Vapor Recompression in Batch Distillation for Separation of Ternary Zeotropic Mixtures: Feasibility and Analysis

Thesis submitted in partial fulfilment of the requirements of the degree of

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by
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This is to certify that the thesis entitled **“Vapor Recompression in Batch Distillation for Separation of Ternary Zeotropic Mixtures: Feasibility and Analysis”** being submitted by **Mrs. Radhika Gandu** for the award of the degree of Doctor of Philosophy (Ph.D.) in Chemical Engineering to the **National Institute of Technology, Warangal, India** is a record of the bonafide research work carried out by him under our supervision. The thesis has fulfilled the requirements according to the regulations of this Institute and in our opinion has reached the standards for submission. The results embodied in the thesis have not been submitted to any other University or Institute for the award of any degree or diploma.

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This is to certify that the work presented in the thesis entitled **"Vapor Recompression in Batch Distillation for Separation of Ternary Zeotropic Mixtures: Feasibility and Analysis"** is a bonafide work done by me under the supervision of Dr. Uday Bhaskar Babu Gara, and the co-supervision of Dr. A Seshagiri Rao and is not submitted elsewhere for the award of any degree.

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(**Radhika G**)

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Chapter 4 Performance evaluation of a vapor recompression in batch distillation with a side withdrawal

Chapter 5 Exploring the economic and environmental feasibility of vapor recompression in middle vessel batch distillation

ABSTRACT

Distillation is a widely used unit operation to separate liquid mixtures in chemical and process industries. However, distillation uses a substantial amount of energy in the chemical process industry, increasing the amount of greenhouse gases (e.g., CO_2 emissions) discharged into the environment. Several factors have motivated the revived interest in improving the thermodynamic efficiency of distillation operations. The most prominent ones are growing fossil fuel costs, the long-term availability of fossil fuels, and CO_2 emissions. Therefore, distillation is a natural operation for enhancement due to its high energy consumption. The need to develop cleaner, smaller and more energy-efficient technologies for the distillation operation has been characterized as process intensification.

Various heat-integrated distillation arrangements have been investigated to minimize energy consumption and maximize cost savings. Two significant heat-integrated schemes used in distillation are internal heat integration, e.g., internally heat integrated distillation column (HIDiC), and external heat integration, e.g., vapor recompression column (VRC). The VRC is the most preferred scheme for separating close boiling mixtures. However, significant work has been focused on separating close boiling mixtures for continuous and batch distillation columns. Perhaps very few works report the introduction of VRC in batch distillation to separate ternary zeotropic wide boiling mixtures. Therefore, the thesis explores the feasibility of VRC in batch distillation for separating ternary zeotropic mixtures. This research work proposes a systematic design, operational, and simulation approaches for vapor recompressed batch distillation columns to improve the performance.

The single-stage vapor recompressed multicomponent batch distillation (SiVRMBD) structure consists of a compressor that drives at variable speed. Aiming to assure the optimal use of internal heat energy, a manipulation approach manipulates the overhead vapor and the external heat to the reboiler. However, to separate high-temperature lift mixtures in SiVRMBD, a significant compression ratio (CR) is required, which causes wear and tear difficulties in the compressor. As a result, the technique's implementation has practical constraints. Therefore, a novel optimal multi-stage vapor recompression approach is proposed, so each compressor operates at a minimum CR, affecting compression work and process costs.

Aiming to improve further the economic and environmental prospect, in this work, we investigate the feasibility of single-stage and multi-stage VRC schemes in unconventional batch distillation columns, namely, batch distillation with a side withdrawal and middle vessel batch distillation.

Keywords: Multi-stage vapor recompression, Single-stage vapor recompression, Ternary zeotropic mixtures, Economics, CO_2 emissions, Simulation experiments

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ABBREVIATIONS

BDS	Batch distillation with a side withdrawal
BF	Bottom Flashing
CBDS	Conventional batch distillation with a side withdrawal
CR	Compression Ratio
CO_2	Carbon dioxide
CMBD	Conventional Multicomponent Batch Distillation
CMVBD	Conventional middle vessel batch distillation
DVRC	Double-stage vapor recompression
DWC	Divided Wall Column
DoVRMBD	Double or two-stage vapor recompressed multi component batch distillation
FoVRMBD	Four-stage vapor recompressed multi component batch distillation
GHG	Green House Gas emissions
GSPI	Gain Scheduling Proportional Integral Controller
HIDiC	internally heat integrated distillation column
ISE	Integral square error
M&S	Marshall and swift index
NT	Number of trays
ODE	Ordinary Differential Equations
PI	Proportional Integral controller
SVRC	Single-stage vapor recompression
SiVRMBD	Single-stage vapor recompressed multi component batch distillation
SiVRBDS	Single-stage vapor recompressed batch distillation with a side withdrawal
SiVRMVBD	Single-stage vapor recompressed middle vessel batch distillation
TAC	Total Annual Cost
TCI	total capital investment
TOC	total annual operating cost
TrVRMBD	Three-stage vapor recompressed multi component batch distillation
TLE	Total local emissions
TGE	Total global emissions
VRC	Vapor Recompression Column
VRMBD	vapor recompressed multi component batch distillation

NOMENCLATURE

C	Concentration of the component
c	Total number of components,
C_P	Heat capacity
C_R	Compression ratio
C%	The mass percentage carbon in fuel
D	Distillate rate
D_C	Column diameter
$e(t)$	Error
H	Enthalpy
h_{Proc}	Enthalpy of steam delivered to the process
H^r	Heat of reaction
k	Vapor-liquid equilibrium coefficient
K_C	Controller gain
K_p	Process gain
L	Liquid flow rates in the column
L_c	Column height
H	Liquid holdup
P_i	Compressor inlet pressure
P_{out}	Compressor outlet pressure
Q_{Comp}	Compressor duty
Q_{Cons}	Total energy consumption
Q_{CV}	Heat released by compressed vapor
Q_E	Externally supplied heat to the reboiler
Q_n	Heat loss
Q_{Proc}	Process heat duty
Q_{Fuel}	The heat duty from fuel burnt
Q_R	Reboiler duty
$R\ or\ Rc$	Reflux flow rate
R_f	Multiplication factor (0 for nonreactive stage & 1 for reactive stage)
r	Reaction rate

S	Side stream flow rate
T	Temperature
$T_{n_{Tc}}$	Compressed overhead temperature
T_{in}	Inlet temperature
T_{out}	Outlet temperature
ΔT_T	Temperature difference between compressed overhead vapor and reboiler liquid
ΔT_B	Temperature difference between reboiler liquid and overhead vapor
U	Overall heat transfer coefficient
V	Vapor flow rates in the column
$V_{n_{Ti}}$	Vapor flow rate to condenser
$V_{n_{TC}}$	Vapor flow rate to compressor
$\mathrm{W}_{\mathrm{comp}}$	Compressor work
W_{GT}	The power (electricity) delivered by a gas turbine
x	Mole fraction of component in liquid stream
y	Mole fraction of component in vapor stream
z	Mole fraction of component in feed stream
τ_I	Integral time
λ	Latent heat
λ_{Proc}	Latent heat of steam delivered to the process
μ	Polytropic coefficient
δ	Stichiometric coefficient
ε	Volume or weight of catalyst
η_{GT}	The efficiency of gas turbine
η_C	The Carnot factor

Chapter 1

Introduction and Objectives

Chapter 1

1. Introduction and Objectives

1.1. Introduction and Literature review

Fossil fuels encounter more than 80% of prior energy necessities in the world (Mulia-Soto and Flores-Tlacuahuac, 2011). In the United States, approximately 80% of the greenhouse gas (GHG) emissions are generated by fossil fuels (US energy information administration, 2008). Global warming results from the raucous discharge of GHG (e.g., CO_2 emissions) to the environment would fetch favorable outcomes in some world regions, such as more extended growing seasons and mild winters. However, a considerably higher rate of the world's residents is possible to face the dangerous consequences, including the rise in receding of glaciers, loss of biodiversity, sea level, reduction of glaciers, environmental change, etc.

The scientific acquaintance of environmental change is now apparent to take proper measures to prepare for it and restrict it. However, it is reasonable to state that reducing GHG emissions requires firm regional and global responsibilities, technical invention, and human willpower. Several technological developments are advancing to satisfy the environmental targets as agreed in the Kyoto Protocol, including exploring renewable energy sectors, inventing novel energy-efficient technologies, and enhancing energy efficiency in the current process units. The present work improves batch processing energy, economic, and environmental performance.

Thermal integration in distillation column

Distillation is the most widely used mature unit operation for separating liquid mixtures in the chemical and process industries. It accounts for around 95% of fluid separation in the chemical and associated sectors. In the United States, approximately 400,000 distillation columns are in process (Diez et al., 2009) and account for 10% of the industrial energy consumption (Salamon and Nulton, 1998). Furthermore, 60% of the energy used by the chemical industry was for Distillation independently (Diez et al., 2009). On the other hand, Distillation uses a substantial amount of energy in the chemical process industry, increasing the amount of greenhouse gases discharged into the environment. But, the overall thermodynamic efficiency of a traditional distillation operation is about 5–20% (De Koeijer and Kjelstrup, 2000). Several factors have motivated the revived interest in improving the thermodynamic efficiency of distillation operations. The most prominent ones are growing fossil fuel costs, the long-term availability of fossil fuels, and GHG emissions. The chemical process sector requires a minimum TAC and CO_2 emissions in energy-efficient systems. Therefore, Distillation is a natural operation for enhancement due to its high energy consumption. In fact, there is a high association between energy consumption, total annual cost, and CO_2 emissions to the environment in the distillation process. The need to develop cleaner, smaller, and more energy-efficient technology for the distillation operation have been characterized as process intensification.

Continuous Flow Distillation

Different thermally coupled continuous distillation arrangements have been explored to minimize energy consumption and maximize cost savings in the literature. For illustration, the divided-wall column is shown in Figure 1.1 (Petlyuk et al., 1965; Dünnebier and Pantelides, 1999; Kolbe and Wenzel, 2004; Kaibel et al., 2006; Kiss and Rewagad, 2011) have been successfully commercialized after decades of research investigation. More than 100 DWC unit operations are operating worldwide (Dejanovic et al., 2010), and the number continues to grow. Introducing a heat pump-assisted scheme to a continuous distillation column has been an appealing option in enhancing energetic and environmental performance. As a result, electrically driven vapor recompression types, namely the vapor recompression column (VRC), closed-cycle heat pump (CC), and bottom flashing (BF), are widely used heat pumps for continuous distillation columns. In the VRC (Figure 1.2), the top tray vapor is compressed to a required temperature in a compressor, and then the internal energy released by the compressed vapor is utilized as energy for reboiler liquid. The CC scheme implicates a refrigerant used to transfer energy between the top vapor and the bottom liquid (Figure 1.3). The BF schematic diagram is shown in Figure 1.4, where the bottom stream is expanded and then utilized as a condenser coolant. After that, it was compressed and returned back to the column.

The heat pumping configurations mentioned above, the vapor recompression column is the most favored and generally used scheme (Jana and Mane, 2011; Jogwar et al., 2010; Diez et al., 2009; Fonyo et al., 1995; Henley and Seader, 1981). The CC scheme pump is typically picked when the liquid mixture is corrosive (Jogwar and Daoutidis, 2009). In contrast, the BF scheme is likely to use if the bottoms product stream is an appropriate refrigerant (Henley and Seader, 1981).

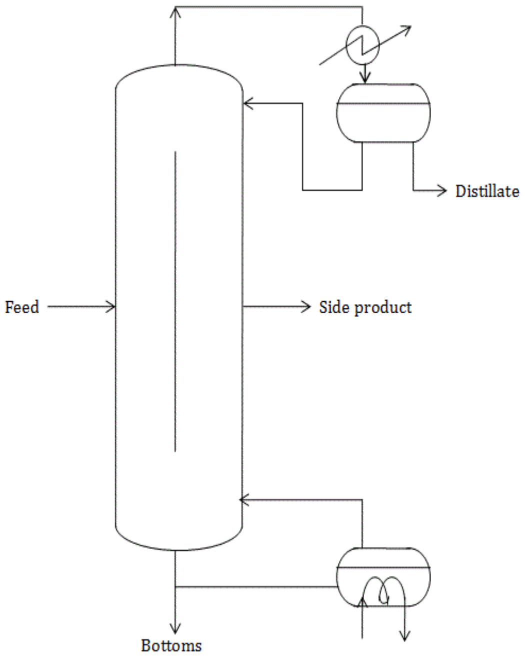

Figure 1.1. The continuous divided wall column.

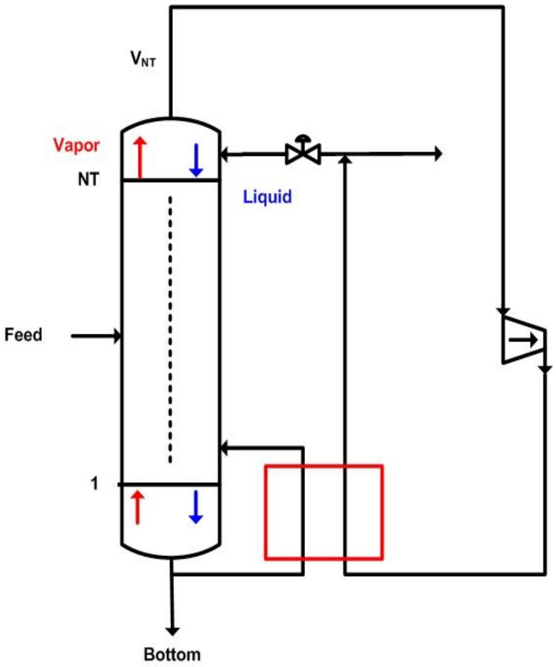

Figure 1.2. The vapor recompressed heat pump-assisted continuous distillation column.

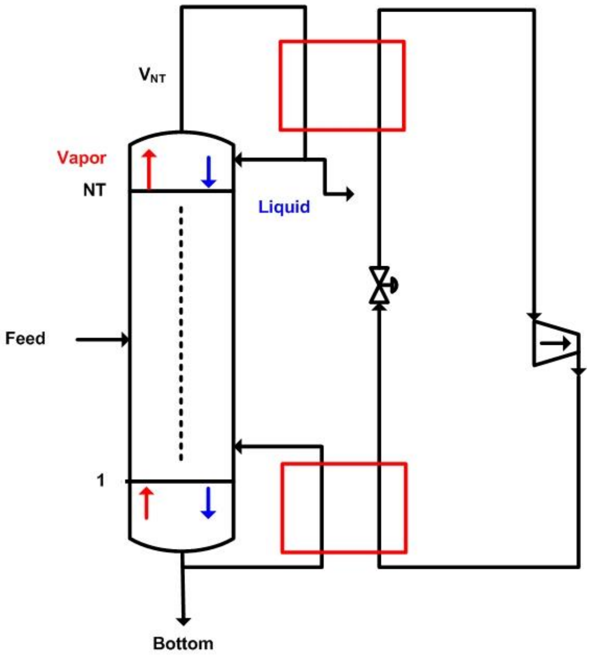

Figure 1.3. The closed-cycle heat pump-assisted continuous distillation column.

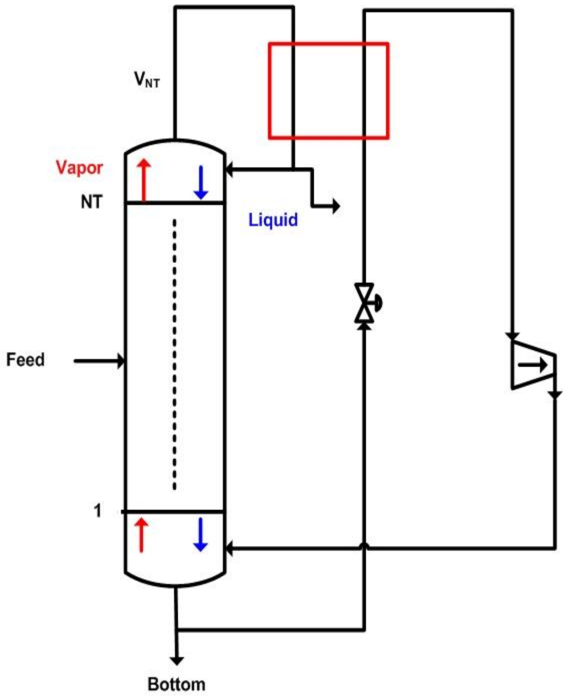

Figure 1.4. The heat pump-assisted bottom flashing continuous distillation column.

Another arrangement, namely the internally heat integrated distillation column (HIDiC), is shown in Figure 1.5 has acquired vast attention. The fundamental idea of the HIDiC is combining heat pump-assisted VRC scheme and the heat integration between two diabatic sections (Suphanit, 2010). Several research groups were dedicated to designing the HIDiC technology (Kiran et al., 2012; Suphanit, 2011; Shenvi et al., 2011; Huang et al., 2008; Fukushima et al., 2006; Gadalla et al., 2005; Lee et al., 2004; Naito et al., 2000; Liu and Qian, 2000).

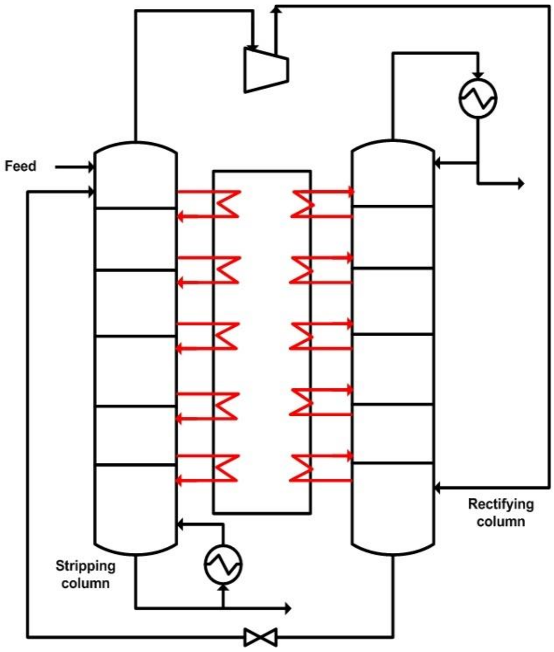

Figure 1.5. The heat integrated distillation column (HIDiC)

Furthermore, various types of HIDiC arrangements, namely the vertical shell-and-tube column and the concentric tray column with heat panel, are reported in the literature (Suphanit, 2010). In addition, a review paper has been published (Jana, 2010) that discusses heat integrated distillation schemes. A systematic comparative analysis has been carried out between the HIDiC and VRC (Schmal et al., 2006). Specifically, the heat pump-assisted schemes appear essential because they are straightforward to present, and the operation is simpler than other heat integration schemes (Annakou and Mizsey, 1995). However, all the above schemes are developed and designed for continuous flow distillation columns. In the literature, few studies have been addressed the heat integration schemes in batch distillation.

Batch Distillation

Batch processing is a vital technology due to the flexibility it offers. For example, multi-component liquid mixtures can be separated in a single batch distillation column. Also, batch distillation handles various feed fractions, difficulty separations, easy separations, and product specifications. This drives batch distillation suitable where the products' demand and a lifetime can vary immensely with time and be tentative, such as fine, pharmaceutical, and specialty chemicals. Although it has some advantages over continuous distillation, batch distillation has several inherent disadvantages: long batch time, increased energy usage, a high temperature in the reboiler, and a complex operation (Demicoli D and Stichlmair J, 2004). In addition, batch distillation is less energy efficient than continuous distillation operation. However, batch distillation has acquired revived interest because of the flexibility offered.

The first heat integration scheme in batch distillation where the rectification section is enclosed by a jacketed reboiler (Takamatsu et al. 1998). Later, organized the concept and described the benefits of the internally heat integrated scheme (Figure 1.6) via numerical simulations (Maiti et al., 2013). The rectification column usually runs at standard pressure in this strategy, and the jacket still operates at elevated pressure. A compressor provides the required temperature driving force for the heat transmitted from the rectifier section to the reboiler.

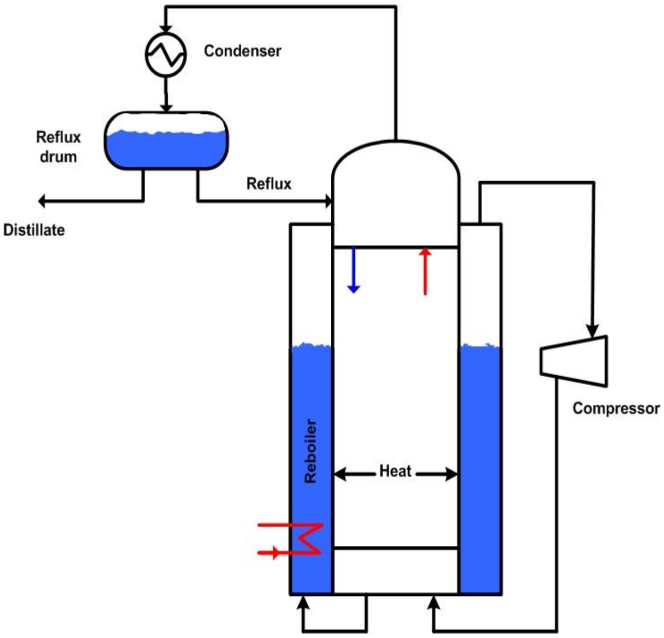

Figure 1.6. The heat integration in batch distillation with a jacketed reboiler.

