Plant-wide Control of Coupled Distillation Columns with Partial Condensers

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Abstract

Conventional distillation control processes use vapor distillate flowrate to control column pressure and condenser heat removal to control the reflux drum level. These intuitive control systems work well for isolated columns or columns with total condensers. However, these controls are not effective when columns with partial condensers occur in series. The pressure and reflux drum level interact in such systems in ways that defeat conventional control systems, rendering them unable to maintain product purities in the presence of large feed flowrate and composition disturbances. This investigation documents a plant-wide control structure that can address this issue by controlling pressure through reflux heat removal rate and reflux drum level by reflux flow rate. This control system demonstrates its capability to handle large disturbances in throughput and feed composition through a series of Aspen simulations. This alternative system is no more complicated than the conventional system and should work on distillation columns of nearly all designs, not just the coupled partial condenser designs for which it is essential.

Common natural gas processing provides a specific example of this alternative control system. Natural gas commonly includes high concentrations of CO₂ that must be removed prior to pipeline or LNG distribution. The existence of a minimum-boiling temperature azeotrope between ethane, virtually always

present in natural gas, and carbon dioxide complicates the separation of CO₂ from the hydrocarbons. This separation commonly employs extractive distillation with high-molecular-weight hydrocarbons. Our recent paper [1] discusses in detail the steady-state economic design of a new extractive distillation strategy for the CO₂-ethane azeotrope separation with three columns. This strategy shows a 5% reduction in capital and 15% reduction in operating costs when compared to optimized versions of the conventional process. The new strategy also produces CO₂ in a liquid rather than a vapor phase, which simplifies transport, storage, and handling. Two columns of the proposed design use partial condensers and are the focus of this investigation.

Keywords: Extractive distillation, CO₂-ethane azeotrope, Simulation, Control, Aspen Plus Dynamics.

1. Introduction

Natural gas has a long-established role as a major primary energy source domestically and internationally. Recently, U.S. natural gas supplies and consumption have increased significantly and US natural gas prices, for the first time, decoupled from petroleum prices. International supply and consumption changes are more measured and in many regions prices remain tied to petroleum prices. By all reasonable accounts, natural gas will remain and probably become an increasingly important primary energy source into the foreseeable future [2-4].

Natural gas processing includes four stages: acid gas removal, dehydration, condensate removal, and liquefaction [5]. The acid gas removal process, or sweetening, removes CO₂ and H₂S from the natural gas. The separation of these gases is crucial for increasing the heating value of LNG, and preventing the solidification of CO₂ during subsequent steps [6-10].

There are several methods for natural gas sweetening, such as chemical absorption with amines, physical absorption, membrane permeation, and cryogenic distillation processes. Of these practices, absorption with amines is the most widely used. The main disadvantages of this process are the significant

consumption of solvent for high CO₂ concentration feedstock and significant safety and operational issues. Moreover, this process produces a low-pressure gaseous CO₂ product that often requires liquefaction for further use. Similarly, the membrane CO₂ removal process produces a low-pressure CO₂ product that suffers from the issue of high pressure drop.

The cryogenic extractive distillation process has the potential to overcome some of the abovementioned issues [11]. To apply this process, two significant challenges must be addressed: first, the sublimation of CO₂ in the demethanizer column, and second, the existence of a minimum-boiling azeotrope between ethane and CO₂. To overcome these challenges, a fraction of the NGL product from the recovery column recycles back as a solvent in the demethanizer column. The solvent contains a mixture of propane and heavier hydrocarbons (C₃+) from the original gas feed and serves as an entrainer by taking CO₂ to the bottom of the column, thus reducing the freezing point of CO₂ and preventing solid CO₂ formation. Moreover, the solvent aids in breaking the CO₂-ethane azeotrope in the extractive distillation column.

There is a rich literature on the subject of azeotropic systems. Doherty and Malone [12], and Stichlmair and Fair [13] provide notable examples of azeotropic separation phase equilibrium for both binary and ternary systems. Hong and Kobayashi [14] provide vapor-liquid equilibrium (VLE) data for the separation of CO₂ and ethane using *n*-C₅ as a solvent in the extractive distillation process. Torres-Ortega [15] investigate an alternative thermally coupled sequence (CEDSR) configuration based on a cryogenic extractive distillation system to enhance the carbon dioxide removal with lower total annual cost. They also study the effect of different liquefied hydrocarbon fractions as an entrainer, the greenhouse gases generation, the theoretical control properties, and the thermodynamic efficiency of the proposed system. They conclude that the proposed configuration has better performances than the conventional chemical absorption process. Lastari et al. [16] study the effects of solvent-to-feed ratio, feed tray locations, and solvent composition on the energy demand of the extractive distillation for azeotrope separation of CO₂ and ethane.

