DETERMINING THE OPERATION LIMITS OF TWO DISTILLATION COLUMNS

By

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ABSTRACT

The two distillation columns of Wacker Institute pilot plant at the Chattanooga State Community College are investigated for stable operating regions, flooding phenomena that occurs in the bubble-cap tray distillation column, weeping phenomena that occurs in the sieve tray distillation column, and comparison of operation between the two distillation columns. With the use of a distributed control system (DCS) and glass equipment in the pilot plant, these phenomena are analyzed visually and with the help of instrumentation readings. The energy usage and production limits of both distillation columns are discussed. The flooding of bubble-cap trays occur before reaching production goals due to a flaw inside the column. The weeping of sieve trays does not allow the distillation column to operate efficiently at low flow rates. The bubble-cap tray distillation column uses less energy to achieve the same production goals as the sieve tray distillation column.

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Special thanks to WACKER Chemie AG for providing the Wacker Institute training facility for future chemical operators, allowing me to train Wacker's chemical operators in distillation, and giving me the opportunity to do research and analysis of distillation columns at the Wacker Institute pilot plant.

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CHAPTER I

INTRODUCTION

1.1 Wacker Institute Pilot Plant

The Wacker Institute Pilot Plant located at the Chattanooga State Community College (CSCC) is a state of the art training facility for chemical operators and students. The pilot plant, built by De Dietrich Process Systems, includes distillation columns and utilities used for the separation of ethanol and water. The training process in the pilot plant consists of being able to read process and instrumentation diagrams (P&IDs), follow standard operating procedures, understand the distributed control system (DCS), operate the DCS software, start-up, continuous operation, troubleshooting, and shut-down of the pilot plant. The start-up includes checking all utilities, equipment, instrumentation, and control valves for safe operation. The continuous operation is where the chemical operators and students are trained on being able to run a distillation process while meeting production goals using the least amount of energy possible. Once the operators are comfortable with the system's operation, troubleshooting scenarios are introduced to show them what to do when things go wrong, how to fix the problem, and how to bring the pilot plant back to the same conditions before the problem occurred. Shut-down of the pilot plant consists of shutting down the two distillation columns which begins with stopping feed and steam supply to the columns, shutting down all the pumps in tank farm area, decreasing coolant flow, shutting down the boiler, and closing all manual valves necessary to bring the pilot plant to the same conditions found at start-up.

Through several months of operating the pilot plant, many flaws in the process has been found. One of the main flaws has been excessive entrainment and flooding of the trays of one of the distillation columns (bubble-cap tray distillation column) before production goals can be reached. Analyzing this flooding phenomenon and why it happens below its design specifications are one of the purposes for this thesis. The weeping of a few trays on the other distillation column (sieve tray distillation column) has been noticed; analyzing this phenomenon, finding its root cause, and the effect it has on the process is another purpose of this research.

Due to having new chemical operators and students every semester, it is important to know the capabilities of the two distillation columns and the parameters at which they can operate efficiently. These parameters would also be very helpful to instructors who are unfamiliar with the distillation process at the pilot plant. This research is intended to determine the most efficient way to operate the distillation columns and find the parameters that allow for this to happen. Also, the work is to compare and contrast the flooding and entrainment behavior with literature information. The literature is very sketchy about the specifics of these phenomena. The work is to carefully document these phenomena in the two distillation columns.

1.2 Continuous Binary Distillation

Binary distillation is the separation of two liquids based on the difference in boiling points of the two liquids. This is done using a distillation column which consists of a reboiler at the bottom of the column used as the heat source to boil the liquid mixture, equally spaced trays used to bring the liquid and vapor phases in contact, and condenser at the top to bring the vapor rising from the last tray back to liquid phase for collection in a tank. From this tank, a portion of the liquid is sent back to the top tray of the distillation column where liquid and vapor contact on each tray is established. This liquid is also called reflux which is used for purification of the low

boiler, or more volatile compound. The other portion of the liquid in the tank is collected as the low boiler product, also called the distillate product. The ratio of the amount of reflux sent back to the top tray to the amount of distillate product collected is called the reflux ratio. The higher the reflux ratio is, the higher the purity of the low boiler, but also the higher the energy cost is. The high boiler, or less volatile compound, left at the bottom of the column can also be collected and is called bottoms product.

Continuous binary distillation is where a mixture in the distillation column is continuously fed with a fresh new mixture to make up for the amount of distillate and bottoms products taken out of the distillation column. This fresh mixture is called the feed, and the tray on which it enters the distillation column is called the feed tray. The feed is pre-heated before entering the feed tray so that the energy required to separate the low boiler from the high boiler entering the column is less, which results in increased efficiency of the separation process in the distillation column. The section of the distillation column below the feed tray is called the stripping section and the section above the feed tray is called the rectifying section. Continuous distillation can be very expensive, contributing to more than 50% of plant operating costs (Cheremisinoff, 2000), due to constant heat supply to the reboiler for boiling the mixture at the high boiler's boiling point and a constant source of coolant supply to the condenser for continuous vapor to liquid phase change. Due to this high cost, distillation columns must be operated as efficiently as possible, meaning using the least amount of energy possible to achieve production goals.

1.3 Background on Bubble-cap and Sieve Trays

The tray hydraulics of bubble-cap and sieve tray distillation columns has been extensively studied by Smith (1963).

Smith's (1963) study on tray hydraulics of bubble-cap trays states:

> The bubble-cap tray is the best known vapor-liquid contacting device. Through the years it has been a standard for the chemical and petroleum industry, and a majority of the existing commercial vapor-liquid contacting devices contain bubble-cap trays. Because of the widespread acceptance of bubble-cap trays and a wealth of operating experience developed on them through the years, designers have in the past been wary of specifying alternate contacting devices having relatively unknown hydraulic characteristics. One unique advantage of bubble-cap trays is the fixed-seal arrangement enabling them to be operated over a wide range of conditions while maintaining constant efficiency. (p.474)

There are other types of vapor-liquid contacting devices such as perforated trays. The most common perforated tray is a sieve tray. The advantage of using sieve trays is the simplicity in their design and a lower cost (Smith, 1963). According to Smith (1963):

> There are two important differences in the way vapor flows up the trays between sieve and bubble-cap trays:
> 1. For the sieve tray, vapor emerges from a large number of small openings (perforations) primarily in a vertical direction.
> 2. For the sieve tray, there is no built-in liquid seal and only vapor flow can prevent liquid passage through the holes. (p. 543)

1.3.1 Flooding

Flooding in a distillation column is a phenomenon where the rate of liquid coming into a tray from a tray above is higher than the rate of liquid leaving that tray through the downcomer, which causes the tray to fill up (become flooded) and the liquid begins to get sent at the tray above.

Smith's (1963) research has found that:

> Flooding on trays may be brought on by either excessive entrainment, where the rising vapor stream carries liquid to the tray above or liquid backup in the downcomer. The true point of flooding is difficult to determine experimentally,

> and "maximum capacity" is usually synonymous with an incipient flooding condition brought on by either of the two phenomena noted above. Regardless of cause, the onset of flooding is detected by a sharp increase in pressure drop and a sharp decline in efficiency.

According to Perry (1997):

> Entrainment in a distillation column is that liquid which is carried with the vapor from one tray to the tray above. It is detrimental in that the effective tray efficiency is lowered because liquid from a tray of lower volatility is carried to a tray of higher volatility, thereby diluting the effect of distillation. Entrainment is also detrimental when nonvolatile impurities are carried upward to contaminate the overhead product from the distillation column. Many experimental studies of entrainment have been made, but few of them have been made under actual distillation conditions. The studies are often questionable because they are limited to the air-water system, and they do not use a realistic method for collecting and measuring the amount of entrainment. (p. 14-28)

There is not much research done on flooding phenomena in tray columns in the recent years since distributed control systems came out. One of the most recent studies is done by Emerson Process Management with the use of their Rosemount 3051S series differential pressure transmitter (Emerson Process Management, 2008). The use of a transmitter to measure the differential pressure across the distillation column can help with detecting when flooding starts. The research predicts flooding only by sharp increase in differential pressure, and the study was done using a packed column (Emerson Process Management, 2008). Most other studies are also done using packed columns since the probability of flooding to occur is much greater due to the packing in between the trays. There are other studies done where mathematical models are developed to predict or estimate flooding capacity in a column using superficial flooding velocities of the vapor and liquid (Piche, Larachi, and Grandjean, 2001). Pop, Dulf, and Festila (2008) studied flooding in a cryogenic separation column and proposes predicting flooding using differential pressure and liquid level in the reboiler data (Pop, Dulf, and Festila, 2008). Still the main purpose in this study was to develop mathematical equations to estimate the flooding point

using data collected from a test column. Other recent studies focus mainly on the pressure drop measurements on distillation columns (Cai, Shariat, and Resetarits, 2009) and computational fluid dynamics on the column trays (Chunjiang and Xigang, 2002). Most of the studies done on flooding only apply to the small experimental columns, packed columns, and estimation using modeling on a computer. There needs to be a better study done in order to observe flooding visually with the use of a glass distillation column and a distributed control system software to analyze exactly what happens to differential pressure and other variables in the tray distillation column.

1.3.2 Weeping

Smith's (1963) research on weeping of sieve trays has found:

> Just as entrainment represents an upper limit to tray operation, excessive flow of liquid through the perforations of a sieve tray represents a lower limit. Liquid passage through the tray may occur to some extent at all vapor rates, but as the rate is reduced, the passage becomes pronounced at the "weep point." Weeping may be fairly uniform across the tray, or it may be localized near the point of liquid entry to the tray. It is important to note that even though some tray bypassing results from weeping, some mass transfer occurs in the vapor zone. The influence of weeping on tray efficiency depends on the fraction of total liquid downflow that weeps; thus, for cases of low liquid flow a small amount of weeping can be relatively serious.
>
> As vapor rate is reduced below the weep point, serious liquid drainage begins at the "dump point." Dumping is easily observed visually and is characterized by a definite drop in tray efficiency. Below the dump point operation may be unstable and efficiency so low that effective separation is difficult, if not impossible. (p. 547-548).

1.3.3 Stable Operating Region

It is important to note the qualitative effect of liquid and vapor loads on bubble-cap tray performance as limited by tray dynamics (Smith, 1963). A performance chart has been developed by William L. Bolles (1963) to illustrate the limit of each dynamic factor for a typical bubble-cap tray (Smith, 1963), and is shown in figure 1.1:

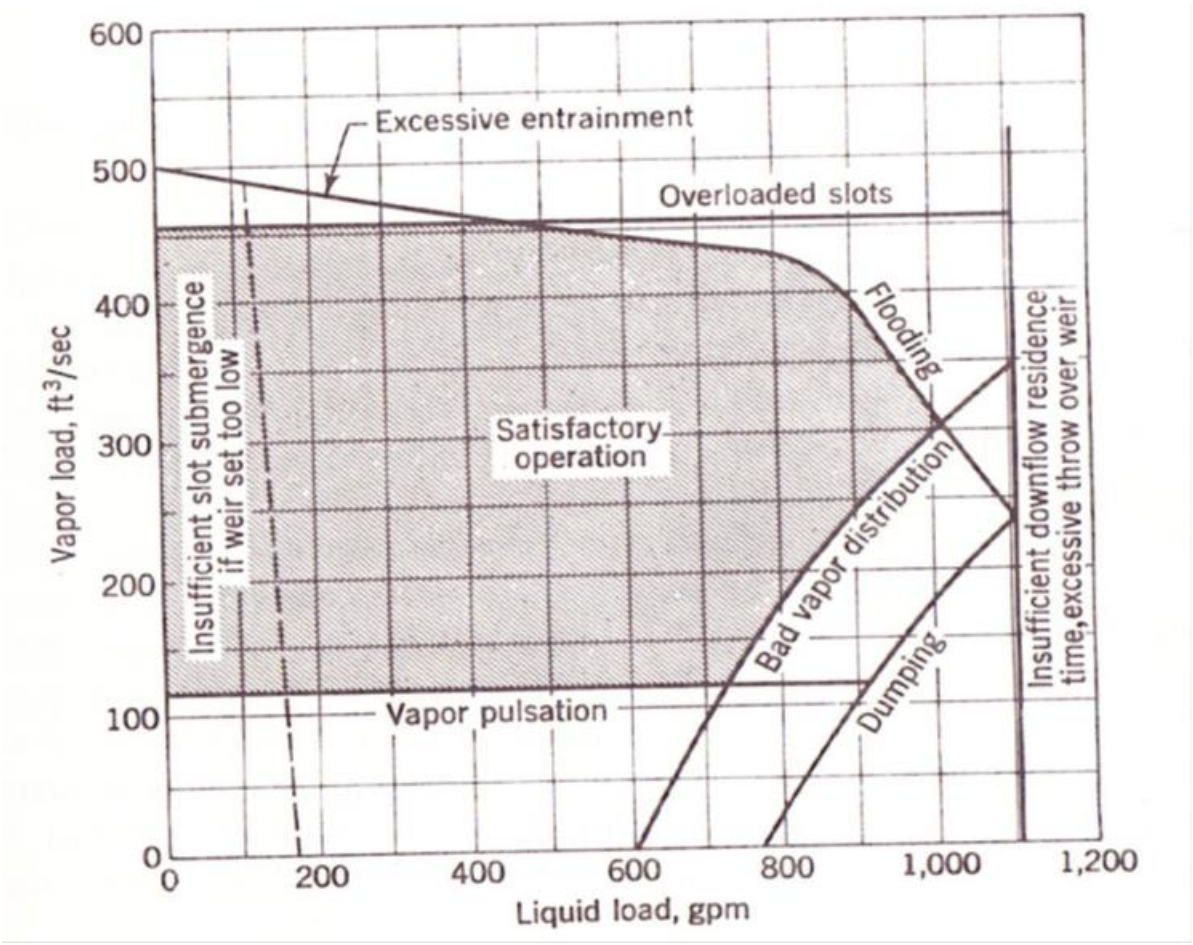

Figure 1.1

Typical Bubble-cap Tray Performance Chart (Smith, 1963)

The area of satisfactory operation is shown surrounded by excessive entrainment, overloaded slots, flooding, insufficient downflow residence time, excessive throw over weir, bad vapor distribution, dumping, and vapor pulsation (Smith, 1963). This satisfactory region is developed mainly for design purposes and can also be called stable operating region. The only unsatisfactory region that is of interest in this research is the excessive entrainment/flooding

region, which is the upper limit of satisfactory operation. A similar stable operating region chart was developed for sieve tray distillation columns (Smith 1963), which is shown below:

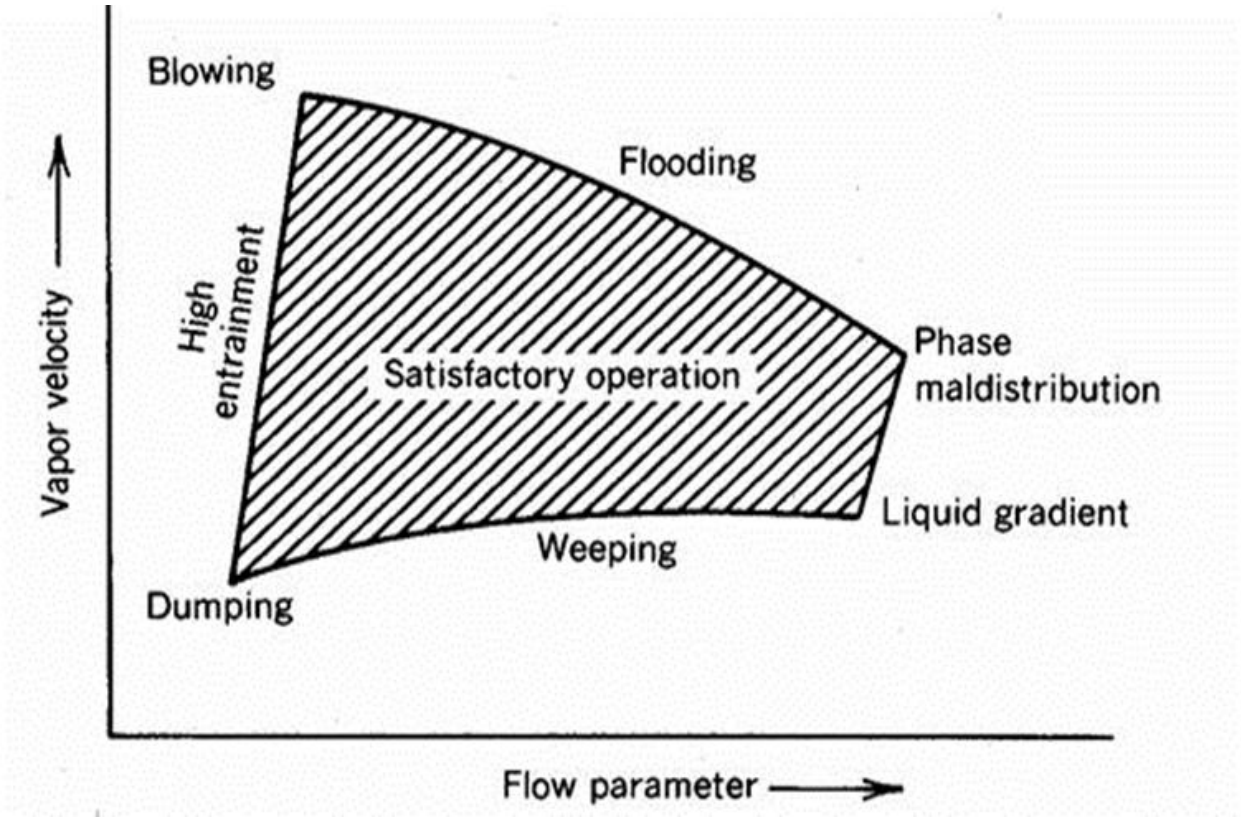

Figure 1.2

Stable Operating Region for Sieve Trays (Smith, 1963)

The vapor velocity term on the y-axis of figure 1.2 refers to the rate of vapor flow from the distillation column into the condenser. The flow parameter on the x-axis refers to the rate of liquid flow into the distillation column which consists of feed and reflux. For the purpose of this research; only the flooding, entrainment, and weeping regions are of interest for the unsatisfactory operation of the sieve tray distillation column.

1.4 Research Objectives

This research thesis is done and applies specifically to the bubble-cap tray and sieve tray distillation columns at the Wacker Institute pilot plant. The research objectives of these distillation columns are:

1. Analyze the flooding phenomenon in bubble-cap tray distillation column and find the region of flooding with respect to liquid and vapor flow rates.
2. Develop a stable operating region chart for the bubble-cap tray distillation column using the results from flooding and satisfactory operation to compare with figure 1.1.
3. Analyze weeping phenomenon in sieve tray distillation column and the effect of it on the distillation process.
4. Develop a stable operating region chart for the sieve tray distillation column and compare with figure 1.2.
5. Find the parameters at which the distillation columns operate most efficiently and compare the differences in operation of the two distillation columns.

CHAPTER II

EQUIPMENT

2.1 Introduction

This chapter will discuss the equipment used to analyze the flooding phenomena, weeping phenomena, and the stable operating region of the two distillation columns. The overall system in the Wacker Institute pilot plant consists of analytical instrumentation, distributed control system (DCS), utilities, tank farm area, bubble-cap tray distillation column, sieve tray distillation column, and control instrumentation. The distillation columns are used to separate an ethanol (EtOH) and water mixture. The bubble-cap distillation column and sieve tray distillation column consist of identical equipment of the same size aside from the type of trays they have. For this reason, only one of the column's equipment will be shown and discussed. The following sections of this chapter will discuss in detail all of the equipment in the overall system mentioned above. One important difference in these distillation columns compared to distillation columns in most plants in the United States is that the first tray (Tray 1) is the lowest tray closest to the reboiler and the last tray (Tray 20) is the highest tray closest to the condenser. This is opposite of standard tray numbering used in the United States where tray 1 is the highest tray closest to the condenser at the top of the column, and tray numbers increase going down the column to the reboiler. The schematic of the overall system is shown in figure 2.1:

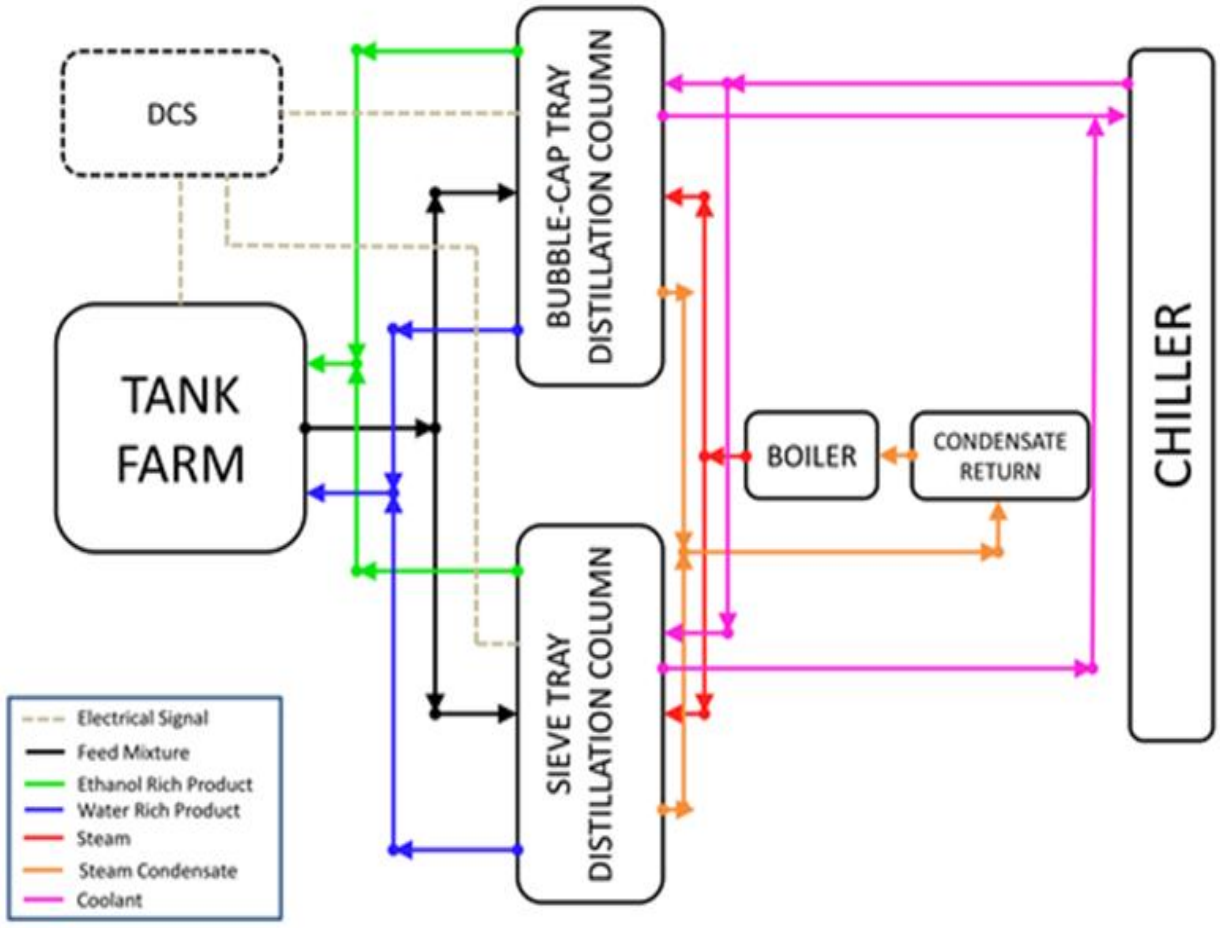

Figure 2.1

Schematic of the Overall System

2.2 Analytical Instrumentation

Density meter and refractometer make up the analytical equipment used to analyze feed, ethanol (EtOH) -rich product, and water-rich product concentrations of EtOH. The density meter is made by Anton Paar model DMA 500 and was used to measure the density and top distillate product concentration of alcohol by weight. After an approximately 2 mL sample is collected from the distillation columns it is first brought to room temperature (20-25 ^{o}C), then a syringe is used to put the sample in the density meter where density and concentration of alcohol by weight

is measured in less than 2 minutes. A picture of the density meter used is shown below in figure 2.2:

Figure 2.2

Density Meter

The refractometer is used for measuring concentration of the bottom water-rich product. It consists of the refractometer itself and a chiller unit which uses an ethylene glycol-water as a coolant to keep the refractometer at a constant temperature for the most accurate measurement. The refractometer gives a refractive index number of the sample being measured, and then a

"refractive index" to "ethanol concentration" graph is used to get the value of ethanol concentration in the mixture. A picture of the density meter used is shown below in figure 2.3:

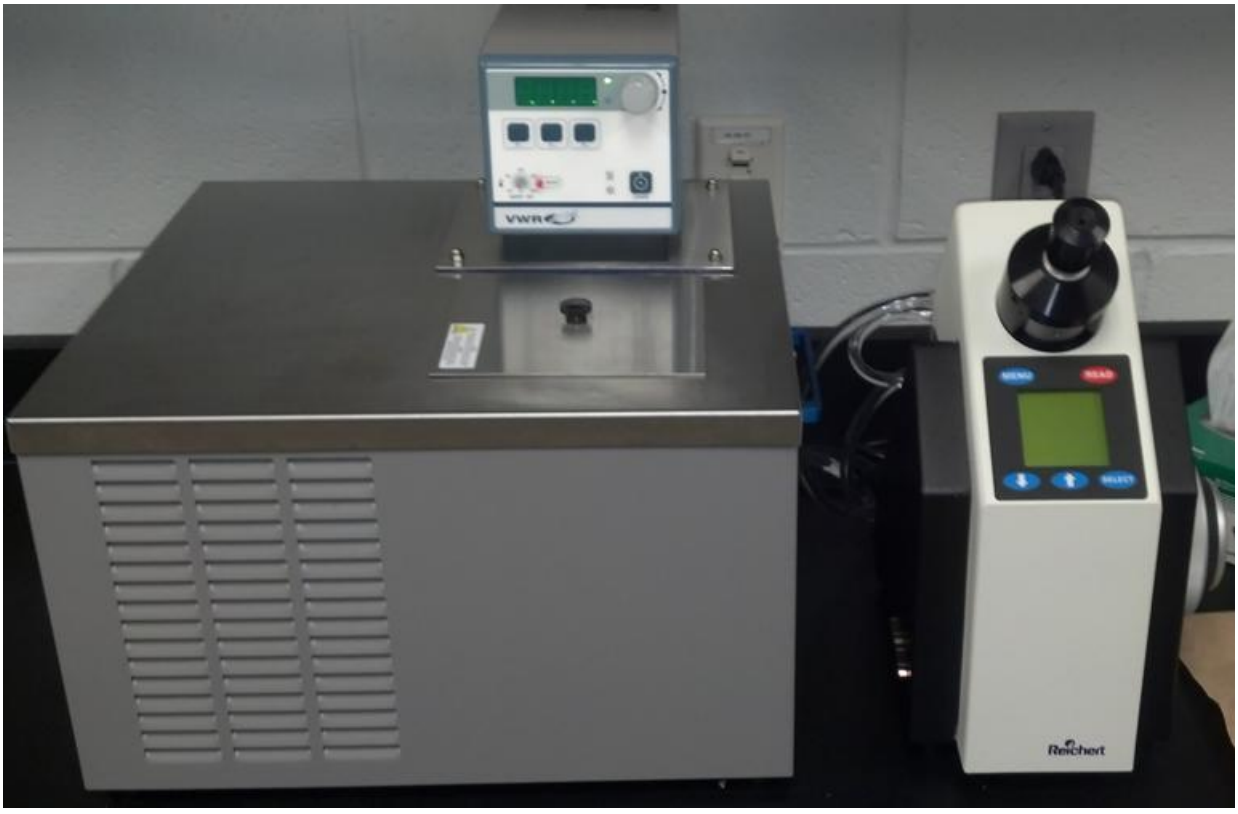

Figure 2.3

Refractometer (right) and Chiller (left)

The chiller unit for the refractometer is the bigger device on the left side of the refractometer shown in figure 2.3.