First to configure the variable speed VRC scheme (Figure 1.7) in regular reactive batch column for separation of close-boiling mixtures (Johri et al., 2013). Explored an adaptive VRC approach for separating ternary closed-boiling mixtures in a batch distillation with a side withdrawal and middle vessel batch columns without vapor bypass (Babu et al., 2012). It was observed from the literature that the VRC is more feasible for separation of close boiling mixtures in batch distillation due to the low-temperature difference between reboiler temperature ($\Delta T_B = T_B - T_{NT}$), i.e., the difference between the reboiler temperature (T_B) and top tray temperature (T_{NT}). As a result, the compressors require low compression; this gives improved energy and TAC (Total annualized cost) savings. The majority of work on VRC was focused on the separation of close boiling mixtures.

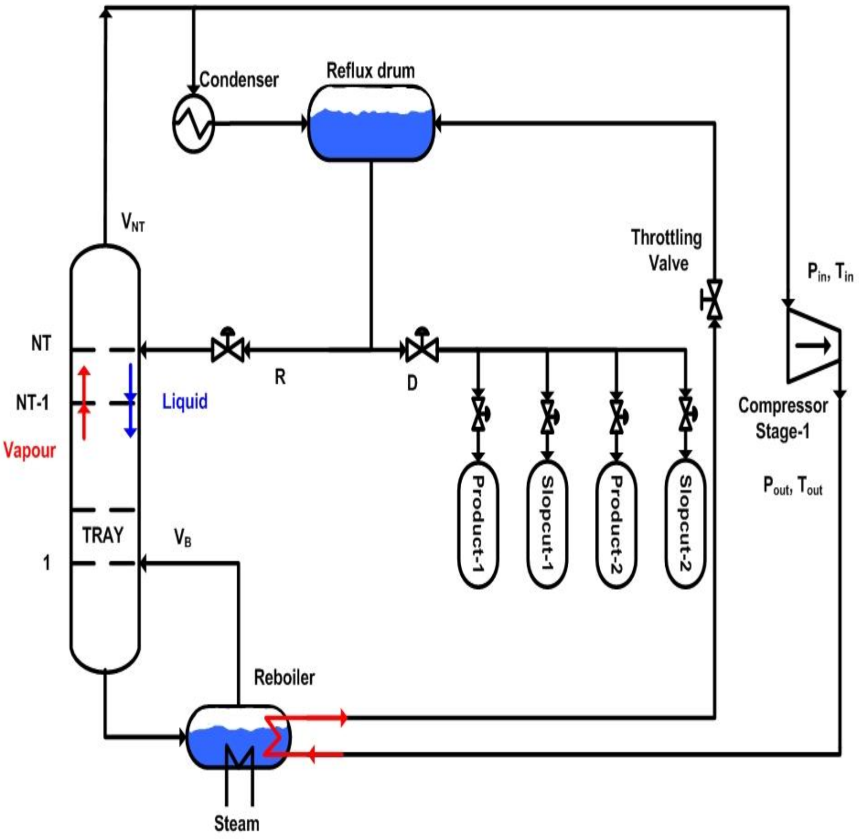

Figure 1.7. The vapor recompression scheme in multicomponent batch distillation

VRC approach can enhance the performance of batch distillation for many separation systems, including binary and multi-component mixtures. The separation of multi-component zeotropic mixtures was easier in batch distillation. Still, it has two significant problems: high-temperature difference in column and increased relative volatility, which leads to high compressor power or CR. However, for moderate binary wide-boiling mixtures, (Khan et al., 2012; Parhi et al., 2019; Vibhute et al., 2020) demonstrated that VRC improves economically and energetically. Very few papers dealing with the VRC in batch distillation for separation of zeotropic wide boiling mixtures have emerged so far, so this research area is only commencing to be explored.

1.2 Motivation of work

In a conventional batch distillation operation, the column top temperature is smaller than the column bottom temperature. Therefore, the heat is added at the highest temperature (i.e., reboiler), and heat is rejected at the lowest temperature (i.e., condenser) points in the batch column. This leads to heat degradation from a higher to a lower temperature level. This is the principal reason for the low thermodynamic efficiency of the conventional distillation column. To enhance the performance, the rejected heat from a condenser must be reutilized to reduce the supply of thermal energy from an external source to the reboiler. An obvious way to accomplish this goal is to couple the condenser and the reboiler, representing the primary source and energy sink, respectively. Since the heat source is colder than the heat sink, reusing the heat rejected in the condenser of a batch distillation column must increase its temperature by some heat pump arrangement.

The VRC is more feasible for separating close boiling mixtures in batch distillation. The majority of work on VRC was focused on the separation of close boiling mixtures in the literature. The zeotropic mixtures have various applications in industries like it is used in the Organic Rankine cycle as a working fluid. Most literature is available to separate binary/multi-component zeotropic mixture in continuous distillation using VRC. However, few papers have dealt with VRC in batch distillation to separate binary zeotropic wide boiling mixtures. Perhaps there is no work reporting the introduction of single-stage and multi-stage vapor recompression scheme in batch distillation for separating ternary zeotropic wide boiling mixtures. Therefore, the thesis explores the feasibility of VRC in batch distillation for separating ternary zeotropic mixtures. This research work proposes a systematic design, operational and simulation approaches for vapor recompressed batch distillation columns to improve the performance.

1.3 Objectives of the work

Batch distillation has long been recognized as less energy-intensive than continuous distillation. Therefore, heat integration emerges to be the most effective strategy to improve the performance of batch distillation in terms of operating cost and emission level. The present research explores the feasibility of a vapor recompression scheme in batch distillation for separating zeotropic wide boiling ternary mixtures.

The objectives of this dissertation are set as follows:

1. Investigating the impacts of vapor recompression on open-loop and closed-loop batch distillation.
2. Introducing the multi-stage vapor recompression approach in batch distillation.
3. Performance evaluation of a vapor recompression in batch distillation with a side withdrawal.
4. Exploring the economic and environmental feasibility of vapor recompression in middle vessel batch distillation.

Organization of the thesis

This dissertation is organized into six chapters. An overview of the state of current research in the area of heat-integration in distillation columns with key objectives of the present research work is presented in **Chapter 1.**

Chapter 2: Investigating the impacts of vapor recompression on open-loop and closed-loop batch distillation

In this work, tight composition control and, in parallel, the operation is integrated with vapor recompression scheme (VRC) is proposed to achieve energy savings and maximum product at a specified high purity for separating the ternary zeotropic mixture in batch distillation. Firstly, the model representing a ternary system of hexanol/octanol/decanol has been simulated to analyze the process's open loop and close loop dynamics. Secondly, the open-loop and closed-loop operations are integrated with single-stage VRC scheme to achieve energy savings. Single-stage VRC is operated at a very high compression ratio (CR) due to the significant temperature difference of the top and bottom streams in a batch distillation column. To further improve the thermodynamic efficiency of single-stage VRC, double-stage compression without intercoolers between the stages of VRC is proposed.

Chapter 3: Introduction of multi-stage vapor recompression approach in batch distillation

The batch distillation industry requires a minimum TAC and CO_2 for any investments in heat integrated systems, such as VRC. Consequently, the design conditions for implementing VRC should be chosen such that the energetic performance is maximum at minimum TAC. In this work, heat pump-assisted vapor recompression design approaches are proposed to separate ternary wide boiling mixtures in batch distillation to reduce TAC and CO_2 emissions. Hexanol/octanol/decanol ternary wide boiling mixture model system was selected in this work. First, a systematic simulation approach was developed to determine the optimal trays based on the lowest energy and TAC reduction. In a SiVRMBD, a high CR is necessary throughout the batch operation, which is practically not feasible for the batch process industry resulting in compressor wear and tear. Therefore, a novel optimal multi-stage vapor recompression approach is proposed to identify the optimum number of compressors with the same design and operating conditions as SiVRMBD, so the batch distillation works at the lowest possible CR throughout while conserving the most TAC.

Chapter 4: Performance evaluation of a vapor recompression in batch distillation with a side withdrawal

The chapter is focused on improving the performance of batch distilaltion with a side withdrawal (BDS) for the separation of ternary zeotropic mixtures using VRC. In this study, two ternary zeotropic wide boiling mixtures were investigated: hexanol/octanol/decanol (This system has a high wide boiling/zeotropic property because of the significant temperature difference between the lightest and heaviest components) and methanol/ethanol/1-propanol (This system has a moderate wide boiling/zeotropic property because of the moderate temperature difference between the lightest and heaviest components).

Chapter 5: Exploring the economic and environmental feasibility of vapor recompression in middle vessel batch distillation

This chapter is focused on improving the performance of middle vessel batch distillation for the separation of ternary zeotropic mixture, i.e., methanol/ethanol/1-propanol using VRC.

Chapter 6: Conclusions and future work

This chapter presents the general conclusions and perspective of future work

Chapter 2

Investigating the impacts of vapor recompression on open-loop and closed-loop batch distillation

Chapter 2

2. Investigating the impacts of vapor recompression on open-loop and closed-loop batch distillation*

In this chapter, tight composition control and, in parallel, the operation is integrated with vapor recompression scheme (VRC) is proposed to achieve energy savings and maximum product at a specified high purity for separating the ternary zeotropic mixture in batch distillation. Firstly, the model representing a ternary system of hexanol/octanol/decanol has been simulated to analyze the process's open loop and close loop dynamics. Secondly, the open-loop and closed-loop operations are integrated with single-stage VRC scheme to achieve energy savings. Single-stage VRC is operated at a very high compression ratio (CR) due to the significant temperature difference of the top and bottom streams in a batch distillation column. To further improve the thermodynamic efficiency of single-stage VRC, double-stage compression without intercoolers between the stages of VRC is proposed.

2.1 Introduction

In chemical and associated industries, separation operations consume roughly 50–70% of the energy used in large-scale chemical manufacturing. In addition, distillation is employed for about 95% of all fluid separations, and it accounts for nearly 3% of the total energy consumption in the world (parhi et al., 2020)**Error! Reference source not found.**. Therefore, distillation operation has excellent potential for heat integration, and many researchers have been working to improve the thermodynamic efficiency of distillation operation for the past century.

Batch distillation is unsteady in nature in which the distillate composition changes with time (Treybal, 1980). This type of operation is commonly used when the priority is to achieve very high product purity from expensive and valuable resources used in pharmaceutical industries for fine chemical production. Its flexibility makes it more preferable than continuous distillation as it can handle different feed compositions by switching a few operational parameters (Jana et al., 2018).

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* This work has been published in *chemical product and process modeling*, https://doi.org/10.1515/cppm-2020-0045

Maintaining tight composition is a primary challenging task when the operation is unsteady in nature. Some online instruments are available, like Chromatograph for measuring composition, but they are expensive, provide delayed response, and require high maintenance costs (Fieg et al., 2002). Researchers are pumping up their ideas to control the composition with an advanced control strategy to get a faster and minimum delay in response to overcome this problem. Here controller comes into the picture to control the distillate composition at the desired value throughout the operation. Obviously, the reflux flow rate has to increase continuously to maintain high and constant purity in batch distillation (Mathew et al., 2014). A reflux drum is a distribution point where condensed vapors are stored for maintaining sufficient reflux and distillate flowrate. Therefore, it's also acquired the same attention as composition control and required a control structure to maintain enough level in the drum; otherwise, it may affect the product purity as well as cause expensive operational problems (Luyben et al., 2020).

The degree of control or controller performance depends not only on controller design but also on feed specification, like the behavior of feed mixture (azeotropic and zeotropic) (Warter et al., 2004). Zeotropic mixture is easier to separate, but it has two major problems (a) chances of having a high-temperature difference in column and (b) high relative volatility. A low relative volatility system gives a slow response, so easy to achieve control at high purity, while the system with high relative volatility gives a quick response and significant deviation from desired purity. Therefore, it becomes more complex to control its specific purity (Fuentes and Luyben, 2010).

Batch distillation is also enumerated as an energy-inefficient operation as it consumes higher energy than continuous distillation and becomes a challenging task for the upcoming generation. Hype in the demand of energy motivates engineers to come up with highly efficient technology like direct vapor recompression (VRC) scheme (Babu et al., 2012), operates using variable speed compressor in which the latent energy of overhead vapor stream is utilized at reboiler junction instead of wasting to surrounding, and it also overcomes the condenser heat duty. Implemented to open and closed-loop to compare energy consumption during the batch operation with and without the VRC scheme (Luyben, 2019).

Many kinds of research have been carried out in distillation with the binary and multi-component mixture, but most are done for an azeotropic mixture using continuous and batch distillation. The VRC scheme is more economical for close boiling mixture, a system that requires high heat load, low-pressure drop, and low process temperature (Felbab et al., 2013). Eco-efficient VRC assisted pressure-swing distillation process for the separation of a maximum-boiling azeotrope (Shi P et al., 2020) in which 44.71% of thermodynamic efficiency has been observed. Heterogeneous binary azeotropic dividing-wall distillation columns steady-state performance is significantly enhanced due to VRC assistance (Shi et al., 2015). Design and control of VRC assisted extractive distillation for separating n-hexane, and ethyl acetate stated lower operating and total annual costs and higher thermal efficiency with VRC assistance. The results are comparably better than pressure swing distillation (Feng et al., 2020).

On the other side, the zeotropic mixture has acquired the same attention as an azeotropic mixture because of its complexity in controlling tight composition due to high relative volatility. The zeotropic mixture has a variety of applications in industries like it is used in the Organic Rankine cycle as a working fluid to enhance thermal efficiency (Wang et al., 2017). Many works of literature are available for the separation of binary/multi-component zeotropic mixture in continuous and batch distillation. The optimal control reflux policy is used to separate binary non-ideal zeotropic mixture (Stojkovicet et al., 2018). Optimal design for separating multi-component zeotropic mixture reduces total annualized cost (Zou et al., 2012). Comparing and controlling three batch distillations for separating ternary zeotropic mixture stated that only the selected control structure can give better performance (Fanaei et al., 2012), multistage VRC for reactive distillation (Kumar et al., 2013). Performance investigation of VRC batch distillation to separate the ternary zeotropic mixture in which multistage VRC study is carried out and stated that double vapor recompression gives 52% of energy saving by increasing thermodynamic efficiency (Nair et al., 2017). However, for ternary zeotropic mixtures, rarely reported in the literature in the area of tight composition control and energy saving using VRC scheme.

As per the author’s knowledge, no work has been reported in the open literature for constant composition control in batch distillation for separating ternary zeotropic mixture integrated with the VRC scheme. Therefore, in this paper, batch distillation assisted with single-stage and double-stage VRC scheme is implemented with selected controller structure to control product composition and holdup in the reflux drum.

2.2 Conventional Multicomponent Batch Distillation column: Open loop operation

Conventional Multicomponent Batch Distillation (CMBD) or open-loop operation is shown in Figure 2.1 to separate a ternary zeotropic feed mixture of hexanol/octanol/decanol with high intermediate composition as given in Table 2.1. The column is divided into two sections rectifying and stripping section. It consists of a reboiler at the bottom of the column and a condenser mounted at the top with a reflux drum.

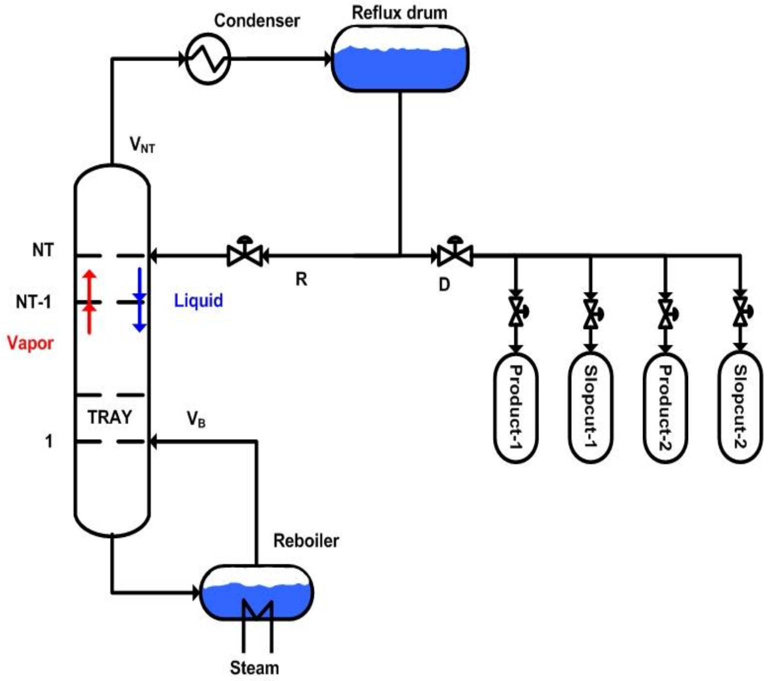

Figure 2.1. Conventional Multicomponent Batch Distillation

Table 2.1. Operating condition and specification of batch distillation column

System	hexanol/ octanol/ Decanol
Feed composition	0.15/0.7/0.15
Number of trays	22
Fresh feed charged (reflux drum/ trays/ reboiler), kmol	0.26/ 0.0118×22/ 12.48
Reboiler duty, KJ/min	2690
Column pressure, bar	1.01325
Tray efficiency, %	80
Distillate flowrate (open loop), kmol/min	0.0337
Time step, min	0.001

CMBD or open-loop operates in two modes, the startup phase and the production phase **(**Babu et al., 2012). In the startup phase, the system attains a steady state where the lighter component (hexanol) purity reaches maximum purity. Once the system attains a steady state after that immediate production phase starts. The production phase is a sequential operation in which the products are withdrawn along with slop-cuts. If the feed mixture consists of 'n' number of components, then production operation produces 'n' product with desired purity and 'n-1' slop cut with less purity. Slopcut part can be recycled back into the system if its amount is very high to increase the production amount, and it is obvious that the ternary mixture gives three products and two slop cuts (Luyben, 1988).

The distillation model has been created with the help of ODEs and algebraic equations (Jana et al., 2018) in the MATLAB environment using the first principle approach are given below. Accordingly, the MESH (Material balance, vapor-liquid Equilibrium, mole fraction Summation and Heat balance) equations are derived as:

♦ ***Reboiler*** (subscript ‘ B ’):

Total mole balance: $\dot{m}_B = L_1 - V_B = -D$ (2.1)

Component mole balance: $\dot{m}_B \dot{x}_{B,j} = L_1 x_{1,j} - V_B y_{B,j}$ (2.2)

Energy balance: $\dot{m}_B \dot{H}_B^L = Q_R + L_1 H_1^L - V_B H_B^V$ (2.3)

Equilibrium: $y_{B,j} = k_{B,j} x_{B,j} = \dfrac{\gamma_{B,j} P_{B,j}^0}{P_T} x_{B,j}$ (2.4)

Summation: $\sum_{j=1}^{C} x_{B,j} = 1$ (2.5a)

$\sum_{j=1}^{C} y_{B,j} = 1$ (2.5b)

♦ ***Intermediate trays*** (subscript ‘n’):

Total mole balance: $\dot{m}_n = L_{n+1} + V_{n-1} - L_n - V_n$ (2.6)

Component mole balance: $\dot{m}_n \dot{x}_{n,j} = L_{n+1} x_{n+1,j} + V_{n-1} y_{n-1,j} - L_n x_{n,j} - V_n y_{n,j}$ (2.7)

Energy balance: $\dot{m}_n \dot{H}_n^L = L_{n+1} H_{n+1}^L + V_{n-1} H_{n-1}^V - L_n H_n^L - V_n H_n^V$ (2.8)

Equilibrium: $y_{n,j} = k_{n,j} x_{n,j} = \dfrac{\gamma_{n,j} P_{n,j}^0}{P_T} x_{n,j}$ (2.9)

Summation: $\sum_{j=1}^{C} x_{n,j} = 1$ (2.10a)

$$\sum_{j=1}^{C} y_{n,j} = 1$$

(2.10b)

♦ ***Condenser and reflux drum*** (subscript ‘ D ’):

Total mole balance: $\dot{m}_D = V_{nT} - R - D$ (2.11)

Component mole balance: $\dot{m}_D \dot{x}_{D,j} = V_{nT} y_{nT,j} - (R + D) x_{D,j}$ (2.12)

Equilibrium: $y_{D,j} = k_{D,j} x_{D,j} = \frac{\gamma_{D,j} P^0_{D,j}}{P_T} x_{D,j}$ (2.13)

Summation: $\sum_{j=1}^{C} x_{D,j} = 1$ (2.14a)

$$\sum_{j=1}^{C} y_{D,j} = 1$$

(2.14b)

Here, C denotes the total number of components, D the distillate rate, H^L_n the enthalpy of a liquid stream leaving *n*th tray, H^V_n the enthalpy of a vapor stream leaving *n*th tray, $k_{n,j}$ the vapor-liquid equilibrium coefficient with respect to *n*th tray and *j*th component, L_n the flow rate of a liquid stream leaving *n*th tray, m_n the liquid holdup on *n*th tray, $P^0_{n,j}$ the vapor pressure of component *j* in *n*th tray, P_T the total pressure, Q_R the reboiler duty, R the reflux rate, V_n the flow rate of a vapor stream leaving *n*th tray, $x_{n,j}$ the mole fraction of component j in a liquid stream leaving *n*th tray, $y_{n,j}$ the mole fraction of component j in a vapor stream leaving *n*th tray and $\gamma_{n,j}$ the activity coefficient of component *j* in *n*th tray. The dot symbol (**.**) is used to denote the time derivative. The time derivative of the multiplication of two variables, say m and x, is represented by $\dot{mx}$ ($= d(mx)/dt$).