Tavan et al. [17, 18] discuss the application of feed splitting technique in extractive distillation for the CO₂-ethane azeotrope separation. They observed that feed-splitting scheme has superior performance in terms of total energy requirement and environmental impacts compared with the conventional method. Tavan et al. [19] investigate the application of the dividing-wall column (DWC) process for the azeotropic separation of CO₂ and ethane at low temperatures and compare the economic and environmental aspects of the DWC process with the conventional process. Tavan et al. [20, 21] explore the application of reactive absorption (RA) to break the CO₂-ethane azeotrope with low energy demand and compare their results with the conventional extractive distillation system. They optimize diethanolamine (DEA) flow rate, DEA inlet temperature, and feed inlet stage with respect to process energy requirement. In another investigation [22], Tavan et al. investigate CO₂-ethane azeotropic mixture as the feed of a reforming process to convert CO₂ and ethane to syngas (H₂ and CO). They recommend Mg-type catalysts and a minimum steam/HC ratio of 1.5 to avoid coke formation during reforming process.

These methods, however, have a significant drawback: the high energy requirement and/or equipment cost leads to penalties in the operating and capital expenses (OpEx and CapEx), as well as long payback period times [23]. A previously disclosed novel extractive distillation system reduces these costs for CO₂-ethane separation using a three-column configuration that meets the desired purification with 5% less capital costs and 15% less energy requirement compared to conventional systems. This new process also produces CO₂ as a liquid product, which avoids the significant amount of energy required for liquefaction [1].

This new process has one total condenser column and two partial condenser columns. Conventional distillation control systems follow intuitive reasoning and control pressure by manipulating vapor distillate flowrate and reflux drum level by manipulating condenser heat removal. However, the interaction among the pressure, reflux drum level and tray temperature control loops cause the conventional control structure to fail in maintaining the purity of the products in the presence of large

disturbances in throughput and feed compositions when the vapor distillate stream feeds another distillation column.

The main purpose of this investigation is to develop an effective control structure that can address the issue of plant-wide control of distillation column systems that have interconnected partial condenser and total condenser columns. Aspen Plus and Aspen Dynamics tools provide rigorous steady-state and dynamic simulations.

2. Thermodynamic Analysis

Figure 1 exhibits the phase equilibrium diagram of the binary mixture of CO₂-ethane at the pressure of 20 bar. The boiling points of CO₂ and ethane at this pressure are 254.13 and 265.81 K, respectively. The figure clearly shows that CO₂ and ethane form a minimum-boiling homogenous binary azeotrope at 0.65 mole fraction CO₂ at 247.71 K. The azeotrope occurs because of the strong molecular repulsion forces between ethane and CO₂. Methods to separate binary azeotropes fall into two categories:

1. *Azeotropic distillation systems*- which require the addition of a third chemical component, or entrainer, that shifts the azeotropic composition. The azeotropic distillation systems function in two ways. A light entrainer forms on of at least two distillate or overhead components in the “azeotropic” column. These generally immiscible components separate in a decanter. One phase refluxes the first column, whereas the second phase feeds another distillation column to regenerate the solvent, which recycles to the first column. A heavy entrainer forms one of at least two components in bottoms of the extractive column. These generally miscible components feed a separate distillation column, which recycles the entrainer to the first column. These methods constitute azeotropic distillation and extractive distillation, respectively.
2. *Pressure-swing distillation*- which does not require the addition of a third component (entrainer) to shift the vapor-liquid equilibrium. Two columns operating at two different pressures can achieve separation if changes in pressure significantly alter the relative volatilities.

Figure 2 shows that pressure changes do not shift the vapor-liquid equilibrium of ethane-CO₂ mixture considerably, indicating that pressure-swing distillation is generally an inefficient method for this separation. However, Figure 3 clearly demonstrates that high molecular weight hydrocarbons such as n-butane change the VLE of the system. Figure 3(a) shows that the addition of the entrainer decreases the freezing point of CO₂ to -67.3 °C, which prevents the solidification of CO₂ at low temperatures. Figure 3(b) illustrates that all the residue curve lines start at the minimum-boiling azeotrope and move toward the highest boiling component n-C₄. Therefore, extractive distillation is a viable method for the separation in the presence of a miscible mixture and a homogenous azeotrope.

3. Process Flowsheet

Figure 4 shows the flowsheet for the proposed alternative distillation sequence. The feed stream comprises a bottoms stream from a de-methanizer column and includes carbon dioxide, ethane, and higher molecular weight hydrocarbons. The process contains three columns: the CO₂ recovery column, the solvent recovery column, and the concentrator column. In this process, not all the CO₂ exits at the overhead of the extraction column. The bottom product of the extraction column, which contains some of the CO₂ in the fresh feed along with most of the ethane and heavier hydrocarbons, feed the second column. This column also produces high-purity solvent as the bottoms stream. The recovery column distillate vapor is an azeotropic mixture, which recycles to the initial feed. The recovery column bottoms stream is the ethane product.