2.3 Distributed Control System (DCS)

The DCS consists of a main I/O control panel (MIOP), remote I/O panels (RIOP) for the tank farm area (RIOP-T110), bubble-cap tray distillation area (RIOP-T120), sieve tray distillation area (RIOP-T220), Proplus programming computer station, operator control station,

two field terminals for the plant area, variable frequency drives (VFDs) by Siemens for all the pumps and utilities in the pilot plant, and DeltaV DCS software package by Emerson. The DCS was designed and built by De Dietrich Process Systems (DDPS). The control system diagram is shown below in figure 2.4:

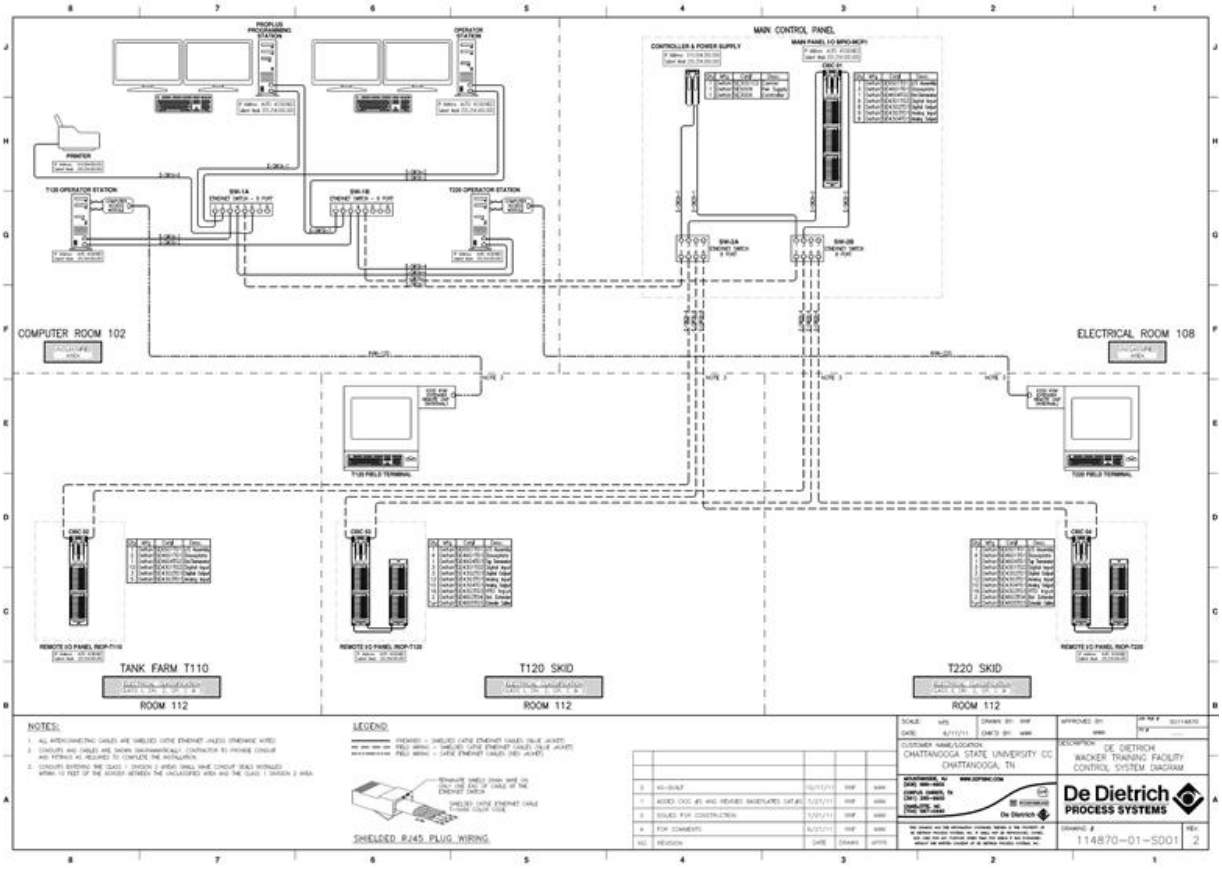

Figure 2.4

Control System Diagram

2.3.1 Input-Output Control Panels

The main I/O control panel (MIOP) components include two power supply switching regulators from a 120/240 VAC input to a 24 VDC output, DeltaV system power supply, DeltaV

SD Plus PID controller, Ethernet, relays, and DeltaV I/O Charms. A labeled picture of the MIOP is shown below in figure 2.5:

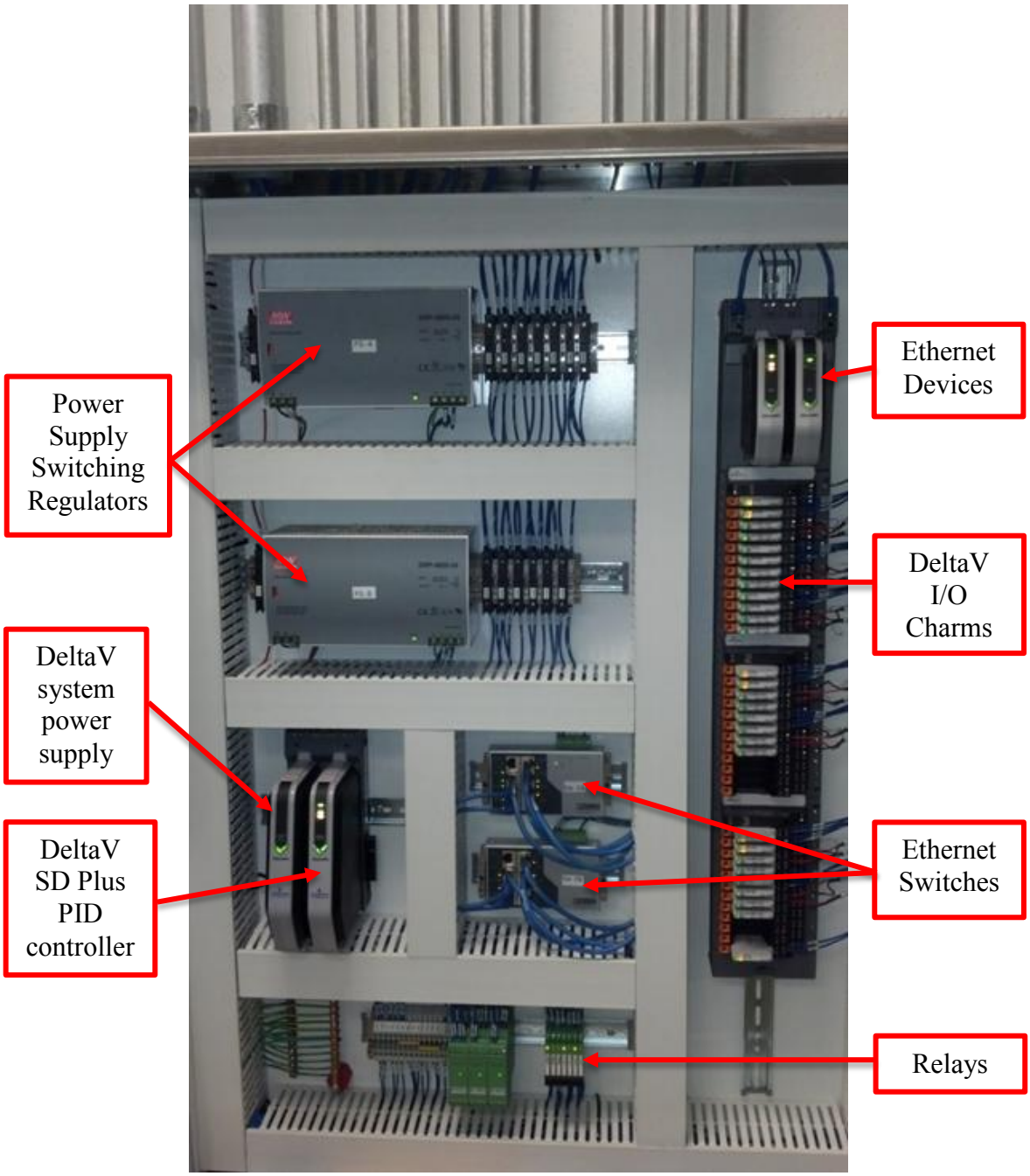

Figure 2.5

Main I/O Control Panel (MIOP)

The remote I/O panels (RIOP) are very similar to the MIOP except they only have the DeltaV I/O Charms, relays and Ethernet devices. A labeled picture of one of the RIOPs is shown below in figure 2.6:

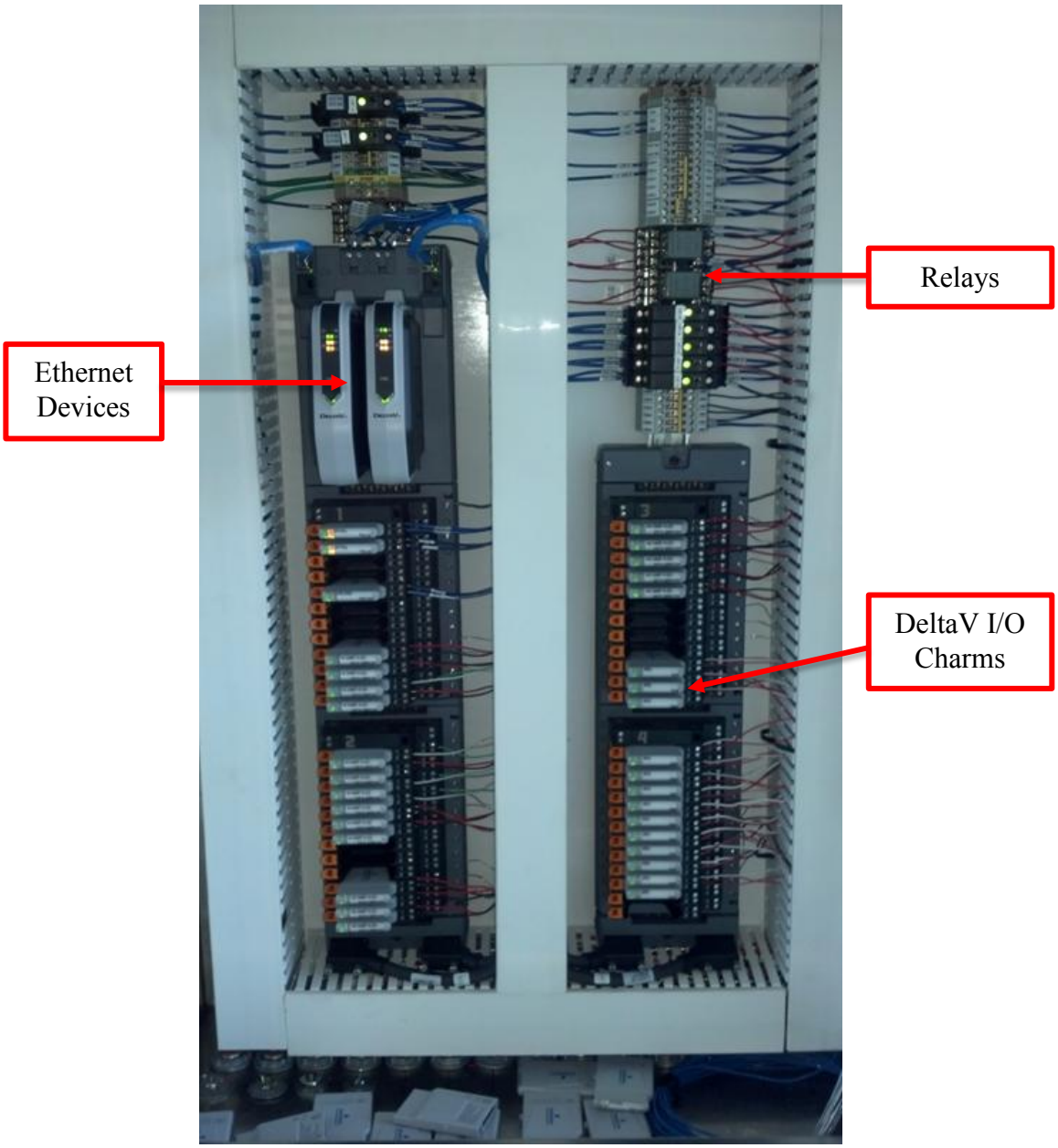

Figure 2.6

Remote I/O Control Panel (MIOP)

2.3.2 DeltaV DCS System Software

The DeltaV DCS system software package by Emerson is the computer software program used to control the distillation process in the pilot plant. The Proplus programming station, operator station, and both of the remote terminals in the plant area all use this software to run the pilot plant. The software package provides means for writing, editing, and maintaining logic code for processing inputs and driving outputs from programmed sequences. It also provides means for developing operator interface screens for control, annunciation, and monitoring of the process (DDPS, 2011).

DeltaV software has four different interface screens where the process can be monitored or the process conditions can be adjusted. The first screen interface is the overview of the overall process and is shown in figure 2.7:

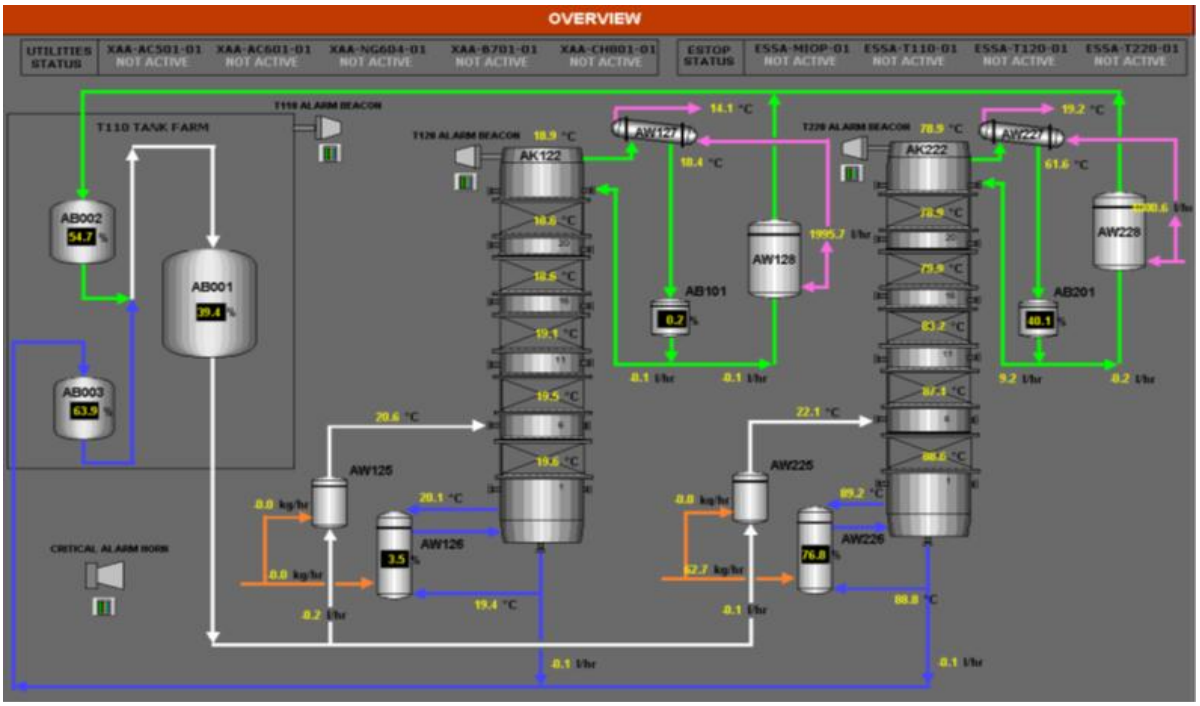

Figure 2.7

Overview Screen Interface of DeltaV DCS system software

Figure 2.7 shows left to right the tank farm area (T110), bubble-cap tray distillation area (T120), and the sieve tray distillation area (T220) mentioned in the introduction section of this chapter. This overview screen is used only to monitor the process variables and cannot be used to make any changes in the process conditions. The process lines are ethanol-rich stream in green, ethanol-water feed mixture stream in white, steam in orange, vent gas in yellow, water-rich stream in blue, and coolant is shown by the pink lines.

The figure below shows the tank farm area (T110) main screen:

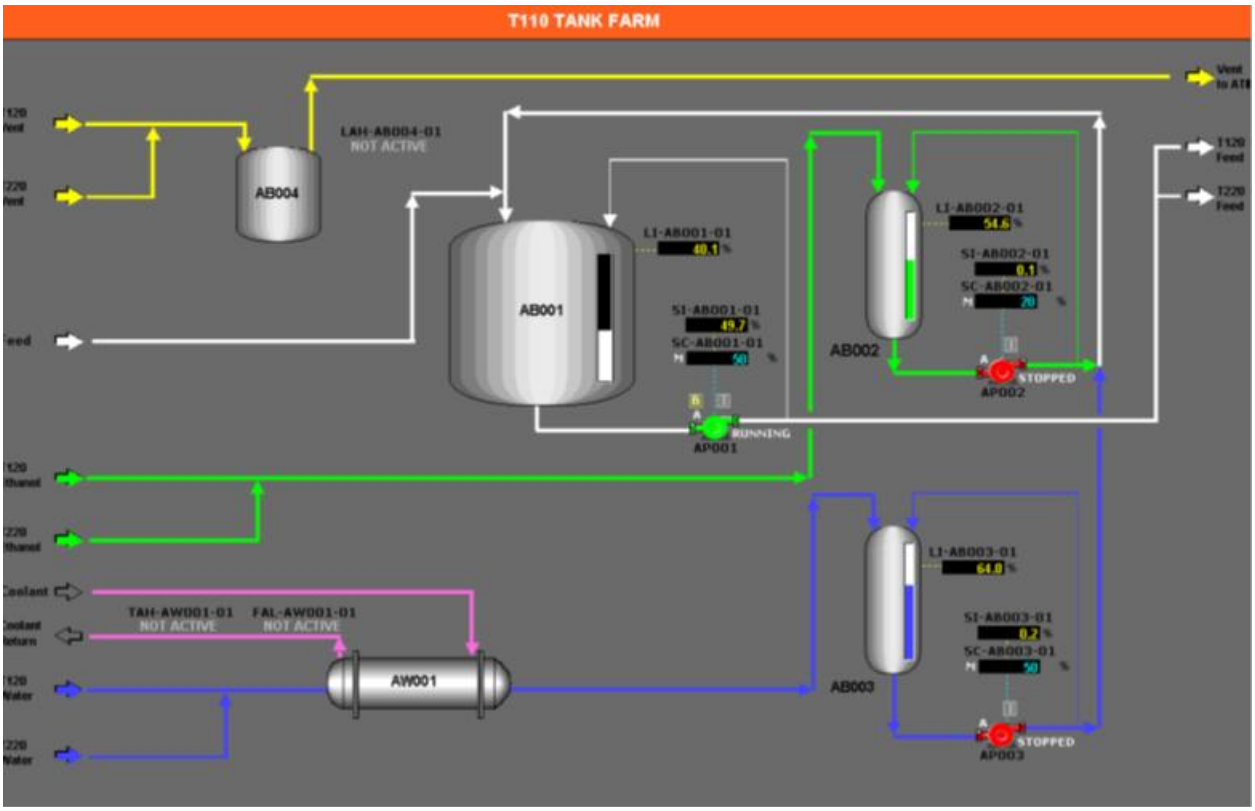

Figure 2.8

T110 Screen Interface of DeltaV DCS system software

Figure 2.8 shows the T110 area and the variables that can be changed from the screen. The yellow numbers represent the process variables as also seen in figure 2.7 since these are just

readings from the transmitters in the system and cannot be changed. The blue numbers are control variables and can be adjusted. The control variables on figure 2.8 are the speed of the pumps in terms of percent power. The green color of the pump indicates that it is running and the red indicates that it is stopped.

The two distillation column screens are identical except for the numbering of the equipment. The bubble-cap tray distillation area has a “1” and sieve tray has a “2” for the first number in the equipment identification. Since the only difference is that and the type of trays, only one of the screens is shown for demonstration, shown in figure 2.9:

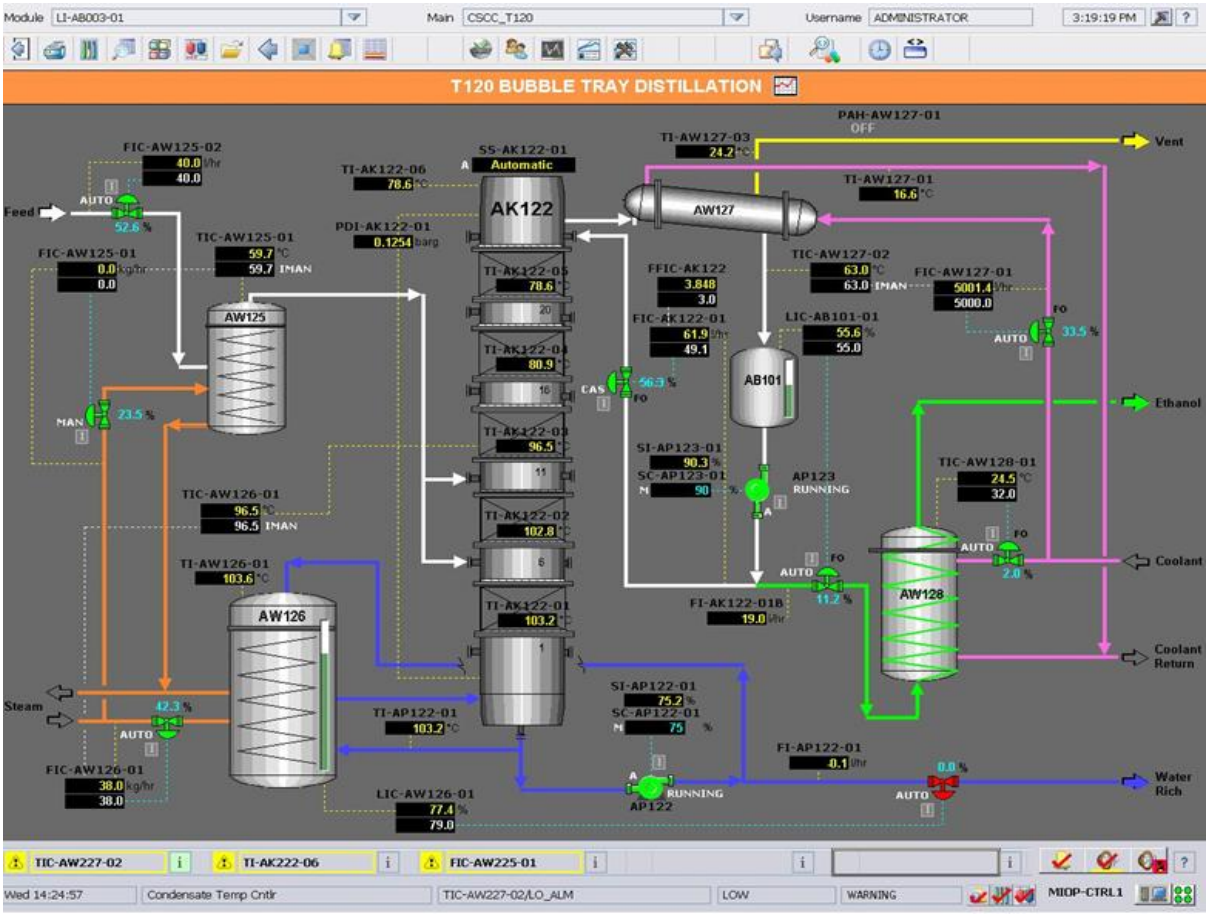

Figure 2.9

T120 Screen Interface of DeltaV DCS system software

The dotted lines on figure 2.9 indicate the control signals. The color coding matches the same ones used for the variables. The white numbers shown on the screen shot are set-points. The blue dotted lines are connected from the valves to the black boxes indicate a control loop. On manual mode (MAN), the percent valve opening can be adjusted with the blue control variable. On automatic mode (AUTO), the control loop activates and the white colored set-point can be adjusted. These include flow rates and levels on the tanks. The white dotted lines going from one black box to another indicate the cascade controls. On cascade mode (CAS), a secondary control takes over and another variable set-point can be controlled. This is the case for the two temperature controls (TIC) and the reflux ratio control (FFIC).