The dynamic model equations above mentioned are used throughout the thesis work for batch distillation columns.

2.3 Conventional Multicomponent Batch distillation column: Closed loop operation

Normally distillate composition (X_D) is controlled by manipulating either reflux flowrate or reflux ratio (Fileti et al., 2000; Alvarez-Ramirez et al., 2000). To regulate distillate composition with reflux rate, it is important to maintain holdup in reflux drum too, therefore, to ensure the complete control of distillation operation two controllers have been implemented to the system namely, PI (proportional controller) and GSPI (Gain Scheduling Proportional Integral Controller) (Adari and Jana, 2008). In this work, two control structures are proposed to control distillate compositions and holdup in the reflux drum is shown in Figure 2.2. Here, constant composition control of distillate product compositions (X_{D1}-hexanol and X_{D2} -octanol mole fractions) by manipulating reflux flow rate (R_C, Kmol/min). The holdup in the reflux drum (H_D, Kmol) is controlled by manipulating distillate flow rate (D, Kmol/min).

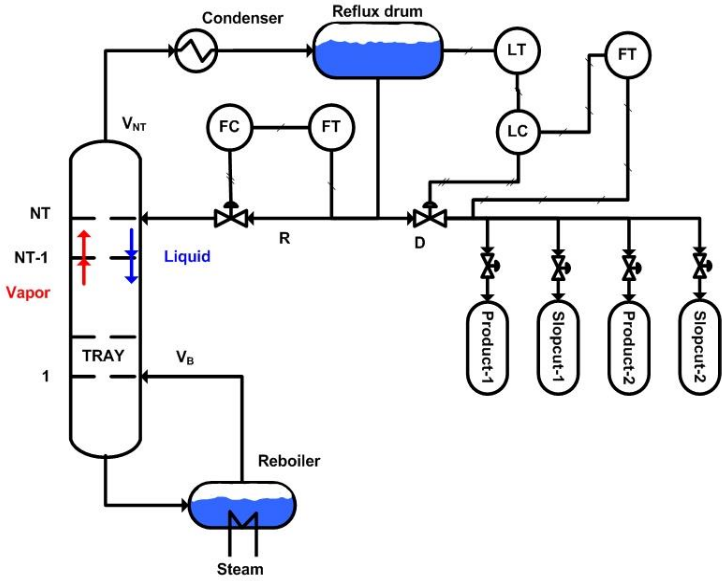

Figure 2.2. Closed loop Multicomponent Batch Distillation

PI controller: Classical controller as defined in equation (2.15) is most commonly used flow controllers. The controller receives error e(t) as input variable i.e. difference between distillate composition set point (X_{Dsp}) and process output (X_D). The error signal ($e(t)$) then goes into the PI control loop where it gets multiplied by the proportional and integral constant (τ_I).

$$u(t) = u_{bias} + K_c e(t) + \frac{K_c}{\tau_I}\int_0^t e(t)dt \qquad (2.15)$$

Where, K_c is controller gain. Poor response of PI controller due to unsteady nature of batch distillation therefore, to maintain good control it is required to vary the gain value of the controller hence variable gain controller like GSPI controller is more suitable than PI.

GSPI controller: A nonlinear controller, and a feedback control loop that calculates an error signal by taking the difference between the output of a system, which is the distillate composition and holdup in the reflux drum, and the set point. The time varying nature of batch distillation makes it more compatible to use the GSPI controller because in batch distillation process gain (K_{PG}) varies with time (t). Therefore, to maintain control in the operation variable gain or scheduling gain (K_{GS}) controller is preferable (Iwakabe et al., 2006; Seborg et al., 2010).

$$K_{GS}(X_{D,i}) = \frac{K_{CG}(X_{DSP,i}) \times K_{PG}(X_{DSP,i})}{K_{PG}(X_{D,i})} \tag{2.16}$$

Where, i is representing the number of components. Both the gains are characterized as the functions of the scheduling variable $X_{D,i}$ and $X_{Dsp,i}$ refers to the steady-state of $X_{D,i}$. Accordingly, the gain-scheduled proportional controller (GSPI) is adopted to manipulate the reflux rate (R_C) as:

$$R_C = R_{bias} + K_{GS}(X_{D,i})\left(e + \frac{1}{\tau_I}\int_0^t edt\right) \tag{2.17}$$

The gain of the GSPI scheme has two different forms, depending on the product purity concerning its desired value (i.e.$X_{DSP,i}$), as:

(i) When $X_{D,i} > X_{DSP,i}$

$$K_{GS}(X_{D,i}) = K_{C0}\frac{1 - X_{DSP,i}}{1 - X_{D,i}} \tag{2.18}$$

Where, K_{C0} is the initial controller gain.

(ii) When $X_{D,i} < X_{DSP,i}$

$$K_{GS}(X_{D,i}) = K_{C0} \tag{2.19}$$

2.4 Open-loop CMBD integrated with VRC

In CMBD energy is provided by reboiler at a constant rate to the bottom of the column and that energy is lost from the top of the column through the condenser therefore, the top of the column is acts as an energy source while the reboiler section act as a sink (Babu and Jana, 2014). Now, to reduce the energy consumption either run the column at optimum reboiler duty or develop an efficient method to integrate heat of the distillation. Vapor recompression (VRC) is one the energy efficient technique to utilize the energy available in the system at reboiler (Luyben et al., 2016; Parhi et al., 2019) as shown in Figure 2.3 is assisted with a mechanical heat pump driven by electricity in which the top tray vapor stream is compressed isentropically and thus increases pressure and temperature. The latent heat of compressed outlet stream is used to supply heat to the reboiler (sink) section before its expansion to its normal temperature and pressure using throttling valve (Cao et al., 2014).

An interesting fact in distillation is that temperature in the top of the column (T_{in}) is less than the temperature in the bottom (T_B) of the distillation column means source temperature is less than sink temperature (Parhi et al., 2019). According to heat transfer law, heat is always transfer from higher temperature to lower temperature region, therefore, to take an advantage of latent heat of source, it is required to maintain a driving force $\Delta T_T = T_{out} - T_B$ of 15 ^{o}C (Luyben WL., 2019) for heat transfer by increasing the source temperature than sink at every time step so that an optimal amount of latent heat can transfer to sink. This driving force of 15^{0} C is maintained by a variable speed compressor with an estimated compression ratio (C_R). C_R is a major factor in VRC, the number of compressor stages depends on the C_R. If C_R is less than 4 then one compressor is enough, but C_R is greater than 4 it is necessary to bring C_R to at least 4 under its limitation with addition of compressor. Therefore, C_R profile for single stage VRC (SVRC) is shown in Figure 2.3 is not

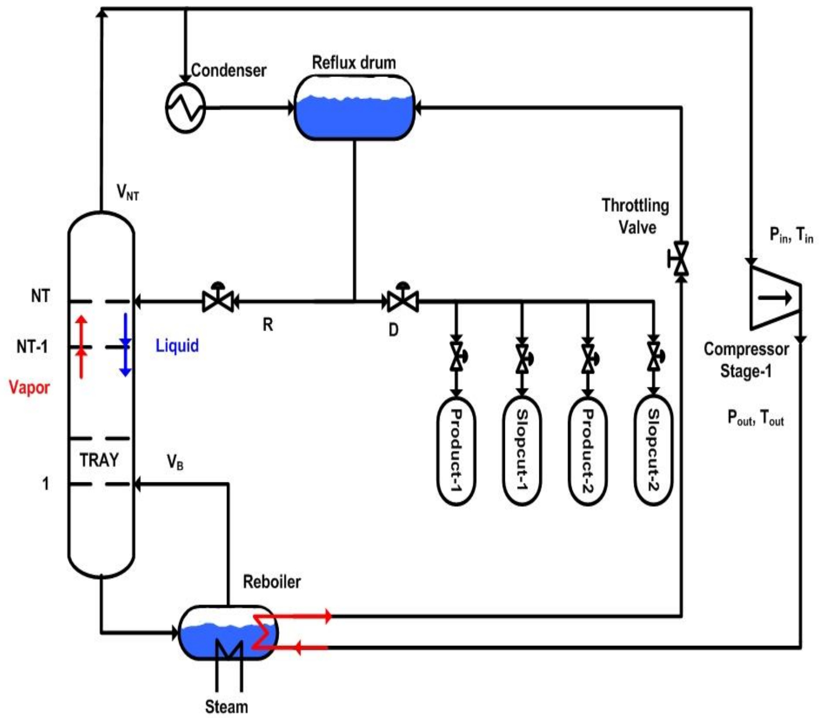

Figure 2.3. VRC scheme with Multicomponent Batch Distillation: Single stage

feasible because of very high C_R (>20), to counter this problem double stage VRC (DVRC) is proposed as shown in Figure 2.4 (Mokhatab and Poe., 2012).

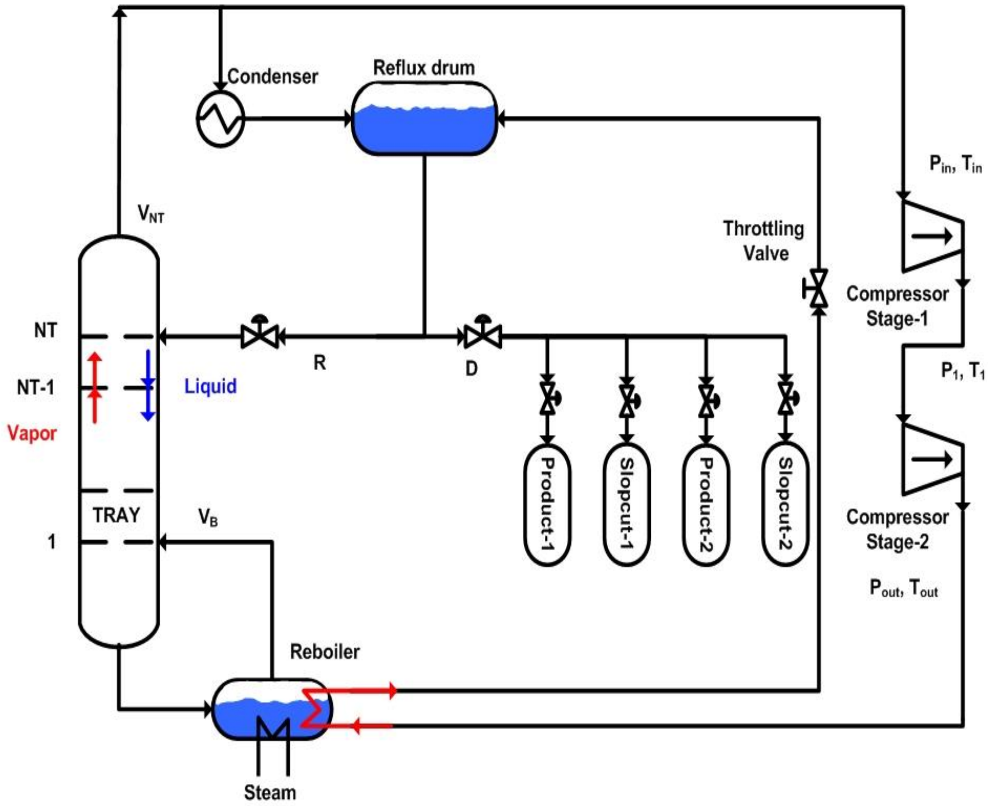

Figure 2.4. VRC scheme with Multicomponent Batch Distillation: Double stage

To attain complete condensation of vapor stream in the reboiler, C_R has to manipulate continuously for batch operation through variable speed compressors (Xu et al., 2018). In VRC scheme, latent heat generated by compression of overhead vapor stream has the potential to transfer energy by internal source Q_{CV} so that heat load by an external source (Q_E) can be minimized and this concept can reduce external energy consumption. Based on internal, external sources it exists in two general cases (Xu et al., 2017).

Two manipulation criteria are employed to maintain (i) temperature difference, i.e., $\Delta T_T = T_{out} - T_B$ of atleast $15^0 C$ for total condensation in reboiler and (ii) same reboiler heat duty. (i) The vapor is compressed so that the compressed overhead vapor and bottom liquid (T_B) have a reasonable temperature difference ($\Delta T_T = 15^0 C$) for total condensation in the reboiler. To maintain $\Delta T_T = 15^0 C$ for total condensation of overhead vapor in the reboiler-condenser and to avoid compressor operation at maximum CR throughout the batch operation, a variable speed compressor (manipulation of CR) has been used.

Manipulation of CR:

We know the ΔT_T,T_B and T_{in} (top tray temperature), then calculate the compressor outlet temperature (T_{out}) from Equation (2.20), and then find out the corresponding CR to maintain constant value which means CR is variable (Equation 2.22).

$$\Delta T_T = T_{out} - T_B \tag{2.20}$$

$$T_{out} = \Delta T_T + T_B \tag{2.21}$$

$$CR = \frac{P_{out}}{P_{in}} = \left[\frac{T_{out}}{T_{in}}\right]^{\left(\frac{\mu}{\mu-1}\right)} \tag{2.22}$$

Step 5: The energy is released by the compressor vapour (Q_{CV}) is calculated from the following equation.

$$Q_{CV} = V_{NT} \times \lambda \tag{2.23}$$

(ii) An iterative vapor flow rate manipulation has been employed to accomplish the same dynamical performance between the standard CBDS and SiVRBDS, i.e., maintaining the same reboiler heat duty (Q_R).

Manipulation of vapor flow rate:

If the energy released by the compressed vapour (Q_{CV}) exceeds the required reboiler heat duty, split the top vapor into two parts: One part (V_{Comp}) goes to the compressor to raise the temperature and the rest (V_{Cond}) to the condenser. If the compressed vapor latent heat (Q_{CV}) is less than the required reboiler heat duty, external heat (Q_E) must be supplied to the reboiler. Compressor work (W_{comp}) is calculated by Equation (2.27). The mathematical manipulation scenarios are presented below.

$$V_{NT} = V_{Comp} + V_{Cond} \tag{2.24}$$

If $Q_{CV} > Q_R$

$$V_{Comp} = Q_R/\lambda \tag{2.25}$$

$$V_{Cond} = V_{NT} - V_{Comp} \tag{2.26}$$

$$W_{comp} = \frac{(3.03\times10^{-5})\mu\, V_{comp}\, T_{in}}{\mu-1}\left[\left(\frac{P_{out}}{P_{in}}\right)^{\frac{\mu-1}{n\mu}} - 1\right] \tag{2.27}$$

$$Q_E = 0 \tag{2.28}$$

else

$$Q_E = Q_R - Q_{CV} \tag{2.29}$$

end

Where, $\mu -$ polytropic coefficient, P_{in} & P_{out} – inlet and outlet pressures of compressor, respectively

The performance indicator of VRC system is the total energy consumption given below

$$\text{Energy savings, \%} = \frac{Q_{CMBD} - Q_{cons,VRMBD}}{Q_{CMBD}} \times 100 \tag{2.30}$$

2.5 Closed-loop CMBD integrated with VRC

The effect of controlling the composition on energy savings is investigated. Figure 2.5 is representing a close loop with SVRC scheme due to its infeasibility of compression ratio, one more variable compressor is added to enhance the efficiency of the VRC scheme is shown in Figure 2.6.

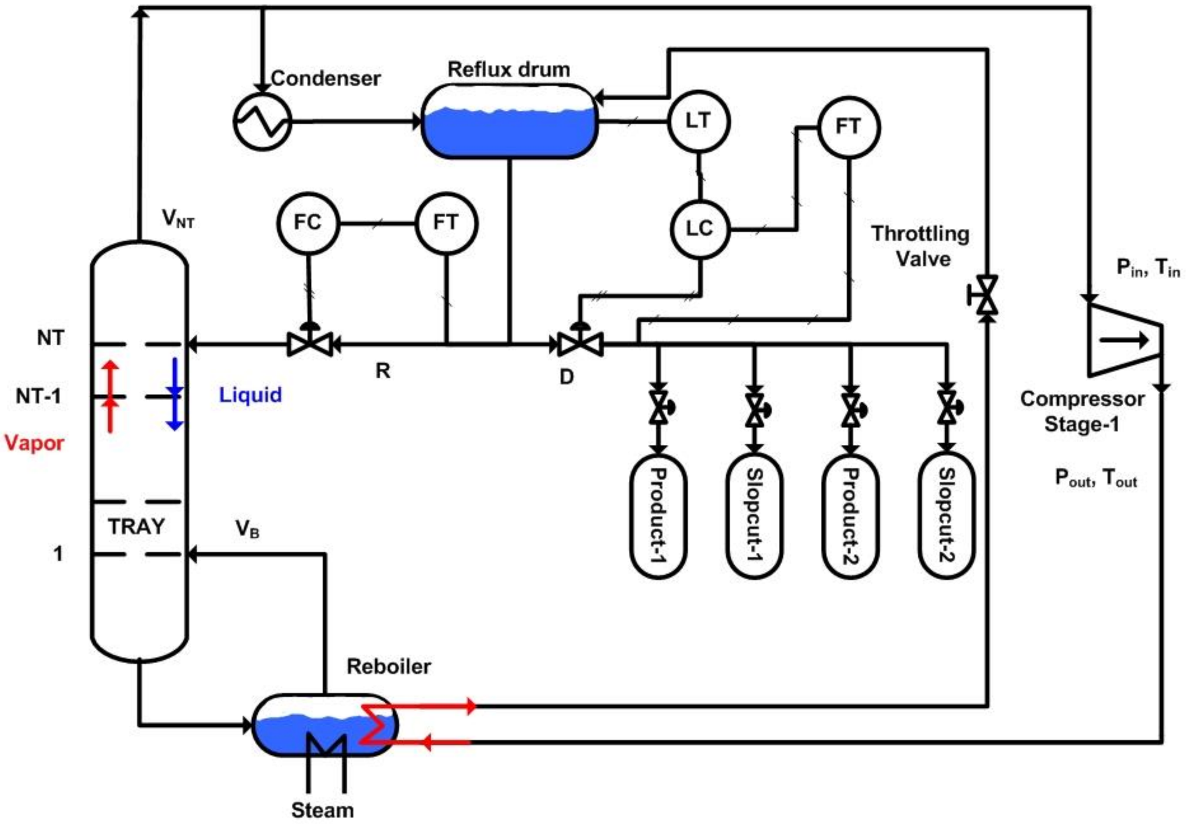

Figure 2.5. Close loop Multicomponent Batch Distillation with VRC scheme: Single stage

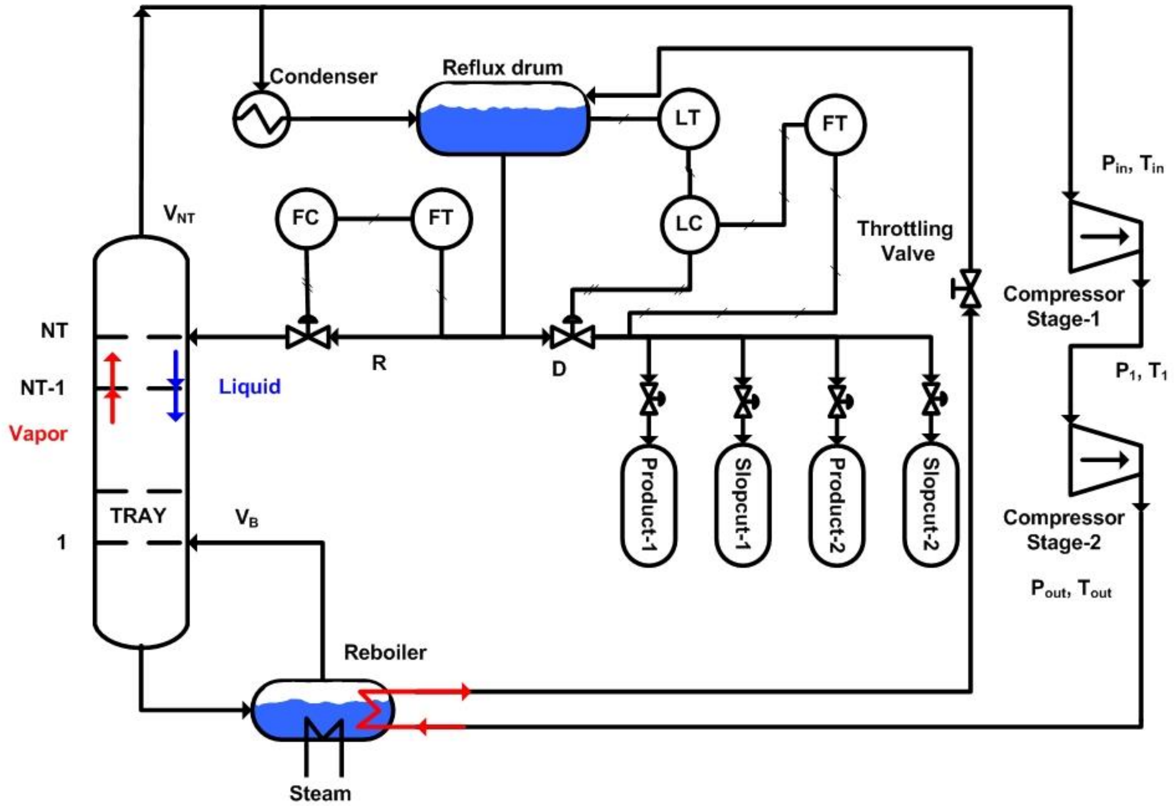

Figure 2.6. Close loop Multicomponent Batch Distillation with VRC scheme: Double stage

2.6 Results and Discussion

2.6.1 Open-loop Dynamics of CMBD

Startup phase: once feed is charged in the column, heat is supplied to the bottom of the column to vaporize the feed mixture. The lighter component purity gradually increases and reaches to steady state. The startup phase runs at total reflux condition means no withdrawal of product in this phase. Here the given system has reached the steady state purity 0.999 of lighter component in 149.93 min.