3.1. Extractive Column

The extractive column has 39 stages and operates at the pressure of 24 atm. The fresh feed gas and the solvent stream enter at stages 36 and 5, respectively. The solvent in the extractive column alters the relative volatility between ethane and CO₂, driving ethane to the bottom of the column and CO₂ overhead. The section above the entrainer feed location in the column separates the entrainer and CO₂. The section between the entrainer feed stage and the fresh feed stage prevents ethane from going up the column. The

section below the fresh feed location prevents CO₂ from going down the column. The extractive column delivers a CO₂-rich distillate (95.7 mol %) with impurities of 2.9 mol % ethane and 1.3 mol % propane. The bottoms stream carries most of the ethane, some of the CO₂ in the fresh feed, and heavier hydrocarbons to the solvent recovery column.

3.2. Solvent Recovery Column

The solvent recovery column has 37 stages and the bottom stream of the extractive column enters as a feed on stage 15. The distillate has 0.3 mol % *n*-C₃, whereas the bottoms stream has 0.05 mol % ethane and the remainder heavier hydrocarbons. This stream splits (after heat exchange) into the NGL product and the solvent feed streams, the latter of which recycles back to the first column. The NGL stream passes through a sequence of traditional distillation columns for C₃, C₄ and C₅ recoveries. The distillate is a mixture of almost 80 mol % ethane and 20 mol % CO₂ that needs to be concentrated before recycling to the extractive column.

3.3. Concentrator Column

The third column has 43 stages and the distillate of the recovery column enters on stage 10. This column produces an ethane-rich stream (99.5 mol %) as the bottom product. The azeotropic mixture of ethane and CO₂ goes overhead with the CO₂ concentrated up to 46 mol %.

4. Plant-wide Control

Process control investigations extensively analyze distillation control. Distillation columns differ in operating conditions, product purity specifications, difficulty of separation, differences in feed contents, column constraints, and local economic parameters, requiring many different control schemes [24]. Steady-state analyses greatly outnumber transient analyses [25-39].

Partial condenser column control is challenging because of the interaction among the reflux drum level, pressure and tray temperature in the control loops. The literature dealing with the control of a distillation

column with a partial condenser is limited, and to the best of the authors' knowledge, there is no analysis covering the control of distillation column systems that have interconnected partial condensers and a total condenser column. Such systems introduce unique control problems, as discussed later.

Buckley [40] and Shinskey [41] pioneer studying the design and control of distillation columns with partial condensers. Luyben [24] examine the fundamental dynamic relationship of a distillation column with partial condenser and proposed three alternatives for the control of distillation columns with partial condensers. Hori and Skogestad [42] investigate alternative control structures for a cryogenic deethanizer column with a partial condenser and implemented the best control structure on a real column. Luyben [43] demonstrates a realistic model for distillation columns with partial condensers producing both liquid and vapor products. He reports that the model assuming a fixed flowrate of cooling water provides the most realistic performance predictions to disturbances. Grassi [44] set the standard configuration for the control of extractive distillation columns and suggests a control structure that uses single-end temperature control for both columns with the solvent flowrate ratioed to feed flowrate.

The overall molar balance around the reflux drum of a distillation column with partial condenser leads to,

$$\frac{dN_R}{dt} = L_c - R \quad (1)$$

where N_R is the molar holdup of liquid in the reflux drum, L_c is the flowrate of the outgoing liquid from the condenser, and R is the reflux flowrate. The following equation relates the liquid flowrate to the condenser heat duty,

$$L_c = \frac{Q_c}{\Delta H_v} \quad (2)$$

where ΔH_v is the latent heat of vaporization of the vapor distillate. Inserting L_c from Eq. (2) with Eq. (1) gives,

$$\frac{dN_R}{dt} = \frac{Q_c}{\Delta H_v} - R \quad (3)$$

- 1 The above equation shows how reflux flowrate and condenser heat removal directly affect the reflux
 2 drum level while the overhead vapor flowrate does not.
- 3 Moreover, applying the overall mole balance around the vapor space in the reflux drum gives,

$$\frac{V_d}{RT_R} \frac{dP}{dt} = V_N - L_C - D \quad (4)$$

- 4 which equates to,

$$\frac{V_d}{RT_R} \frac{dP}{dt} = V_N - \frac{Q_c}{\Delta H_v} - D \quad (5)$$

- 5 where V_d is volume of the vapor, T_R is the reflux drum temperature, V_N is the vapor flowrate leaving the
 6 top tray, D is the vapor distillate flowrate, and R is the ideal gas law constant. Eq. (5) clearly shows that
 7 both distillate flowrate and condenser heat removal affect the pressure. This is the main cause of the
 8 interaction between the level and pressure loops in a partial condenser. The parameter V_N indicates the
 9 load disturbance.

