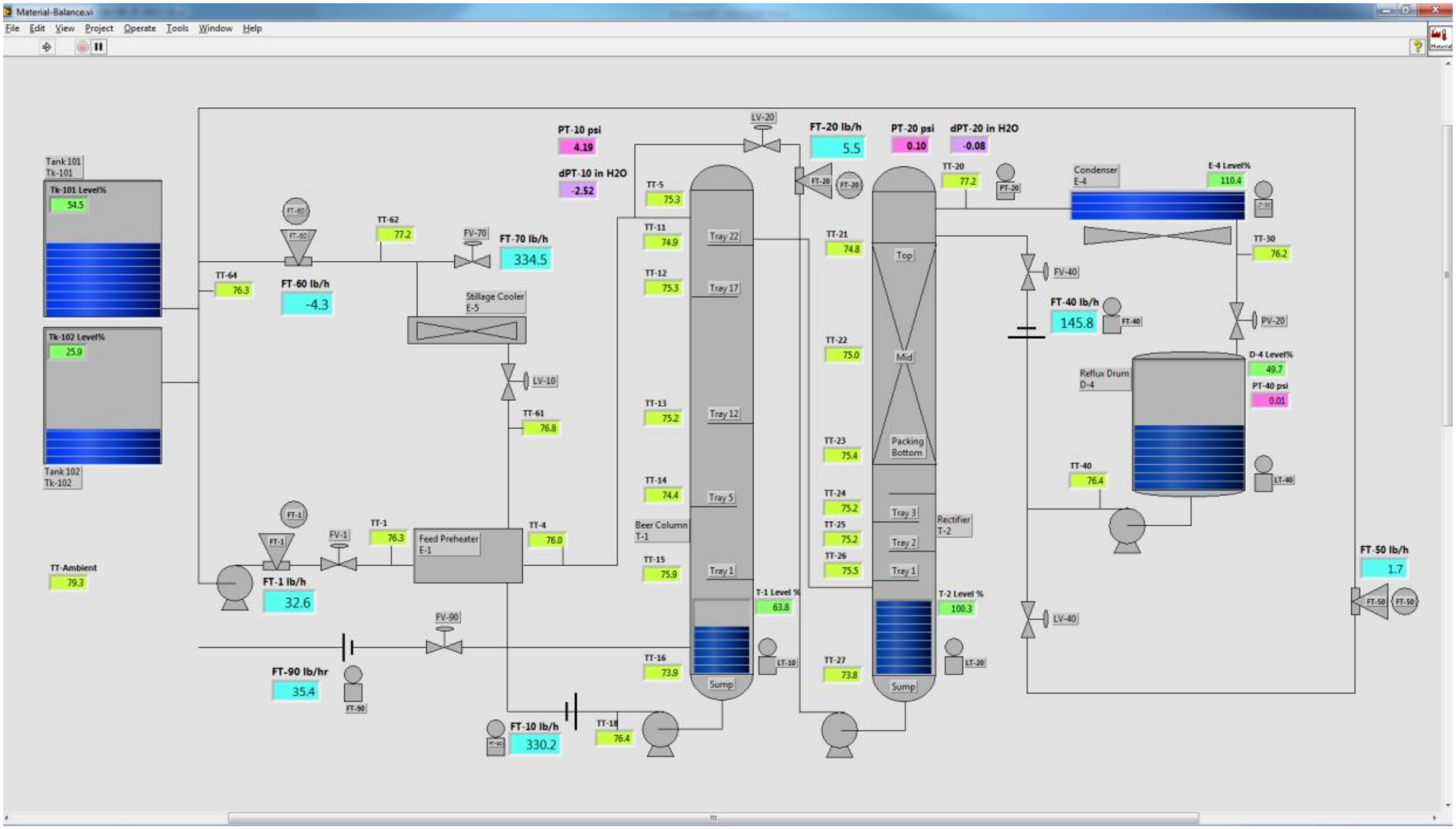


Figure B10.2: Process monitoring windows: a) Flow and level visualization

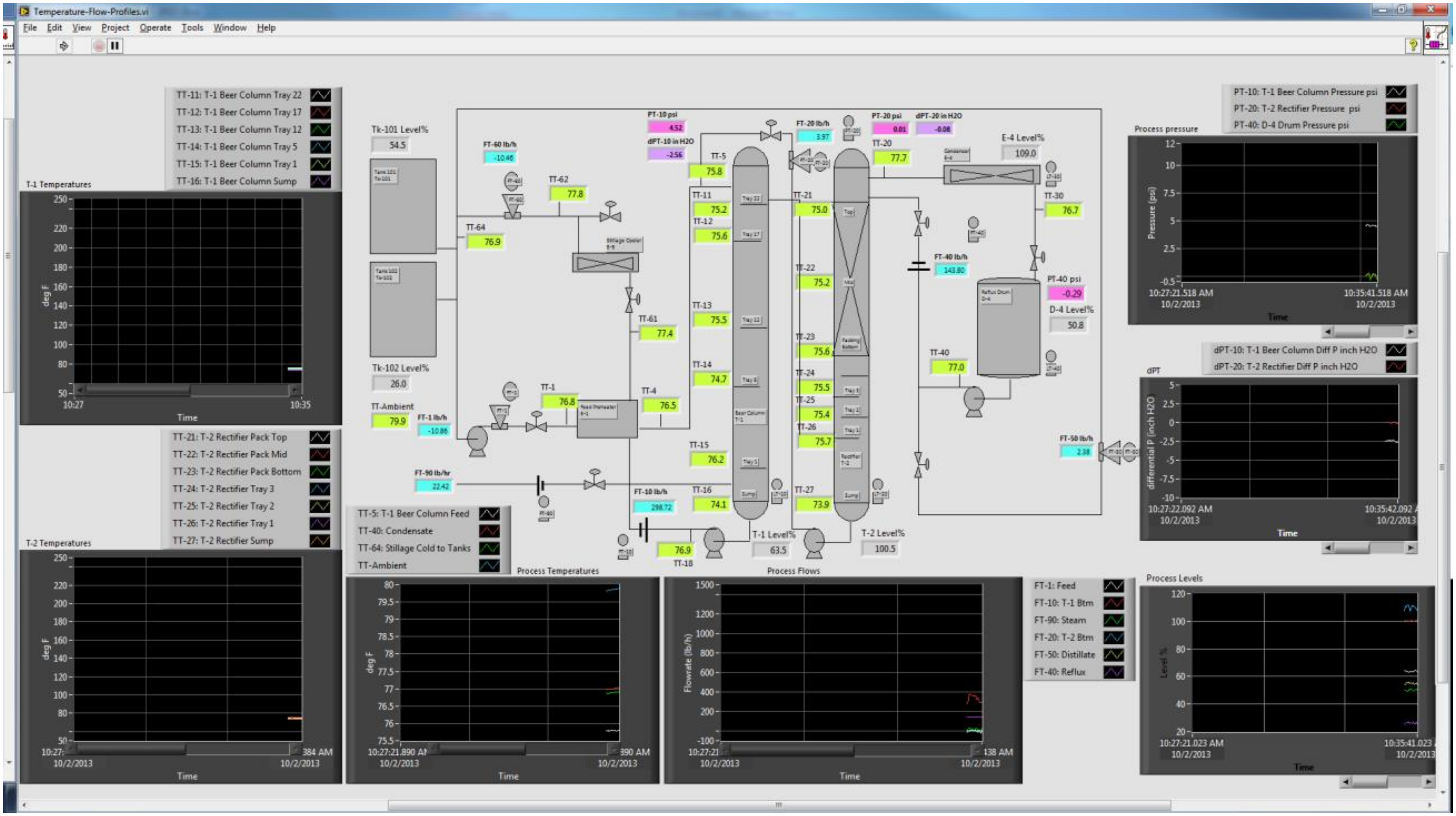


Figure B10.3: Process monitoring windows b) Process variable strip charts

Appendix B11

Instrument Calibration Table

				Low Range	High Range	Units	Equation Constants m	c
Flow	FT-1	Feed flow	Amp to lb/h	0	4800	lb/h	300000	-1200
	FT-90	Steam	Amp to deltap (inch H_2O)	0	150	inch H_2O	9375	-37.5
			deltap to lb/h	(deltap^0.5)*47.093				
	FT-10	Stillage	Amp to deltap (inch H_2O)	0	150	inch H_2O	9375	-37.5
			deltap to lb/h	(deltap^0.5)*289.77				
	FT-50	Distillate	Amp to lb/h	0	250	lb/h	15625	-62.5
	FT-40	Reflux	Amp to lb/h	0	250	lb/h	15625	-62.5
	FT-60	Stillage	Amp to lb/h	0	4800	lb/h	300000	-1200
	FT-20	T-2 bottoms stream	Amp to lb/h	0	600	lb/h	37500	-150
Level	LT-10	T-1 beer column level	Amp to % level	0	60	% level	-18349	195.78
	LT-20	T-2 Rectifier level	Amp to % level	0	100	% level	-10405	147.53
	LT-40	Reflux drum level	Amp to % level	0	100	% level	-21786.49	226.58
	LT-30	Condenser level	Amp to % level	26	54	% level	-9815.5	157.44
	Tk-101	Tk-101 level	Amp to height (ft)	0.5	12.5	ft	750	-2.5
	Tk-102	Tk-102 level	Amp to height (ft)	0.5	12.5	ft	750	-2.5
Pressure	PT-10	T-1 beer column top pressure	Amp to psig	0	30	psig	1875	-7.5
	PT-20	T-2 rectifier top pressure	Amp to psig	0	30	psig	1875	-7.5
	dPT-10	T-1 differential pressure	Amp to inch H_2O	0	30	inch H2O	1875	-7.5
	dPT-20	T-2 differential pressure	Amp to inch H_2O	0	30	inch H2O	1875	-7.5
	PT-40	D-4 reflux drum pressure	Amp to psig	0	50	psig	3125	-12.5
	PT-90		Amp to psig	0	200	psig	12500	-50