Production phase: After the startup phase, production of lighter component (Hexanol) starts immediately till an average composition greater than equal to the desired purity 0.99. If the average composition of lighter component is less than the desired purity then distillate amount is collected as slopcut-1, during this region an intermediate component (octanol) composition starts raising gradually and reaches the desired purity 0.98. Once the intermediate component reaches 0.98, immediately production of intermediate component starts till the average composition of the intermediate component is greater than the desired purity. Again, if the composition of intermediate component is less than average composition then slopcut-2 starts till the heavier component Decanol reaches its desired purity as shown in Figure 2.7. Once heavier component reaches its desired purity of 0.90 system terminates and product-3 is collected from the bottom of the column as the entire amount available in batch distillation.

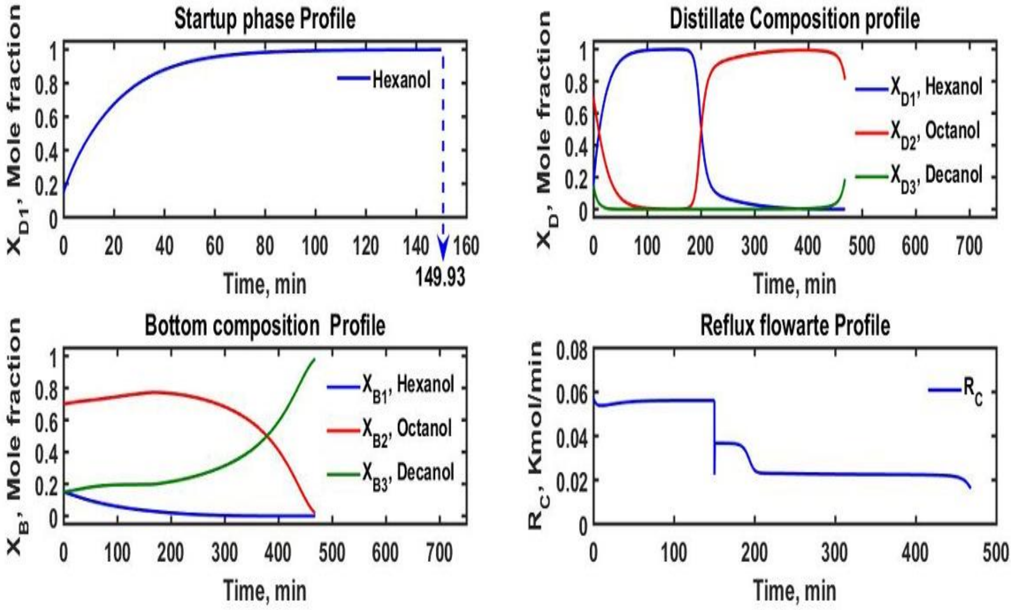

Figure 2.7. CMBD profile throughout the operation

2.6.2 Closed-loop dynamics of CMBD

Tight composition is a challenging task when distillation is done in batch operation because of time varying nature (Monroy-Loperena and Alvarez-Ramirez., 2003). The manipulating variable (M.V), Control variable (C.V) and Setpoint (S.P) are given in Table 2.2. The controllers tuning parameters and performance index values (**ISE**) are shown in Table 2.3. It is clear from the Table that low ISE values are obtained for GSPI controller compared to PI.

Table 2.2. M.V, C.V and S.P specification

System	S. P	C.V	M.V
Hexanol (product-1)	0.999	X_{D1}	R_C
Octanol (product-2)	0.99	X_{D2}	R_C
Holdup in reflux drum	0.26	H_D	D (±25%)

Table 2.3. Tuning parameters for PI and GSPI controller

	PI			GSPI		
	K_{c1}	τ_{c1}	**ISE₁**	K_{c2}	τ_{c2}	**ISE₂**
Product-1	35	2.5	8.24×10^{-6}	94	3	2.20×10^{-6}
Reflux drum holdup for product-1	-10	0.01	7.01×10^{-8}	-10	0.01	9.71×10^{-10}
Product-2	-50	2.5	8.13×10^{-7}	-50	2.5	3.48×10^{-7}
Reflux drum holdup for product-2	-10	0.01	7.42×10^{-8}	-90	0.01	9.20×10^{-10}

If the composition of product-1 is less than average desired purity 0.999 then slop cut-1 starts afterwards. Here controllers work in a sequence: during start-up phase or total reflux condition the controller doesn't work; once system reaches steady state the controller starts immediately to control the composition of product-1 (hexanol); then in slopcut-1 again controller doesn't work; if intermediate component octanol reaches the desired composition 0.99 then controller starts working to control the composition of product-2 (octanol) till the composition less than average desired purity (Kano et al., 2003).

Figure 2.8 representing tight control of lighter component hexanol by manipulating the Reflux flowrate. It is clear that to maintain the very high composition of lighter component reflux flow rate has to increase with time. However, the composition start to decrease and falls immediately due to the non-availability of Hexanol in the system. The holdup in the reflux drum also plays an important role in distillation operation to avoid disturbance in the column during operation it helps to maintain reflux and distillate flowrate throughout the operation, therefore, control of holdup in the reflux drum at a specified level (0.26) as shown in Figure 2.9 is also important and is controlled by manipulating distillate flowrate in a limit of ±25% of distillate flowrate 0.0337 used for CMBD (Seborg., 2010).

As in slopcut-1 region, intermediate component octanol composition reaches the desired composition 0.99 then immediately controller starts to work to control the product-2 composition at its desired purity (0.99) as shown in Figure 2.10 by manipulating reflux flow rate.

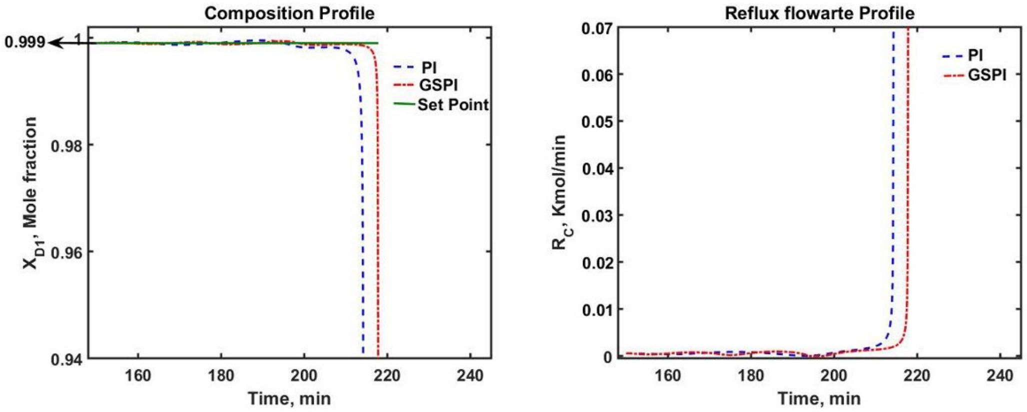

Figure 2.8. Closed loop production phase-1 profile without VRC scheme: Composition control

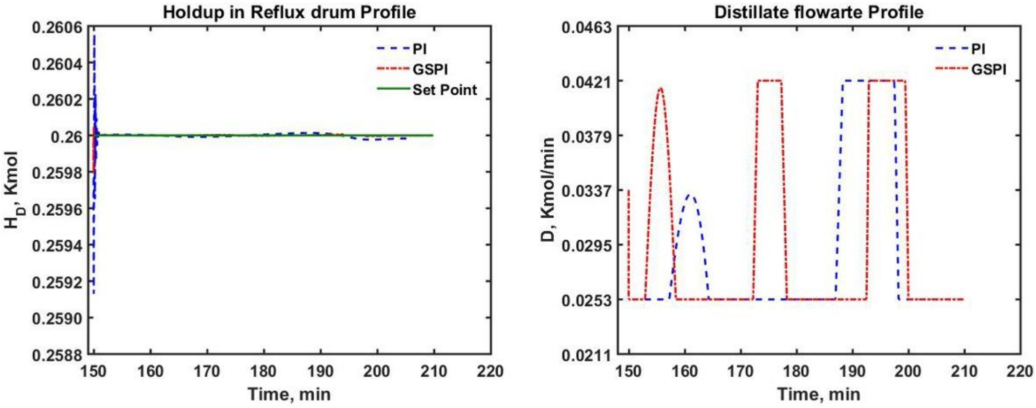

Figure 2.9. Closed loop production phase-1 profile without VRC scheme: Reflux drum holdup control

It is clear from figure that to maintain tight composition of product 2 reflux flow rate is increasing continuously. When the system switch from slopcut to production a sudden variation in the reflux drum can be seen in Figure 2.11 and later that variation is completely countered by proper tuning of controller. It is obvious that at low distillate flow rate, control is quite better than at high distillate flow rate. The results shows the advantage of GSPI controller is works for more time to maintain constant composition and withdraw more product amount as shown in Table 2.4.

2.6.3 CMBD integrated with single and double stage VRC: Open-loop

VRC scheme is implemented to the CMBD system so that the latent heat available in the system as shown in Figure 2.12 could be utilized as an internal heat source for the reboiler section. Initially energy consumed by the reboiler section was 2690 kJ/min at a constant rate throughout the operation. After heat integration, external heat is reduced means that reduction of natural resources as well as less emission of CO_2.

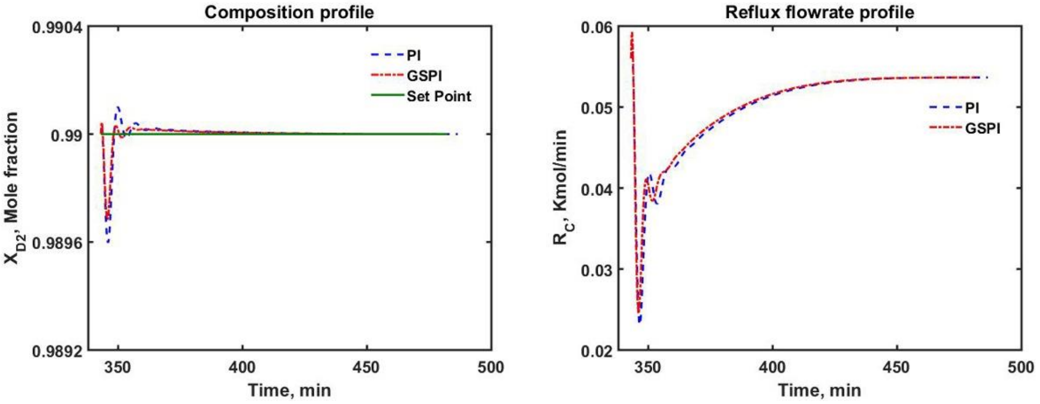

Figure 2.10. Closed loop production phase-2 profile without VRC scheme: Composition control

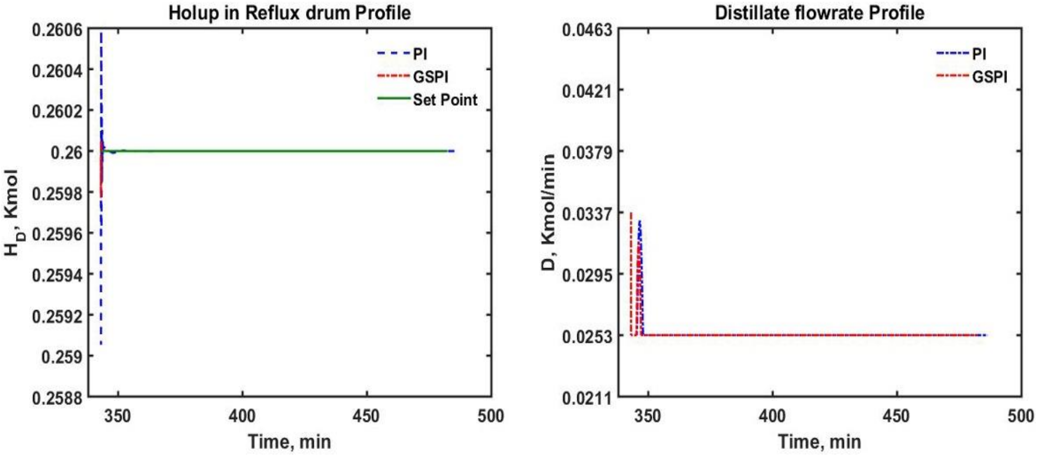

Figure 2.11. Closed loop production phase-2 profile without VRC scheme: Reflux drum holdup control

Table 2.4. Comparison between open loop and close loop product amount and energy consumption in CBD without VRC scheme

		SP purity	TSP, min	P1, Kmol	P1 avg. purity	TP1, min	P2, Kmol	P2 avg. purity	TP2, min	Total batch time, min	Energy Cons, KJ
Open loop	CBD	0.999	149.93	1.24	0.99	36.85	5.03	0.98	150.25	467.66	12.58×10^5
Close loop	PI	0.999	149.93	1.62	0.999	56.02	3.64	0.99	143.39	486.43	13.08×10^5
	GSPI	0.999	149.93	1.77	0.999	60.04	3.53	0.99	139.23	482.30	12.97×10^5

Figure 2.13 shows the compression ratio throughout the operation for SVRC and DVRC scheme as per the limitation of compression ratio as SVRC scheme required more than 20 C_R to compress the vapors completely which is beyond the C_R limits. Therefore, it suggests to add one more compressor (DVRC). In the DVRC scheme C_R is divided equally for both compressor, resulting DVRC is under limitation and is acceptable.

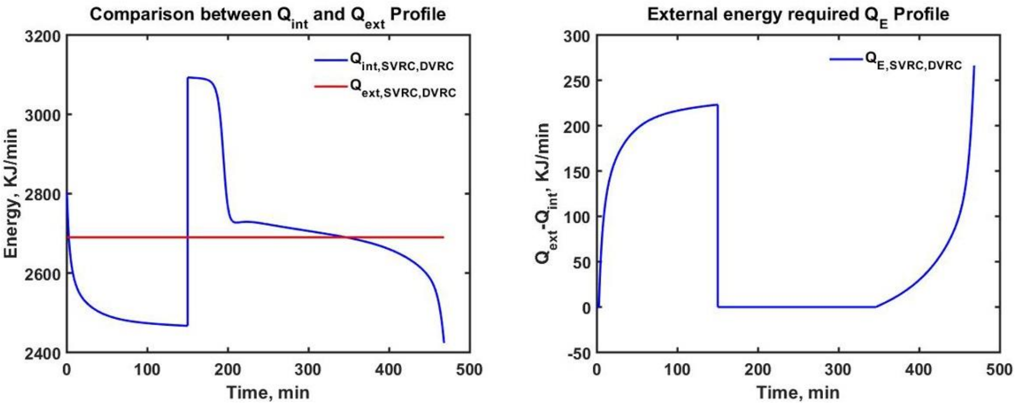

Figure 2.12. Profile with VRC scheme: Energy available

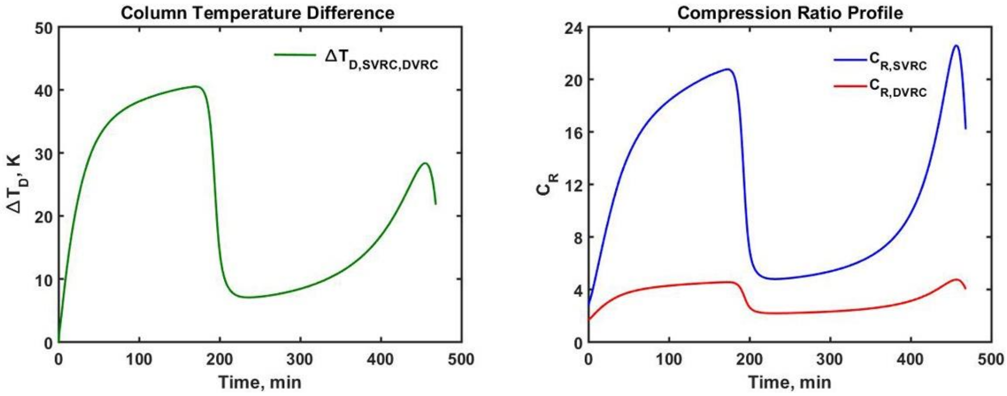

Figure 2.13. Profile with VRC scheme: Temperature difference in column and Compression ratio

2.6.4 CMBD integrated with single and double stage VRC: Closed-loop

In this work, we have implemented two controllers (PI and GSPI) to maintain constant high purity of product. But it may happen at the expense of the high energy consumption depending on the behavior of the system. It is obvious that to control high purity batch time will increase so energy consumption will be more which is considered as a performance indicator. To overcome this energy consumption VRC is integrated with the closed-loop batch operation. Figure 2.14 show the energy available in the system where Q_{ext} is external energy and Q_{int} is energy available in the system. The total auxiliary energy supplied to the system throughout the operation curve has oscillatory behavior during product-1 which is due to high purity control where the system is highly sensitive. Table 2.5 shows the total energy consumption for CMBD, SVRC and DVRC schemes.

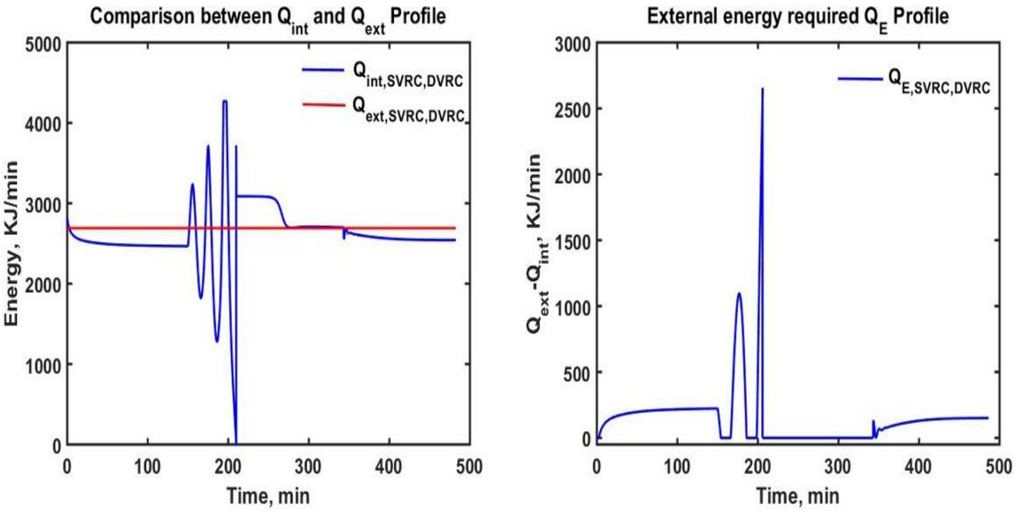

Figure 2.14. Closed loop profile with VRC scheme: Energy available

Table 2.5. Product amount and energy consumption in CMBD with multistage VRC scheme

		SP purity	T_{SP}, min	P_1, Kmol	P_1 avg. purity	TP_1, min	P_2, Kmol	P_2 avg. purity	T_{P2}, min	Total batch time, min	Energy Cons., KJ
SVRC	CMBD	0.999	149.93	1.24	0.99	36.85	5.03	0.98	150.25	467.66	7.485×10^5
	PI	0.999	149.93	1.62	0.999	56.02	3.64	0.99	143.39	486.43	8.153×10^5
	GSPI	0.999	149.93	1.77	0.999	60.04	3.53	0.99	139.23	482.30	8.259×10^5
DVRC	CMBD	0.999	149.93	1.24	0.99	36.85	5.03	0.98	150.25	467.66	7.334×10^5
	PI	0.999	149.93	1.62	0.999	56.02	3.64	0.99	143.39	486.43	8.016×10^5
	GSPI	0.999	149.93	1.77	0.999	60.04	3.53	0.99	139.23	482.30	8.086×10^5

Figure 2.15 represents the compression ratio during PI and GSPI controller (comparatively same) for SVRC and DVRC. It is observed that SVRC has a very high compression ratio which is not feasible. Therefore, as an indication to add one more compressor stage, in double stage VRC, the Compression ratio is under the limitation and acceptable. As distillation operation consumes a significant part of the energy in chemical industries, therefore, even 1% of energy-saving may save many barrels of fossil fuel as an essential source of energy

for distillation is fuel oil which produces an enormous amount of unburned carbon in the atmosphere which leads to global warming. VRC scheme with batch operation has given approximately 36-42% of energy saving with different cases as given in Table 2.6.