- 10 Applying the Laplace transform to rearrange the above equations in the form of transfer functions gives,

$$P(s) = -\left[\frac{RT_R}{V_d \Delta H_v s}\right] Q_c(s) - \left[\frac{RT_R}{V_d \Delta H_v s}\right] D(s) \quad (6a)$$

$$N_R(s) = \left[\frac{1}{\Delta H_v s}\right] Q_c(s) - \left[\frac{1}{s}\right] R(s) \quad (6b)$$

- 11 These equations clearly show that pressure is a function of condenser heat removal and vapor distillate,
 12 but not the reflux flowrate. Moreover, the reflux drum level is a function of condenser heat removal and
 13 reflux flowrate, but not the vapor distillate flow.

The ideal control structure of a distillation column would measure the impurity of the light-key component in the bottoms and the impurity of the heavy-key component in the distillate and manipulate two input variables to maintain the desired impurities in the two product streams. However, this ideal control structure is barely used for distillation columns because composition analyzers; (1) are expensive to purchase affiliated with their high maintenance costs, (2) do not always provide reliable results for online continuous control, and (3) introduce long measurement lags into the control loop when chromatographic methods are applied. However, it is possible to attain very effective control without using direct composition measurement. Single-end inferential control structures commonly provide inferential composition control for distillation columns. Thermocouples are cheap and introduce small dead time in the control loop. Appropriately chosen locations for temperatures provide reliable information about the key components composition.

This investigation explores two control structures. The first structure (CS I) uses conventional controls for partial condenser columns in which vapor distillate flowrate controls column pressure and condenser heat removal controls reflux drum level. The second control structure (CS II) controls column pressure by condenser heat removal and controls drum level by reflux flowrate. Valve pressure drops and pump heads are 3 atm to give reasonable rangeability so that valve saturation does not occur in the face of large increases in feed throughput. The reflux drums and the column bases provide 5 min of liquid holdup when the vessel is half-full at the design conditions.

4.1. Control Structure CS I (Conventional Control Structure)

Figure 5 presents the CS I control structure for the new CO₂-ethane separation process. Proportional controllers with $K_c = 2$ are applied for level loops, whereas conventional proportional-integral actions are used for the remainder of the system.

The CS I control structure includes the following features;

1. Feed and recycle streams are flow-controlled.
2. Solvent is ratioed to feed.
3. Base level in each column is controlled by manipulating the bottoms flow.
4. Reflux drum in the extraction column is controlled by manipulating the distillate flow.
5. Reflux drum in the solvent recovery and the concentrator columns are controlled by manipulating the corresponding condenser heat removal.
6. Pressure in the extraction column is controlled by manipulating the condenser heat removal.
7. Pressure in the solvent recovery column and the concentrator is controlled by manipulating the corresponding vapor distillate flows.
8. The reflux flow in the extraction column is ratioed to the feed.
9. The reflux flows in the solvent recovery and the concentrator columns remain constant.
10. A temperature in each column is controlled by manipulating the corresponding reboiler heat input.

Partial condenser columns (the second and third columns of this process) require both pressure and reflux drum level control. There are several manipulated variables available: distillate flowrate, condenser heat removal and reflux flowrate. In a partial condenser column, the distillate flows from the reflux drum in the form of a vapor stream. The vapor distillate cannot directly control the reflux drum level because the drum level does not depend on the vapor flowrate. Practically, it is also typically quite difficult to meter.

Conventional systems control pressure by manipulating distillate vapor flowrate. There is an interaction between the pressure and control loop in this configuration because any disturbance affecting either loop will propagate to the other loop. For instance, assume the feed composition changes and more CO₂ enters the column. This reduces the reflux drum temperature, and as a result, decreases the heat transfer rate in the condenser for a fixed flowrate of refrigerant. As the condensation rate decreases, the pressure increases, and the pressure controller increases the vapor distillate flowrate. Additionally, the drop in condensation rate decreases the reflux drum level. As a result, the level controller increases the refrigerant flowrate to compensate, which decreases the pressure. This interaction places the level controller and the pressure controller into a cyclic, potentially unstable interaction.

Figure 6(a) shows the temperature profile of the extraction column whereas Figure 6(b) shows the composition profiles of the key components in the extractive column. The slope of the temperature profile (Figure 6(c)) and the slope of the composition profiles (Figure 2(d)), indicate that the temperatures and the key components compositions change rapidly from tray to tray around stage 24 of the extractive column. If the temperature of stage 24 remains at 271 K, the heavy components should not escape out the top, and light components would not drop out the bottom. Figure 7 shows that holding stage 24 at 334 K in the solvent recovery column maintains the purity of the products at the design specs. These simulations use dead-times of 1 min in each temperature loop. Relay-feedback tests use the conservative Tyreus-Luyben tuning settings to obtain the temperature controller tuning constants.