2.4 Utilities

The utilities consist of two air compressors, nitrogen generator, boiler, condensate return system, and the chiller unit. One of the air compressors is only used to feed air into the nitrogen generator, where nitrogen in the air gets separated using molecular sieves. The other air compressor is used for air supply to all the control instrumentation, which are air actuated control valves. A picture of the air compressor is shown in figure 2.10:

Figure 2.10

Air Compressor

The nitrogen generator supplies nitrogen gas to all of the glass tanks and equipment in the pilot plant. Since the process equipment has denatured ethanol in it and is explosive in the presence of oxygen, nitrogen is filled in all the empty space in the tanks for safety purposes. A labeled picture of the nitrogen generator is shown in figure 2.11:

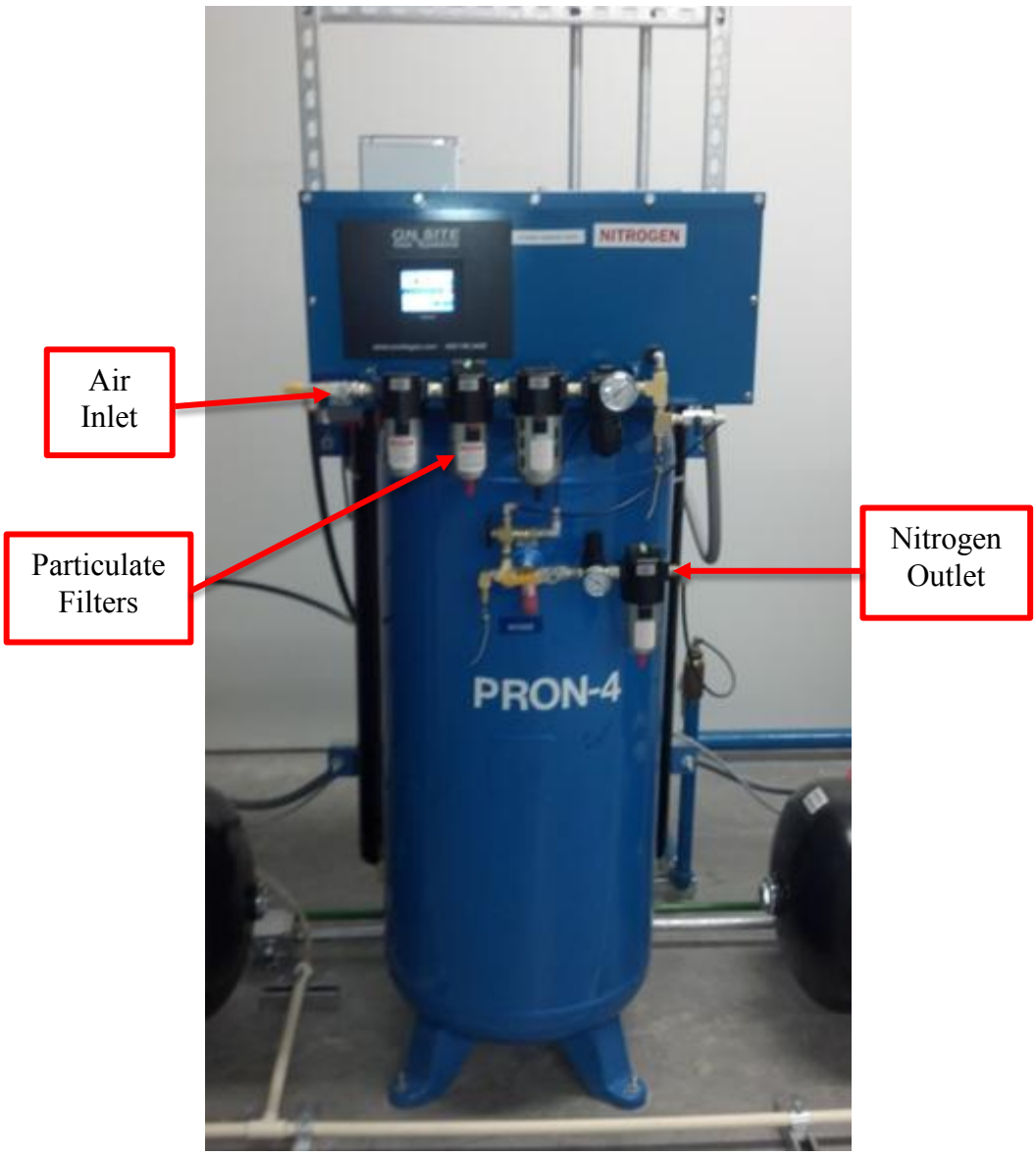

Figure 2.11

Nitrogen Generator

The air enters as shown with the red arrow in figure 2.11 above, goes through three filters to remove particulates in air, then gets sent to the plant after regulating the pressure of the nitrogen. Nitrogen is also used in the start-up of the distillation process to mix the initial ethanol-water mixture.

The boiler in the system is electrically operated and is used to generate a saturated steam supply to the distillation columns from an inlet water supply. The pressure of the steam in the boiler is controlled at approximately 80 psig. A picture of the boiler is shown below in figure 2.12:

Figure 2.12

Boiler

The steam sent from the boiler to the distillation columns is then adjusted to 29 psig (2 barg) using a self-actuated pressure regulator. After the saturated steam goes through the coils in the reboiler of the distillation column it transfers its heat to the ethanol-water mixture and turns into liquid condensate. This condensate then gets sent to a condensate return system where its pumped back into the boiler. The condensate return system is shown in figure 2.13 below:

Figure 2.13

Condensate Return System

The last of the utilities is a chiller unit for cooling purposes. The coolant from the chiller is used in the condensers for the top distillate vapor, distillate coolers, and bottom product cooler. The type of coolant the chiller unit uses is 50% ethylene glycol and 50% water by weight. The ethylene glycol is DOWTHERM SR-1 and the water in the mixture is distilled water. The chiller unit is located outside of the pilot plant; a picture of it is shown in figure 2.14:

Figure 2.14

Chiller

2.5 Tank Farm (T110)

The tank farm (T110) area is where the initial feed mixture is located and where it is sent to the two distillation columns. The 1000 L feed tank (AB001) is filled with approximately 600 L of a 50% distilled water and 50% denatured ethanol (EtOH) by weight. T110 is also the location where the EtOH-rich and water-rich products are sent to and collected. The EtOH-rich product gets collected in the EtOH-rich product tank (AB002). The water-rich product first goes through the bottom product cooler (AW001), then gets collected in the water-rich product tank (AB003). The safety equipment in T110 includes a waste gas separator (AB004) and an activated carbon filter (AF001). In the case of waste gases escaping the top of the distillation columns, they get sent to AB004 and then the volatile compounds get trapped in AF001 before getting sent out to the atmosphere. The process and instrumentation diagram (P&ID) of T110 is shown in figure 2.15:

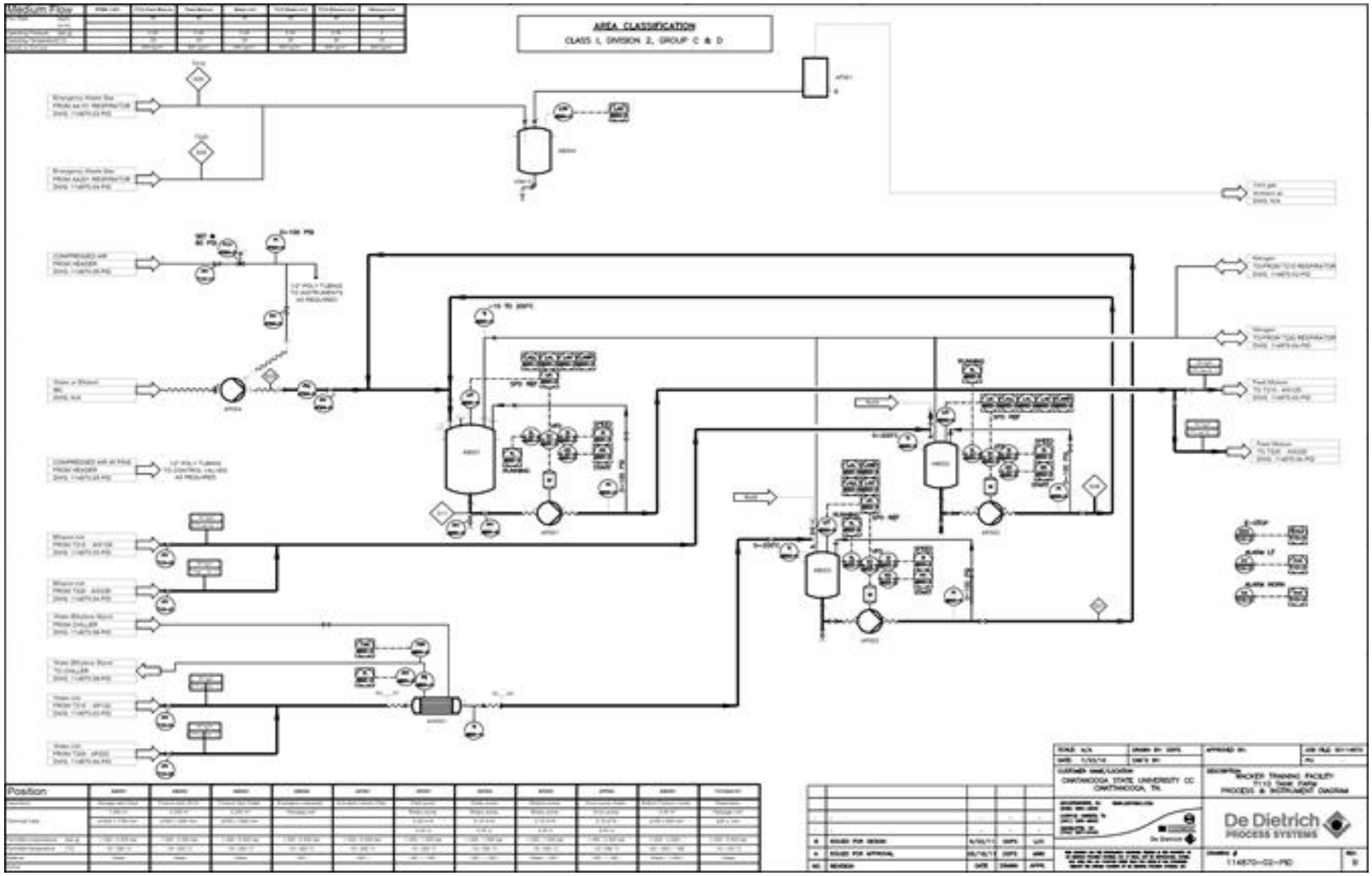

Figure 2.15

P&ID of T110

A clearer and labeled picture of T110 is shown below in figure 2.5.2:

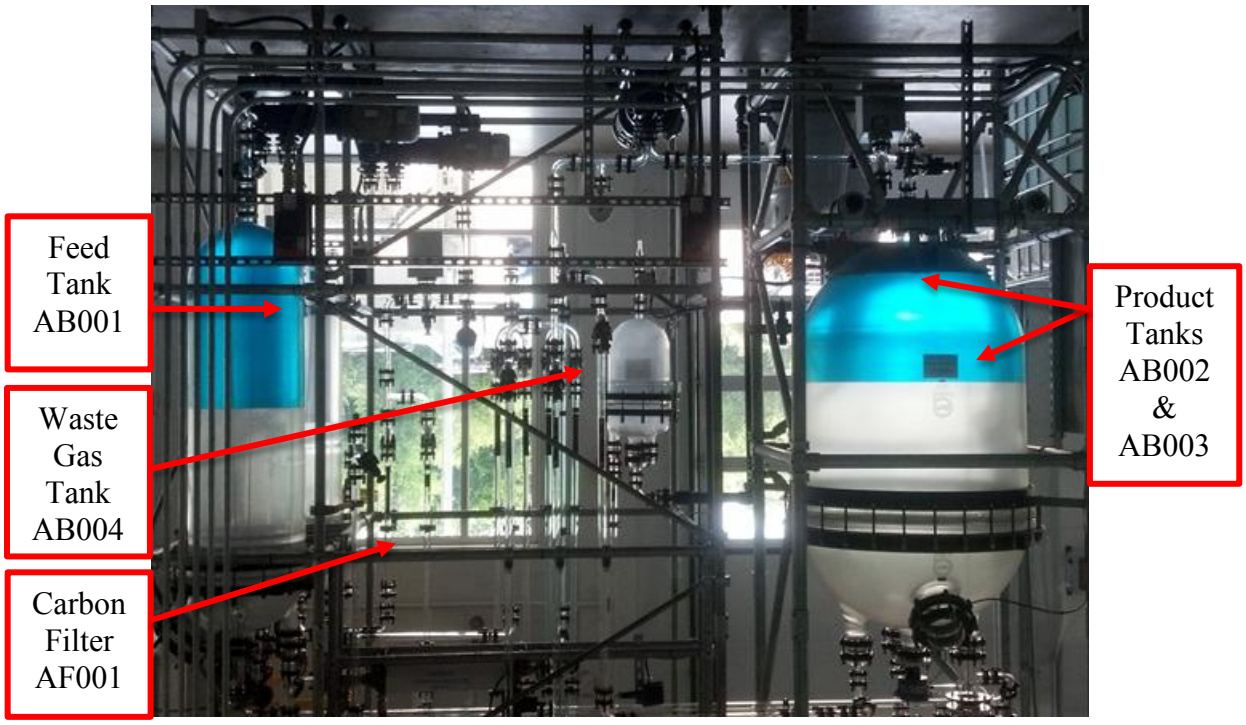

Figure 2.16

Labeled picture of T110

The tanks and pipelines are made of borosilicate glass manufactured by QVF (DDPS, 2011)

2.6 Distillation Columns

There are two distillation column areas as mentioned previously, bubble-cap tray distillation column area (T120) and sieve tray distillation column area (T220). The only difference in the two distillation columns is the type of tray each one has. The height of each distillation column is 6000 mm or approximately 20 ft. The diameter is 200 mm and the number of trays in each column is 20. The feed tray location can either be between trays 5-6 or 10-11 depending on the amount of stripping and rectifying that needs to be applied in the process. The

material of construction of the distillation columns is Borosilicate glass 3.3, which is the same material that was used for all the tanks and pipelines in the T110 area. Tables 2.1 and 2.2 below show a summary of the specifications for the distillation columns:

Table 2.1 Bubble-cap Tray Distillation Column (AK122) Specifications

Bubble-cap Tray Distillation Column (AK122)	
Material of Construction:	Borosilicate glass 3.3
Column Height:	6000 mm
Column Diameter:	200 mm
Number of Trays:	20
Tray Spacing:	208 mm
Operating Pressure:	1 atm
Tray Type:	Crossflow
Tray Material:	Stainless Steel (316Ti)
Tray Diameter:	190 mm
Number of Caps per Tray:	2
Bubble-cap Size:	152 mm X 28.5 mm
Downflow Area:	3226 mm^2

Table 2.2 Sieve Tray Distillation Column (AK222) Specifications

Sieve Tray Distillation Column (AK222)	
Material of Construction:	Borosilicate glass 3.3
Column Height:	6000 mm
Column Diameter:	200 mm
Number of Trays:	20
Tray Spacing:	208 mm
Operating Pressure:	1 atm
Tray Type:	Crossflow
Tray Material:	Stainless Steel (316Ti)
Tray Diameter:	190 mm
Number of Holes per Tray:	66
Hole Diameter:	8 mm
Downflow Area:	3226 mm^2

Pictures of a model of one of the trays for each column are shown below in figures 2.17 and 2.18:

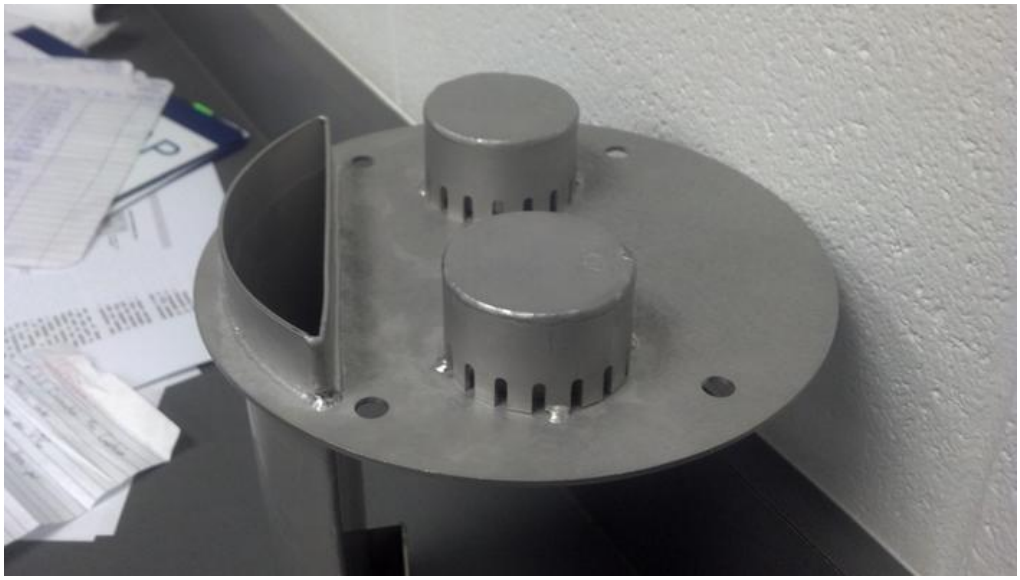

Figure 2.17

Bubble-cap Tray

Figure 2.18

Sieve Tray

The distillation column areas (T120/220) also have other equipment besides the columns their selves. These equipment include a recirculation evaporator (AW126/226), feed pre-heater (AW125/225), condenser (AW127/227), respirator (AA101/201), buffer tank (AB101/201), and distillate cooler (AW128/228). The details of this equipment will be discussed in the following subsections. Since both T120 and T220 have the same exact equipment and only different in their identification number, only one from either column will be discussed. These do not include the various pumps, control valves, and instrumentation which will be discussed in the later sections of this chapter.

2.6.1 Recirculation Evaporator

The recirculation evaporator has a heat exchange surface area of 1 m^2 and included inside it is a stainless steel heating coil for steam to go through. The recirculation evaporator is a type of heat exchanger and also is used for mixing the ethanol-water mixture in order to avoid flash evaporation in the distillation column. This is done using the bottoms pump and also by introducing nitrogen gas to the bottom of the recirculation evaporator. The portion of this device in the P&ID (DDPS, 2011) of T120 is shown in figure 2.19 highlighted in yellow:

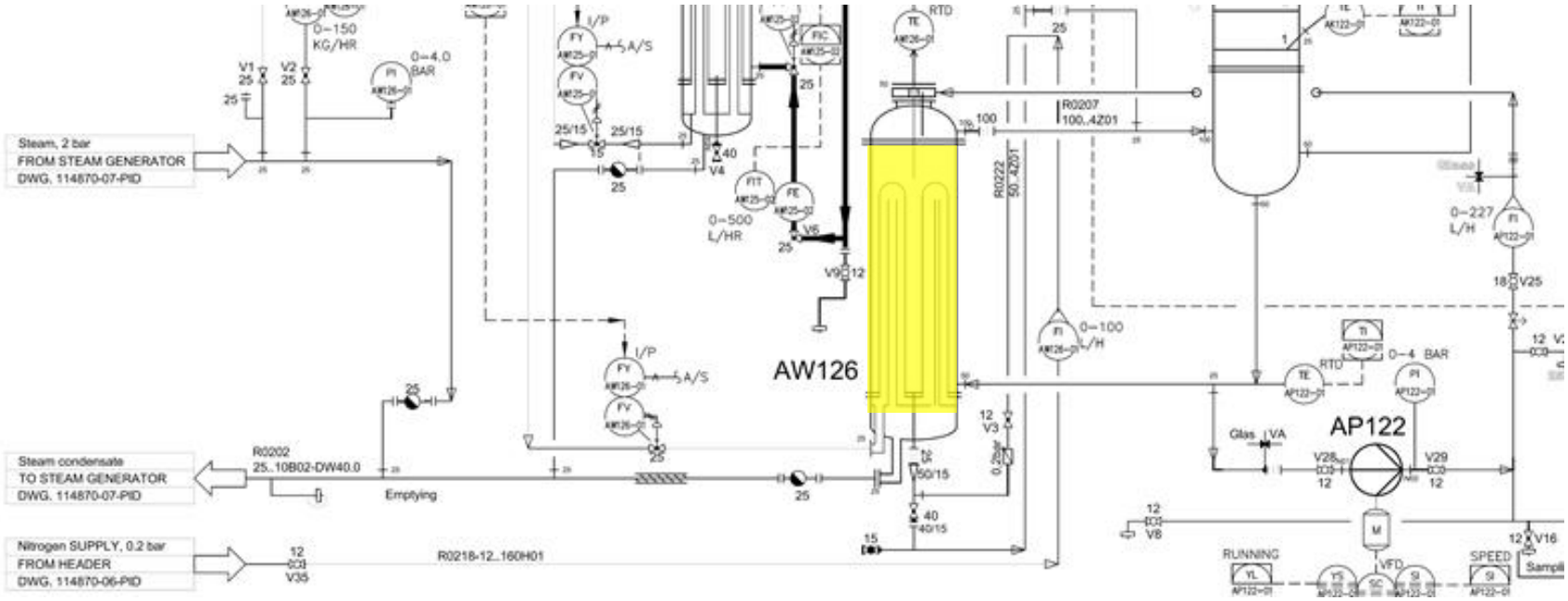

Figure 2.20

Picture of Recirculating Evaporator (AW126) in T120

A picture of AW126 is shown below in figure 2.20:

Figure 2.20

Picture of Recirculating Evaporator (AW126) in T120

2.6.2 Feed Pre-Heater

The feed pre-heater is used in the distillation column to warm the feed mixture closer to its boiling point so that the process runs more efficiently. This device has a heat exchange surface area of 0.4 m^2 and also has a heating coil inside it. The portion of this device in the P&ID (DDPS, 2011) of T120 is shown in figure 2.21 highlighted in yellow:

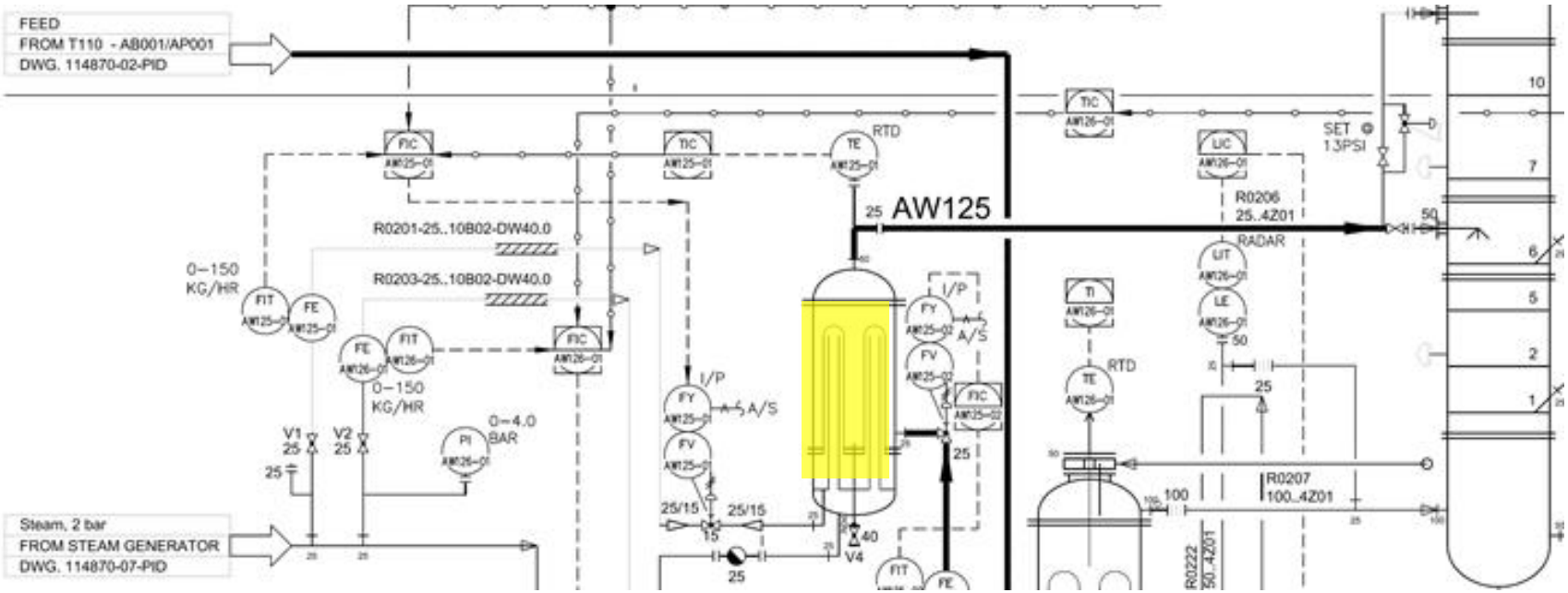

Figure 2.21

P&ID of Feed Pre-Heater (AW125) in T120

A picture of AW125 is shown below in figure 2.22:

Figure 2.22

Picture of Feed Pre-Heater (AW125) in T120

2.6.3 Condenser

The condenser in the distillation column is used to condense the vapors going up the distillation column. This device is a type of counter-flow shell and tube heat exchanger. It uses coolant on the tube side and the vapors from the distillation column condense on the shell side. The heat exchange surface area of this device is 2.5 m^2 and is horizontally tilted in order for the condensate to travel with the aid of gravity. The condenser's top is opened into a vent gas line in case the vapors escape the condenser in the case where cooling rate is insufficient compared to the rate of vapor going up the column. This vent line is connected to a rupture disk and then the respirator. In the case of a vacuum condition in the column, this line is also used to supply nitrogen gas into the column through the condenser. The portion of this device on the P&ID labeled and a picture of it is shown with figures 2.23 and 2.24:

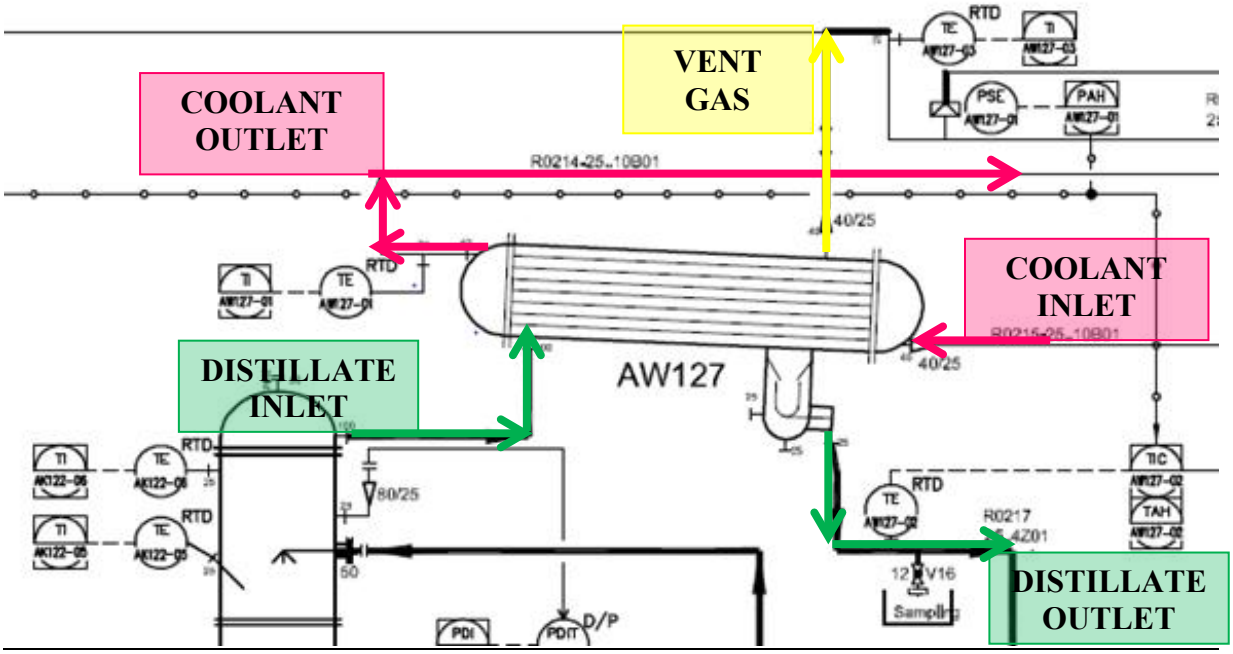

Figure 2.23

Labeled P&ID of Condenser (AW127) in T120

Figure 2.24

Picture of Condenser (AW127) in T120

2.6.4 Respirator

The respirator is a mechanical device with two weights used to keep an equilibrium pressure inside the distillation column. The nitrogen supplied to blanket all the tanks and the distillation column is done through this device. Nitrogen is supplied to the device at 0.04 Barg and enters through one of the inlets with a weight on top of it. In the case of exceeding a certain pressure in the column, this weight closes and the other one lifts to release the gas through the vent gas line and gets sent to AB004. The respirator for T120 (AA101) on the P&ID is shown in figure 2.25:

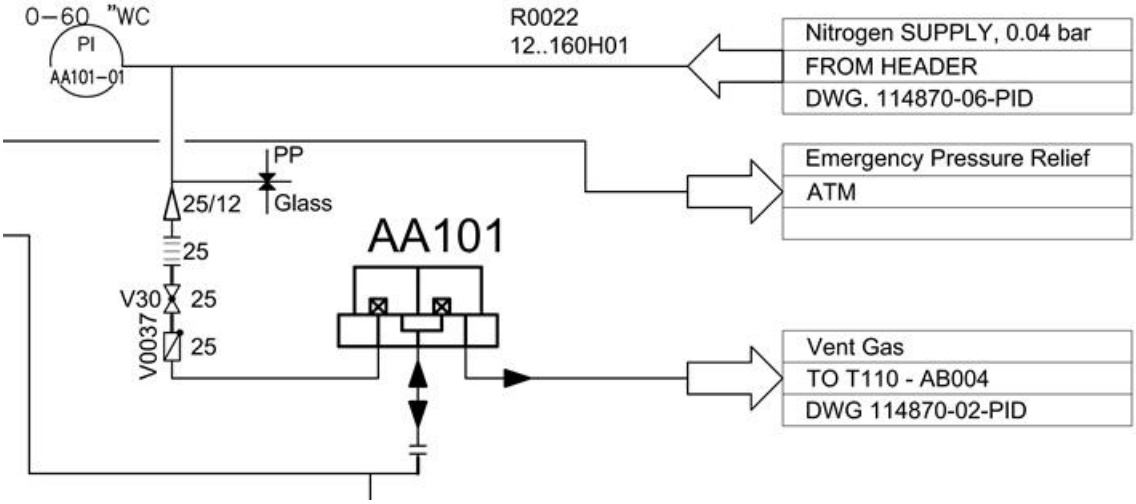

Figure 2.25

P&ID of Respirator (AA101) in T120

Figure 2.25 above illustrates how the nitrogen is supplied at 0.04 bar, which can be checked with the pressure indicator (PI AA101-01), then goes in the left side of AA101 after a manual hand valve. This nitrogen supply then enters the condenser, column, and all the tanks from the middle AA101. The vent gases from the condenser AW127 in an emergency situation would go in through the same place nitrogen enters the condenser, middle portion of AA101, shown by the two directional arrows. A picture of this actual device in T120 can be seen in figure 2.26:

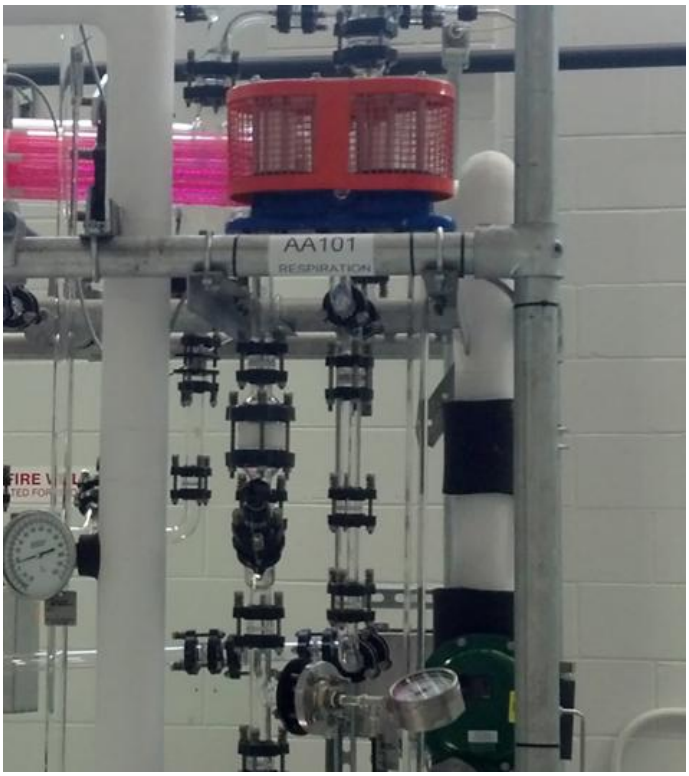

Figure 2.26

Picture of Respirator (AA101) in T120

2.6.5 Buffer Tank

The buffer tank, also called a reflux drum in many distillation applications, is the tank where the top distillate product condensate gets collected. This is a 50 L tank and is the place where reflux splitting takes place. The reflux is sent back to the top of the column and also the distillate EtOH-rich product gets sent to the distillate cooler before getting collected in the product tank AB002 in the tank farm area T110. The level in the tank is controlled in a loop by the distillate EtOH-rich product return valve (LV AB101/201-01) and a constant reflux ratio can be achieved by using the cascade control (FFIC-AK122/222) for the reflux valve (FV AK122/222-01). A portion of this device in the T120 P&ID (DDPS, 2011) is labeled and highlighted in yellow in figure 2.27:

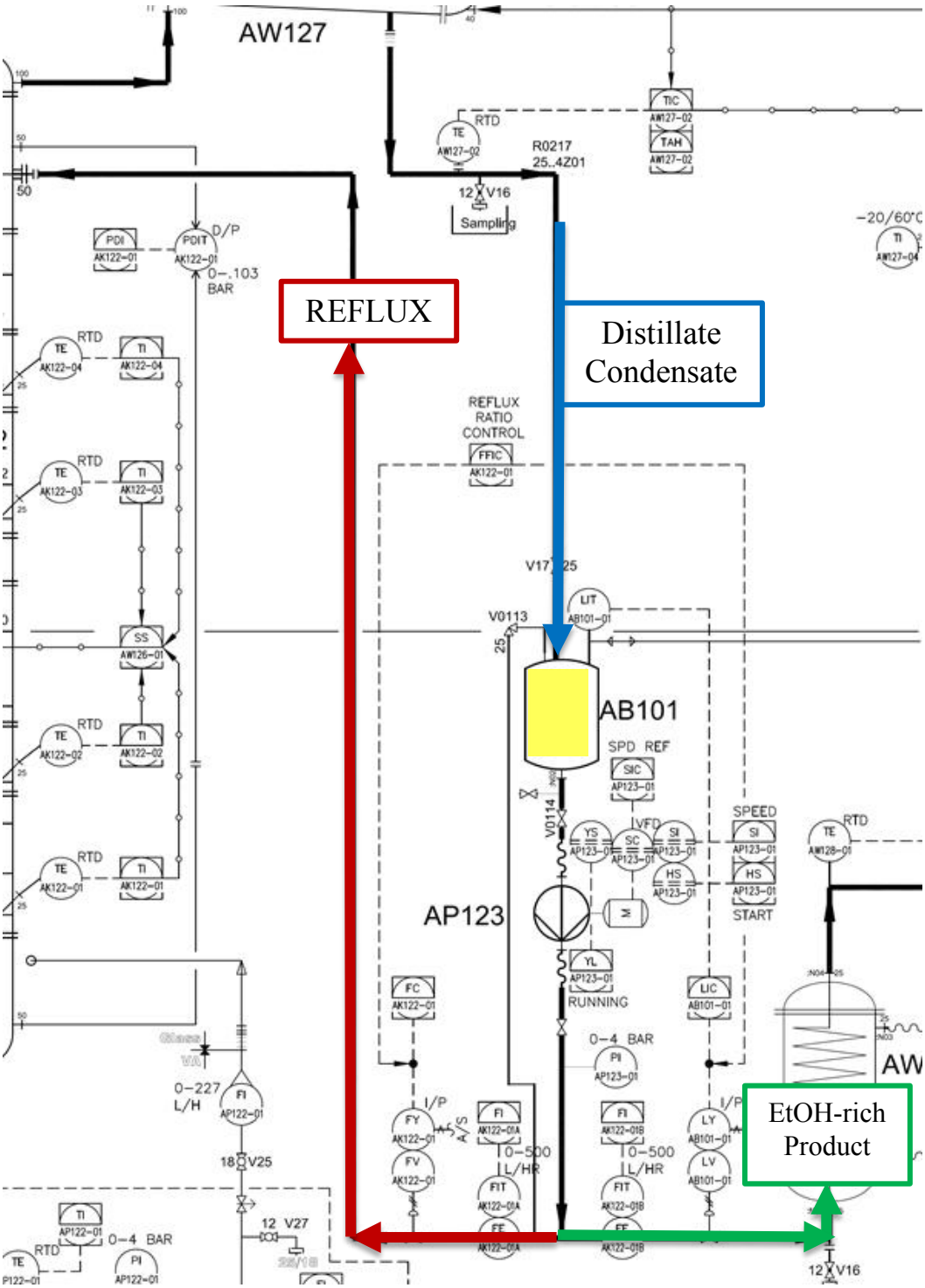

Figure 2.27

Labeled P&ID of Buffer Tank (AB101) in T120

A picture of front and back view of AB101 is shown below in figure 2.28:

Figure 2.28

Picture of Front (Left) and Back (Right) View of Buffer Tank (AB101) in T120

2.6.6 Distillate Cooler

The distillate cooler is another heat-exchanger device that is used to cool the EtOH-rich distillate product and keep it below 32^{o}C before it gets sent to the EtOH-rich product tank AB002 in the tank farm T110. This is also a shell and tube heat exchanger with the coolant on the shell side and the EtOH-rich distillate product on the tube side. The heat exchange area of this device is 1.0 m^2. The P&ID for the distillate cooler AW128 in the bubble-cap distillation column area T120 and its picture is shown in figures 2.29 and 2.6.30 respectively:

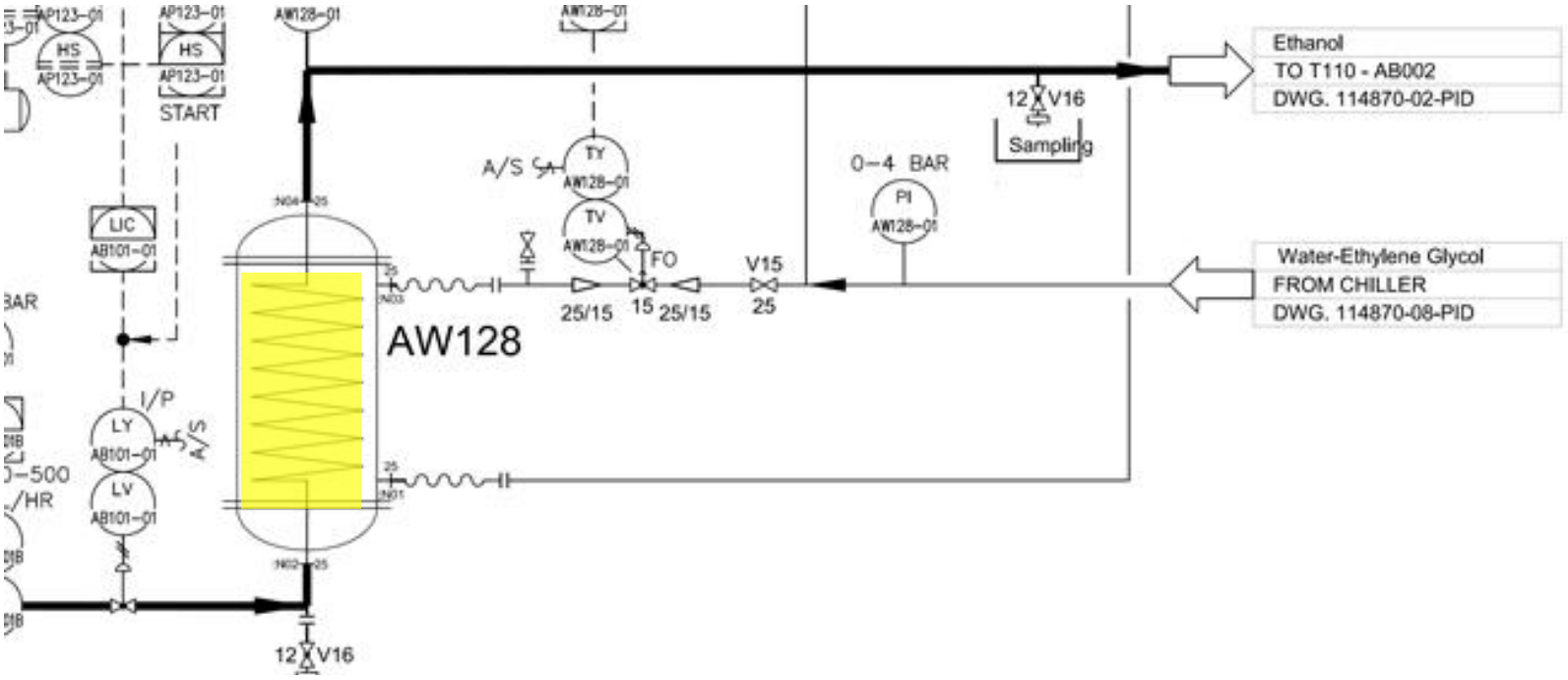

Figure 2.29

P&ID of Distillate Cooler (AW128) in T120

Figure 2.30

Picture of Distillate Cooler (AW128) in T120

2.7 Pumps, Control Valves, and Measuring Devices

2.7.1 Pumps

There are two different types of pumps used in the pilot plant facility, the ones used in the tank farm area T110 and the ones used in the distillation column areas T120 and T220. This section does not include or discuss the pumps for the condensate return system, the boiler, and the chiller unit. The pump used in T110 is a side-channel centrifugal pump with two impellers. It is used for the feed tank (feed pump AP001) and both of the product tanks (Water-rich product tank pump AP002 and EtOH-rich product return pumpAP003), shown below in figure 2.31:

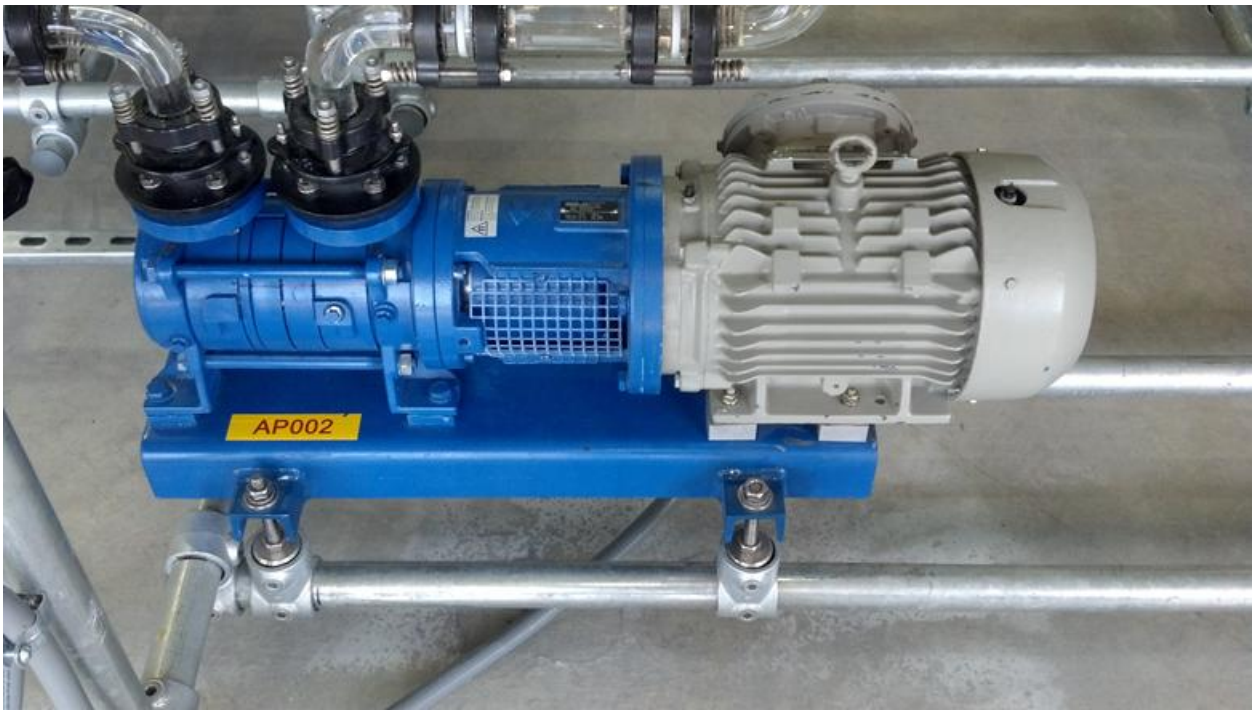

Figure 2.31

Tank Farm T110 Pump

The other type of pump used in the two distillation columns is also a side-channel centrifugal pump except that it only has one impeller. This pump is used for the bottom recirculation/bottom

water-rich product return (called bottoms water pump AP122/222) and for buffer tank recirculation/reflux/EtOH product return (called EtOH distillate pump AP123/223). This pump is shown below in figure 2.32:

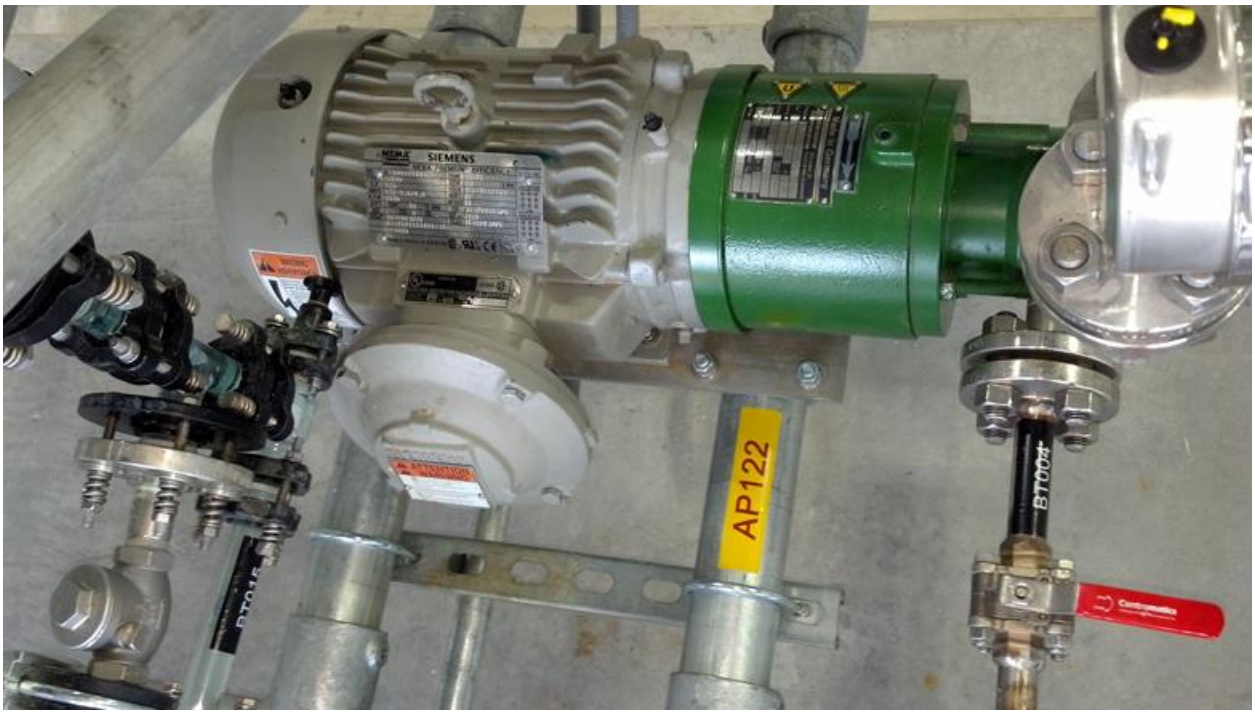

Figure 2.32

Distillation Column Area T120/220 Pump

2.7.2 Control Valves

There are two different types of control valves for the distillation columns; a Fisher 3661 positioner with a Baumann pneumatic control valve and actuator, and a Samson pneumatic valve and actuator. The Fisher/Baumann control valve is used for controlling the steam supply to recirculating evaporators AW126/226, steam to feed-preheaters AW125/225, coolant supply to condensers AW127/227, coolant supply to distillate coolers AW128/228, and bottoms water-rich product return to the water-rich product tank AB003. The Samson control valve is used for

controlling the feed flow rate, reflux, and distillate EtOH-rich product return to EtOH-rich product return tank AB002 for both of the distillation column areas T120 and T220. Pictures of one of each of these valves are shown in figures 2.33 and 2.34:

Figure 2.33

Fisher 3661 Positioner with a Baumann Pneumatic Control Valve and Actuator

Figure 2.34

Samson Pneumatic Valve and Actuator

2.7.3 Measuring Devices

There are total of three different kinds of flow meters used in the distillation process for measuring flow of fluids. The two main ones are a Rosemount 8800D Vortex flowmeter and a Micro Motion F-Series Coriolis flowmeter. The Rosemount Vortex flowmeter is used for indicating steam and coolant flow rates in kg/hr. A picture of this device is shown in figure 2.35:

Figure 2.35

Rosemount 8800D Vortex Flowmeter

The Micro Motion Coriolis flowmeter is used for indicating feed, EtOH-rich distillate product, and reflux flow rates in L/hr for both of the distillation columns. A picture of this device is shown in figure 2.36:

Figure 2.36

Micro Motion F-Series Coriolis flowmeter

The last type of flowmeter used is a Rotameter which is used for indicating the flow rate of bottoms recirculation into recirculating evaporator measured in gal/min. A picture of this device is shown in figure 2.37:

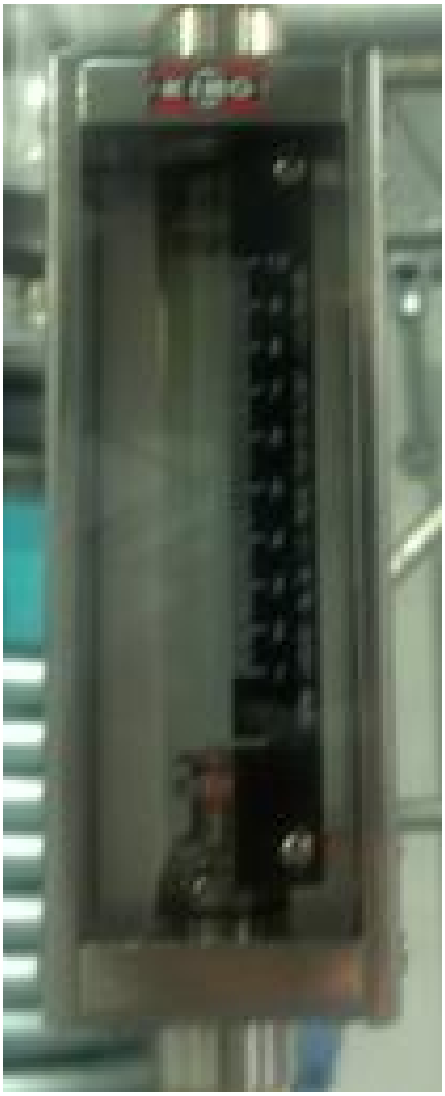

Figure 2.37

Rotameter

There are two different types of level measuring devices used in the distillation process. One of them is a Rosemount 3100 Series Ultrasonic Level Transmitter that is used in the buffer tank AB101/201 in both of the distillation column areas T120/220 and the feed tank AB001 in the tank farm area T110. This device is shown in figure 2.38:

Figure 2.38

Rosemount 3100 Series Ultrasonic Level Transmitter

The other level device is a Rosemount Guided Wave Radar Level Transmitter which is used to determine the level at the bottom of the column/recirculating evaporator and in both of the product tanks in the tank farm AB002 and AB003. This device is shown below in figure 2.39:

Figure 2.39

Rosemount Guided Wave Radar Level Transmitter

Pressure is measured at many places around the distillation column areas, tank farm, and utilities. The most common pressure gauge found is the QVF Pressure Gauge located after the outlet of every pump. Pressure is measured at these pump outlet and then adjusted with manual hand valves to achieve the right pressure for the process. The pressure measurement device that is most useful in analyzing flooding phenomena in the distillation columns is the Rosemount Differential Pressure Transmitter. This device measures the difference in the pressure from the bottom to the top of the distillation column, measured in barg. This pressure transmitter is shown below in figure 2.40:

Figure 2.40

Rosemount Differential Pressure Transmitter

All of the temperature measurements in the distillation columns are measured by resistance temperature device (RTD) made by JMS Southeast. The temperature readings indicated by this device are also crucial in analyzing flooding.

CHAPTER III

PROCEDURES

3.1 Introduction

The flooding phenomena in distillation and determining the stable operating region of the two distillation columns were investigated in this research. The procedure for accomplishing this is split into four sections. The second section of this chapter will describe the procedure for safety which describes checking of all equipment and ensuring that the distillation columns can be started-up safely. Also included in the safety section are the details of the hazardous chemicals used and the procedures followed to minimize the likelihood of an incident happening. The third section describes the start-up process where the procedures for starting up the distillation process with total reflux are described. Continuous distillation is the next section of procedures where feed is being supplied to the column and product streams are being taken out. This section will also describe how the distillation column was brought to its flooding point and other important boundary regions in the stable operating region. The last section of this chapter will discuss the procedures for stopping flooding, bringing the distillation column back to a safe mode, and shutting down the distillation column.

The fourth section of procedures most closely relates to the research topic of this thesis. This is where all the data are recorded and analyzed to study the flooding phenomena as well as the stable operating regions of the two distillation columns.

3.2 Safety

3.2.1 Description of Hazardous Chemicals

The hazardous chemicals in the pilot plant include denatured ethanol for distillation and ethylene glycol for coolant supply to the condensers. The ethylene glycol is always contained within the coolant pipelines where ethylene travels from the plant to the outside chiller, then back to the plant in a continuous cycle. This means there is never any contact with the ethylene glycol. The National Fire Protection Association (NFPA) rates this chemical as code 1 for health hazard (slightly hazardous), code 1 as a fire hazard (flash point above 200 ^{o}F), and code 0 for reactivity (stable). The denatured ethanol used in the distillation process is a flammable liquid and vapor. It is harmful by inhalation, in contact with skin and if swallowed (MSDS, Denatured Ethanol). NFPA rates this chemical as a code 2 for health hazard (hazardous), code 3 for fire hazard (flash point below 100 ^{o}F), and code 0 for reactivity.