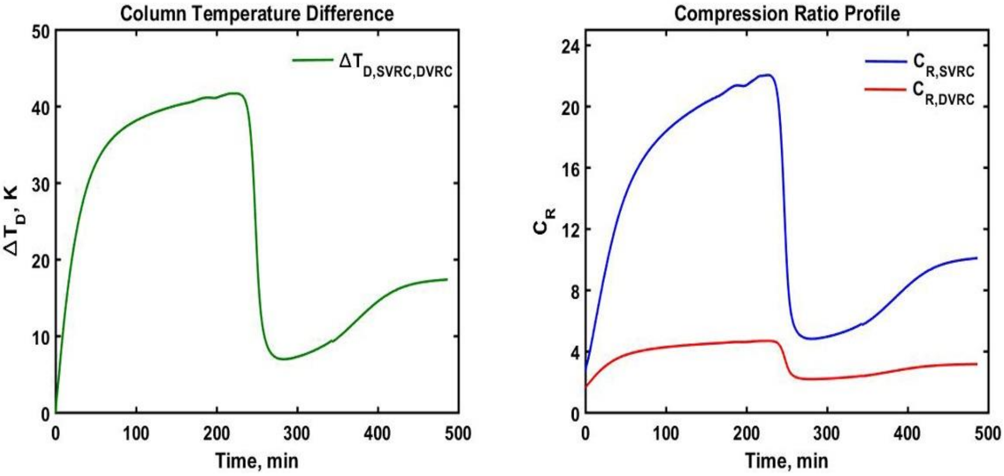

Figure 2.15. Closed loop profile with VRC scheme: Temperature difference in column and Compression ratio

Table 2.6. Comparison between the energy consumption in open loop and close loop with and without VRC scheme.

		Without VRC	With VRC			
			SVRC		*DVRC*	
		Energy cons. KJ	*Energy cons. KJ*	*Energy saving, %*	*Energy cons. KJ*	*Energy saving, %*
Open loop	CMBD	12.58×10^5	7.485×10^5	40.50	7.334×10^5	41.70
Close loop	PI	13.08×10^5	8.153×10^5	37.67	8.016×10^5	38.72
	GSPI	12.97×10^5	8.259×10^5	36.32	8.086×10^5	37.66

2.7 Conclusion

This work investigates tight composition control of high purity, multicomponent batch distillation for separating zeotropic mixture with and without vapor recompression scheme. Further, double-stage variable speed compressors are used in this study to improve the energy consumption under the limitation of compressors. The comparative analysis has been carried out between open-loop and closed-loop with and without vapor recompression schemes. For tight composition control with vapor recompression, the GSPI controller gives better control performance in terms of ISE and more product-1 amount at very high purity (0.999) than conventional PI. Furthermore, it is observed that energy consumption for an open-loop system with VRC is less, around 40% less than the energy consumed by the open-loop system without the VRC scheme. However, energy consumption for a closed-loop with VRC is approximately 36-38% less than a closed-loop without VRC. Therefore, both systems show nearly the same energy savings. Overall observation concludes that the system with GSPI controller integrated with DVRC gives a higher product amount at very high purity than PI and energy savings compared to closed-loop SVRC.

Chapter 3

Introduction of novel multi-stage vapor recompression approach in batch distillation

Chapter 3

3. Introduction of novel multi-stage vapor recompression approach in batch distillation*

3.1 Introduction

Distillation is the most common unit operation for separating liquid mixtures in the chemical processing industry, accounting for over 95% of fluid separation in the chemical and associated sectors (Sinnott et al. 2010). On the other hand, distillation uses a substantial amount of energy in the chemical process industry, increasing the amount of greenhouse gases discharged into the atmosphere. Distillation is one of the natural processes for enhancement due to its high energy consumption. The need to develop cleaner, smaller, and more energy-efficient technology for the distillation process have been characterized as process intensification (Cabrera-Ruiz et al., 2011). The chemical process sector requires a minimum TAC and CO_2 emissions in energy-efficient systems. In fact, there is a high association between energy consumption, total annual cost, and CO_2 emissions to the environment in the distillation process.

Many energy integration techniques have been developed in recent decades, especially for continuous distillation to improve performance (Jogwar and Daoutidis, 2010) Energy may be integrated into distillation operations using internal and external heat integration systems. The heat integrated distillation column (HIDiC) for continuous distillation columns (Naito et al.,2000; Gadalla et al., 2005; wakabe et al., 2003; Suphanit et al., 2010; Shenvi et al., 2011) the most typical example of the former technology, whereas the vapor recompression (VRC) or heat pumping is based on external heat integration (Jana, 2016; Maiti et al., 2011).

In small-scale manufacturing, such as high-value-added products, fine and specialized chemicals, and the pharmaceutical industry, the batch distillation column is the most common unit operation. Batch distillation is characterized by its flexibility since a single column can separate and produce numerous different types of high purity components from a multicomponent feed.

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On the other hand, batch distillation consumes significantly more energy than continuous distillation. VRC is a technology that can improve batch distillation performance in many separation systems, including binary and multicomponent mixtures. Because the temperature difference between the column $\Delta T_B = T_B - T_{NT}$) i.e., the difference between the reboiler temperature (T_B) and top tray temperature (T_{NT}) is small in separations involves close boiling mixtures, VRC has a strong potential for separations of close boiling liquid binary multicomponent mixtures. The majority of research on VRC was focused on the separation of closed boiling liquid mixtures (Kiran et al., 2015; Babu et al., 2012; Modla and Lang, 2013). However, for moderate wide-boiling binary mixtures, where ΔT_B is moderate, (Khan et al., 2012; Parhi et al., 2019; Vibhute et al., 2020) shown that VRC in conventional batch distillation is economically and energetically appealing. In the published literature, VRC in batch columns for separating multicomponent high, wide boiling/zeotropic mixtures has received very little attention. A two-stage VRC scheme was recently presented to improve the performance of a ternary wide boiling mixture, i.e., hexanol–octanol–decanol separation. The highest boiling component is not conspicuous in the feed composition, thereby acting as a close boiling mixture. Despite having a high CR, the proposed solution saves 52 percent of energy and 12.21 percent of TAC (Nair et al., 2017).

When applied to an existing classical batch distillation column, the parameters that have the most impact on the assessment of the vapor recompression heat pump economic and environmental performance are the temperature difference of the column, as well as the column capacity and compression ratio (CR). In addition, the number of theoretical trays (NT) has a significant impact on ΔT_B in heat pump-assisted vapor recompressed batch distillation systems (VRBD) when compared to conventional multicomponent batch distillation (CMBD). CMBD and VRBD grow when the NT increases, batch time decreases, total energy consumption decreases (less fuel consumed = less CO_2 emissions), and TAC increases (capital cost increases and operating cost decreases). However, in a cost-benefit comparative analysis between CBD and VRBD, TAC savings rise to a maximum (due to minimal capital investment) and then decline to a minimum when the number of trays grows (due to high capital investment). As a result, while designing a heat pump-assisted batch distillation system, the number of trays must be chosen so that the maximum TAC and CO_2 savings are realized compared to conventional batch distillation systems. Furthermore, to reduce TAC and CO_2 emissions, vapor recompressed batch distillation's operating and design conditions should be adjusted for separating wide boiling mixtures (high-temperature lifts). This fact motivated us to investigate the operational and economic feasibility of the energy-integrated VRC technology in batch distillation to separate high wide boiling ternary components.

First, for conventional multicomponent batch distillation (CMBD) and single-stage vapor recompressed multicomponent batch distillation (SiVRMBD), a systematic simulation approach was developed to determine the optimal number of stages based on the lowest energy and TAC reduction. The authors used SiVRMBD and CMBD to show how the stages affect performance. Furthermore, it has been observed that in SiVRMBD, a substantial compression ratio (CR) is necessary throughout the batch operation, resulting in compressor wear and tear. As a result, there are some restrictions to putting the SiVRMBD technique into practice. If CR exceeds 3.5 throughout the operation at any time instant, multi-stage vapor recompression was performed in this work (Chew et al., 2014). As a result, the authors proposed a novel optimal multi-stage vapor recompression approach for batch distillation that lowers the CR, reduces wear and tear, and improves heat pump performance in high-temperature lift scenarios. Finally, the influence of different feed compositions on the performance of the suggested VRC schemes was studied.

To the best of the authors' knowledge, this is the first paper to present a systematic approach for determining the optimal number of stages based on VRC performance evaluation indices, as well as a novel optimal multi-stage VRC approach to improve the performance and practical feasibility for separation of ternary zeotropic wide boiling mixtures.

3.2 Performance evaluation indices

3.2.1 Energy savings

In batch distillation, energy consumption is a key performance measure for evaluating the energy efficiency of heat pump assisted VRCs. The sum of the total reboiler heat duty ($\mathrm{Q_R}$) plus three times the compressor duty ($\mathrm{Q_{comp}}$) yields total energy consumption of vapor recompressed batch distillation ($\mathrm{Q_{cons,VRMBD}}$)

$$\mathrm{Q_{cons,VRMBD}} = \mathrm{Q_R} + 3\mathrm{Q_{comp}} \tag{3.1}$$

The conventional multicomponent batch distillation energy consumption ($\mathrm{Q_{conc,CMBD}}$) is calculated from:

$$\mathrm{Q_{conc,CMBD}} = \text{Batch time } \times \mathrm{Q_R} \tag{3.2}$$

When comparing vapor recompressed multi component batch distillation (VRMBD) to CMBD, the corresponding energy savings are

$$\text{Energy savings, \%} = \frac{Q_{CMBD} - Q_{cons,VRMBD}}{Q_{CMBD}} \times 100 \tag{3.3}$$

3.2.2 Total annual cost

TAC has been chosen as one of the most important indicators of economic performance. The TAC is determined by the following Equation

$$\text{TAC}\left(\frac{\$}{\text{year}}\right) = \text{TOC} + \left(\frac{\text{TCI}}{\text{payback period}}\right) \tag{3.4}$$

TOC stands for total annual operating cost, while TCI stands for total capital investment. The capital investment includes the installation cost of apparatus (reboiler, condenser, column shell, column trays, intercoolers and compressors). The operating costs includes the electricity, cooling water and steam (Babu et al., 2012). In this study, the modernised price of the Marshell and Swift (M&S) index of 1704.9 was used .While heat pumps offer a high energy efficiency rating for separating high temperature lifts, it is difficult to achieve economic benefits over a shorter payback period. Additional costs such as reflux drums, pumps, valves, and piping can be eliminated because they are far less expensive than column vessels and heat exchangers. The TCI and TOC were calculated using correlation methods from (Douglas, 1988) are given below:

Column shell (MOC: SS)

Installed cost (\$) = $\left(\frac{M\&S}{280}\right)101.9\,D_c^{1.066}L_c^{0.802}(c_{in}+c_m c_p)$

where, D_c is the column diameter (ft), L_c the column height (ft), $M\&S$ =950, and the coefficients c_{in}=2.18, c_m=3.67 and c_p=1.0.

■ *Column tray* (Type: bubble-cap, MOC: SS)

Installed cost (\$) = $\left(\frac{M\&S}{280}\right)4.7\,D_c^{1.55}L_c(c_s+c_t+c_m)$

where, the coefficients c_s=1, c_t = 1.8 and c_m=1.7.

■ *Heat exchanger* (MOC: SS)

Installed cost (\$) = $\left(\frac{M\&S}{280}\right)101.3\,A^{0.65}(c_{in}+c_m(c_t+c_p))$

where, A is the heat transfer area (ft^2), and the coefficients c_{in}=2.29, c_m=3.75, c_t=1.35 and c_p=0.

■ *Compressor*

Installed cost (\$) = $\left(\frac{M\,\&\,S}{280}\right)517.5\,(bhp)^{0.82}\left(2.11+F_d\right)$

where F_d = 1.0. This expression is valid in the range of 30 < bhp < 10000.

3.2.3 Greenhouse gas emissions

Batch distillation is a high-energy unit operation that contributes significantly to greenhouse gas (CO_2) emissions. Reducing CO_2 emissions is a must and a challenge for the chemical processing industry to meet the Kyoto Protocol's environmental goals. Gadalla et al., (2005) developed a simple model for computing CO_2 emissions from an integrated heat distillation system, and we used the same model in our investigation. The heat pump-assisted VRC scheme requires steam to boil the liquid in the reboiler and power (electricity) for the compressor, whereas the CMBD scheme uses steam to boil the liquid in the reboiler. As a result, there are two utility driving systems for steam and power generation: steam boilers and turbines. We examined carbon dioxide emissions using natural gas as heating fuels in this work.

In this work, we compare the emission levels by using natural gas as heating fuels in both the utility devises. In the utility system, CO_2 emissions are related to the amount of fuel burnt by the following stoichiometric equation as (Smith and Delaby, 1991; Gadalla et al., 2005):

$$\mathrm{C_n H_m} + \left(\mathrm{n} + \frac{\mathrm{m}}{4}\right)\mathrm{O_2} \rightarrow \mathrm{nCO_2} + \frac{\mathrm{m}}{2}\mathrm{H_2O} \tag{3.5}$$

Air must be supplied to burners in excess to ensure complete combustion. It is true that the amount of CO can be reduced to negligible levels by excess supply of air, proper burner design, regular maintenance and adequate control. Here, we assume that all carbon in the fuel is reacted to CO_2.

In a heating devise, CO_2 emissions are related as:

$$\mathrm{CO_2\ flow\ rate\ (kg/hr)} = \frac{Q_{\mathrm{Fuel}}}{\mathrm{NHV}} \times \frac{\mathrm{C\%}}{100} \times \alpha \tag{3.6}$$

Where, Q_{Fuel} represents the heat duty from fuel burnt (kJ/hr), NHV the fuel net heating value (kJ/kg), C% the mass percentage carbon in fuel, and α the ratio of CO_2 and carbon molar masses (= 3.67).

CO_2 emissions from steam boiler

The fuel is combusted when mixed with air, producing flue gases that can be used as a heat source in the utility systems. Actually, the flue gases release heat when they are cooled from the flame temperature (T_{FT}) to the stack temperature (T_{Stack}). For a steam boiler, the flame temperature (T_{FTB}) and T_{Stack} are adopted as 1800^0C and 160^0C, respectively (Smith and Delaby, 1991).

The steam may be produced in a steam boiler either at the temperature required by the process or at a higher temperature and then throttled. In the later scheme, the boiler feed water is added after expansion (desuperheating) in order to maintain the steam quality.

Making a heat balance around the boiler, we obtain:

$$Q_{\mathrm{Fuel}} = \frac{Q_{\mathrm{Proc}}}{\lambda_{\mathrm{Proc}}}(h_{\mathrm{Proc}} - h_{\mathrm{FW}})\frac{T_{\mathrm{FTB}} - T_0}{T_{\mathrm{FTB}} - T_{\mathrm{Stack}}} \tag{3.7}$$

Where, λ_{Proc} and h_{Proc} denote the latent heat (kJ/kg) and enthalpy (kJ/kg) of steam delivered to the process, respectively, Q_{Proc} the process heat duty (kW), h_{FW} the enthalpy (kJ/kg) of feed water (FW) and T_0 the ambient temperature (= 25^0C). Now, by using Eq. (5.2), one can calculate the CO_2 emissions from steam boiler.

CO_2 emissions from gas turbine

This section deals with the model for combustion of a fuel in a gas turbine. This utility system can typically provide both heat as well as power to the process. However, here we employ it to generate power for the compressor unit integrated in the heat integrated scheme.

The amount of fuel burnt to supply Q_{Proc} can be calculated from:

$$Q_{\mathrm{Fuel}} = \frac{Q_{\mathrm{Proc}}}{\eta_{\mathrm{GT}}}\,\frac{1}{1-\eta_{\mathrm{C}}} \tag{3.8}$$

Where, the efficiency of gas turbine

$$\eta_{\mathrm{GT}} = \frac{T_{\mathrm{out}} - T_{\mathrm{Stack}}}{T_{\mathrm{out}} - T_0} \tag{3.9}$$

and the Carnot factor

$$\eta_{\mathrm{C}} = \frac{T_{\mathrm{in}} - T_{\mathrm{out}}}{T_{\mathrm{in}} + 273} \tag{3.10}$$

Here, T_{in} is the inlet temperature to the gas turbine (i.e., after combustion chamber, ^{0}C) and T_{out} the outlet temperature from the gas turbine (^{0}C). These two temperatures vary according to the turbine design. As suggested by Smith and Delaby (1991), T_{in} of 1027^{0}C and T_{out} of 720^{0}C are adopted.

The power (electricity) delivered by a gas turbine, W_{GT} (kW) is estimated by applying a typical mechanical efficiency of 90% from:

$$W_{\mathrm{GT}} = 0.9\,\eta_{\mathrm{C}}\,Q_{\mathrm{Fuel}} \tag{3.11}$$

It is interesting to note that so far we discussed an approach for calculating the 'local emissions' from a gas turbine. The power generated from the gas turbine is either consumed in the process itself or exported to other consumers. In both cases, the central power station, which is situated somewhere else, has the possibility of reducing electricity production by the amount generated by the gas turbine. Thus, certain amounts of fuels can be saved at the central power plant, thereby reducing the CO_2 emissions. This, in turn, leads to a saving in the emissions at the central power station. Hence, the central power plant together with the utility system is suggested (Smith and Delaby, 1991) to be considered as one unit in emission calculations. The corresponding CO_2 emissions are called 'global emissions' and expressed as:

$$\text{Global emissions} = \underset{\text{process plant}}{\text{emissions from}} - \underset{\text{power station}}{\text{emissions saved at central}} \tag{3.12}$$

It should be noted that the same fuel is used in both the utility systems and the central power station when we compare the emission levels between the VRC and CBD columns.

The above mentioned performance indices are used in the subsequent studies in the thesis.

3.3 Conventional multi component batch distillation column (CMBD)

The topology of an unsteady state conventional multicomponent batch distillation column

(CMBD) was designed to separate ternary wide boiling point mixtures in this study. The dynamic model was developed using the framework presented in Figure 3.1. CMBD dynamics are made up of algebraic-differential equations that are applied in (Jana et al., 2018).

This model contains the following simplification assumptions: The molar vapor holdup is negligible in comparison to the molar liquid holdup, the liquid and vapor are perfectly mixed on each tray, the delay time between plates was ignored, and atmospheric pressure, the nonlinear Francis–Weir formula for tray hydraulics, and the NRTL thermodynamic model for vapor-liquid compositions are all taken into account. The following is a step-by-step systematic, iterative simulation approach (Table 3.1) for determining Energy and TAC for various trays.

Table 3.1. Approach 1: A systematic design, operation and simulation approach

Step 1: Input information on the column: The column specifications (weir length and weir height), feed specifications (composition, flow rate and temperature), tray efficiency, initial conditions such as liquid compositions, liquid holdup on all trays etc.,)

Step 2. Specify the average composition for all products

Step 3: The liquid feed is introduced into the reboiler with the initial feed composition specified

Step 4: Choose the number of trays (NT)

Step 5: The reboiler receives a constant heat input (fixed reboiler heat duty) and the vapor begins to rise.

Step 6: The vapor passes through the perforations in the plate and into the condenser. The vapor from the top tray begins to condense, and the reflux drum begins to fill up.

Step 7: Total reflux (start-up phase) commences, implying that all condensed liquid from the reflux drum is introduced to the top tray column.