4.1.1. CS I Results

Simulated large disturbances in feed flowrate and composition help evaluate the effectiveness of this control structure. In the first case uses 20% increases in feed flowrate at time equal 0.5 h with the results shown in Figure 8 by solid lines. Manipulating the reboiler heat inputs controls the temperature of each column. Figure 8(a) shows that distillate purity in the extractive column x_{CO_2} remains constant and close to the specification. However, bottoms purity in the concentrator column x_{C_2} drops significantly (Figure 8(c)). This occurs because condenser heat removal controls column pressure. The higher throughput decreases reflux drum temperature, which decreases heat transfer rate in the condenser for a fixed flowrate of the refrigerant. Therefore, column pressure begins to increase, which leads to increases in the vapor distillate flowrate. The decrease in the condensation rate results in a decrease of the reflux drum level. The decrease in the reflux drum level makes the level controller compensate by increasing the refrigerant flowrate, which leads to a pressure decrease. This coupling between the level controller and the pressure controller causes large and rapid changes in the vapor distillate flowrate. Since this vapor distillate is the feed of another column, any variation in this stream propagates to the downstream process and upsets it as well. As a result, the bottoms purity x_{C_2} in the third column drops from 99.5% to 92%.

Feed composition perturbations lead to similar results. The simulations present results where the ethane mole fraction in the feed increases by 5% with a corresponding 5% decrease in CO₂ mole fraction at time equal 0.5 h (Figure 8 - dashed lines). As indicated, it takes more than 15 hours for the temperature controller of the extractive column $TC_{1,24}$ to return the temperature to the desired level after this transient. During this time, the distillate purity in the extractive column, x_{CO_2} , becomes unstable and the purity of the bottoms product in the concentrator column reaches 100%. As already discussed, this poor control performance is because the pressure of the columns with partial condensers are controlled by their vapor distillate flowrates, which vary rapidly during perturbations in the feed. Any variation in the vapor distillate flow upsets the downstream process, causes the valve to saturate, and loses control.

4.2. Control structure CS II

Good distillation column control prevents rapid pressure changes. A rapid pressure increase decreases vapor flowrates through the trays, which may cause dumping and weeping. A rapid pressure decrease of increases vapor flowrates, which may cause flooding. Any rapid pressure change also presents potential safety and environmental hazards. Controlling the pressure with vapor distillate leads to large and rapid changes in the flowrates of distillate. If the distillate feeds a downstream unit, which as in this case, these large distillate flowrate variabilities represent significant product purity disturbances and upsets. To avoid this issue, condenser heat removal controls pressure and reflux flowrate controls reflux drum level in this system. Additionally, the distillate flowrate remains proportional to the reflux flowrate to maintain a constant reflux ratio.

The proposed control structure CS II is shown in Figure 9. Figure 10 exhibits the faceplate of CS II. Note that in the controller faceplate, the “FCs” controller is on cascade mode, indicating that the controller receives its setpoint signal from multiplier “S/F”. Similarly, “FCD2” and “FCD3” are in the cascade mode and receive their setpoints from multipliers “D₁/R₁” and “D₂/R₂”, respectively. The following sections describe why these control loops require cascade control. All level controllers are proportional action with a gain of 2. Conventional PI systems control the remaining loops. Relay-feedback test and

Tyres-Luyben tuning rules provide controller tuning constants in the column temperature loops given in Table 1.

4.2.1. Extractive column

The main features of the extractive column control loops are pressure control through condenser heat removal and the reflux drum and bottom level control through the distillate and the bottoms flows, respectively. The column has a total condenser, which eliminates interaction between the reflux drum level and pressure.

Similar to CS I structure, stage 24 of the extractive column provides inferential temperature control, which is ultimately controlled by manipulating the reboiler duty. There are two possibilities to determine the second control degree of freedom: (a) holding the reflux ratio constant, or (b) ratioing the reflux flowrate to feed flowrate. When disturbances in feed flowrate occur, both choices are equivalent because all flows ratio directly with throughput. However, when disturbances occur in feed composition, the ratios vary in a different way when the two products are kept at their design specs. The reflux-to-feed ratio structure provides the second control degree of freedom in the following analysis

The reflux ratio in the extractive column is 4.61. When column reflux ratios exceed 3, it is conventional to control the reflux drum level by regulating the reflux flowrate. However, level control using the reflux prohibits the use of a reflux-to-feed control scheme, which is the effective control structure for feed composition disturbances. On the other hand, manipulating the distillate to control the drum level leads to large alterations in the distillate flowrate, which can upset downstream processes. Assuming that disturbances do not occur rapidly and frequently, the selection of a reflux-to-feed ratio structure handles disturbances more effectively, without rapidly changing the distillate flow rate.

4.2.2. Solvent Recovery and Concentrator Columns

The recovery and concentrator columns have partial condensers, which causes the main difference between the two control structures. CS II differs from CS I structure in the following ways. The rest of the loops are unchanged.

- The reflux flowrate controls the reflux drum level in the solvent recovery and the concentrator columns.
- Condenser heat removal controls pressure in the solvent recovery column and the concentrator columns.
- The reflux ratios remain constant in the solvent recovery and the concentrator columns by reflux flowrate control.