For the protection from these hazardous chemicals the pilot plant is equipped with all the necessary equipment. Protection from the health hazards includes having a safety shower and an eye wash station easily accessible inside the pilot plant. Eye protection equipment such as safety glasses or goggles are always worn when inside the pilot plant area and near any of these chemicals. Safety gloves are worn when handling samples and during analytics. The following measures are taken for protection from the fire hazard of denatured ethanol:

- All of the equipment inside the pilot plant are intrinsically safe (explosion proof).
- Oxygen gas is removed from all devices operating with denatured ethanol and replaced with nitrogen gas.
- Fire extinguishers and fire water hose is located inside the pilot plant in the case of an explosion or fire.

The DeltaV DCS software is programmed with many interlock conditions to stop all equipment in the case of a dangerous or hazardous condition in the pilot plant. This includes closing all steam valves in the case of denatured ethanol and/or ethylene glycol reaching above normal temperatures, shutting down operating of all equipment and opening coolant control valves in the case of the chiller and the nitrogen generator failing to operate properly.

3.2.2 Equipment Check for Safe Operation

Before starting up the distillation columns, the safety systems are checked to ensure safe operation of the process. The very first and most important device to check is the operation of the nitrogen generator shown in figure 2.11. All inlet and outlet valves, the pressure inside the tank, oxygen concentration, and pressure of the nitrogen gas leaving the nitrogen generator into the pilot plant are checked. The regulators for the nitrogen supply to the distillation columns are checked to make sure the supply to the recirculating evaporators for mixing is at 0.4 barg, nitrogen blanketing to the top of the distillation columns is at 0.04 barg, and the necessary manual valves are open in order for the nitrogen gas to enter the respirator. This process ensures that there are no leaks and nitrogen is inside all tanks and the distillation columns. These devices are checked every hour of operation as necessary.

The second most important device to check for safety is the chiller unit, shown in figure 2.14, for coolant supply. The supply of coolant to the pilot plant from the chiller is checked by the flow indicating controls on the distillation columns (FIC AW127-01 and FIC AW227-01) from the T120 screen interface of DeltaV DCS (figure 2.9) and the T220 screen interface. These flow rates are indicated by the vortex flow meters (figure 2.35) for the coolant supply and should read approximately 200 L/hr when the plant is not in operation. This check only ensures that the pump for the chiller unit is working properly and coolant is being circulated from the chiller to

the distillation columns. To check to see if the compressor for the chiller is working and the coolant is cooled further once it heats up to a set-point temperature set in the chiller unit control panel, a process history of the RTD temperature indicator from the outlet of the condenser (AW127 or AW 227) must be checked from the DeltaV DCS process history. This temperature indicator for T120 is shown in figure 2.23 labeled TI AW127-01. The process history time axis is expanded to show the past 8 hours to see if the temperature on the y-axis has been rising to the set-point on the chiller and then dropping in temperature after it reaches this point. This process ensures that the chiller is working properly and the compressor will operate to cool the coolant once it heats up in the process.

The waste water pit is checked to make sure that the gate valve is closed and no chemicals will be sent to the city sewer line in the case of a large spill. This is followed by checking the air compressor (figure 2.10) for air supply to the control valves in the pilot plant. This consists of checking the oil level in the compressor and draining the condensate built up in the air dryer of the air compressor. Checking of the operation of all control valves, pumps, and measuring devices is the next and final step for safety. The control valves are opened to 100% and back down to 0% using DeltaV DCS and physically checked to ensure they open and close all the way. The pumps are started then stopped while physically checking that they work, followed by checking all flow meters, RTDs, level transmitters, and differential pressure transmitter to ensure they indicate normal readings for the current conditions of the tanks and the distillation columns. The last equipment to be checked is the operation of the boiler shown in figure 2.12 and the condensate return system shown in figure 2.13. The water levels in the condensate return tank, blowdown tank, and boiler are checked before starting the boiler. After the boiler reaches operating pressure (approximately 80 psig), the steam supply ball valve is

opened about a quarter turn every 15 minutes to avoid excessive water knocking on the pipes and steam is circulated in the steam pipes. The pipes and valves are checked for leaks and the condensate return pipelines are checked to make sure all valves are open in order for the built up condensate to get sent to condensate return system. A very important safety mark is to remember to check all manual (hand operated) valves for the correct position before operating any equipment.

3.3 Start-Up

3.3.1 Tank Farm T110 Preparation

The start-up procedure begins with the preparation of raw mixture in the feed tank AB001 in the tank farm area T110. The end products from the previous distillation are contained in the tanks AB002 (EtOH-rich) and AB003 (Water-rich). These product tanks are lowered to 20% level, as indicated by the level transmitters shown in figure 2.39, by sending the EtOH and the water to AB001. AB001 is then recirculated at a pressure of 0.5 barg at the feed pump AP001 for good mixing. After 15 minutes of mixing, a sample is collected to analyze the amount of EtOH in the mixture using the density meter (see Section 2.2, figure 2.2). The feed mixture in AB001 should have approximately 50 wt% EtOH. If this is not the case, then this is brought to a 50 wt% EtOH by adding more from AB002 or AB003 as necessary to accomplish this concentration of EtOH. Then the mixing and sampling steps are repeated until it is confirmed that the mixture in AB001 is 50 wt% EtOH.

3.3.2 Filling up the Bottom of Distillation Column

After the mixture in tank farm T110 is prepared, the cooling system is started by adjusting the coolant flow to the condenser AW127/227 (FIC AW127/227-01) on automatic

mode to 1000 L/hr and to distillate cooler AW128/228 (TIC AW128/228-01) on manual mode to 2%. This is followed by filling up the bottom of the distillation column with a 50 wt% denatured ethanol-50 wt% distilled water mixture to a safe level before starting up. The feed pump pressure is adjusted to 1.5 barg and feed valve FIC AW125/225-02 is opened to start filling the bottom of the distillation column using the feed line. Once the level indicator for the bottom level in distillation column (LIC AW126/226-01) is over 70%, the bottoms pump AP122/222 is started for recirculation and the recirculation rotameter (see figure 2.37) is adjusted to read 0.8 gal/min using the manual valve on the recirculation line. The feed valve is shut once there is enough mixture at the bottom of the distillation column which is physically indicated by mixture crossing over from the recirculating evaporator AW126/226 to the bottom of the column (see figure 2.20). This "cross-over" level is necessary for well mixed EtOH-water mixture and is done to avoid flash evaporation which is an unsafe condition in the distillation column. Nitrogen is also supplied to AW126/226 at 0.4 barg to help with mixing, but this is stopped after approximately 10 minutes.

3.3.3 Heating and Total Reflux

Once complete mixing is achieved, heating of the system is initiated. Manual valves for steam supply to AW126/226 are opened slowly and pressure of the steam is adjusted to 2 barg (29 psig) using a self-actuated pressure reducing regulator. The steam control valve FIC AW126/226-01 is first opened manually to approximately 20%, and then placed on automatic mode to have a steam flow rate ranging from 15 to 35 kg/hr. After the evaporator reaches boiling point (82-85 ^{o}C), which takes approximately 10 to 20 minutes depending on the steam flow rate, and the top tray (20th Tray) reaches approximately 78 ^{o}C shortly after boiling (1-5 minutes), coolant flow rate is increased to 4000 L/hr to ensure all the vapors get condensed in the

condenser, and EtOH distillate pump AP123/223 is started and recirculation in the buffer tank AB101/201 (see Section 2.6.5) is started by adjusting the pressure after the pump to 1.5 barg. At this time, condensate starts filling up the buffer tank and reflux control valve (FIC AK122/222-01) is opened to begin total (100%) reflux. The reflux valve is first opened on manual mode to 40% to 50% then put into automatic mode and given a set-point flow rate. The set-point flow rate of this reflux is adjusted to where the level of liquid in the buffer tank stays constant, which depends only on the flow rate of the steam going into the recirculating evaporator. The higher the steam flow rate is, the higher the reflux flow rate must be to keep a constant level in the buffer tank. Since there is no flow meter to measure the rate of condensate coming from the condenser, the flow rates of steam and reflux must be adjusted on a trial and error basis.

3.4 Continuous Distillation

3.4.1 Reflux Splitting / Reflux Ratio

Once total reflux has being continued for approximately 30 minutes, reflux splitting can begin. Reflux splitting is where the EtOH-rich distillate product control valve (LIC AB101/201-01) is opened and EtOH-rich distillate product gets sent to the distillate cooler (see Section 2.6.6), where temperature of the product is kept at or below 32 ^{o}C, followed by getting sent to the EtOH-rich product tank AB002. The control valve mentioned is opened manually to get a certain flow rate (15 to 25 L/hr) and the reflux flow rate is decreased (45 to 75 L/hr) in order to start out with a 3:1 reflux ratio and still keep the buffer tank at a constant level. Once this is accomplished manually and the mentioned variables (reflux ratio and buffer tank level) are steady, EtOH-rich distillate product control valve is placed on automatic control where the level in the buffer tank can be controlled at a set-point level adjusted by LIC AB101/201-01, followed by placing the reflux flow control valve on cascade mode where the reflux ratio control (FFIC AK122/222-01)

goes in control. The reflux ratio control set-point is set to 3 initially. The way this control works is that it receives the flow rate of EtOH distillate product as indicated by the Micro Motion flow meter (see figure 2.36), multiplies this flow rate by the reflux ratio control set-point, and sends the result of this mathematical operation as a set-point flow rate to the reflux flow control valve. The result of this is that the level of the buffer tank always stays constant (+/- 1%) and the reflux ratio stays constant (+/- 1% uncertainty) throughout the distillation process.

3.4.2 Introducing Feed

Feed can be introduced from two different locations on both distillation columns as was mentioned in section 2.6. Feed location can either be tray 5 or 10. The feed tray location also has an effect on the location where flooding begins as will be discussed later. Feed can begin to be introduced once the temperature at the recirculating evaporator reaches above 95 ^{o}C. This helps the stripping section of the column (section below the feed location) to work more efficiently due to most of the EtOH being separated from the feed mixture before making its way down to the bottom of the distillation column.

Once the condition mentioned above is reached, feed control valve (FIC AW125/225-02) is opened manually to 50%, and then set to automatic mode where a set-point flow rate of 30 L/hr is set for the initial feed flow rate. After feed flow is achieved, steam to the feed-preheater is started by opening steam valve (FIC AW125/225-01) to 30%. Since the vortex flow meter for this steam line is oversized and is unable to get a reading on steam flows less than 8 kg/hr, the valve is left on manual mode. This control valve is adjusted up or down to keep a constant average temperature of 60 ^{o}C (as indicated by TIC AW-125-01) for the feed entering the column. Pre-heating the feed to this temperature brings the feed closer to its flash point (approximately 82 ^{o}C). This leads to EtOH being separated from water much faster and helps less EtOH to go all

the way down the column, which leads to decreased temperatures at the bottom and lower purity of water-rich product. So it can be said that pre-heating the feed closer to its flash point leads to a more efficient distillation process. The reason for not pre-heating the feed to its flash point is for safety reasons. The feed is supplied to the feed pre-heater and then sent to the feed tray in a one inch glass pipe. The glass pipe is not pressure rated to handle any possible vapors coming from the feed pre-heater and will most likely shatter in the event of a high temperature for the feed stream.

3.4.3 Collecting Bottoms (Water-rich) Product

Water-rich bottoms product is ready to be collected once the temperature of the recirculating evaporator reaches 100 ^{o}C. This temperature can be 1 to 3 degrees higher in certain cases depending on the pressure at the bottom of the column, and also very small amount of acetic acid in the denatured EtOH staying at the bottom of the column due to having higher boiling point than water (118 ^{o}C). Once this condition is accomplished, the level indicating control valve for water-rich bottoms product (LIC AW126/226-01) is opened to approximately 20% on manual mode, where the product gets sent to a condenser heat exchanger AW001 in the tank farm T110 to drop the temperature of product from 100 ^{o}C to approximately 30 ^{o}C, followed by being sent to and collected in the water-rich product tank AB003. This percentage of valve opening is then adjusted to achieve proper level in the recirculating evaporator. Once proper level is reached, the valve is adjusted to get a flow rate of water-rich bottoms product to mass balance in the column. This means that the EtOH-rich product plus the water-rich product flow rates should add up to approximately the feed flow rate entering the column. This will not be exact since volumetric flow rates do not balance, but it will be very close. The mass balance can also be easily calculated by taking samples of the products and the feed, then using the

density reading from the density meter results to convert the volumetric flow rates into mass flow rates. After the water-rich bottoms product return valve has been manually adjusted, it is placed in automatic mode where a level set-point in the recirculating evaporator is set. This allows the bottom of the column to keep a constant level by adjusting LIC AW126/226-01 automatically.

3.4.4 Stable and Unstable Operating Conditions

The goal of the distillation process is to have a feed flow rate of 80 kg/hr, achieve greater than or equal to 90 wt% EtOH as top distillate product at a flow rate of 40 kg/hr, and less than or equal to 1% wt% EtOH in the bottoms water-rich product stream at a flow rate of 40 kg/hr while using the least amount of energy as possible.

Stable operating conditions are defined as achieving the purity levels mentioned above at any inlet and outlet flow rates. This is accomplished by increasing the feed flow rate and steam in such a way that the bottom of the distillation column remains at 100 ^{o}C, the middle region in the range of 80 ^{o}C to 85 ^{o}C, and the top at 78 ^{o}C. This procedure ensures that the products are meeting the production purity goals. The following steps are followed in order to accomplish this and reach the production flow rate goals:

- Increasing feed flow rate only when feed tray is increasing in temperature above 82 ^{o}C.
- Increasing feed flow rate in small increments (maximum of 10 L/hr each time)
- Increase steam rate with small increments (1-5 kg/hr) only when bottom of column decreases in temperature below 100 ^{o}C.
- Decrease reflux ratio by small increments (maximum of 0.5 each time) when:
    - Top product purity is greater than 90 wt% EtOH

- Feed Flow rate is high (50-70 L/hr)
- Reflux flow rate is greater than 60 L/hr on bubble-cap tray and 120 L/hr on sieve tray distillation columns.

Unstable operating conditions consist of not having the proper temperature in the distillation column which results in poor purity levels, weeping, and flooding of the distillation column trays. The weeping point of the sieve tray distillation column's trays is determined by physically seeing the liquid falling through the perforations of the sieve tray. An image of this phenomenon is shown in figure 3.1:

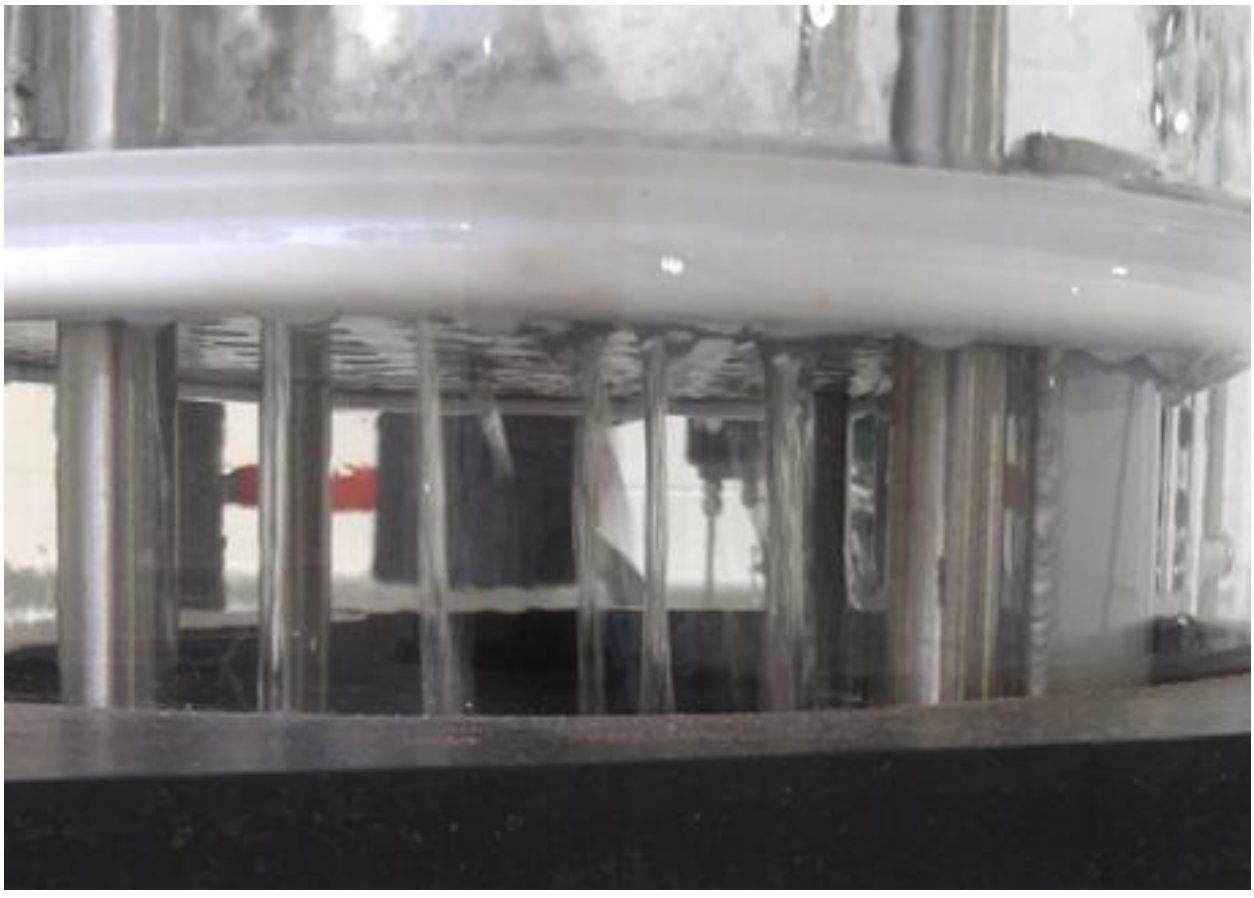

Figure 3.1

Weeping of a Sieve Tray

The procedure to find the weeping point is to keep the distillation column in total reflux where no feed is introduced and no product is being taken out of the column. The next step is to adjust the steam flow rate to the recirculating evaporator and reflux flow rate to where the buffer tank level stays constant (see Section 3.3.3). The steam flow rate then is reduced, while keeping reflux flow rate constant, by increments of 1 kg/hr until weeping on all 20 trays are visually observed as shown above in figure 3.1. After one weeping point is determined, the reflux flow rate is decreased or increased to another constant value and steam rate is again decreased until weeping is observed.

The flooding point of the distillation column involves more than just a visual inspection. Flooding in the bubble tray distillation column can be observed by mainly sudden drops in differential pressure across the column followed by continuous increase. Since the distillation column is made out of glass, it can also be visually observed for confirmation that the tray is actually flooding. A picture of 3 flooded trays is shown in figure 3.2:

Figure 3.2

Flooding of 3 Bubble-cap Trays

The white foam covering the trays shown above in figure 3.2 is what is described as flooding of the trays. This white foam visual is the result of turbulence of the liquid inside that tray. This flooding phenomenon will be better observed with the video footages shown in the next chapter. The procedure followed to find the flooding points are:

1. Start up the distillation column as described in this chapter and proceed to continuous distillation.
2. Keep a constant reflux ratio while increasing feed and steam flow rates and under stable operating conditions.
3. Visually inspect the distillation column periodically to ensure no flooding is occurring.
4. Keep increasing flow rates until flooding is observed visually and through process variables such as differential pressure of the column.
5. Take samples of top distillate EtOH-rich product to analyze changes in purity.
6. Record the results.

3.5 Shut-Down

3.5.1 Stopping Flooding

The following steps are done in order to reduce and then stop the flooding of the trays. It is important to never cut off all steam supply to stop flooding. This would cause all of the ethanol on the top trays to fall to the bottom of the column where it will flash vaporize due to the temperature at the bottom being very high. All of the ethanol will flash vaporize, go rapidly in the condenser, and escape through the emergency waste gas line (vent gas) since the condenser would not be able to handle condensing the vapor at that velocity. The flooding of the trays must be slowed down at a slow and controlled pace. The steps to successfully accomplish this are shown below:

1. Change all controls still in cascade mode to automatic or manual mode.
2. Decrease steam flow rate to the recirculating evaporator (FIC AW126/226-01) with increments of 5-10 kg/hr.

3. Close the steam supply to the pre-heater valve (FIC AW125/225-01) followed by closing the feed control valve (FIC AW125/225-02) to stop feeding in new mixture into the column.
4. Decrease reflux flow rate to accommodate the decrease in steam rate to keep the constant level in the buffer tank AB101/201.
5. Close the distillate EtOH-rich product return valve (LIC AB101/201-01) in order to avoid collecting poor quality product.
6. Close the bottoms water-rich product return valve (LIC AW126/226-01).
7. Keep the distillation columns operating on total reflux which is the same condition when the column is first starting up (see Section 3.3.3).
8. Decrease steam and reflux flow rates further if the column's flooding trays have no signs of improving.

3.5.2 Shutting Down the Distillation Columns and Pilot Plant

The shutdown procedure is done in the order shown below:

1. All automatic and cascade controls are put back into MANUAL mode
2. Steam to the feed pre-heater AW125/225 is shut-down
3. Feed valve is closed followed immediately by closing steam to the recirculation evaporator AW126/226. **Note:** The manual valves for steam flow must also be closed in that order to assure no steam is escaping through the control valves.
4. Distillate EtOH-rich product return control valve is closed (LIC AB101/201-01).
5. Reflux control valve is remained open but decreased to about 30% to cool down the trays of the distillation column AK122/222. Keep the valve open until the

temperature of the 20th tray gets below 65°C then stop distillate pump AP123/223 followed by closing the reflux valve.

6. The bottoms water-rich return control valve is also remained open in order to keep the bottom level of AK122/222 and AW126/226 from filling up. The valve is remains open until level gets to 72% (or as low as possible while having the heating coils covered with liquid), then the bottoms pump AP222 is shut down followed by closing the bottoms water-rich return valve (LIC AW126/226-01).
7. The coolant valves remain open for at least 10 minutes; if there is no more condensation inside the condenser AW127/227, the coolant flow rate can be reduced to 200 L/hr.
8. Shut down the boiler, do an automatic blowdown, close steam supply valve, and refill the blowdown tank back with water.
9. Shut down all remaining equipment still running on tank farm T110.
10. Close all manual valves except the ones used for adjusting pressure outlet of pumps (recirculation valves) and nitrogen blanketing into the distillation columns

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Introduction

This chapter presents the data collected and analyzed to explore flooding phenomena in the bubble-cap tray distillation column, weeping in sieve tray distillation column, determine the stable operating conditions, and compare the differences between the two distillation columns. The columns and their operation were described in Chapter III of this thesis. The flooding phenomenon only applies to the bubble-cap tray distillation column and weeping only applies to the sieve tray distillation column. These phenomena seem to be due to larger tray spacing between certain trays and not having the proper downcomer in those trays. The columns were started up using the procedures shown in the previous chapter and several experiments were conducted to gather the data consisting of concentrations, densities, flow rates, temperatures, and the differential pressure across the distillation columns. The differential pressure was very important in predicting the initiation stage of flooding in the bubble-cap tray distillation column. The weeping points of the sieve trays were observed visually and the data were collected to analyze the effects of this phenomenon on the distillation process. The concentration, density, and flow rates of the liquid streams in the distillation process were used to develop a stable operating curve for each distillation column. These data were also used to make production curves and to compare the energy usage versus the production rate for the two distillation

columns. The production goals and some constant average values in the process are listed in table 4.1:

Table 4.1 Production Goals

Production Goals	Concentration EtOH wt%	Density (kg/L)	Flow rate (kg/hr)	Flow rate (L/hr)
Feed	50	0.914	80	88
Distillate	90	0.814	40	49
Bottoms	1	0.998	40	40

All of the analyses are done based on these production goals. The steam supply to the distillation columns is at a pressure of 2 barg. The pressure in the column is approximately 1 atm, but the differential pressure from the bottom to the top of the columns can vary anywhere from 0 to 0.15 barg.