Step 8: The liquid begins to fall from the down comers, indicating that the plates have sufficient liquid holdup.

Step 9: Operational *Steps 5–7* are repeated until the lightest component purity in the reflux drum achieves its maximum and the composition remains unchanged (does not change with time). The operation is then said to have reached a steady state.

Step 10: When the operation has reaches a steady state, the column operation is switched from entire reflux (start-up phase) to production phase, with the distillate stream being removed.

Step 11. Choose a different distillate flow rate (*Step 10*) and, repeat the simulations if the average composition specifications of all products are not met.

Step 12. Calculate the energy from Equation (2) and TAC from Equation (3.4), if the average composition specifications of all products are met.

Step 13. Repeat *Steps 5* through *12* with a different number of trays (NT).

Step 14. Tabulate the Energy and TAC values for various NT

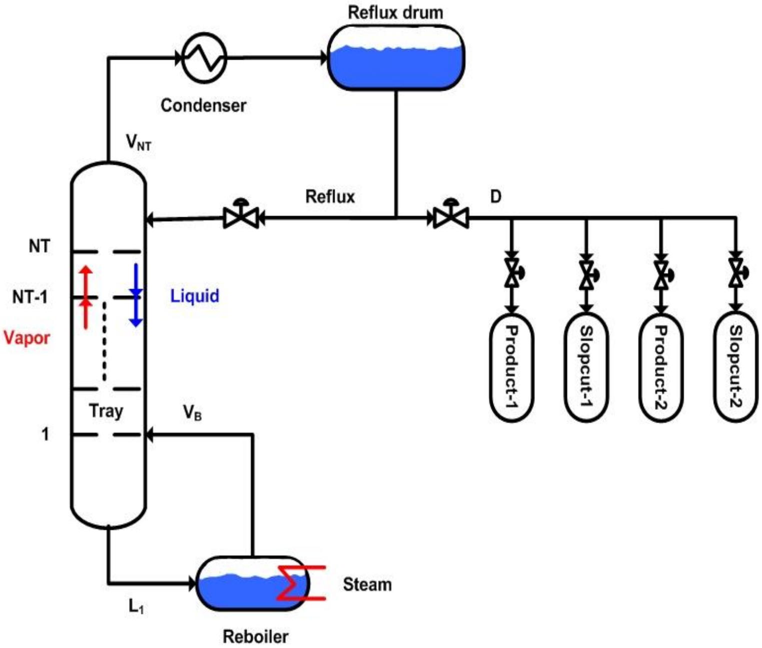

Figure 3.1. Schematic diagram of conventional multicomponent batch distillation (CMBD)

3.4 Single-stage vapor recompressed multi component batch distillation (SiVRMBD)

Figure 3.2 shows the principle operation of a VRC with a single compressor in multicomponent batch distillation conceptually. The primary goal of this VRC is to recover heat from top vapor, lowering hot and cold utility consumption. SiVRMBD is made up of an extra compressor that is separate from the standard CMBD.

The optimum design parameter, that is, the number of trays (NT) was identified using a following step by step systematic iterative simulation approach (Table 3.2) based on maximal TAC savings compared to CMBD.

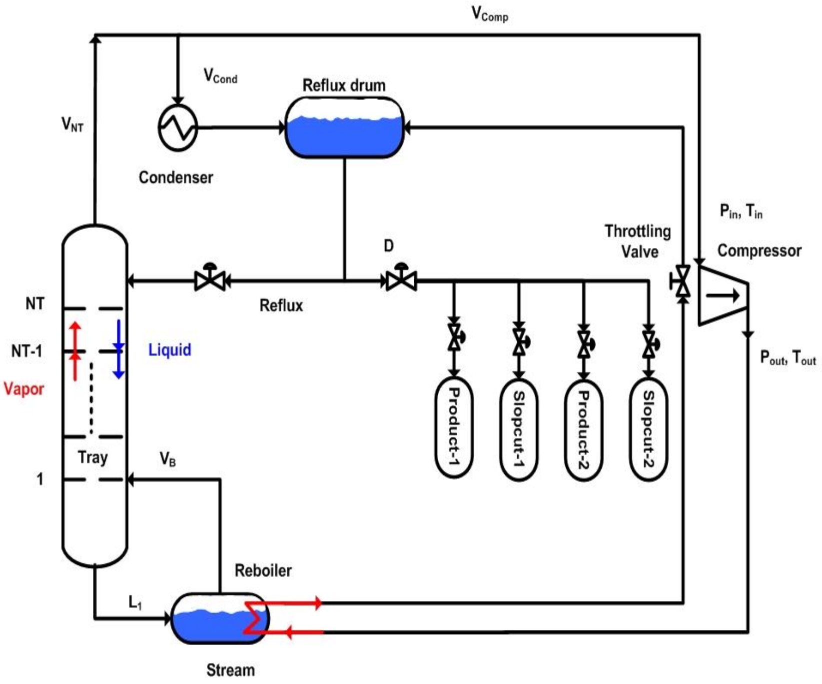

Figure 3.2. Schematic diagram of single-stage vapor recompressed multicomponent batch distillation (SiVRMBD)

Table 3.2. Approach 2: A systematic design, operation and simulation approach to identify the optimal number trays

Step 1: Carry out *Steps 1* through *6* of Approach 1**.**

Step 2: The vapor flow through the plate holes and reaches to the top tray.

Step 3: The compressor receives the vapor from the upper tray. The upper tray vapor ($\mathrm{V_{NT}}$) is compressed to the necessary pressure or temperature (temperature lift). The latent heat (λ) is released by compressed vapor, which is then used as heat input to the reboiler and condensed against the reboiler liquid.

- The vapor is compressed so that the compressed overhead vapor and bottom liquid ($\mathrm{T_B}$) have a reasonable temperature difference ($\Delta\mathrm{T_T}$) for total condensation in the reboiler-condenser. Because of the unstable nature of SiVRMBD, the compressor must run at maximum CR throughout the batch operation in order to maintain a $\Delta\mathrm{T_T}$ of at least $15^0 C$. To maintain $\Delta\mathrm{T_T} = 15^0 C$ for total condensation of overhead vapor in the reboiler-condenser and to avoid compressor operation at maximum CR throughout the batch operation, a variable speed compressor (manipulation of CR) has been used.

Manipulation of CR: We know the ΔT_T,T_B and T_{in} (top tray temperature), then calculate the compressor outlet temperature (T_{out}) from Equation (3.13), and then find out the corresponding CR to maintain constant value which means CR is variable (Equation 3.15).

$$\Delta T_T = T_{out} - T_B \tag{3.13}$$

$$T_{out} = \Delta T_T + T_B \tag{3.14}$$

$$CR = \frac{P_{out}}{P_{in}} = \left[\frac{T_{out}}{T_{in}}\right]^{\left(\frac{\mu}{\mu-1}\right)} \tag{3.15}$$

Step 4: Use the following Equation to calculate the energy released by compressor vapor.

$$Q_{CV} = V_{NT} \times \lambda \tag{3.16}$$

An iterative scenario including vapor flow rate modification has been used to achieve the same dynamical performance between the standard CMBD and SiVRMBD while retaining the same reboiler heat duty of 2690 kJ/min.

Manipulation of vapor flow rate: Split the top vapor into two halves if the latent heat released by the overhead vapor (V_{NT}) exceeds the required reboiler heat obligation. One part goes to the compressor (V_{Comp}) to raise the pressure, then to the bottom receiver for condensation and the rest goes to the condenser (V_{Cond}). Compressor work (W_{comp}) is calculated by Equation (3.20). If the compressed vapor latent heat (Q_{CV}) is less than the required reboiler heat duty, external heat (Q_E) must be supplied to the reboiler.

$$V_{NT} = V_{Comp} + V_{Cond} \tag{3.17}$$

If $Q_{CV} > Q_R$

$$V_{Comp} = Q_R/\lambda \tag{3.18}$$

$$V_{Cond} = V_{NT} - V_{Comp} \tag{3.19}$$

$$W_{comp} = \frac{(3.03\times10^{-5})\mu\, V_{comp}\, T_{in}}{\mu-1}\left[\left(\frac{P_{out}}{P_{in}}\right)^{\frac{\mu-1}{n\mu}} - 1\right] \tag{3.20}$$

$$Q_E = 0 \tag{3.21}$$

else

$$Q_E = Q_R - Q_{CV} \tag{3.22}$$

end

Where, μ – polytropic coefficient, P_{in} & P_{out} – inlet and outlet pressures of compressor, respectively

Step 5: The complete condensed liquid at high pressure is then throttled to top tray pressure and reaches the reflux drum. A throttling valve depressurizes the high pressure condensed liquid before it is returned to the reflux drum.

Step 6: *Steps 7* through *14* of Approach 1 should be repeated.

Finally, Calculate the Energy savings from Equation (3.3) and TAC savings from:

$$\text{TAC savings, \%} = \frac{\text{TAC}_{\text{CMBD}} - \text{TAC}_{\text{SiVRMBD}}}{\text{TAC}_{\text{CMBD}}} \times 100 \qquad (3.23)$$

3.5 Multi-stage vapor recompressed multicomponent batch distillation

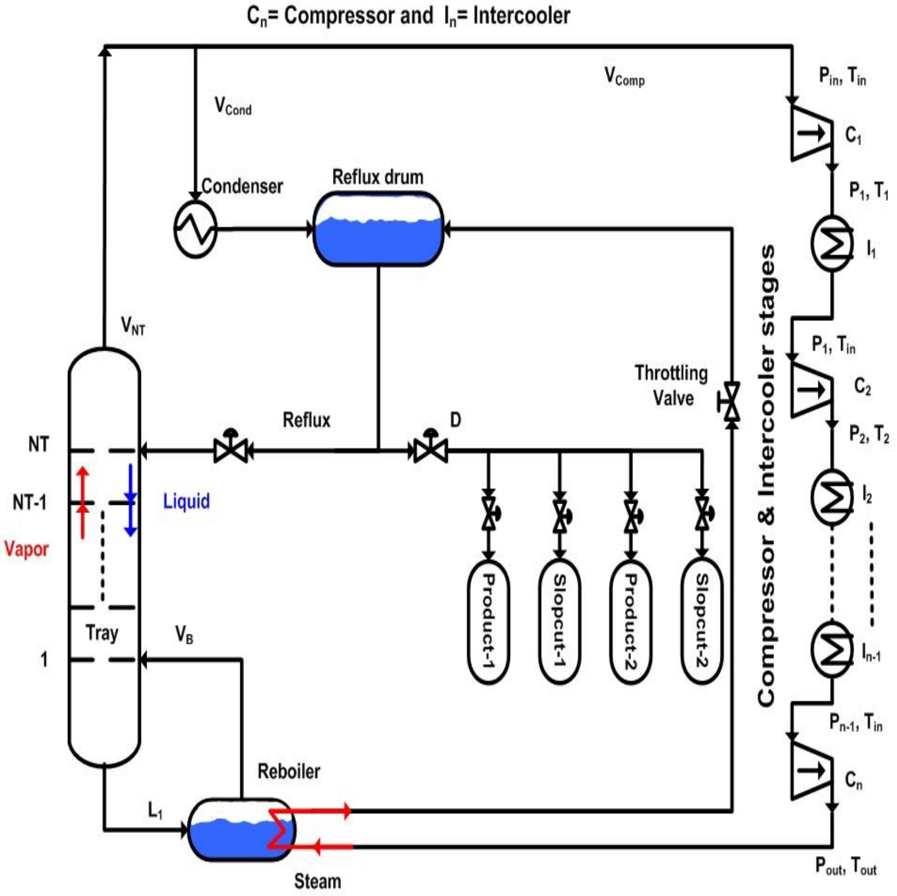

Figure 3.3. Schematic diagram of multi-stage vapor recompressed multicomponent batch distillation

To separate high-temperature lift mixtures in single-stage vapor recompressed batch distillation, a large compression ratio (CR) is required, which causes wear and tear difficulties in the compressor. As a result, the technique's implementation has practical constraints. In this work, we proposed a novel optimal multi-stage vapor recompressed approach and was used throughout the batch operation if the pressure ratio of SiVRMBD at any time step exceeds 3.5. Therefore, each variable speed compressor must run at a minimum CR of 3.5. However, if many compressors are required, it may not be the ideal option because the capital costs can grow excessively high. The goal of the proposed approach was to determine the optimal number of compressors, so each compressor operates at a minimum CR of 3.5, which affects compression work and capital costs. As a result, the proposed optimal multi-stage vapor recompression technique improves heat pump performance while lowering the CR. A schematic diagram of a multi-stage VRC system with intercoolers is shown in Figure 3.3.

A multi-stage VRC configuration consists of two or more compressors with intercoolers. In two-stage VRC configuration (consists of two compressors in series with intercooler between them), the overhead vapor mixture (V_{NT}) at atmospheric pressure P_{in} taken into the compressor 1 (C_1) and is compressed to a high pressure P_1. This pressure is intermediate between intake pressure (P_{in}) and delivery pressure P_2. Hence this is known as intermediate pressure ($P_1 = P_{opt}$). The compressed vapor mixture is then passed into an intercooler1((I_1). In the intercooler1, the mixture is cooled to initial temperature (T_{in}) at constant pressure by circulating cold water. The cooled mixture from the intercooler1 is then taken into the compressor 2 (C_2). In the compressor 2, the vapor mixture is further compressed to a required high pressure (P_2). The optimal pressure of the first compressor is the intake pressure of the second compressor, and the optimized pressure of the second compressor is the inlet pressure of the third compressor, and so on for multiple compressors. In multi-stage compression, assumptions are made to calculate the compressors' work: the pressure remains constant during suction and delivery, intercooling takes place at a steady pressure at each stage, compression method is the same for each stage, and no pressure drop between the two stages.

The sum of the work for each stage in an n-stage compression process is the total work, and estimated horsepower (hp) for an isentropic centrifugal compressor is computed by the following Equation.

$$W_{comp,n} = \frac{(3.03\times10^{-5})\,\mu\, V_{comp}\, T_{in}}{\mu-1}\left[\left(\frac{P_{opt}}{P_1}\right)^{\frac{\mu-1}{\mu}} - 1\right] + \frac{3.03\times10^{-5}\mu\, V_{comp}\, T_{in}}{\mu-1}\left[\left(\frac{P_2}{P_{opt}}\right)^{\frac{\mu-1}{\mu}} - 1\right] + \ldots \qquad (3.16)$$

n – number of compressors, $W_{comp,n}$ – total work of compressors, μ – polytrophic index, P_{opt} – optimum pressure, P_1– first stage compressed pressure, P_2 – second stage compressed pressure, T_{in} – inlet temperature, V_{comp} – vapour flow rate to compressor

Differentiating Equation (3.16) with regard to P_{opt} and setting the resulting expression equal to zero yields the optimum value of P_{opt}. The optimal pressure therefore becomes:

$$P_{opt} = (P_{in}\ P_{out})^{1/n} \tag{3.17}$$

As a result, the pressure ratio of each stage of the compressor is the same $(CR_1 = CR_2 = \cdots)$ and compression work at each stage is the same $\left(W_{comp,1} = W_{comp,2} = \cdots\right)$, and energy conservation is maximised. As a result, Equation (3.18) is used to determine the compression ratio at each stage, as well as Equation (3.19) for the overall work done by the compressors.

$$CR_n = \left(\frac{P_{out}}{P_{in}}\right)^{\frac{1}{n}}\ ;\ n = 1,2,3,4,5\ \ldots\ldots \tag{3.18}$$

$$W_{comp,n} = \frac{n\ (3.03\times10^{-5})\mu\ V_{comp}\ T_{in}}{\mu-1}\left[\left(\frac{P_{out}}{P_{in}}\right)^{\frac{\mu-1}{n\mu}} - 1\right] \tag{3.19}$$

In this Equation, the pressure (inlet pressure, P_{in} and outlet pressure, P_{out} is in lb_f/ft^2, and the vapor inflow rate to the compressor (V_{comp}) ft^3/min.

A novel simulation approach to determine the optimal number of compressors so that each compressor CR is less than 3.5 ($CR_{spec} < 3.5$) throughout the batch operation is given below.

Table 3.3. Approach 3: Proposed approach for identification of optimum number of compressors

Step 1: Perform *Steps 1* through *6* of Approach 1 using the ideal column design parameter (number of trays) and operational (distillate flow rate) parameter.

Step 2. The vapor flow through the plate holes and reaches to the top tray.

Step 3. Assume two-stage vapor recompression (DoVRMBD; n=2), which means that system consist of two compressors and a single intercooler between compressors.

Step 4. Perform *Step* 3 to *Step* 6 of Approach 2 (In *Step 4*, use Equation (3.18) with n=2 instead of Equation (3.12))

Step 5. Throughout the batch operation

> If $CR_n > CR_{spec}$ at any point throughout the batch operation. Assume three-stage vapor recompression (TrVRMBD; n=3), and repeat the *Step 4* and *Step 5* (In *Step 4*, use Equation (3.18) with n=3 instead of Equation (3.12))

Step 6. If $\mathrm{CR_n} < \mathrm{CR_{spec}}$, Stop the procedure; otherwise, continue *Steps 3* through *7* until the desired CR is achieved.

Step 7: Calculate the TAC and CO_2 emissions.

3.6 Results and discussion

The hexanol/octanol/decanol ternary wide boiling system was investigated in this work. This system is high wide boiling/zeotropic because the temperature difference between the lightest and heaviest components is significant. The column specifications utilized in computer simulations are listed in Table 3.4. The computer program was developed and simulated in MATLAB with a time step of 0.001. In the simulations, the initial compositions in all stages and reflux drum are equal to that of the feed mixture. The distillation column was designed for the specified average product purity of product 1 (hexanol – 98%), product 2 (octanol – 95%), and product 3 (decanol – 90%).

Table 3.4. System and column specifications

System	**Hexanol/octanol/decanol**
Total feed (H_{B0}), kmol	13
Tray efficiency, %	80
Heat input to the still pot, kJ/min	2690
Initial holdup on trays (H_{NT}), kmol	0.0118×NT
Initial condenser holdup (H_D), kmol	0.26
Initial reboiler holdup (H_B), kmol	$H_{B0} - H_{NT} - H_D$
Hexanol composition (steady state), mole fraction	0.999

3.6.1. Base case: An equimolar feed composition (0.33 – 0.33 – 0.34)

3.6.1.1. Single-stage vapor recompressed multicomponent batch distillation Vs CMBD

We demonstrated how the number of stages has the most significant impact on assessing the vapor recompression heat pump energy and economic performance when applied to an existing classical batch distillation column. Figure 3.4 and Table 3.5 provide the comparative analysis outcomes for the CMBD and SiVRMBD columns from approaches 1 and 2, respectively, to examine the impact of the factor as mentioned above. Figure 3.4 shows that as the number of trays increases, batch duration decreases while the temperature difference in the column increases. Therefore, an economic study is required to identify the appropriate number of theoretical trays.

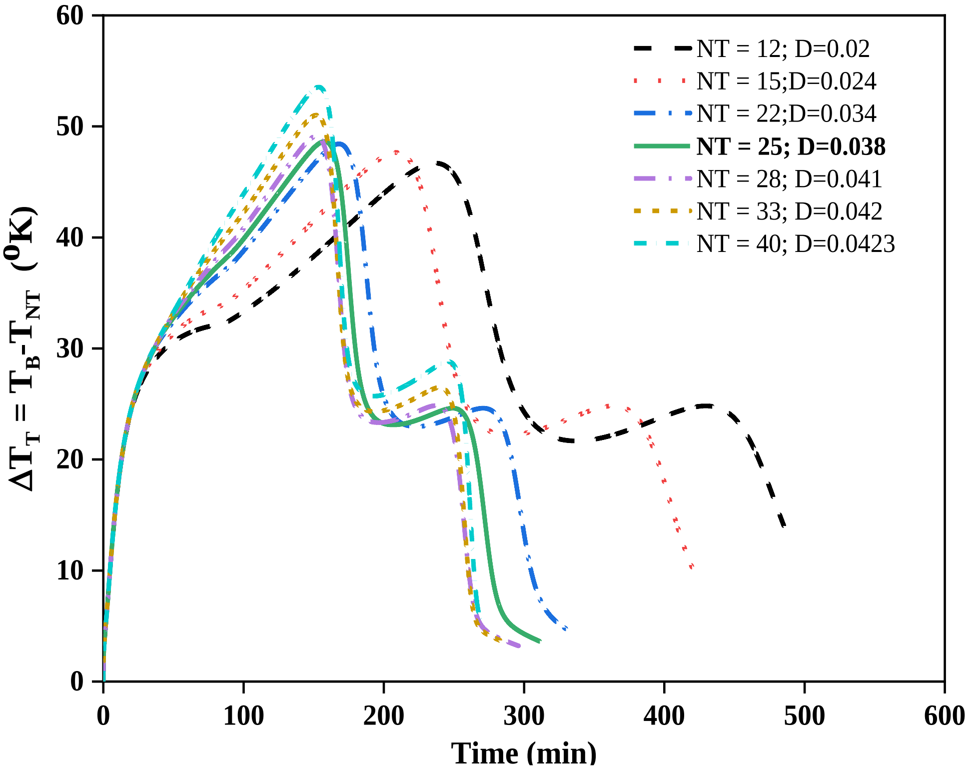

Figure 3.4. *Base case*: Temperature difference of the column profile at various trays throughout the batch operation

Table 3.5 shows that TAC savings grow as the number of trays increases from 12 to 25 due to low capital investment, whereas TAC savings drop as the number of trays increases above 25 due to increasing capital expenditure. It is evident from Table 3.5 that the largest TAC and energy savings are achieved with 25 trays. As a result, we have chosen the optimum number of trays of 25 in our subsequent simulations after examining the influence of equilibrium trays on separation, avoiding the usage of additional trays that would increase the initial investment expenses. The dynamical behavior of the batch distillation process is shown in Figures 3.5(a) (reflux drum composition) and (b) (reboiler composition) at an optimal number of stages of 25.