4.2.3. CS II Results

In the analysis below, transient analyses of large feed disturbances (20% alterations in the flowrate of the fresh feed, and 20% alterations in feed composition) illustrate the performance of the control structure CS II. Figure 11 illustrates all three columns results for a 20% disturbance in feed flowrate. The dashed lines represent feed stream flowrate decreases (-20%) and the solid lines represent increases (+20%). These graphs clearly show that the temperatures and product purities are well controlled.

Figure 12 presents all the three columns result for feed composition disturbances. The dashed lines represent ethane feed content increases from 0.46 to 0.51 (mole fraction) with a corresponding decrease in the CO₂ feed content from 0.32 to 0.27. The solid lines represent ethane feed content decreases from 0.46 to 0.41 mole fraction with a corresponding increase in the CO₂ feed content from 0.32 to 0.37. As observed with respect to the feedrate changes, this scheme provides good control of product purities and temperatures for all the three columns.

4.2.4. Generalization

The underlying issues illustrated in these specific examples pertain to all partial-condenser column controls. The alternative control system is not as traditional or intuitive and the conventional systems, but its control logic, sensors, and loops are no more complicated or difficult to install. Additionally, the control systems here should be just as effective for any partial condenser column or, for that matter, any distillation column, regardless of whether such columns occur in series and independent of whether they treat azeotropic systems. In these ways, they represent potentially more robust approaches to column control than the conventional approaches.

5. Conclusions

Dynamic control using conventional configurations fail to maintain product purities in processes that involve interconnected partial condensers. Specifically, conventional distillation columns control pressure by manipulating vapor distillate flowrate and control reflux drum level should by manipulating condenser heat removal. This control strategy is effective for an isolated distillation column. However, this scheme fails to provide effective control of interconnected columns, specifically columns with interconnected partial condensers. An alternative control scheme controls pressure through condenser heat removal rate and reflux drum level by reflux flow rate. This scheme effectively controls product purities in the face of even large perturbations in feed flowrate and composition. This alternative system is no more complicated than the conventional system and should work on distillation columns of nearly all designs, not just the coupled partial condenser designs for which it is essential.

A recently proposed CO₂-ethane azeotropic distillation system serves as an example of this control system. The design has one total condenser column and two partial condenser columns. The interaction among the pressure, reflux drum level, and tray temperature control loops make the control of partial condenser columns more complex than total condenser columns. Dynamic analyses indicate that, contrary to expectation, the conventional control structure cannot maintain the purity of the two product streams at

the desired values. An effective plant-wide control structure based on inferential single-end temperature control is presented for this process. The main features of the control loops of the partial condenser columns in the proposed scheme are reflux drum level control through reflux flowrate, pressure control through heat condenser removal and distillate flowrate ratioed to the reflux flowrate. Transient analyses show that the proposed control structure maintains CO₂ and ethane product purities in the face of large disturbances (20%) in throughput and feed composition. Similar control systems should

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References

- [1] E. Ebrahimzadeh, J. Matagi, F. Fazlollahi, L.L. Baxter, Alternative extractive distillation system for CO₂-ethane azeotrope separation in enhanced oil recovery processes, *Applied Thermal Engineering*, 96 (2016) 39-47.
- [2] R.G. Newell, S. Iler, The global energy outlook, in, National Bureau of Economic Research, 2013.
- [3] B. Bazooyar, N. Hallajbashi, A. Shariati, A. Ghorbani, An Investigation of the Effect of Input Air Upon Combustion Performance and Emissions of Biodiesel and Diesel Fuel in an Experimental Boiler, *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, 36 (2014) 383-392.
- [4] B. Bazooyar, A. Shariati, A Comparison of the Emission and Thermal Capacity of Methyl Ester of Corn Oil with Diesel in an Experimental Boiler, *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, 35 (2013) 1618-1628.
- [5] D. Elliot, W.R. Qualls, S. Huang, J.J. Chen, R.J. Lee, J. Yao, Y. Zhang, Benefits of integrating NGL extraction and LNG liquefaction technology, in: 2005 AIChE Spring National Meeting, Conference Proceedings, 2005, pp. 1943-1958.
- [6] S.M. Safdarnejad, J.D. Hedengren, L.L. Baxter, Plant-level dynamic optimization of Cryogenic Carbon Capture with conventional and renewable power sources, *Applied Energy*, 149 (2015) 354-366.
- [7] R.C. Jensen MJ, Bergerson D, Hoeger CD, Frankman DJ, Bence CS, Baxter LL Prediction and validation of external cooling loop cryogenic carbon capture (CCC ECL) for full-scale coal-fired power plant retrofit, *Int J Greenhouse Gas Control*, 42 (2015) 200-212.
- [8] S.M. Safdarnejad, L. Kennington, L.L. Baxter, J.D. Hedengren, Investigating the impact of Cryogenic Carbon Capture on power plant performance.