Appendix B12

Tuning Parameters

Service	***Valve***	K_c	τ_i
Feed Flow	FV-1	0.25	0.05
Steam Flow	FV-90	2	0.1
Waste	FV-70	0.3	0.3
T-1 Level	LV-10	-3	2.5
T-2 Level	LV-20	-6	4
E-4 Level	PV-20	0.4	3
Direct T-2 pressure	PV-20	-1	4
Cascade T-2 pressure	PV-20	4	2
D-4 Level	LV-40	-8	2
Reflux Flow	FV-40	0.25	0.05
Cascade T-2 Temperature	FV-40	-0.4	2

Appendix B13

Start Up and Shut Down Procedure for Pilot Plant

Instrumentation readiness steps:

1. Connect instrument power and Ethernet cable connection on cDAQ9188 in north and south junction box. Check for any loose wiring connections. When all connections are complete – close junction box and check for tight closed seal.
2. Turn computer on and follow instructions in Appendix B9 MAX Operating instructions, Section 1 to set up the hardware.
3. Open LabView 2011 and click on ASU-Operation-Reflux-Control-Main program to bring up the Main Process front panel (Figure B9.1). From the Sub-VI folder find Material Balance (Figure B9.2) and Temperature Pressure Profile (Figure B9.3) VIs.
4. On internet explorer set up the security camera tabs for both the process and storage area view

Connections to the Pilot Plant:

1. Hose connection to the sparger assembly is made. The other end of the hose is connected on to D-4 top.
2. Sparger assembly is immersed in 50 gallon plastic tub.

Utility Readiness:

1. Switch ON the boiler and boiler pump. Make sure the feedwater line valve is open. Make up water tank should have water.
2. Check the pressure readout. MAIN pressure setting should be 140 psig. The DIFF setting should be set to 5 psi. LIMIT is set to 145 psig.
3. Perform boiler blowdown by opening the blowdown valve fully for 10-12 seconds. a The McDonnell Miller low water control and the auxiliary low water cut-off water column should also be drained.
4. Valve at boiler outlet should be opened slowly to let pressurized steam into steam line.
5. Turn the air compressor ON. The set pressure on the compressor output should be 120 psig.

Instrument readiness and calibration check:

1. Air line valve is opened slowly. Air line blowdown valve (on north and south island) are opened for 10 to 12 seconds. Blowdown valves on air tubing on the air tube bank are opened for 10-12 seconds to let any condensate out.
2. Individual air connections to each control valve on North and South air islands are opened. The upstream supply pressure is 120 psig. The downstream pressure on the air regulator should be 30 psig.

3. All control valves are checked for proper function. Valve movement is assessed with open and closing each valve. The %open control on front panel was changed from 0% (closed) to 100% (fully open) over a few cycles.
4. All transmitters should read 4 mA (+/- 0.2 mA) to indicate zero readings. If transmitters are off by a large margin they might need recalibration or replacement.
5. Blowdown valve on steam line should be opened for 10 to 12 seconds to let any condensed liquids out. Close the valve when pressurized steam comes out from the valve.

Steam Inlet

1. Before letting the steam in, check to make sure all vents in the process (T-2 OH vent, D-4 vent) are open.
2. Steam control valve FV-90 open position should be approximately 25%. Open the globe valve slowly to let pressurized steam into the beer column. Steam flowrate set point was 250 lb/h. Temperatures along the tower should start rising in quick succession.
3. The steam makes its way to the rectifier and overhead through the condenser into the condensate receiver. The vents can be closed at this point.
4. The steam condenses and a liquid level should begin to form in the beer column, the rectifier, the condenser E-1, and the condensate receiver. As levels appear LT-10, LT-20, LT-30 and LT-40 are put on level control with appropriate set points (usually between 20 to 30%)

Feed Inlet:

1. Valves are lined up on the storage tank lines for recirculation. P-101 is turned on and the discharge pressure is adjusted by throttling the recycle valve. Discharge pressure was 55 psi. The content in Tk-101 or Tk-102 (whichever was used as feed) was recirculated for 15 minute prior to diverting to the process area.
2. Stillage recombination line valves are checked – they should be closed. The feed is introduced into the beer column in increments starting from 500 lb/h and increasing to 1700 lb/h.

Reflux and Distillate:

1. Once a level is steadily maintained in D-4, a reflux into T-2 is diverted at approximately 100 lb/h (the flowrate should be small enough to ensure a steady level in D-4. With a high flowrate the condensate receiver runs the risk of emptying out).
2. With a steady level in T-2 bottoms from T-2 is directed to T-1 as reflux. A flowrate of 200 lb/h.
3. Column temperatures drop as ethanol concentrates in the columns.
4. Once the column refluxes are set in place, product samples should be analyzed for

ethanol content. The sample point upstream of the condensate receiver was used for product sampling.

Shut Down:

1. Scale back on the feed in decrements till a zero feed flowrate. Turn off the feed pump.
2. Reduce the steam flowrate in stages to zero. Before the steam flowrate is completely turned off the vents on the condensate receiver and overhead lines are opened to avoid vacuum build up inside the equipment.
3. Valves are lined up to operate in recombination mode. All liquid is pumped out of the columns and condensate receiver and sent back to the storage area.

VITA

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