4.2 Bubble-cap Tray Distillation Column

4.2.1 Analysis of Flooding Phenomena

The flooding phenomena in the bubble-cap tray distillation column were analyzed using three constant reflux ratios and following the procedure from Chapter III to try to reach the production goals shown in table 4.1. The three experiments will discuss the actions taken leading to flooding and negative effects of the flooding on the process. These experiments which include the feed tray used, feed rate, and the reflux ratio at the time of flooding are shown below:

1. Experiment of March 8, 2012
    a. Feed Tray: Tray 10

b. Feed Rate: 40 L/hr (37 kg/hr)

c. Reflux Ratio: 3:1

In this experiment, the 6th Tray had excessive entrainment the whole time until 15:00. The video footage of this heavy entrainment can be found at:

http://doiop.com/Ozkaya-Vid-01

At approximately 15:00, tray 6 temperature suddenly rose to 100 ^{o}C (boiling point of water) after steam rate @ 2 barg was raised from 37 kg/hr to 38 kg/hr. This means that all the ethanol that was entrained in that tray was suddenly removed as shown by the following video footage:

http://doiop.com/Ozkaya-Vid-02

This event also led to a sudden differential drop at approximately 15:05 as it can be seen from the red trend line in figure 4.1. Also from figure 4.1, the temperature of tray 6 also began oscillating leading up to the sudden differential pressure drop. The sudden differential drop caused sudden temperature spike of the trays above tray 6. The trend lines shown in figure 4.1 are labeled as:

- Tray 1 temperature – Black line
- Tray 6 temperature – Dark blue line
- Tray 11 temperature – Brown line
- Tray 16 temperature – Pink line
- Tray 20 temperature – Light blue line
- Differential pressure – Red line

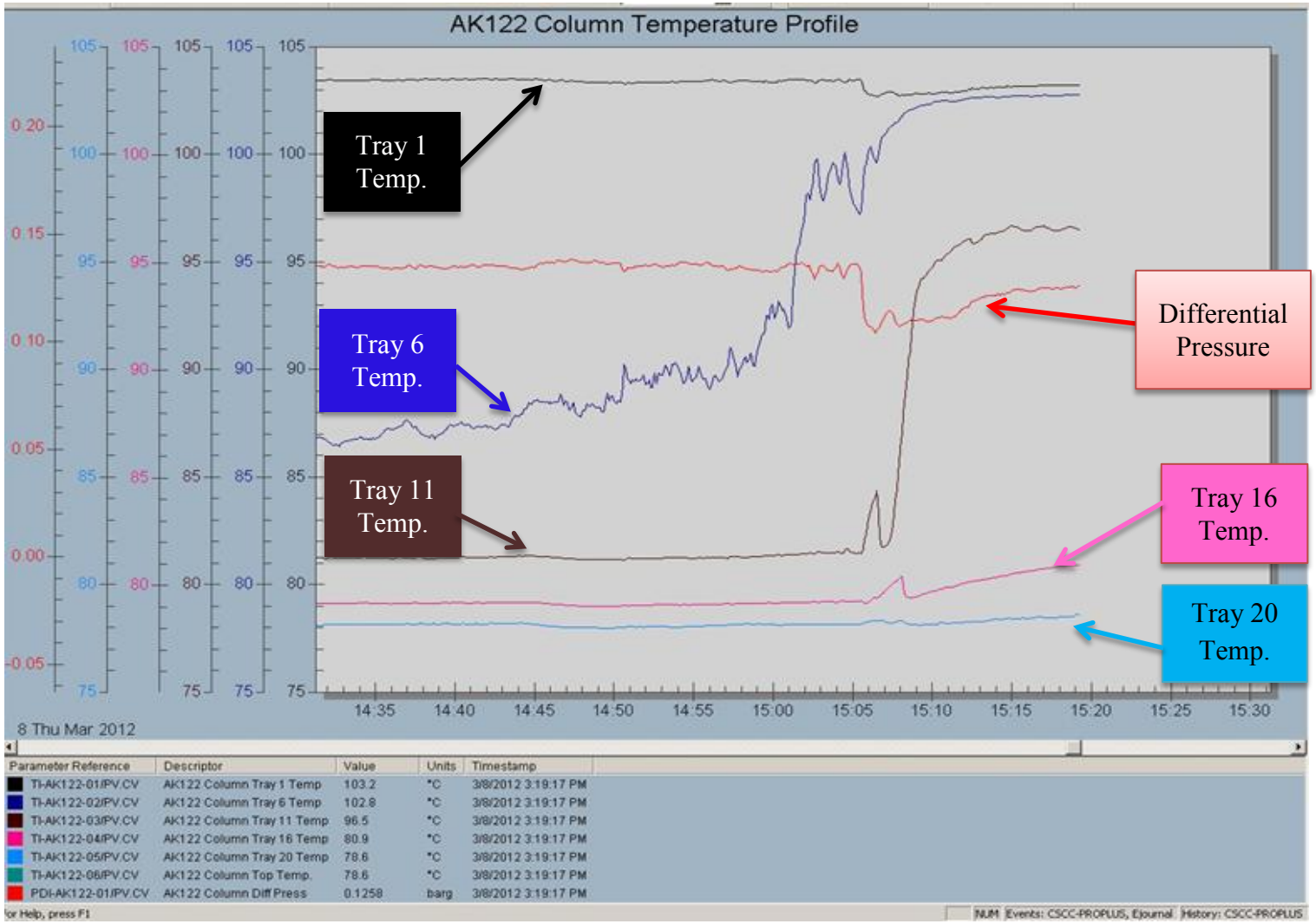

Figure 4.1

Temperature and Differential Pressure Profile of AK122 for Experiment 1

The ethanol that was removed from tray 6 shot its way up the column where all 5 top trays began flooding. This can be noticed from the graph where trays 11 and 16 temperatures suddenly started to rise with the differential pressure rising from the time 15:07 on. The screen snap shot of the DeltaV DCS screen at 15:19 is shown in figure 4.2. This snap shot of the DeltaV DCS screen shows the process variables at the time when flooding started to occur. The feed flow rate at this time was 40 L/hr (shown by FIC-AW125-02) and steam flow rate was 38 kg/hr (FIC-AW126-01). Description of the lines and values in figure 4.2 is stated below:

- Feed flow – White solid line
- Steam flow – Orange solid line
- Water-rich bottoms product flow – Blue solid line
- EtOH-rich distillate product flow – Green solid line
- Reflux flow – White solid line
- Coolant flow – Pink solid line
- Vent gas – Yellow solid line
- Control variables – Blue values
- Process variables – Yellow values
- Set-Points – White values
- Control loop connections – Dashed lines (blue, yellow, and white)

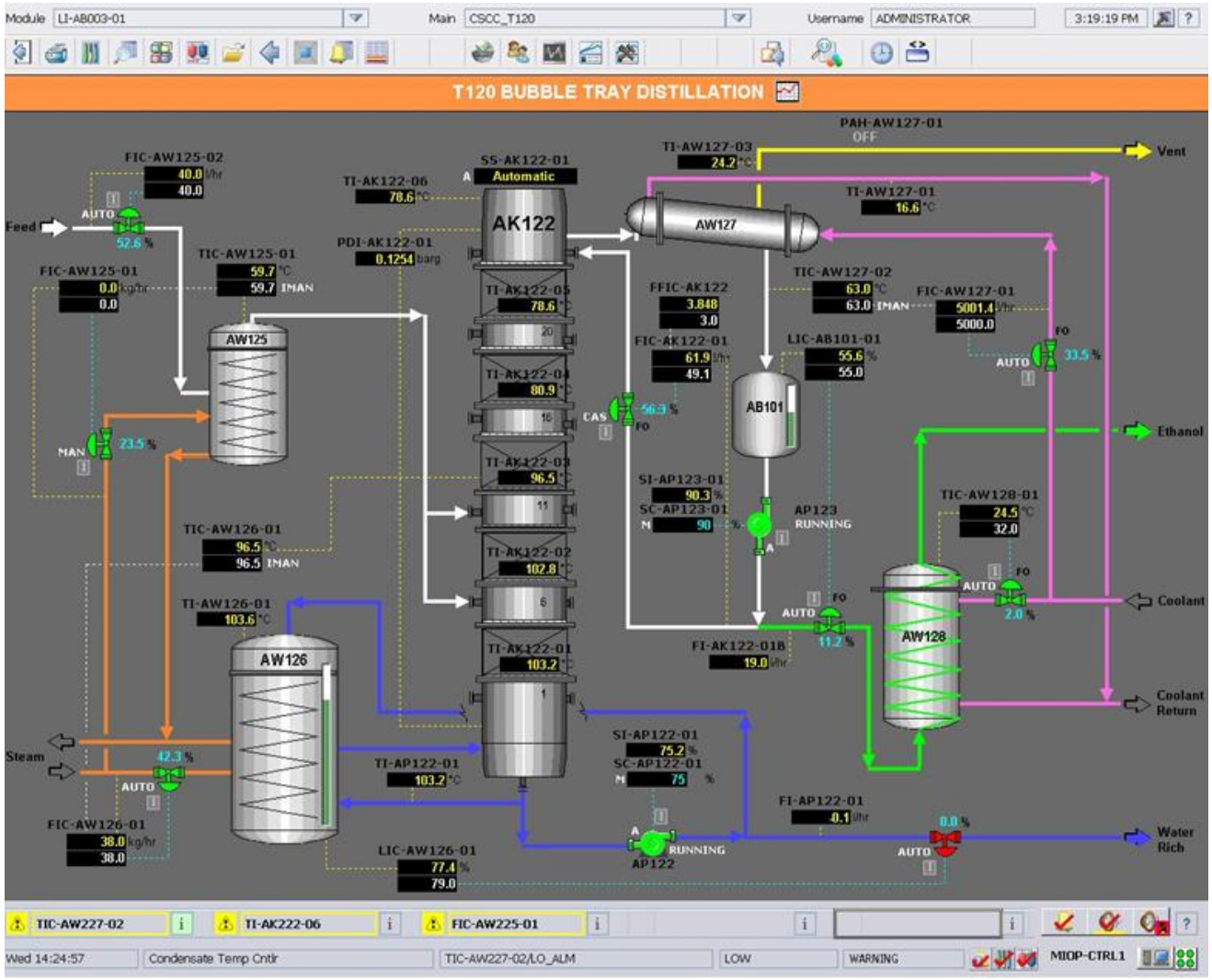

Figure 4.2

Screen Snapshot of T120 at 15:19 for Experiment 1

The video footage of the top of the column fully flooded was recorded at 16:55 and can be found at:

http://doiop.com/Ozkaya-Vid-03

The flow conditions leading up to this event is shown in figure 4.3. As soon as the top five trays started to flood, the bottoms flow rate (AP122 Flow) indicated by the blue line went down to 0 L/hr. This was due to the reflux and feed being trapped at the top of the column because of the flooding of the trays. This lead to very small amount of liquid coming down the column, causing the control valve for bottoms water-rich product return (LV-AW126-01) to shut since the bottom level of the column was being automatically controlled at 79% level. This is shown by the snapshot of the PCS screen in figure 4.2 and the flow conditions in figure 4.3.

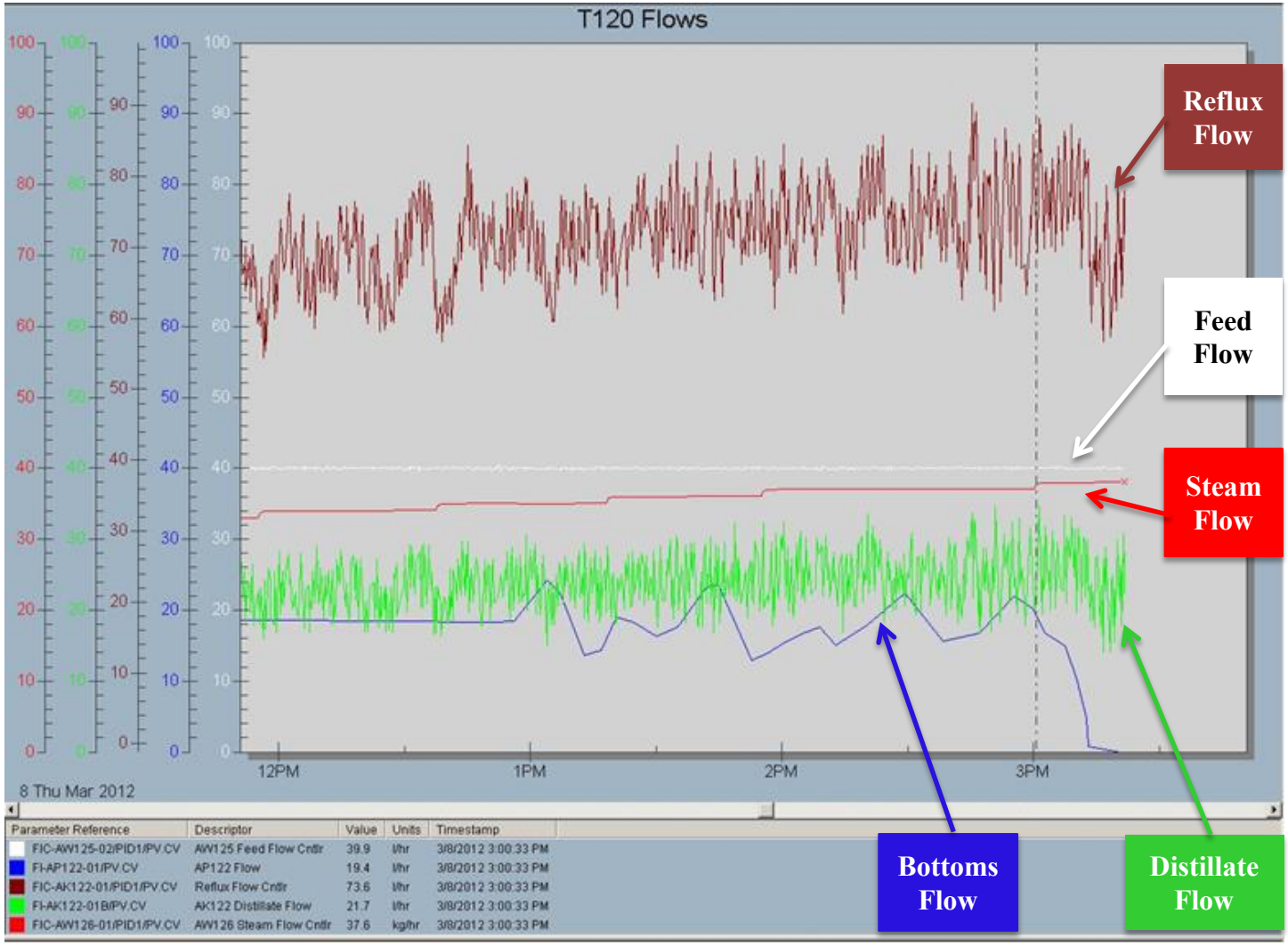

Figure 4.3

Flow Conditions of AK122 for Experiment 1

Actions Taken Leading to Flooding

- Steam flow rate being increased to 38 kg/hr
- Average reflux flow rate increasing to 75 L/hr

Negative Effects on the Process

- The purity of the top product dropped from 91.6 wt% EtOH at 14:25 PM to 89.1 wt% EtOH at 15:21 PM, then to 86.0 wt% EtOH at 15:35 PM.
- Tray temperatures of column became too high.
- Liquid level at the bottom of the column got too low

2. Experiment of March 7, 2012
    a. Feed Tray: Tray 10
    b. Feed Rate: 60 L/hr (55 kg/hr)
    c. Reflux Ratio: 2:1

On this experiment, the first abnormal drop in differential pressure and rise in tray temperatures was noticed at approximately 15:55 (see figure 4.4). Soon after this abnormal behavior, the differential pressure starts to spike up and down at approximately 16:05 (see figure 4.4). This resulted in sudden rise in the temperature of the trays as the differential pressure drops suddenly since pressure and temperature are directly related. This condition was the initiation of flooding in trays 16, 17, 18, and 19. The video footage of this initiation stage can be seen at:

http://doiop.com/Ozkaya-Vid-04

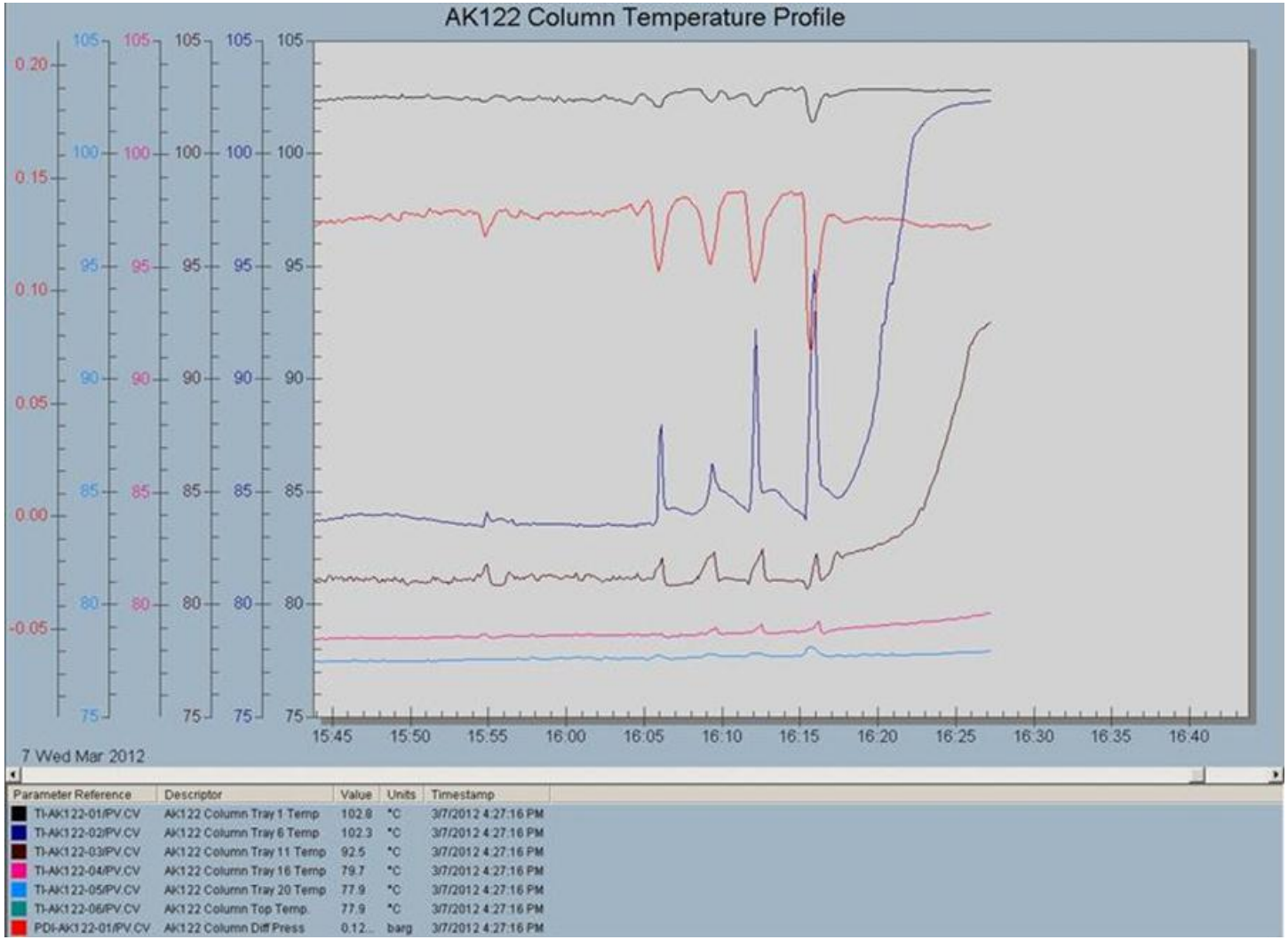

Figure 4.4

Temperature and Differential Pressure Profile of AK122 for Experiment 2

The flow conditions leading up to this event is shown below in figure 4.5:

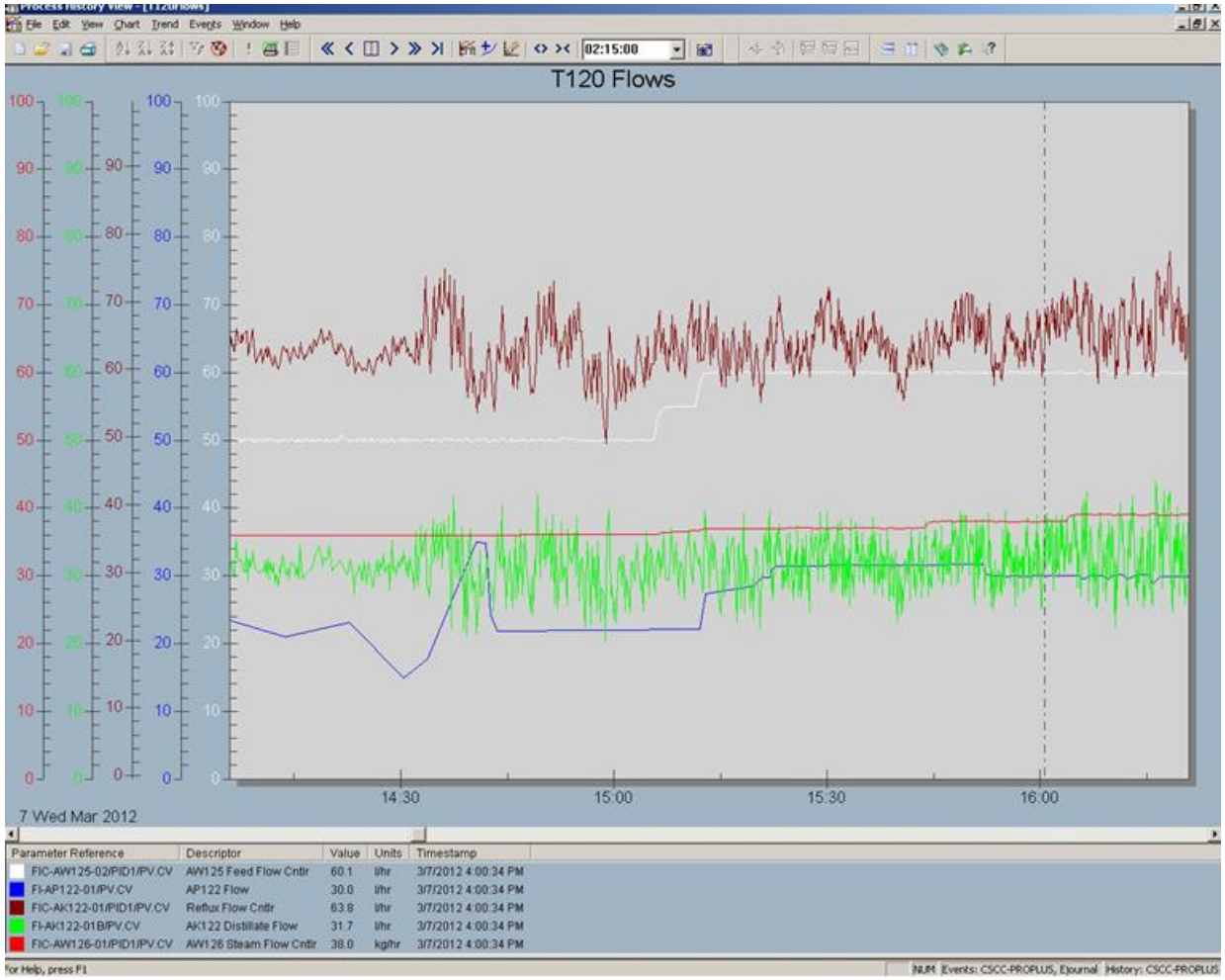

Figure 4.5

Flow Conditions of AK122 for Experiment 2

Keeping the process conditions same resulted in those 4 trays to be fully flooded at around 16:17. This can be seen in figure 4.4 by when differential pressure gets steady and trays 6 and 11 suddenly rise in temperature due to entrainment of the reflux in the top of the column. The video footage of this event can be found in the following link:

http://doiop.com/Ozkaya-Vid-05

Actions Taken Leading to Flooding

- Steam flow rate being increased to 39 kg/hr
- Average reflux flow rate increasing to 66 L/hr

Negative Effects on the Process

- The purity of the top product dropped from 90.7 wt% EtOH at 16:02 to 89.4 wt% EtOH at 16:25.
- Temperatures at the stripping section of column became too high.
- Liquid level at the bottom of the column got too low

3. Experiment of March 19, 2012
    a. Feed Tray: Tray 5
    b. Feed Rate: 70 L/hr (64 kg/hr)
    c. Reflux Ratio: 1.5:1

The temperature and differential pressure profile of the bubble-cap tray distillation column during this experiment is shown by figure 4.6:

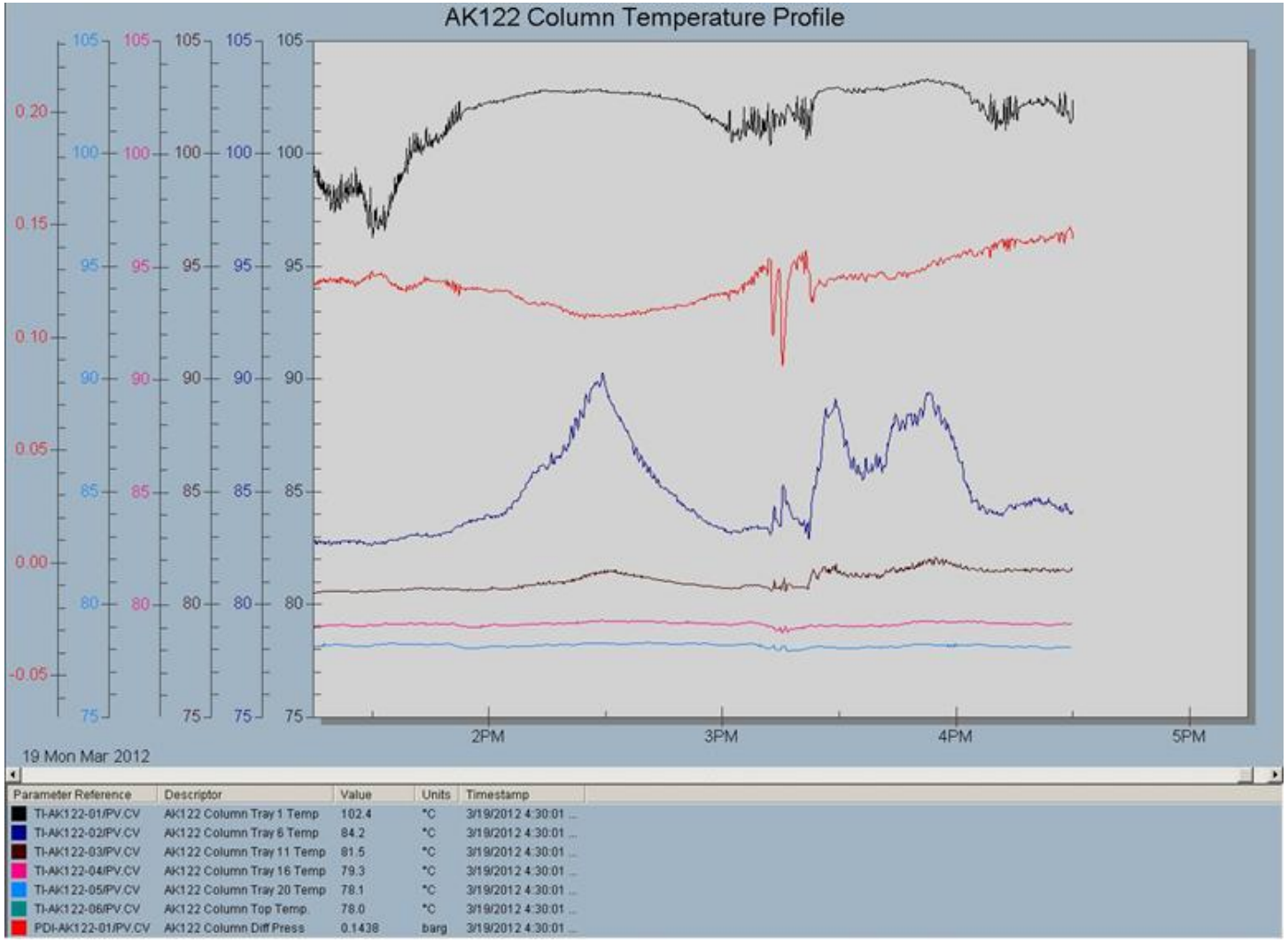

Figure 4.6

Temperature and Differential Pressure Profile of AK122 for Experiment 3

Following the temperature and differential pressure trends in figure 4.6, a spike and oscillation can be noticed around 15:15 on the differential pressure and temperatures of the trays. This was the initiation of flooding at the top of the bubble tray distillation column. The video footage of this event at 15:15 can be seen at the following link:

http://doiop.com/Ozkaya-Vid-06

The flow conditions for this experiment are shown in figure 4.7. These figures show how the distillation column is brought to stable operation and end up in flooding. The feed flow rate is increased when tray 6 begin to increase in temperature. This is continued until tray 6 reaches a stable temperature meaning that the tray is at steady state conditions. Once this is the case, feed flow rate is continued to be increased. Once tray 6 temperature begins to decrease, steam flow rate is increased to keep tray 6 at a stable temperature around 82 ^{o}C again. This process is described in the procedures (see Section 3.4) and is continued until flooding is observed. The differential pressure and the tray temperatures oscillate during the time of flooding, which is a phenomenon that should be investigated further.