Table 3.5. Effect of number of trays on the performance of SiVRMBD: comparative analysis

Performance indices	**Number of stages/Distillate flow rate (kmol/min)**						
	12/0.02	15/0.024	22/0.034	**25/0.038**	28/0.041	33/0.042	40/0.0423
TAC ($/yr)							
CMBD×10^4	2.3723	2.598	2.9587	3.1064	3.2511	3.4865	5.994
SiVRMBD×10^4	2.3842	2.5524	2.8936	3.01	3.1636	3.44	6.038
%, Savings	-0.5	1.755	2.2	**3.1**	2.69	1.33	-0.73
Energy (kJ)							
CBD×10^5	13.08	11.35	8.9726	8.37	7.96	7.62	7.28
VRBD×10^5	9.4176	8.1853	6.3184	5.84	5.553	5.5235	5.59
%, Savings	28	27.88	29.5	**30.2**	30.2	27.51	23.21

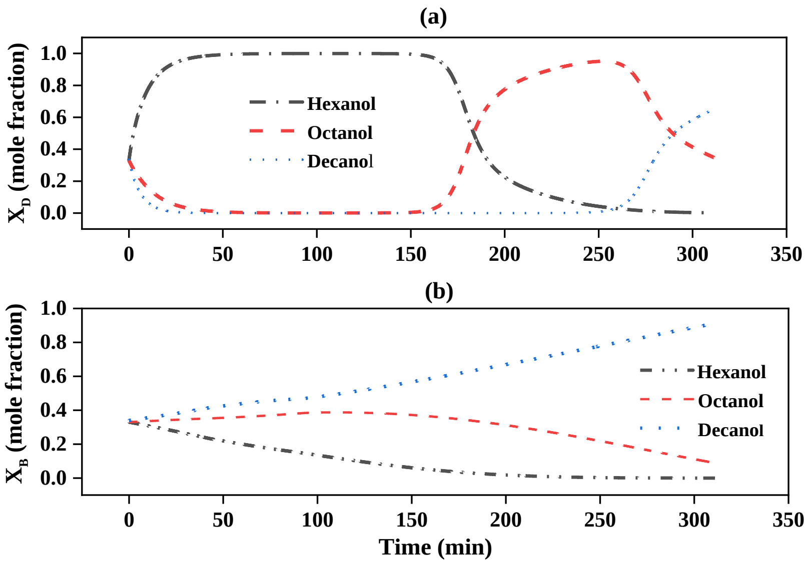

Figure 3.5. *Base case*: Dynamic composition profile throughout the batch operation at optimum number of trays (a) reflux drum (b) reboiler

In 78.01 minutes, the lightest component (hexanol) approaches the steady-state composition of 0.999 (start-up phase). The production phase then begins at a constant distillate flow rate of 0.038 kmol/min to allow for proper separation, which means that the specified purities of the product are achieved at this distillate flow rate. The batch distillation process took a total of 334.80 minutes. Figure 3.6 shows the CR profile of SiMVRBD with 25 stages throughout the batch operation, and it was clear that the CR is very high throughout the batch operation; nevertheless, such a high CR is not practically feasible in the batch distillation. Figure 3.7 and Figure 3.8 shows the energy and vapor flow rate manipulations during the batch operation, respectively.

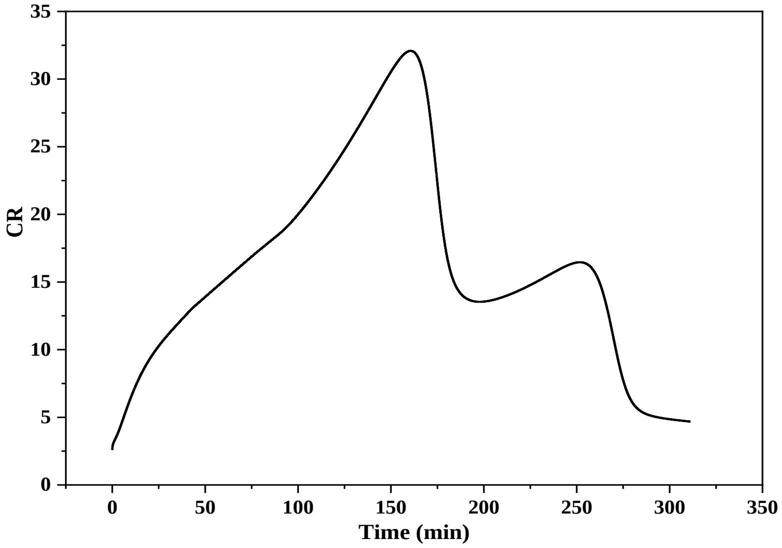

Figure 3.6. *Base case*: single-stage (SiVRMBD) compression ratio (CR) profile during batch operation at optimum number of trays

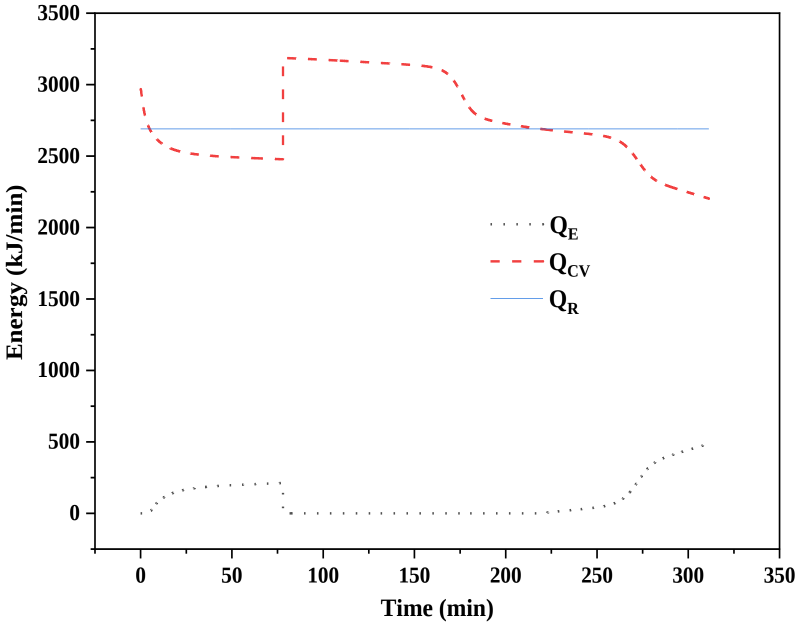

Figure 3.7. *Base case*: single-stage (SiVRMBD) energy profile during batch operation at optimum number of trays

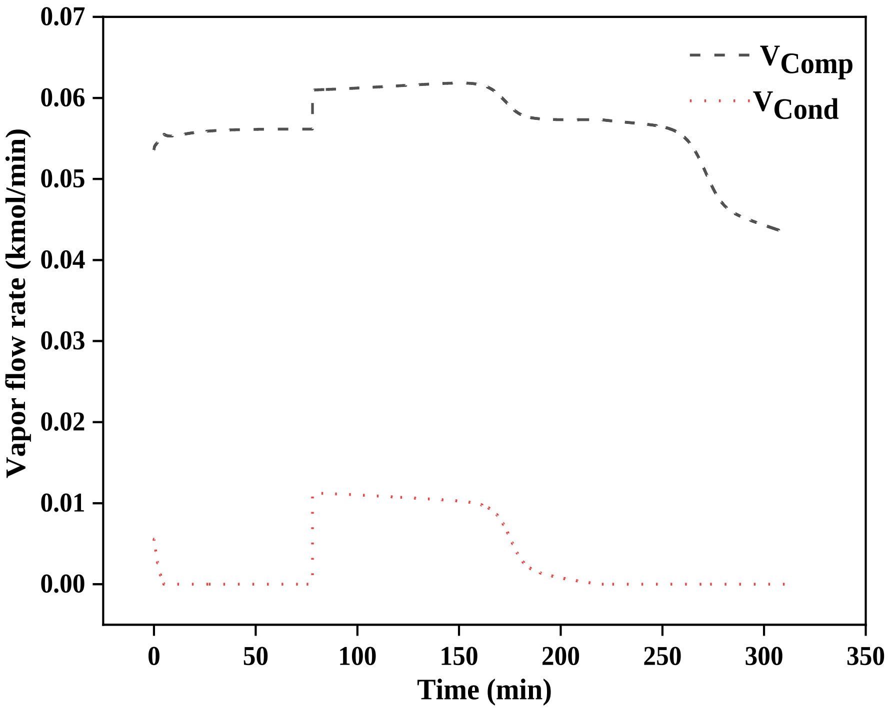

Figure 3.8. *Base case*: single-stage (SiVRMBD) vapor flow rate profile during batch operation at optimum number of trays

3.6.1.2. Multi-stage vapor recompressed multicomponent batch distillation vs SiVRMBD & CMBD

The compressor must run at the specified CR (<3.5) during the batch operation to limit compressor workload, eliminate wear and tear concerns, and have practical feasibility. Therefore, we proposed a novel multi-stage VRC approach to solve the drawbacks of the SiVRMBD. The proposed approach (Table 3.3) determines the number of compressors required to achieve a minimum CR of 3.5 throughout the batch operation and a minimum TAC and CO_2 emissions under optimal design parameters, that is, 25 trays.

Figure 3.9 shows the CR profiles of multi-stage vapor recompression. The SiVRMBD and DoVRMBD (double-stage compression) are operated at high CR during batch operation. However, the batch operation is run with CR< 3.5 throughout using multi-stage vapor recompression with three-stage compressors and two intercoolers in between (TrVRMBD). It was clear from Figure 3.9 that the multi-stage vapor recompression system requires at least three compressors during the batch operation to keep the CR below 3.5. Even though the CR of four and five-stage compressors remain below 3.5, capital costs will rise, operational complexity will grow, and TAC reductions may not be realized.

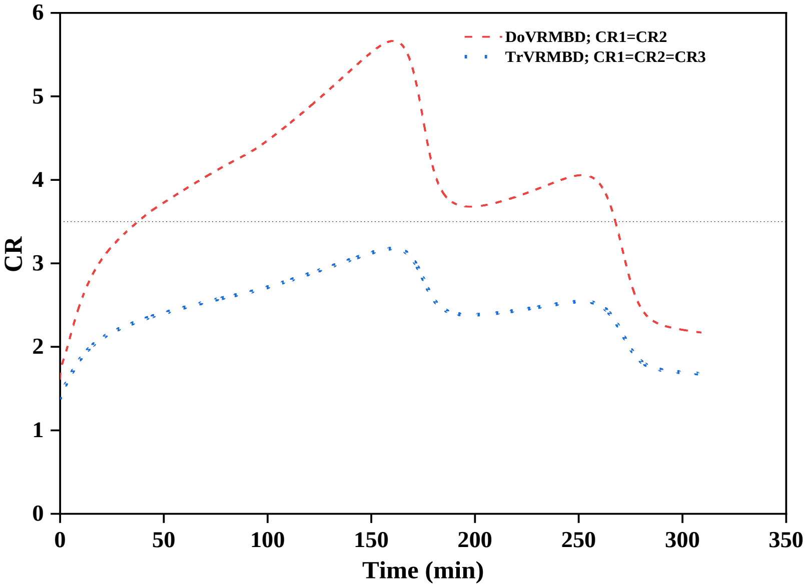

Figure 3.9. *Base case*: Compression ratio (CR) profile of multi-stage vapor recompression during batch operation

Table 3.6. *Base case*: Economic and environmental performance comparison

Performance Indices	CMBD	SiVRMBD	multi-stage VRC	
			DoVRMBD	TrVRMBD
Cost ($/yr)				
TCI $\times10^5$	2.70	3.46	3.45	3.42
TOC$\times10^3$	12.4	7.11	6.96	6.95
TAC$\times10^3$	3.04	3.01	3.04	2.98
%, Savings		3.10	3.33	**3.49**
CO_2 Emissions (ton/yr)				
Steam boiler	125.39	4.63	4.63	4.63
gas turbine		89.68	87.43	86.70
Total local emissions (TLE)	125.39	94.31	92.06	91.33
%, TLE savings		24.78	26.58	27.16
Emissions saved at power station		-63.49	-61.90	-61.38
Total global emissions	125.39	26.18	25.53	25.31
%, TGE savings		79.12	79.64	**79.81**

Table 3.6 compares economic and environmental performance for CMBD, SiVRMBD, and multi-stage VRC, respectively. Table 3.6 shows that when compressor stages increase, total operating costs decrease, and total capital investment reduces. With three-stage vapor recompression, the minimum TAC is also plainly visible. Also, it was observed that CO_2

emissions decreases globally with three-stage vapor recompression. The results show that compared to CMBD, the proposed TrVRMBD process reduces TAC savings by 3.49%, and CO_2 emissions by 79.81%. Therefore, TrMVRBD was the best choice in terms of economics, environment, and practical feasibility compared to SiMVRBD and DoMVRBD.

3.6.2. Influence of feed composition on the performance of VRC: *Multi-stage stage vapor recompressed multicomponent batch distillation vs SiVRMBD & CMBD*

The influence of feed composition on the performance of a single-stage and multi-stage vapor recompressed multicomponent batch distillation column was investigated. In this study, we have considered three different feed compositions as follows:

- *Case 1*. The intermediate component (octanol) is predominant (0.15– 0.7– 0.15)
- *Case 2*. The lightest component (hexanol) is predominant (0.7– 0.15– 0.15)
- *Case 3*. The heaviest component (decanol) is predominant (0.15– 0.15– 0.7)

For a fair comparison, the simulations for *Cases 1*, *2*, and *3* were run using the same design and operational variables as the *Base case*, with the optimal number of trays, that is, 25. To attain the required average composition specifications, just the distillation flow rate was identified (Approach 1).

3.6.2.1. Case 1

The total period of batch distillation was 411.034 minutes to attain the specified average purities with a constant distillate flow rate of 0.042 kmol/min. Figure 3.10 (a) shows the CR profile of SiMVRBD throughout the batch operation, and it was clear that the CR is exceptionally high throughout the batch operation; nevertheless, such a high CR is not feasible in batch distillation. Therefore, to restrict compressor workload and avoid wear and tear concerns, the compressor must run below specified CR (< 3.5) during the batch operation. The proposed multi-stage vapor recompression a determines the best number of compressors to ensure that they run at a minimum CR of 3.5 during batch operation and achieve minimum TAC. It is evident from Figure 3.10 (b) that the multi-stage vapor recompression system requires at least three compressors during the entire batch operation to keep CR below 3.5. Table 3.7 compares the economic and environmental performance of the CMBD, SiVRMBD, and multi-stage VRMBD, respectively. It is clear from Table 3.7 that the minimum TAC clearly appears at three-stage compression. Furthermore, it is evident from Figure 3.10 (b) and Table 3.7, that the optimum number of compressors are three to ensure that the compressors are operated below 3.5 CR. The results show that compared to CMBD, the proposed TrVRMBD process reduces TAC savings by 10.12%, and CO_2 emissions by 84.21% globally. Therefore, we determined that TrMVRBD is the best alternative in terms of economic, environmental, and practical feasibility compared to SiMVRBD and DoMVRBD.

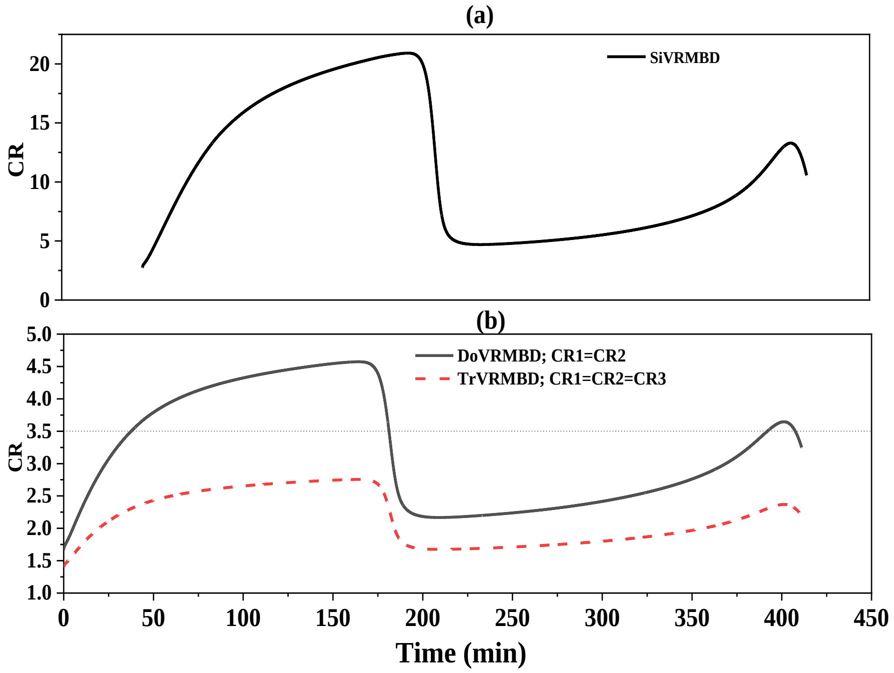

Figure 3.10. *Case 1*: Compression ratio (CR) profile throughout the batch operation (a) single-stage compression (b) multi-stage compression

Table 3.7. *Case 1*: Economic and environmental performance comparison

Performance Indices	**CMBD**	**SiVRMBD**	**multi-stage VRC**	
			DoVRMBD	**TrVRMBD**
Cost ($/yr)				
TCI ×10^5	2.707	3.337	3.335	3.336
TOC×10^3	13.176	5.903	5.837	5.815
TAC×10^4	3.121	2.815	2.807	2.805
%, Savings		9.80	10.06	**10.12**
CO_2 Emissions (ton/yr)				
Steam boiler	133.32	4.306	4.306	4.306
gas turbine		74.142	72.591	72.083
TLE	133.32	78.448	76.897	76.389
%, TLE savings		41.16	42.32	42.70
Emissions saved at PS		-52.492	51.394	51.035
Total global emissions	133.32	21.649	21.196	21.048
%, TGE savings		83.76	84.10	**84.21**

3.6.2.2. Case 2

With a constant distillate flow rate of 0.0442 kmol/min, batch distillation took 300 minutes to achieve the specified average purities. Figure 3.11 (a) depicts the CR profile of SiMVRBD throughout the batch operation, and it is clear that the CR is exceptionally high throughout the batch operation; nevertheless, such a high CR is not feasible in batch distillation. Therefore, the compressor must run at less than the specified CR (3.5) during the batch operation to limit compressor workload and avoid wear and tear concerns.

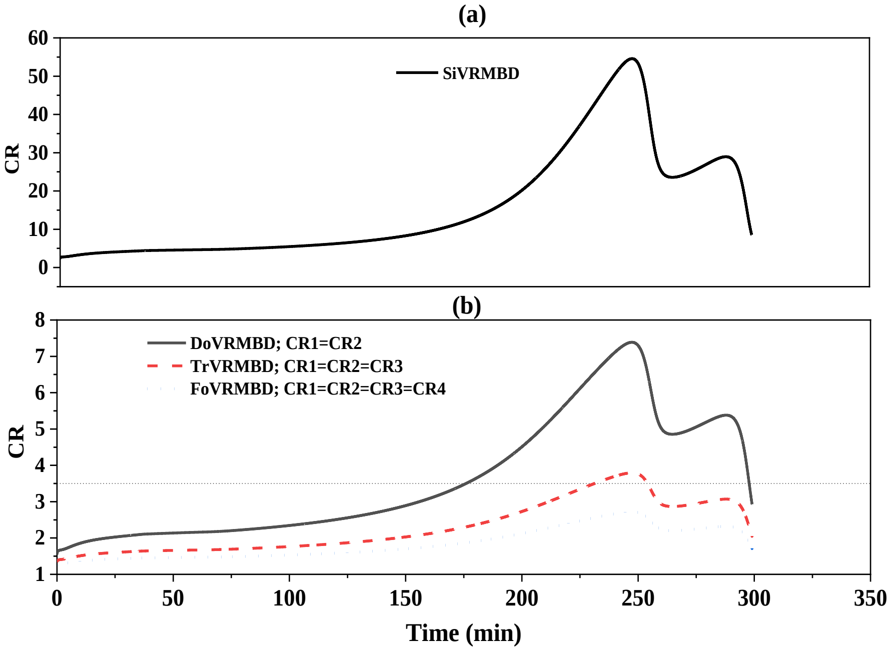

Figure 3.11. *Case 2*: Compression ratio (CR) profile throughout the batch operation (a) single-stage compression (b) multi-stage compression.