- [9] B.A. Fazlollahi F, Ebrahimzadeh E, Baxter LL. , Design and analysis of the natural gas liquefaction optimization process- Energy Storage of Cryogenic Carbon Capture (CCC-ES), Energy, in press (2015).
- [10] E. Ebrahimzadeh, P. Wilding, D. Frankman, F. Fazlollahi, L.L. Baxter, Theoretical and experimental analysis of dynamic plate heat exchanger: Non-retrofit configuration, Applied Thermal Engineering, 93 (2016) 1006-1019.
- [11] B. ZareNezhad, N. Hosseinpour, An extractive distillation technique for producing CO₂ enriched injection gas in enhanced oil recovery (EOR) fields, Energy Conversion and Management, 50 (2009) 1491-1496.
- [12] M.F. Doherty, M.F. Malone, Conceptual design of distillation systems, McGraw-Hill Science/Engineering/Math, 2001.
- [13] J. Stichlmair, J.R. Fair, Distillation: principles and practices, Vch Verlagsgesellschaft MbH, 1998.
- [14] J.H. Hong, R. Kobayashi, To break an azeotrope. 1. The use of n-pentane to break the carbon dioxide-ethane azeotrope, for carbon dioxide EOR gas processing, Industrial & Engineering Chemistry Process Design and Development, 25 (1986) 736-741.
- [15] C.E. Torres-Ortega, J.G. Segovia-Hernández, F.I. Gómez-Castro, S. Hernández, A. Bonilla-Petriciolet, B.-G. Rong, M. Errico, Design, optimization and controllability of an alternative process based on extractive distillation for an ethane-carbon dioxide mixture, Chemical Engineering and Processing: Process Intensification, 74 (2013) 55-68.
- [16] F. Lastari, V. Pareek, M. Trebble, M.O. Tade, D. Chinn, N.C. Tsai, K.I. Chan, Extractive distillation for CO₂-ethane azeotrope separation, Chemical Engineering and Processing: Process Intensification, 52 (2012) 155-161.
- [17] Y. Tavan, S. Shahhosseini, S.H. Hosseini, Feed-splitting technique in the extractive distillation of CO₂-ethane azeotropic process, Separation and Purification Technology, 122 (2014) 47-53.
- [18] R. Hasanvandian, Y. Tavan, Techno-Economic Analysis of the Carbon Dioxide (CO₂)-Ethane Azeotrope Separation for Natural Gas Processes, Energy Technology, 3 (2015) 570-578.
- [19] Y. Tavan, S. Shahhosseini, S.H. Hosseini, Design and simulation of ethane recovery process in an extractive dividing wall column, Journal of Cleaner Production, 72 (2014) 222-229.
- [20] Y. Tavan, S.H. Hosseini, A novel application of reactive absorption to break the CO₂-ethane azeotrope with low energy requirement, Energy Conversion and Management, 75 (2013) 407-417.
- [21] Y. Tavan, A. Tavan, Performance of conventional gas sweetening process to remove CO₂ in presence of azeotrope, Journal of CO₂ Utilization, 5 (2014) 24-32.
- [22] Y. Tavan, A note on use of the CO₂-ethane azeotrope as new feed of reforming process using mathematical modeling, Journal of Natural Gas Science and Engineering, 21 (2014) 275-282.
- [23] A.A. Kiss, R.M. Ignat, Innovative single step bioethanol dehydration in an extractive dividing-wall column, Separation and Purification Technology, 98 (2012) 290-297.
- [24] W.L. Luyben, Alternative Control Structures for Distillation Columns with Partial Condensers, Industrial & Engineering Chemistry Research, 43 (2004) 6416-6429.
- [25] W.L. Luyben, Control of the maximum-boiling acetone/chloroform azeotropic distillation system, Industrial & Engineering Chemistry Research, 47 (2008) 6140-6149.
- [26] W.L. Luyben, Effect of solvent on controllability in extractive distillation, Industrial & Engineering Chemistry Research, 47 (2008) 4425-4439.
- [27] I.-L. Chien, K.-L. Zeng, H.-Y. Chao, Design and control of a complete heterogeneous azeotropic distillation column system, Industrial & Engineering Chemistry Research, 43 (2004) 2160-2174.
- [28] L.M. Rueda, T.F. Edgar, R.B. Eldridge, A novel control methodology for a pilot plant azeotropic distillation column, Industrial & Engineering Chemistry Research, 45 (2006) 8361-8372.
- [29] W.L. Luyben, Plantwide control of an isopropyl alcohol dehydration process, Aiche Journal, 52 (2006) 2290-2296.
- [30] S. Arifin, I.-L. Chien, Design and control of an isopropyl alcohol dehydration process via extractive distillation using dimethyl sulfoxide as an entrainer, Industrial & Engineering Chemistry Research, 47 (2008) 790-803.