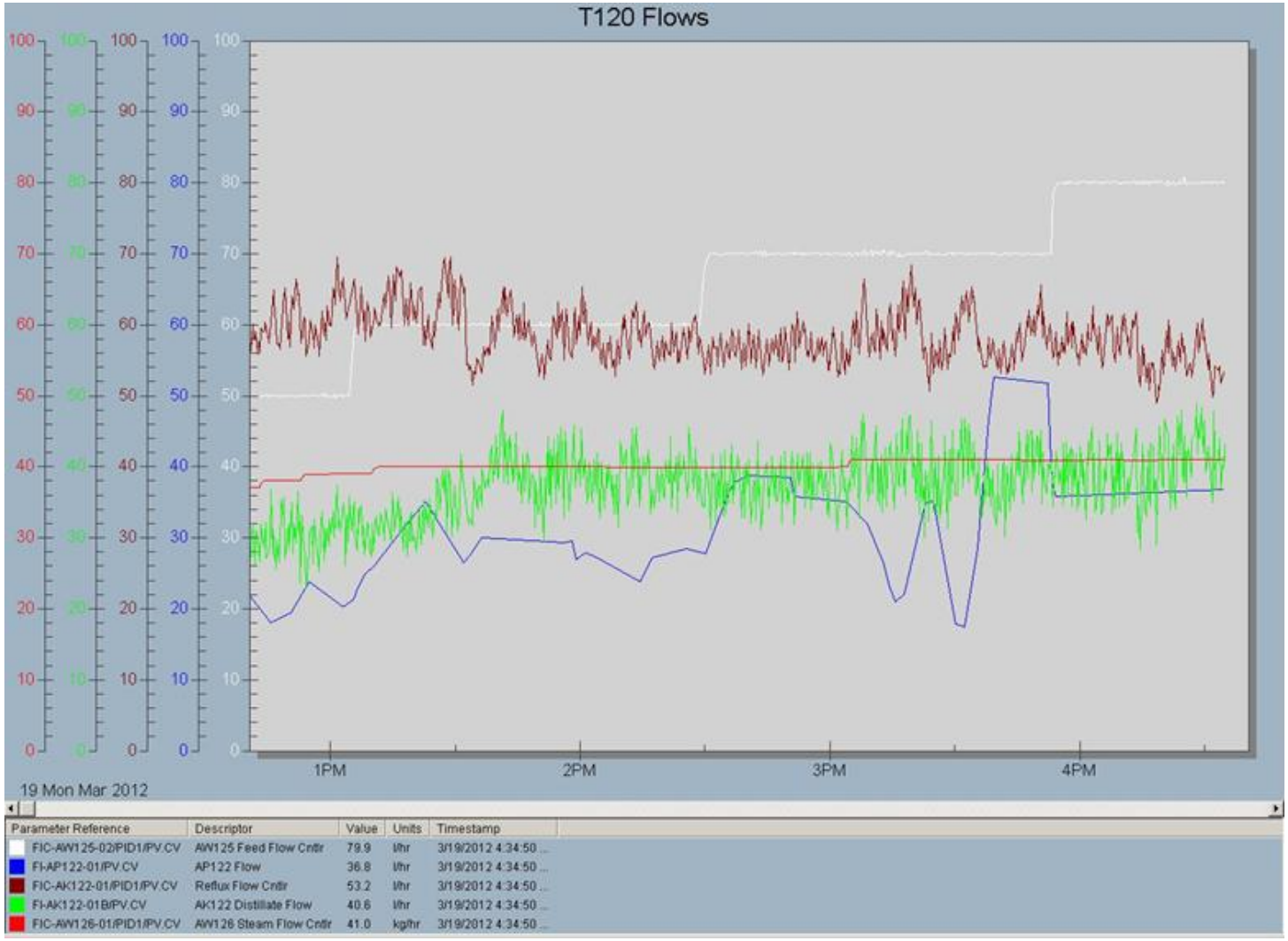

Figure 4.7

Flow Conditions of AK122 for Experiments 3

Actions Taken Leading to Flooding

- Steam flow rate being increased to 40 kg/hr

Negative Effects on the Process

- The purity of the top product dropped from 90.6 wt% EtOH at 15:30 to 90.2 wt% EtOH at 16:00, then to 88.5 wt% EtOH at 16:15.

4.2.2 Stable Operating Curve

The stable operating region analysis was accomplished by running the distillation column 100 different days. The stable conditions were achieved by following the procedure described in Section 3.4.4. The table shown below consists of the average flow rates for the steam, feed, reflux, distillate, and bottoms product found for the stable operation, and flooding (unstable operation) of the bubble-cap tray distillation column (AK122):

Table 4.2 AK122 Average Flow Rates for Stable Operation and Flooding

Bubble-cap Tray	Steam Flow Rate (kg/hr)	Feed Flow Rate, F (L/hr)	Reflux Flow Rate, R (L/hr)	Distillate Flow Rate, D (L/hr)	Bottoms Flow Rate, B (L/hr)	Reflux Ratio, R/D
Stable Operation	25	30	48	16	14	3
	27	32	54	18	15	3
	30	40	55	22	19	2.5
	32	48	54	27	22	2
	34	53	60	30	24	2
	35	65	56	37	29	1.5
	36	70	59	39	32	1.5
Flooding	38	40	75	25	16	3
	39	60	66	33	28	2
	40	70	60	40	31	1.5

The flooding point flow rate values are the results obtained from the flooding analysis experimentation shown previously. Developing a stable operating region consists of calculating the total amount of vapor going up the distillation column into the condenser and the total amount of liquid coming back down the distillation column. Since the density of the liquid and vapor streams are different, this analysis cannot be done using the volumetric flow rate results shown in table 4.2.1. The only way to accomplish the analysis is by using either mass or molar flow rates. The density readings taken for the feed, distillate EtOH-rich product, and bottoms water-rich product were used to convert the volumetric flow rates into mass flow rates for the analysis. The average densities shown previously in the production goals table (see Table 4.1) were used for this conversion. The results from this are shown below in table 4.3:

Table 4.3 AK122 Average Flow Rate Conversions from L/hr to kg/hr for Stable Operation and Flooding

Bubble-cap Tray	Steam Flow Rate (kg/hr)	Feed Flow Rate, F (kg/hr)	Reflux Flow Rate, R (kg/hr)	Distillate Flow Rate, D (kg/hr)	Bottoms Flow Rate, B (kg/hr)	Liquid Flow Rate, F+R (kg/hr)	Vapor Flow Rate, R+D (kg/hr)	Reflux Ratio, R/D
Stable Operation	25	27	39	13	14	66	52	3
	27	29	44	15	15	73	59	3
	30	37	45	18	19	81	63	2.5
	32	44	44	22	22	88	66	2
	34	48	49	24	24	97	73	2
	35	59	45	30	29	105	75	1.5
	36	64	48	32	32	112	79	1.4
Flooding	38	37	61	20	16	98	81	3
	39	55	54	27	28	109	81	2
	40	64	49	33	31	113	81	1.5

The liquid flow rate (F+R) from table 4.3 is found by adding the amount of feed (F) and reflux (R) entering the distillation column. The vapor flow rate (R+D) entering the condenser is calculated by adding the amount of reflux and distillate leaving the buffer tank AB101. The reason for this is that since the level in AB101 is being automatically controlled by the distillate EtOH-rich product return valve and reflux flow valve is in cascade control to always keep a constant reflux ratio, the amount of vapor going into the condenser and turning into liquid to fall into the buffer tank must be same rate as what is coming out of the buffer tank if the level is staying constant. This concept is also explained in the procedure for total reflux and continuous distillation (see Sections 3.3.3 and 3.4.1). A graph was developed using the total vapor and liquid rates in the bubble-cap tray distillation column (AK122), shown in figure 4.8:

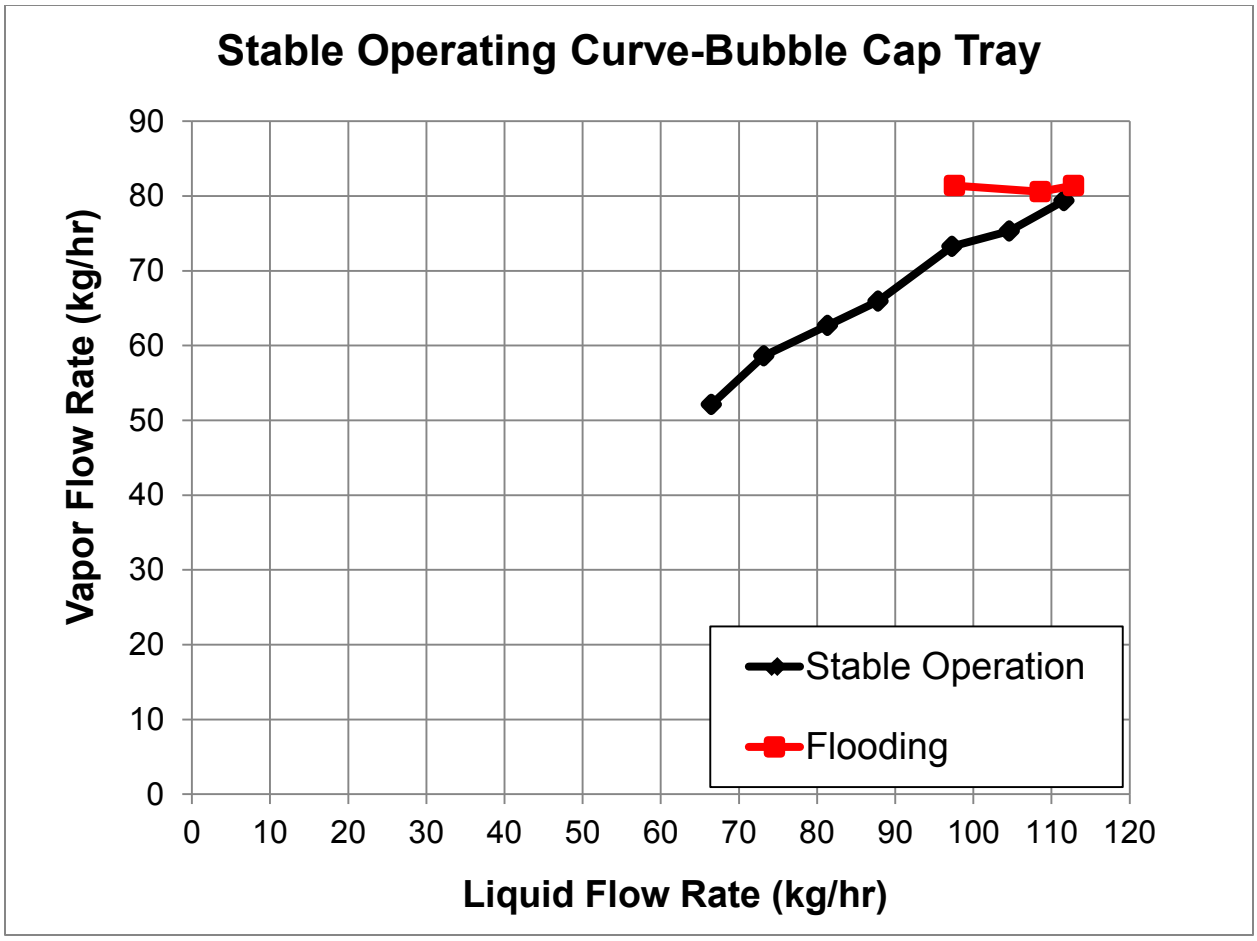

Figure 4.8

Stable Operating Curve for AK122

The black line in figure 4.8 is the stable region for operation (or stable operation region). The red line indicates flooding of the distillation column, or unstable operating region. The flooding region of AK122 is shown to be at a vapor flow rate of approximately 81 kg/hr. This region represents an upper limit for the operation of the bubble-cap tray distillation column.

Using the stable operating region results and average volumetric flow rate values from this region shown in table 4.2, a production curve was developed using the steam mass flow rate as the independent variable and liquid volumetric flow rates in the column (feed, reflux, distillate

EtOH-rich product, and bottoms water-rich product) for stable operation. This production curve for AK122 is shown in figure 4.9. The production curve shown above will be very useful for anyone operating the bubble-cap tray distillation column. For any given steam flow rate, the production curve in figure 4.9 will give the approximate liquid flow rate the distillation column should have for stable operation. It will also help operators of the distillation column to figure out what the production flow rates (EtOH-rich distillate and water-rich bottoms products) should be at a given feed and steam flow rate for stable operation. This is usually not that easy for operators to figure out since not all volumes are additive (for example EtOH and water) and the production flow rates will not always add up to the amount of feed going into the distillation column. Also, the more volatile compound (EtOH) with less density will have a higher volumetric flow rate then the less volatile compound with higher density using a 50/50 by weight mixture in the feed. This flow rate difference of the products is shown in figure 4.9.

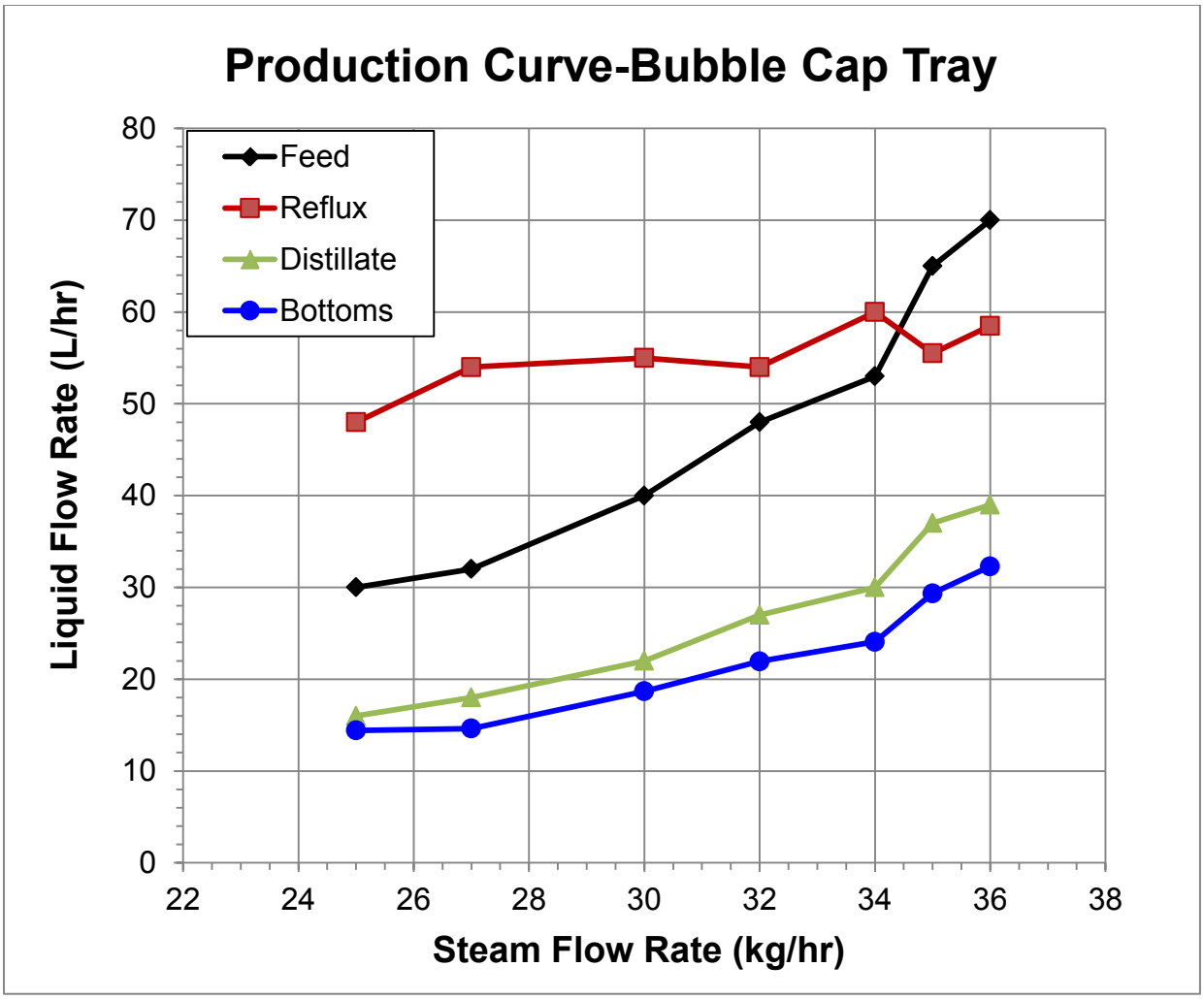

Figure 4.9

Production Curve for AK122

The y-axis of the figure above shows the liquid flow rates going in and out of the bubble-cap tray distillation column in L/hr. The x-axis shows the steam flow rate sent to the recirculating evaporator AW126. The reflux flow line, shown by the red line, does not increase steadily like the other flow lines due to reflux ratio decreased throughout the stable operation of the bubble-cap tray distillation column. Since the reflux flow rate is lowered due to lower reflux ration, the reflux flow line crosses the feed flow line in figure 4.9 because the feed flow rate becomes

higher than the reflux flow rate. The black line is the feed flow rate, the green line is the EtOH-rich distillate product flow rate, and the blue line is the water-rich bottoms product flow rate. The minimum steam flow rate is 25 kg/hr and maximum is 36 kg/hr for stable operation.

4.3 Sieve Tray Distillation Column

4.3.1 Analysis of Weeping Phenomena

The weeping points of the sieve tray distillation column (AK222) were found by visually inspecting the sieve trays at total reflux and checking the purity of the top distillate EtOH-rich product. The procedure for this analysis is described in Section 3.4.4. The classification of weeping for AK222 was to have all trays having liquid going down from their perforations. This is the case for trays 6, 11, and 16 at all times throughout the distillation process, but this inconsistency will be explained later in the chapter. The results for weeping points at total reflux are shown below in table 4.4:

Table 4.4 AK222 Weeping Points at Total Reflux

Steam Flow (kg/hr)	**Reflux Flow (l/hr)**	**Reflux Ratio**	**EtOH wt%**
20	30	Infinity	89.8
28	50	Infinity	90.9
32	70	Infinity	92.1
32	80	Infinity	92.2
34	100	Infinity	93.5
36	120	Infinity	94.6

From the weeping point results shown in table 4.4, the EtOH purity of the top distillate product meets the production goal purity (greater than 90 wt% EtOH) for all of the steam and reflux flow rate combinations except for the lowest combination (20 kg/hr steam and 30 L/hr reflux). It is also noticed that the purity of the top distillate product increased as the reflux flow rate increased. As a result, the weeping point analysis at total reflux did not have a significant effect on the distillate purity. This was only the results for keeping total reflux at a constant reflux flow rate and decreasing steam until weeping is visually observed. Operating the sieve tray distillation column through a normal start-up has shown that at low flow rates of steam (less than 35 kg/hr) and reflux (less than 70 L/hr) leads to low concentrations of EtOH on the top distillate product (less than 90 wt%).

4.3.2 Stable Operating Curve

The stable operating region analysis was done following the same guidelines discussed for the bubble-cap tray distillation column results in section 4.3.2. There is no flooding that occurs in the sieve tray distillation column due to other upper limits which will be discussed in the later sections. Table 4.5 consists of the average flow rates for the steam, feed, reflux, distillate, and bottoms product found for the stable operation of the sieve tray distillation column (AK222):

Table 4.5 AK222 Average Flow Rates for Stable Operation

Sieve Tray	Steam Flow Rate (kg/hr)	Feed Flow Rate, F (L/hr)	Reflux Flow Rate, R (L/hr)	Distillate Flow Rate, D (L/hr)	Bottoms Flow Rate, B (L/hr)	Reflux Ratio, R/D
Stable Operation	40	45	78	26	20	3
	45	50	84	28	23	3
	48	55	93	31	25	3
	50	60	99	33	28	3
	52	65	108	36	30	3
	54	70	109	39	32	2.8
	55	80	119	44	37	2.7
	58	90	125	50	42	2.5

Table 4.5 above shows the recommended operation of the distillation column on total reflux highlighted in green and the rest of the table indicates the average flow rates of stable operation of the sieve tray distillation column. Converting the flow rates into mass flow rates, as it was done for AK122 in section 4.2.2, results in the table of average flow rates for AK222 shown in table 4.6:

Table 4.6 AK222 Average Flow Rate Conversions from L/hr to kg/hr for Stable Operation

Sieve Tray	Steam Flow Rate (kg/hr)	Feed Flow Rate, F (kg/hr)	Reflux Flow Rate, R (kg/hr)	Distillate Flow Rate, D (kg/hr)	Bottoms Flow Rate, B (kg/hr)	Liquid Flow Rate, F+R (kg/hr)	Vapor Flow Rate, R+D (kg/hr)	Reflux Ratio, R/D
Stable Operation	40	41	63	21	20	105	85	3
	45	46	68	23	23	114	91	3
	48	50	76	25	25	126	101	3
	50	55	81	27	28	135	107	3
	52	59	88	29	30	147	117	3
	54	64	89	32	32	153	121	2.8
	55	73	97	36	37	170	133	2.7
	58	82	102	41	42	184	142	2.5

The calculation of these results including the total liquid and vapor flow rates were done as described in section 4.2.2 for AK122. Just as it was done for AK122 in section 4.2.2, the stable operating curve and the production curve developed for AK222 using tables 4.5 and 4.6 are shown in figures 4.10 and 4.11.

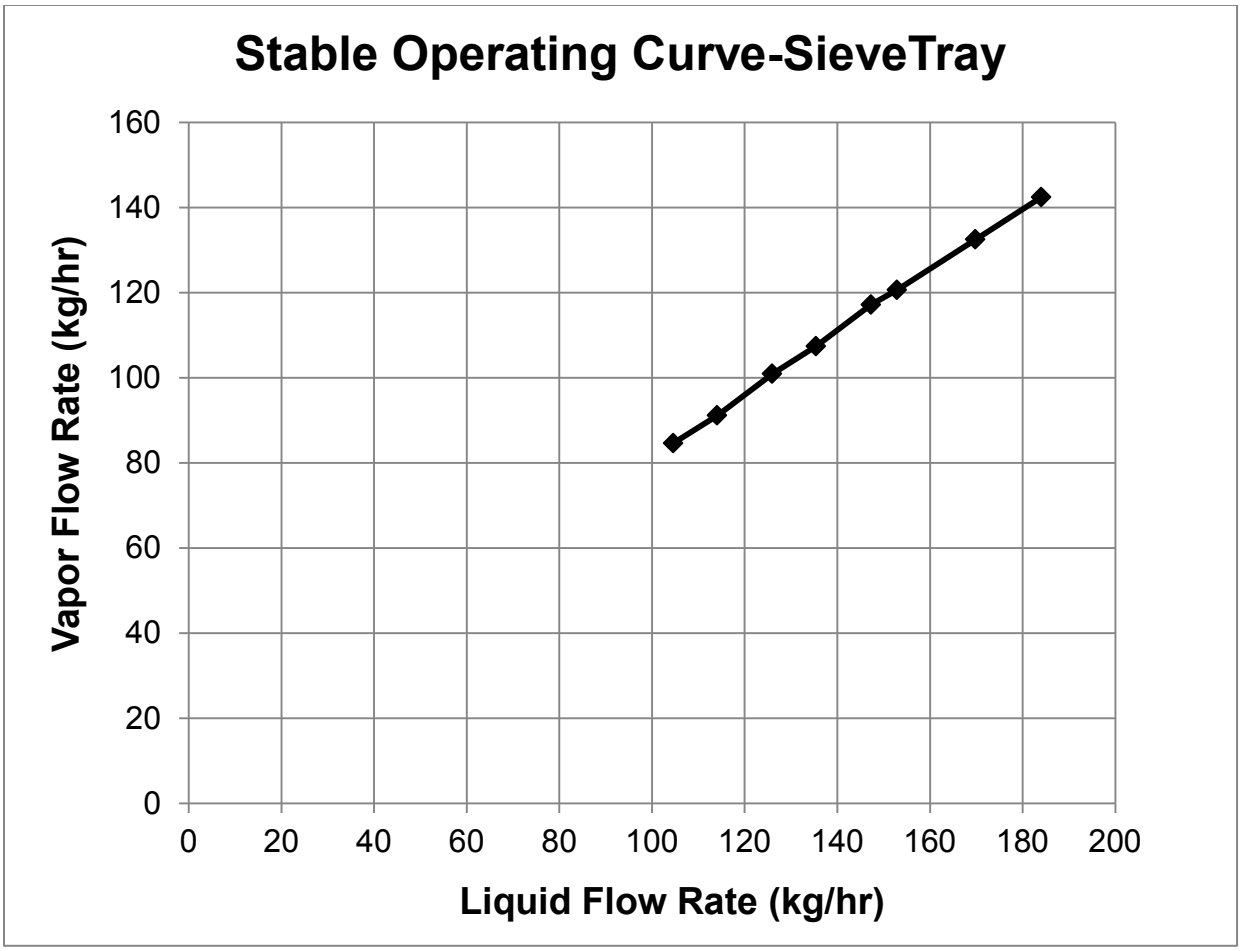

Figure 4.10

Stable Operating Curve for AK222

The vapor flow rate in kg/hr is on the y-axis and the liquid flow rate in kg/hr is on the x-axis of figure 4.10. The vapor flow rate is the total amount of vapor going into the condenser AW226, turning into a liquid, and being collected in the buffer tank AB201. This flow rate is found by the addition of reflux and EtOH-rich distillate product flow rates as described previously. The liquid flow rate is the addition of feed and reflux flow rates. The stable operating curve in figure 4.10 begins at a liquid flow rate of 105 kg/hr and a vapor rate of 85 kg/hr. The curve has a linear behavior and ends at a liquid flow rate of 184 kg/hr and a vapor rate of 142 kg/hr.