- [31] W.L. Luyben, Design and control of a fully heat-integrated pressure-swing azeotropic distillation system, *Industrial & Engineering Chemistry Research*, 47 (2008) 2681-2695.
- [32] W.L. Luyben, Comparison of extractive distillation and pressure-swing distillation for acetone-methanol separation, *Industrial & Engineering Chemistry Research*, 47 (2008) 2696-2707.
- [33] W.L. Luyben, Control of the Heterogeneous Azeotropic n-Butanol/Water Distillation System, *Energy & Fuels*, 22 (2008) 4249-4258.
- [34] W.L. Luyben, Control of a column/pervaporation process for separating the ethanol/water azeotrope, *Industrial & Engineering Chemistry Research*, 48 (2009) 3484-3495.
- [35] Y.C. Wu, I.-L. Chien, W.L. Luyben, Two-stripper/decanter flowsheet for methanol recovery in the TAME reactive-distillation process, *Industrial & Engineering Chemistry Research*, 48 (2009) 10532-10540.
- [36] Y.-C. Wu, I.-L. Chien, Design and control of heterogeneous azeotropic column system for the separation of pyridine and water, *Industrial & Engineering Chemistry Research*, 48 (2009) 10564-10576.
- [37] K.-Y. Hsu, Y.-C. Hsiao, I.-L. Chien, Design and control of dimethyl carbonate– methanol separation via extractive distillation in the dimethyl carbonate reactive-distillation process, *Industrial & Engineering Chemistry Research*, 49 (2009) 735-749.
- [38] J.R. Phimister, W.D. Seider, Semicontinuous, pressure-swing distillation, *Industrial & Engineering Chemistry Research*, 39 (2000) 122-130.
- [39] J.R. Phimister, W.D. Seider, Semicontinuous, middle-vessel, extractive distillation, *Computers & Chemical Engineering*, 24 (2000) 879-885.
- [40] P.S. Buckley, *Techniques of process control*, Wiley, 1964.
- [41] F. Skinskey, *Distillation control: For productivity and energy conservation*, (1984).
- [42] A.E.S. Hori, B.S. Skogestad, Control structure selection of a deethanizer column with partial condenser, in: *Proceedings of European Congress of Chemical Engineering (ECCE-6)*, 2007, pp. 16-20.
- [43] W.L. Luyben, Realistic Models for Distillation Columns with Partial Condensers Producing Both Liquid and Vapor Products, *Industrial & Engineering Chemistry Research*, 51 (2012) 8334-8339.
- [44] V.G. Grassi II, Process design and control of extractive distillation, in: *Practical distillation control*, Springer, 1993, pp. 370-404.

Table 1. Controller Tuning Parameters

	$T_{C_{1,24}}$	$T_{C_{2,24}}$	$T_{C_{3,22}}$
controlled variable	stage 24 temperature in extractive column	stage 24 temperature in extractive column	stage 22 temperature in extractive column
manipulated variable	reboiler heat input $Q_{b,1}$	reboiler heat input $Q_{b,2}$	reboiler heat input $Q_{b,3}$
setpoint	271.86 K	334.72 K	270.30 K
transmitter range	240-340 K	300-400 K	240-340 K
OP	15.12 MW	90.47 MW	21.32 MW
OP range	0 to 30.24 MW	0 to 180.95 MW	0 to 42.66 MW
dead-time	1 min	1 min	1 min
K_C	3.71	1.8	3.8
τ_I	9.8	10.7	7.2

Figure(s)

Figure 1. CO₂-ethane at 20 bar.

Figure 2. T - xy (a) and xy diagram (b) of the azeotrope mixture of ethane-CO₂ over a range of pressures and compositions.

(a)

Figure 3. phase envelope (a) and residue curve map (b) for the mixture of ethane, carbon dioxide and n-butane at 20 bar.

Figure 4. The flowsheet for the proposed CO₂-ethane azeotrope separation.

Figure 5. The conventional control structure (CS I) of the new extractive distillation process.

Figure 6. Temperature profile (a), composition profile, (c) temperature slopes and composition slopes (d) in the extractive column.

Figure 7. Temperature profile (a), composition profile, (c) temperature slopes and composition slopes (d) in the solvent recovery column.

(a)

(b)

(c)

Figure 8. (a) Extractive column, (b) Recovery column, and (c) Concentrator column responses in the face of fresh feed flowrate and composition disturbances: Solid lines are for the case of +20% increase in feed flowrate, and dashed lines are for the case of -5% decrease in ethane composition and +5% increase in CO_2 composition in the fresh feed content.

Figure 9. The proposed control structure (CS II) of the new extractive distillation process.

Figure 10. Control structure CSII and the faceplate.

(a)

(b)

Figure 11. (a) Extractive column, (b) Recovery column, and (c) Concentrator column responses in the face of $\pm 20\%$ fresh feed flowrate disturbances.

(a)

(b)

(c)

Figure 12. (a) Extractive column, (b) Recovery column, and (c) Concentrator column responses in the face of fresh feed composition disturbances.