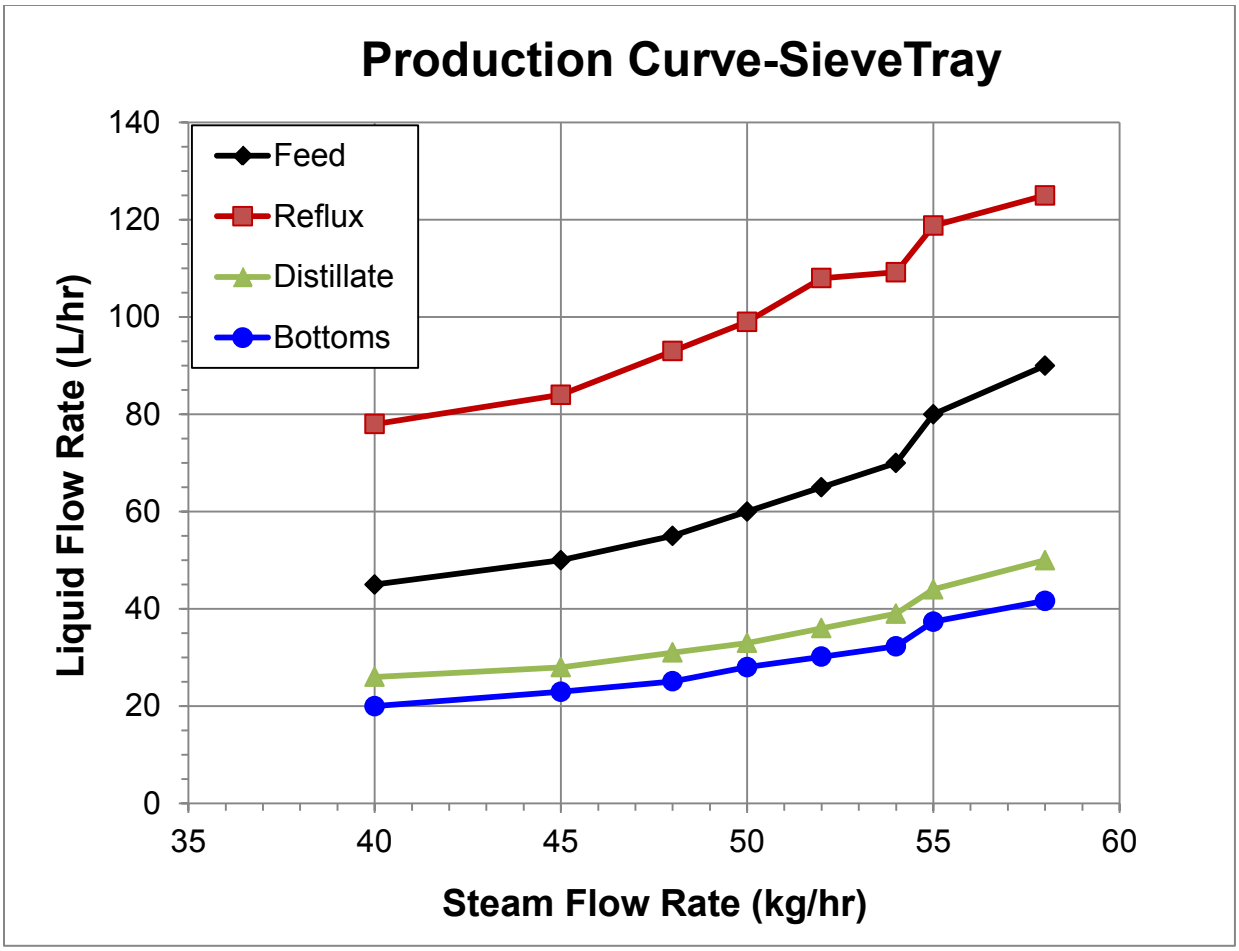

Figure 4.11

Production Curve for AK222

The production curve above in figure 4.11 shows the liquid flow rates (y-axis) and steam flow rates (x-axis) for stable operation of the sieve tray distillation column (AK222). All of the flow lines including feed, reflux, EtOH-rich distillate product, and water-rich bottoms product flows increase as the steam rate to the recirculating evaporator AW226 increases. The minimum steam flow rate is 40 kg/hr and the maximum is 58 kg/hr for stable operation of AK222.

4.4 Comparison of the Distillation Columns

The bubble tray distillation column (AK122) and the sieve tray distillation column (AK222) have many differences in their stable operating regions. Each distillation column has its advantages and disadvantages. The comparison of each column's stable operating curve is shown below in figure 4.12:

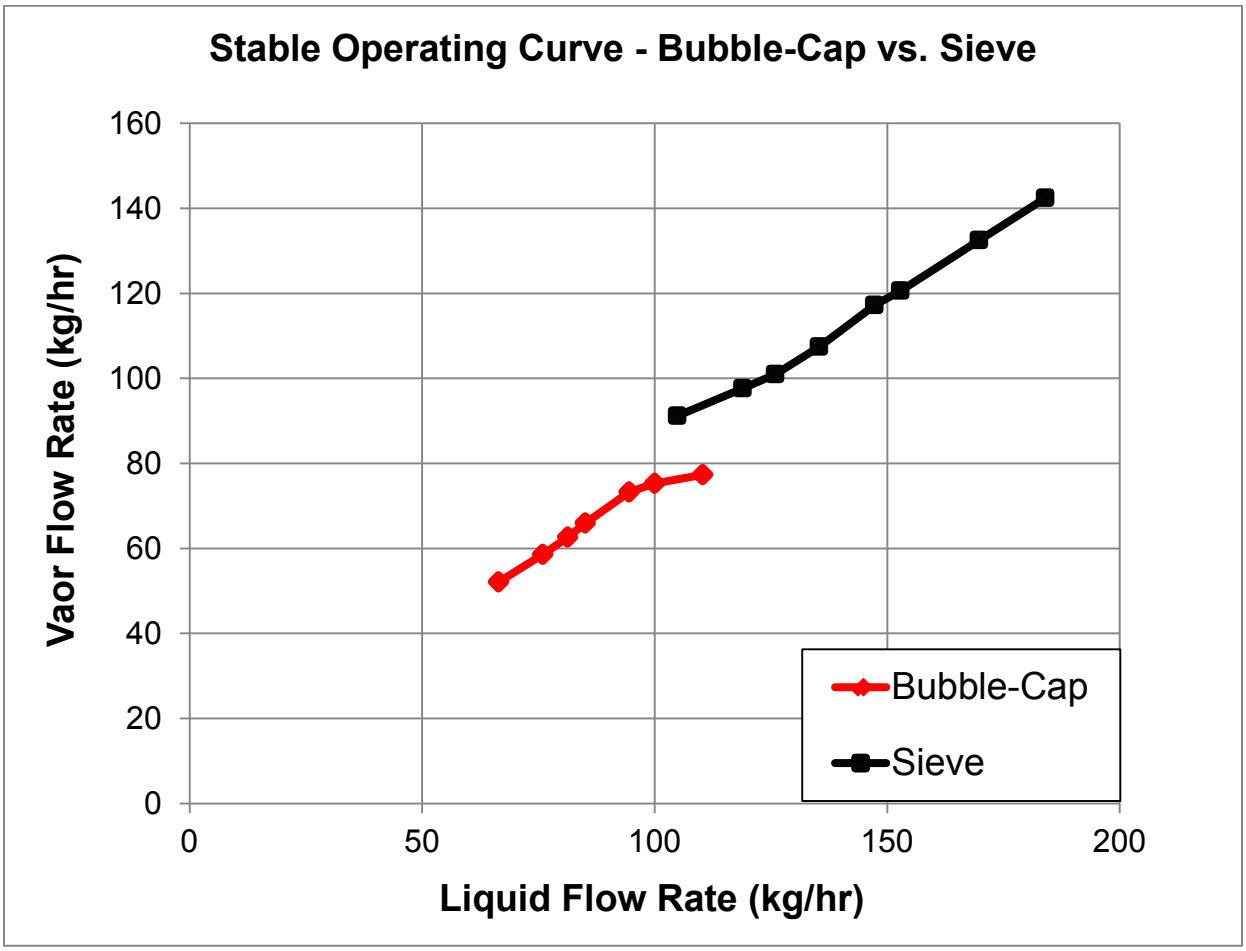

Figure 4.12

Stable Operating Curve Comparison of Bubble-Cap and Sieve

where the red line represents the stable operating curve for the bubble-cap tray distillation column (AK122) and the black line represents the stable operating curve for the sieve tray

distillation column (AK222). The stable operating region for AK222 is at much higher flow rates than AK122 as indicated by figure 4.12. AK122 can achieve higher purity of EtOH-rich distillate product and water-rich bottoms product at much lower flow rates and reflux ratio than AK222. On the other hand, AK222 can handle much higher flow rates into the distillation column and higher production rates of both products. To compare the energy usage of the distillation column to the production rate it can achieve, a plot of the steam flow rate and the EtOH-rich distillate product flow rate is developed to compare AK122 and AK222. Since the production rates of EtOH-rich distillate product and water-rich bottoms product are approximately equal to each other in terms of mass, only the distillate product rate is used in the graph in terms of mass flow rate. This graph is shown in figure 4.13:

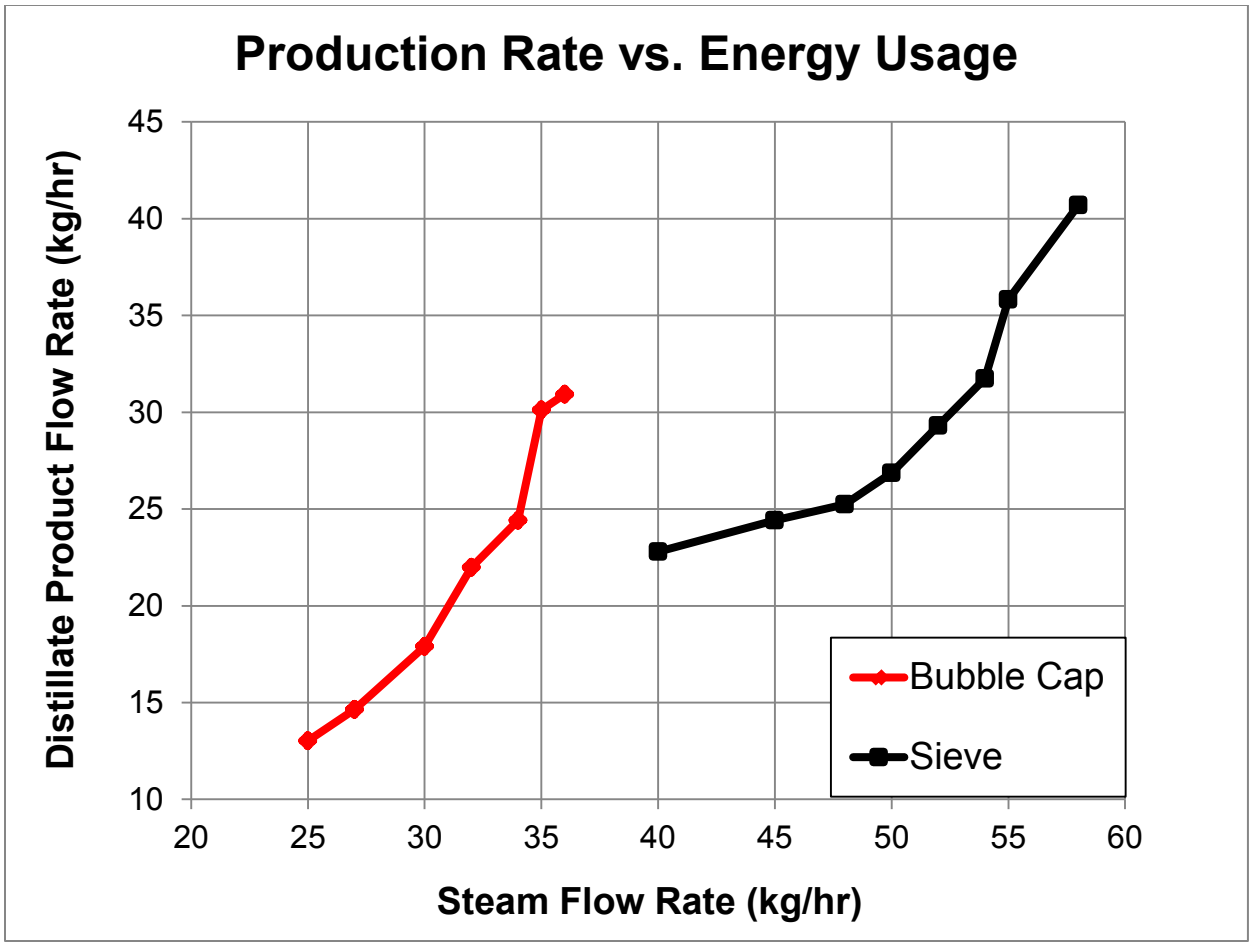

Figure 4.13

Production Rate vs. Energy Usage Comparison of Bubble-Cap and Sieve

4.5 Discussion of the Results

Both of the distillation columns, AK122 and AK222, have identical equipment and dimensions, varying only on the type of tray inside the distillation column. The results shown in the previous sections show how different type of a tray in the column results in very different operating regions.

From figure 4.8, AK122 (bubble-cap) is shown to operate efficiently through very low liquid and vapor flow rates and continues that way until flooding region is reached around 81

kg/hr vapor rate, which is equivalent to approximately 100 L/hr condensate entering the buffer tank AB101. Table 4.2 shows that the reflux ratio can be as low as 1.5 and AK122 (sieve) can still achieve the production goal concentration of EtOH wt% in both of the products (see Table 4.1). This advantage of AK122 is offset by flooding which occurs before the distillate and bottoms products' flow rates can reach the production goals shown in table 4.1 and represents an upper operating limit for the distillation column. The flooding analysis from section 4.2.1 shows that the flooding occurs when the differential pressure in the column rises above 0.13 barg, suddenly drops and rises in the initiation stage of flooding, and continues to rise once all of the top 5 trays are fully flooded. This phenomenon results in reflux being thrown into the condenser AW127 without making its way down the column. The first two experiments (reflux ratio of 3 and 2) were done using tray 10 as the feed tray and the last experiment with a reflux ratio of 1.5 was done using tray 5 as the feed tray. The two experiments using tray 10 as the feed tray had problems with heavy entrainment in tray 6 throughout the distillation process before flooding occurred. The flooding of the top trays was initiated by sharp rise in the lower tray temperatures (trays 6 through 10) as it is seen in figures 4.1 and 4.4. This meant that all of the ethanol being fed into the column was being sent up the column before making its way down to the lower trays. This caused a dangerous situation in the distillation column due to low liquid level at the bottom of the column, high temperatures in the feed tray, and ethanol condensate getting too hot coming out of the condenser into the buffer tank AB101. The third experiment where tray 5 was used as the feed tray, no such sudden temperature rise occurred when the top trays began flooding. In both cases, the distillate product purity dropped suddenly below production standards leading to inoperable conditions for the distillation column. It has also been determined that tray 5 is better to use as the feed tray due to the fact that trays 6 through 10 can flood very

easily at high flow rates when using tray 10 as feed tray, which has never occurred when using tray 5 as the feed tray. The reason for this phenomenon to occur before production goals are met and different behavior while using the higher tray as the feed tray is the design error of certain sections of the distillation column. Even though tray spacing was indicated to be 208 mm is the column specifications (see Table 2.1), the tray spacing between trays 5-6, 10-11, and 15-16 are double the size (416 mm) due to having sectional connections of the column at these locations. The tray spacing in these sections are double the spacing of regular trays, yet the downcomer from at these trays (trays 6, 11, and 16) are the same length and have flat opening at their bottom section leaving a 208 mm air gap from the lower tray to the downcomer. This design flaw results in vapors from the lower trays (trays 5, 10, and 15) to enter the tray above through the downcomer, which is the path of least resistance, and block the liquid on the tray above from easily coming down from the downcomer. At low flow rates; the weight of the liquid on the tray can overcome the force of the vapor going up, but high vapor and liquid rates lead to flooding due to this design flaw of the downcomer. Tray 1, which also has a big air gap below due to bottom of the column and recirculating evaporator AW126 being located there, has a different type of downcomer that has the bottom portion closed and curved up to not allow the vapors rising from AW126 to go through the downcomer. A picture model of this type of downcomer is shown in figure 4.14:

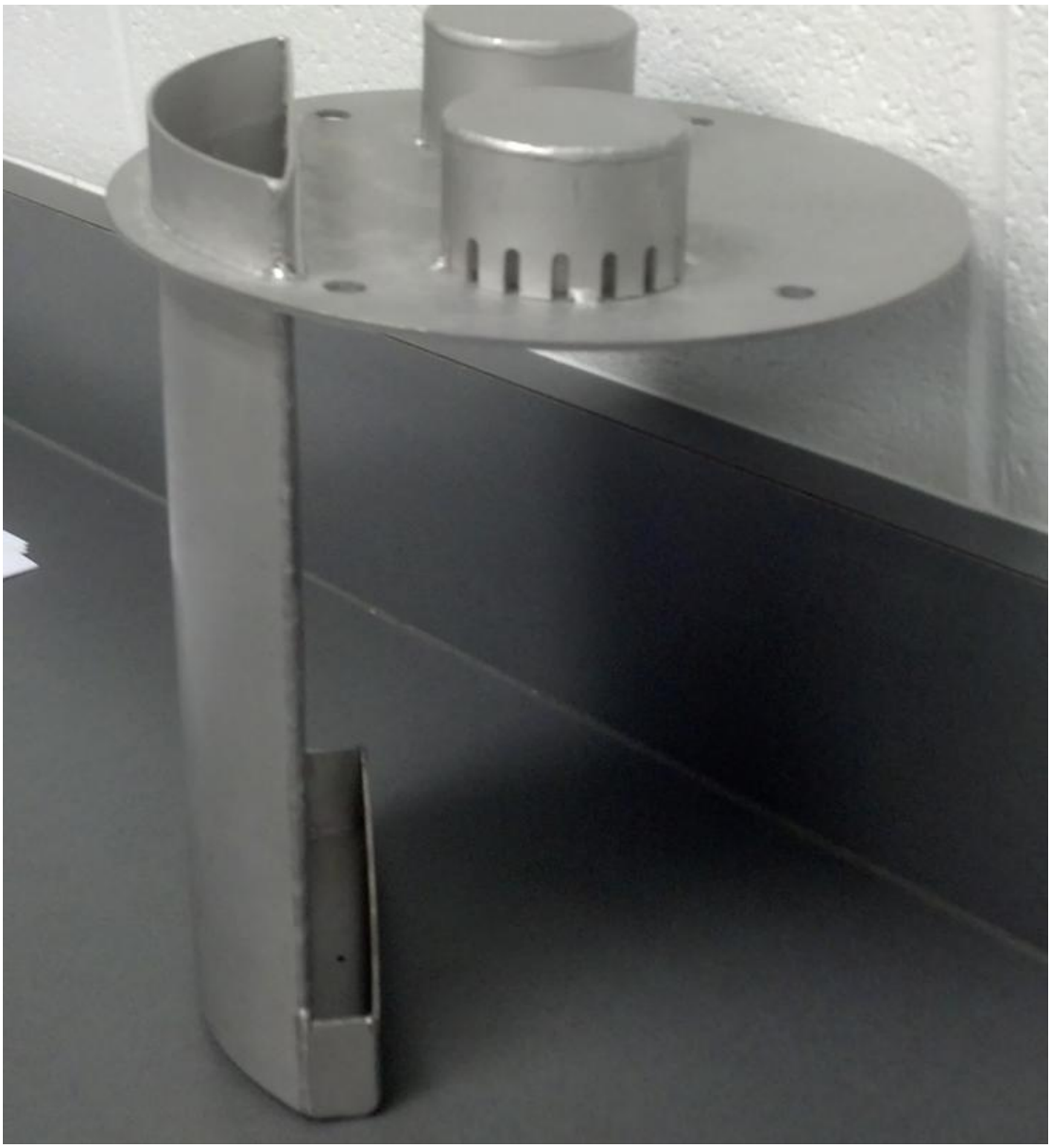

Figure 4.14

Picture Model of Bubble-Cap Tray with Downcomer Used in Tray 1

In order to stay away from flooding the bubble-cap trays, figures 4.8 and 4.9 must be followed, differential pressure of the column must stay below 0.13 barg, and the rate of reflux plus EtOH-rich distillate product flow rates must be less than 100 L/hr.

The weeping effect of the sieve tray distillation column represented the lower operating limit of AK222. The table of results for the experiments ran (see Table 4.4) did not have much use for the stable operating curve due to experiments done only at total reflux. Table 4.4 does show that decreasing flow rates of steam and reflux results in lowered concentration of EtOH of the top EtOH-rich distillate product. Table 4.5 and figure 4.11 show that the minimum amount of steam supply during stable operation is 40 kg/hr compared to 25 kg/hr of AK122. This was calculated by operating the distillation columns in many instances and noticing that steam rates lower than 40 kg/hr on AK222 resulted in product purity of top distillate product to be less than 90 wt% EtOH. This is due to AK222 having sieve trays which have perforations that the liquid can flow through (weeping) instead of going down the downcomer. Steam and liquid rates inside AK222 must be high enough to develop a liquid layer on each tray in order to achieve good mass transfer and separation. Also, due to the same design flaw of the tray spacing and downcomer issue discussed for AK122, the same trays that have excessive entrainment in AK122 have weeping on AK222. This is the case all throughout the operation of AK222 and can be seen for tray 16 at the following website:

http://doiop.com/Ozkaya-Vid-07

Since weeping is only a factor at low flow rates, it is not as bad of a condition as flooding for AK122. AK222 can achieve production goals with no problems, but uses a very high steam flow rate to do so when compared to AK122. The upper limit for operating AK222 is the temperature of the condensate coming from the condenser AW227 into buffer tank AB201 being too high (greater than 70 ^{o}C). The condenser AW227 cannot handle steam flow rates higher than 58 kg/hr, which is the maximum steam rate as shown in table 4.5 and figure 4.11. There is a

flooding region in every distillation column, but AK222 does not reach this region operating the column at its designed production goals.

The graph developed to compare the stable operating regions of both columns (see figure 4.12) show how much higher flow rates at which AK222 can operate. A better comparison is shown by figure 4.13 where the energy usage (steam supply) is compared to the distillate production rate for both columns. This graph indicates that it is more feasible to operate AK122 when production goal of EtOH-rich distillate product flow rate is in the range of 13 to 32 kg/hr, because AK122 is able to use much less steam supply and reflux ratio to achieve this production rate compared to AK222. AK122 can achieve a distillate product rate of 32 kg/hr at 36 kg/hr steam rate while AK222 achieves the same distillate production rate at 54 kg/hr steam flow rate. The advantage of AK222 is when distillate production rate is above 32 kg/hr all the way up to 41 kg/hr. AK122 is almost at its flooding region when trying to produce 32 kg/hr so in terms of production rate, AK222 wins. In terms of efficiency, AK122 is much better than AK222 because of its ability to achieve its production rates using much less energy than AK222.

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

There are five conclusions made in this thesis; these are shown below:

1. The flooding phenomenon in the bubble-cap tray distillation column occurred at a vapor rate of 81 kg/hr and before the production goals could be reached. Flooding is indicated by an increase in differential pressure in the column, rising above 0.13 barg. This phenomenon resulted in inoperable, dangerous conditions inside the distillation column and lowered the top distillate product purity below production goals. The reason for flooding to occur before production goals could be reached was found to be the design flaw of the downcomers on the trays which had double the tray spacing compared to the rest of the trays in the bubble-cap tray distillation column.
2. The stable operation analysis of bubble-cap tray distillation column resulted in development of a stable operating curve. The flow rates in this curve fall way below the performance chart shown in figure 1.1. This was due to the fact that the chart in figure 1.1 was developed for large industrial distillation columns with much higher flow rates. Also, the stable operating curve in figure 4.8 was a line instead of a region, this is because the distillation process has specific purities of the products as production goals and there is only one way to reach these goals. Thus, the distillation process in bubble-

cap tray distillation column is not flexible in its operating conditions and does not have a region of satisfactory operation for these specifications.

3. Weeping phenomenon analysis for the sieve tray distillation column at total reflux showed that efficiency of separation in the column decreased as the vapor and liquid flow rates decreased. This was observed by decrease in the purity of the top distillate product at low flow rates. Weeping was also noticed at the same tray locations as bubble-cap tray distillation column due to the same flaw in downcomers and tray spacing.
4. The stable operating curve was also developed for the sieve tray distillation column which included the minimum and maximum limits of stable operation. The maximum limit was not due to flooding, instead it was the result of the condenser not being able to handle steam flow rates over 58 kg/hr. The stable operating region chart shown in figure 1.2 did not resemble the stable operating curve due to the same reason mentioned above in conclusion 3.
5. The production curves developed for both columns in figures 4.9 and 4.11 were a representation of the capabilities of the distillation columns and the parameters at which they operated most efficiently. The comparison of the columns showed that the bubble-cap tray distillation column uses much less energy to reach the same production goals as the sieve tray, but sieve tray distillation column actually was able to reach production flow rate goals while bubble tray flooded way before reaching the flow rate goals.

5.2 Recommendations

All of the distillation experiments and regular operation of the pilot plant were done using constant production goals, concentration of ethanol in feed mixture (50 wt%), steam pressure (2 barg). For more detailed analysis of the stable operating regions of the distillation columns, it is

recommended that the feed mixture concentration ethanol be changed and the steam pressure be varied to analyze the system to see how different it behaves. It is recommended that pure ethanol instead of denatured ethanol be used in the distillation, which effects the operation due having chemicals such as methanol and acetic acid mixed in the ethanol (MSDS, Denatured Ethanol). Due to bubble-cap tray distillation column flooding at a differential pressure higher than 0.13 barg and steam flow rates between 36-40 kg/hr, alarms should be readjusted to warn the operator that the distillation process is about to become unstable due to flooding of the trays. The flooding and weeping of trays due to the large tray spacing between trays 5-6, 10-11, and 15-16 can be fixed by changing the downcomers of trays 6, 11, and 16 to have a closed bottom section like the ones used for tray 1. It is recommended that this analysis of the distillation column be done again after those downcomers are changed in the distillation columns.

5.3 Future Work

It would be interesting to study the effect of preheating the feed going into to the distillation column (see Sections 1.2 and 3.4.2). Does preheating reduce energy costs? Would it be better to superheat the feed? What kind of impact does preheating or superheating the feed have on energy costs, product quality, and production rate?

The oscillating behavior observed for the differential pressure and tray temperatures in the flooding analysis for bubble-cap tray distillation column should also be studied further to find the causes and effects of this phenomenon. These oscillations are not observed in any other previous studies done on column flooding and should be further studied.

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VITA

Murat Ozkaya was born in Istanbul, Turkey, to the parents of Tugrul and Fusun Ozkaya. He has one older brother named Metehan Ozkaya. He spent most of his childhood living in Izmit, Turkey, before moving with his family to the U.S.A. in February 1998. He started Hixson Middle School in Chattanooga, TN, and went on to graduating from Hixson High School in 2004. After graduation, Murat attended University of Tennessee at Chattanooga where he completed his Bachelors of Science degree in Chemical Engineering in December 2009. Murat's special interest in chemical engineering led him to continuing his education by becoming a graduate assistant for the Chemical Engineering Department at the same university, where he began working exclusively with Dr. Jim Henry on control systems and a remote distillation column in January 2010. The experience he got from control systems and the distillation column helped him become the Manager of Wacker Institute Pilot Plant at Chattanooga State Community College in October 2011, where he began training lead chemical operators employed by WACKER Chemie AG in distillation. Murat completed his Master of Science degree in Chemical Engineering in August 2012. He is currently continuing his work on distillation columns and training future chemical operators at the Wacker Institute Pilot Plant.