Table 3.8 compares the economic and environmental performance of the CMBD, SiVRMBD, and multi-stage VRMBD, respectively. The results show that compared to CMBD, the proposed FoVRMBD process reduces TAC savings by 8.16%, and CO2 emissions globally by 82.72%. When comparing SiMVRBD, DoMVRBD, TrVRMBD, and FoVRMBD, we found that FoVRMBD (multi-stage with four compressors) was the most cost-effective, environmentally friendly, and practically feasible. The proposed multi-stage approach finds the best number of compressors to run at a minimum CR of 3.5 and a minimum TAC under optimal design parameters during the batch operation. Figure 3.11(b) indicates that the multi-stage vapor recompression system requires at least four compressors to keep the CR below 3.5

throughout the batch operation. Therefore, the optimum number of compressors was four to ensure that the compressors operated at less than 3.5 CR.

3.6.2.3. Case 3

The total period of batch distillation was 341.039 minutes to attain the specified average purities with a constant distillate flow rate of 0.0135 kmol/min. The CR profile of SiMVRBD.

Table 3.8. *Case 2*: Comparative comparison of economic and environmental performance

Performance Indices	**CMBD**	**SiVRMBD**	**multi-stage VRC**		
			DoVRMBD	**TrVRMBD**	**FoVRMBD**
Cost ($/yr)					
TCI×10^5	2.711	3.408	3.399	3.397	3.3972
TOC×10^3	13.26	6.315	6.186	6.144	6.1236
TAC×10^4	3.133	2.903	2.884	2.879	2.8772
%, Savings		7.34	7.94	8.11	**8.16**
CO_2 Emissions (ton/yr)					
Steam boiler	132.52	1.380	1.380	1.380	1.380
gas turbine		81.501	79.435	78.762	78.429
TLE	132.52	82.881	80.815	80.142	79.81
%, TLE savings		37.45	39.01	39.52	39.77
Emissions saved at PS		57.703	56.240	55.763	55.528
Total global emissions		23.798	23.195	22.998	22.901
%, TGE savings	132.52	82.04	82.49	82.64	**82.72**

throughout the batch operation is shown in Figure 3.12(a), and it was evident that the CR is very high throughout the batch operation; nevertheless, such a high CR is not practicable in batch distillation. Therefore, the compressor must run at the specified CR (<3.5) during the batch operation to restrict compressor workload and avoid wear and tear concerns. The suggested multi-stage approach determines the best number of compressors to run at a minimum CR of 3.5 during the batch operation. It is evident from Figure 3.12(b) that the multi-stage vapor recompression system requires at least four compressors during the entire batch operation to keep CR below 3.5. The comparative economic and environmental analysis data is provided in Table 3.9. Compared to CMBD, the results show that SiVRMBD and multi-stage vapor recompression techniques did not yield TAC reductions. From an economic aspect, SiVRMBD and multi-stage VRC are not suitable for feed composition with the highest boiling point component. However, the proposed FoMVRBD saves CO_2 emissions globally by 72.26%

compared to CMBD.

3.6.3. Comparison with literature

It is worth noting that the approaches mentioned above can be generally applied for any vapor recompressed batch distillation processes. This section illustrates the effectiveness and advantages of the proposed approaches overwork in the literature.

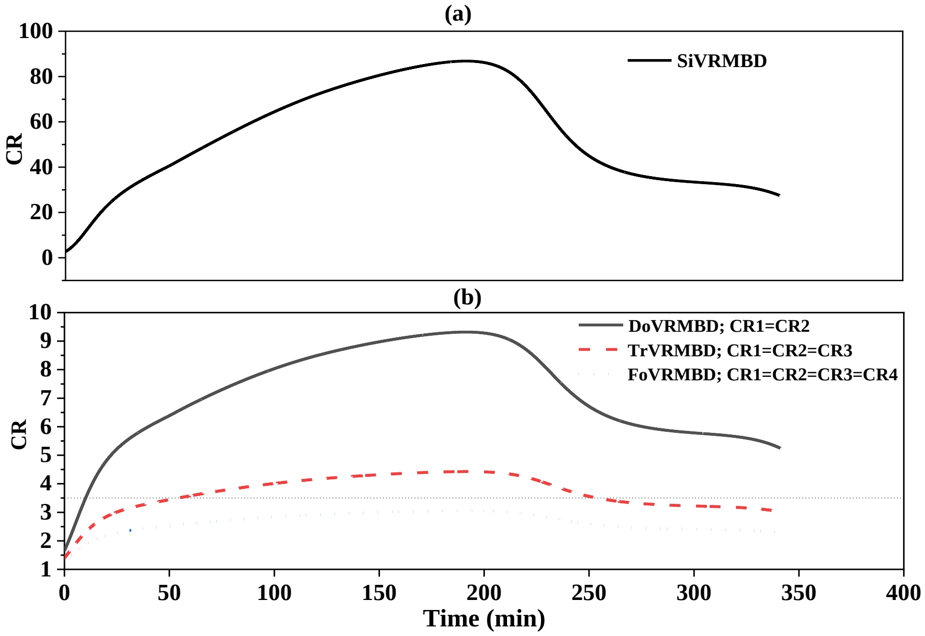

Figure 3.12. *Case3*: Compression ratio (CR) profile throughout the batch operation (a) single stage compression (b) multi-stage compression

Table 3.9. *Case 3*: Comparative comparison of economic and environmental performance

Performance Indices	CMBD	SiVRMBD	multi-stage VRC		
			DoVRMBD	TrVRMBD	FoVRMBD
Cost ($/yr)					
TCI ×10^5	2.707	3.761	3.748	3.747	3.7495
TOC×10^4	1.309	1.060	1.039	1.032	1.0285
TAC×10^4	3.114	3.567	3.538	3.530	3.5282
%, Savings		-14.54	-13.61	-13.35	-13.30
CO_2 Emissions (ton/yr)					
Steam boiler	132.571	8.2217	8.2217	8.2217	8.2217
gas turbine		133.188	128.300	126.724	125.948
TLE	132.571	141.41	136.52	134.94	134.17
TLE savings, %		-6.66	-2.98	-1.78	-1.26
Emissions saved at PS		94.297	90.836	89.721	89.171
Total global emissions		38.891	37.463	37.003	36.776
%, TGE savings	132.571	70.66	71.74	72.08	**72.26**

The dynamics and control of double-stage vapor recompressed batch distillation without intercoolers for separation of high wide boiling hexanol–octanol–decanol system with the middle component dominating in the feed composition (0.15–0.7–0.15) for average product purity specifications of 0.99–0.98–0.9 were investigated by the authors in the literature (Number of trays of 22 are utilized by the authors). In the simulation process, some design and operational parameters of the column configuration (reboiler heat duty, holdup, etc., average product specifications of products) are the same as the literature process to make fair comparisons between the proposed and literature. First, we used Approaches 1 and 2 to determine the optimal number of trays based on the maximal TAC savings when comparing SiVRMBD to CMBD. Table 7 shows the results obtained from Approaches and literature results. It is clear from the results that the suggested Approach 1 yielded the optimum number of theoretical trays of 12, with the maximum TAC savings at given average product purity specifications. Furthermore, the results reveal that the suggested technique saved more TAC (13.32%) than the literature (9.19%), and also saved more energy (43.57%) than the literature (40.50%). As a result of the findings, it was evident that the number of trays significantly impacts the VRC performance.

It is clear from Figure 3.10 that CR of single-stage and double-stage vapor recompression schemes of the proposed and literature are operated greater than 3.5 during the batch operation. We used the proposed multi-stage approach (Approach 3) to further clarify the novelty of the present work over the literature. It is evident from Figure 3.11 that the multi-stage vapor recompression requires at least three compressors during the entire batch operation to keep CR below 3.5. Table 3.10 and 3.11 compares the proposed approach to the literature in terms of cost and environmental impact. Compared to the literature, the results show that the proposed methodology saves more TAC and CO_2 emissions.

Table 3.10. Results with the proposed present work to those of previous work

Performance Indices	**Number of stages/Distillate flow rate (kmol/min)**						
	Proposed						***Literature [21]***
	10/0.025	**12/0.03**	15/0.032	22/0.035	25/0.04	28/0.042	***22/0.0337***
TAC ($/yr)							
CMBD×10^4	2.391	2.447	2.609	2.968	3.116	3.26	*2.968*
SiVRMBD×10^4	2.083	2.121	2.305	2.688	2.827	2.99	*2.696*
%, Savings	12.89	**13.32**	11.65	9.43	9.27	8.28	***9.19***
Energy (kJ)							
CMBD×10^5	16.651	14.464	13.991	12.315	11.323	11.178	*12.58*
SiVRMBD×10^5	9.573	8.161	8.063	7.27	6.63	6.69	*7.485*
%, Savings	42.50	**43.57**	38.51	40.96	41.44	40.15	***40.50***

Table 3.11. Results with the proposed work to those of previous work (radhika et al., 2020): Economic and environmental performance comparison

Performance Indices	**CMBD**	**SiVRMBD**	**multi-stage VRC**	
			DoVRMBD	**TrVRMBD**
TAC ($/yr)				
Literature ×10^4	2.968	2.696	2.694	
Proposed ×10^4	2.447	2.121	2.122	2.1223
%, Savings	17.55	21.32	21.23	
Total gobal CO_2 Emissions (ton/yr)				
Literature ×10^4	132.52	22.467	21.992	
Proposed ×10^5	131.21	21.155	20.726	20.586
%, TGE savings	0.98	5.84	5.83	

3.7 Conclusion

The batch distillation industry requires a minimum TAC and CO_2 for any investments in heat integrated systems, such as VRC. Consequently, the design conditions for implementing VRC should be chosen such that the energetic performance is maximum at minimum TAC. In this work, heat pump-assisted vapor recompression design approaches are proposed to separate ternary wide boiling mixtures in batch distillation to reduce TAC and CO_2 emissions. Hexanol–octanol–decanol ternary wide boiling mixture model system was selected in this paper.

First, a systematic simulation approach was developed to determine the optimal trays based on the lowest energy and TAC reduction. The results show that the optimal number of trays found from a proposed systematic simulation approach is 25. Compared to CMBD, the SiVRMBD process reduces energy by 30.2% and TAC by 3.1% at an optimal number of trays. Furthermore, it has been observed that in a SiVRMBD, a high CR is necessary throughout the batch operation, which is practically not feasible for the batch process industry resulting in compressor wear and tear. Therefore, a novel optimal multi-stage vapor recompression approach is proposed to identify the optimum number of compressors with the same design and operating conditions as SiVRMBD, so the batch distillation works at the lowest possible CR throughout while conserving the most TAC. The results reveal that, compared to CMBD, the suggested TrMVRBD has a CR<3.5 throughout the operation, saving 3.49% of TAC and 79.81% of CO_2 emissions for equimolar feed composition.

Finally, the influence of various feed compositions on the VRC performance was investigated. Overall, the findings showed that, as compared to CMBD, TrMVRBD saves 10.12% TAC and 84.21% CO_2 emissions when the intermediate component is prominent in feed, while FoMVRBD saves 8.16% TAC and 82.72% CO_2 emissions when the lightest component is prevalent in feed. SiVRMBD and multi-stage VRC processes are not cost-effective for feed composition, with the heaviest boiling point component is predominant. Furthermore, we demonstrated the efficacy and advantages of the proposed methods over literature. The results suggest that the proposed method saves more TAC and CO_2 emissions than the literature. It's worth mentioning that the practices described above can be used for any vapor recompressed batch distillation system.

Chapter 4

Performance evaluation of a vapor recompression in batch distillation with a side withdrawal

Chapter 4

4. Performance evaluation of a vapor recompression in batch distillation with a side withdrawal *

The chapter is focused on improving the performance of batch distillation with a side withdrawal (BDS) for the separation of ternary zeotropic mixtures using VRC. In this study, two ternary zeotropic wide boiling mixtures were investigated: hexanol/octanol/decanol (This system has a high wide boiling/zeotropic property because of the significant temperature difference between the lightest and heaviest components) and methanol/ethanol/1-propanol (This system has a moderate wide boiling/zeotropic property because of the moderate temperature difference between the lightest and heaviest components).

4.1 Introduction

Greenhouse gas (i.e., Carbon dioxide (CO_2)) emissions from fossil fuel combustion are one of the numerous severe environmental problems of the current century. For example, distillation is the widely used unit operation in the chemical industry for separating liquid mixtures. It accounts for around 95% of separations in the chemical and allied sectors. But, distillation uses a significant amount of energy, increasing CO_2 emissions released into the atmosphere.

Multi-component liquid mixtures can be separated in a single batch distillation column. Also, multi-component batch distillation handles efficiently for various feed fractions, difficulty separations, easy separations, and various product specifications. This drives batch distillation suitable where the products' demand and a lifetime can vary immensely with time and be tentative, such as fine, pharmaceutical, and specialty chemicals. Although it has some advantages over continuous distillation, batch distillation has several inherent disadvantages: long batch time, increased energy usage, a high temperature in the reboiler, and a complex operation (Demicoli et al., 2004). In addition, batch distillation is less energy efficient than continuous distillation operation. However, batch distillation has acquired revived interest because of the flexibility offered.

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* The one part of the work is submitted to Periodica Polytechnica Chemical Engineering, and second part of the work is Under Review (Asia Pacific Journal of Chemical Engineering)

To solve these issues, several unconventional batch distillation column configurations, like batch distillation with a side withdrawal (BDS) and multivessel vessel batch distillation, have received intense attention both in the industry and academia (Demicoli and Stichlmair, 2004; Skouras and Skogestad, 2004; Cui et al., 2009). Therefore, the unique BDS operation for separating the ternary zeotropic mixture is selected for further investigation in this work. Improving the thermal efficiency of a BDS process, cost savings, and reducing CO_2 emissions is a significant and pricey challenge to satisfy the environmental marks as consented in the Kyoto Protocol. Therefore, to further enhance the performance of BDS, we explore the feasibility of heat integration in BDS.

Recently, numerous heat integration schemes have been formulated to enhance performance, especially continuous distillation. As a result, energy may be integrated internally and externally for distillation operations. The internally heat integrated distillation column (HIDiC) for continuous distillation (Naito et al.,2000; Gadalla et al., 2005; wakabe et al., 2003; Suphanit et al., 2010; Shenvi et al., 2011) is the most typical example of internal heat integration, whereas the vapor recompression (VRC) column is established on external heat integration (Jana, 2016; Maiti et al., 2011).

Few papers dealing with the external heat integration of batch distillation for separation of close-boiling point mixtures have emerged so far, so this research area is only commencing to be explored. A novel external energy integrated vapor recompression column (VRC) approach was proposed for a regular reactive batch column (Johri et al., 2012). Explored an adaptive VRC approach for separating ternary closed-boiling mixtures in a batch distillation with a side withdrawal and middle vessel batch columns without vapor bypass (Babu et al., 2013). The VRC is more feasible for separation of close boiling mixtures in batch distillation due to the low-temperature difference between reboiler temperature ($\Delta T_B = T_B - T_{NT}$), i.e., the difference between the reboiler temperature (T_B) and top tray temperature (T_{NT}). As a result, the compressors require low compression; this gives improved energy and TAC (Total annualized cost) savings. VRC approach can enhance the performance of batch distillation for many separation systems, including binary and multi-component mixtures. The majority of work on VRC was focused on the separation of close boiling mixtures (Babu et al., 2012-2014). The separation of multi-component zeotropic mixtures was easier in batch distillation. Still, it has two significant problems: high-temperature difference in column and increased relative volatility, which leads to high compressor power or CR. However, for moderate binary wide-boiling mixtures, (Vibhute et al., 2020; Parhi et al., 2019; Babu et al., 2013) demonstrated that VRC improves economically and energetically.

Very few papers have appeared to deal with VRC in batch distillation to separate ternary zeotropic mixtures. There is no work reporting on VRC in BDS for separating ternary zeotropic mixtures. To the best of the author's knowledge, this is the first work on the vapor recompression in a batch distillation with a side withdrawal (BDS) for separating a ternary zeotropic wide-boiling mixtures.

BDS is a promising energy-saving alternative to traditional batch distillation. Although the purification of the wide boiling ternary mixture through BDS involves an energy-intensive and cost-effective separation process. It is clear from the literature that the single-stage VRC (traditional VRC), which employs a single compressor, for improving BDS performance in separations for close-boiling multi-component mixtures. In this contribution, we first developed single-stage vapor recompression in BDS (SiVRBDS) and compared it to the conventional multi-component batch distillation (CBDS) to separate ternary zeotropic wide boiling mixtures. Furthermore, SiVRBDS produces a substantial compression ratio (CR) throughout the batch operation. As a result, there are some limitations to applying the SiVRBDS scheme into practice. As a result, the authors proposed a novel multi-stage vapor recompression approach to lower the CR, reduce wear and tear, and reduce process cost and CO_2 emissions in high-temperature lift scenarios. This is a crucial task concerning practical feasibility in industrial implementation.

4.2 Conventional batch distillation with a side withdrawal

The process consists of a distillation tower equipped with a reboiler, trays, and reflux drum. The batch distillation side withdrawal column (BDS) is different from the conventional multocompnent batch distillation column (CMBD). In CMBD, one reflux at the top of the column. But, BDS consists of two refluxes: the top of the column and the middle of the column. The principle operation of BDS is shown in Figure 4.1.

The BDS has adequate for purifying an intermediate-rich component from light and heavy boiling constituents. It consumes less time to achieve the product. This method is more feasible for separating intermediate rich feed compositions and decomposing reactions. The design of BDS is easy and straightforward to control. To improve thermal efficiency and decrease the batch time (Demicoli et al., 2004) established a new workable policy for purifying a middle boiling constituent by batch distillation such as BDS and conducted feasibility analysis through computer-based simulations. (Cui et al., 2009) examined BDS for separating ternary mixture with a decomposing reaction and found that BDS is promising for purifying a middle component from light and heavy components. Different combinations of top and side reflux ratios were also examined. A systematic design, operation and simulation approach of BDS is given Table 4.1.

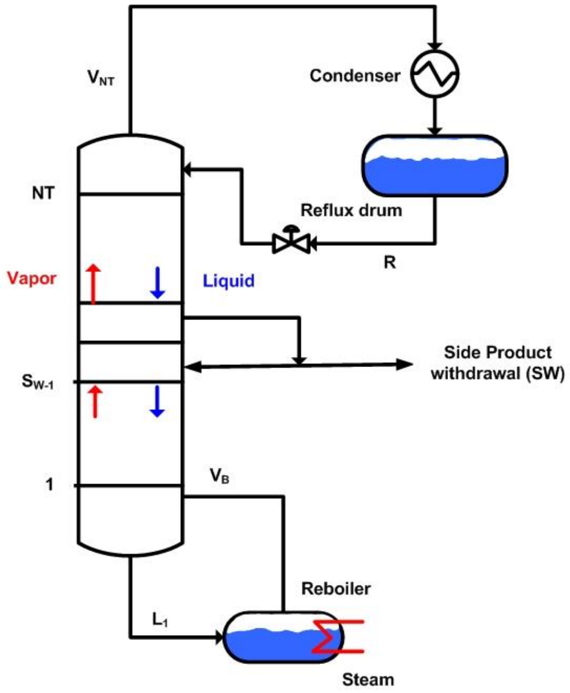

Figure 4.1. Conventional batch distillation with a side withdrawal

Table 4.1. Approach 1: A systematic design, operation and simulation approach of BDS

Step 1: Given input data: The column specifications, feed specifications (composition, flow rate, and temperature), tray efficiency, number of trays, initial conditions such as liquid compositions, liquid holdup on all trays, etc.,) are defined

Step 2: The feed mixture is introduced into the reboiler, trays, and reflux drum

Step 3: constant heat input is provided to the reboiler (fixed reboiler heat duty), and the vapor rises.

Step 4: The vapor departs through the perforations in the plate and into the condenser. The vapor begins to condense, and the reflux drum fills up.

Step 5: Total reflux starts, meaning that all condensed liquid from the reflux drum is sent back to the top tray.

Step 6: The liquid starts to drop from the downcomers, implying that the plates have good liquid holdup.

Step 7: Operational *Steps 5–8* are repeated until the middle component (octanol) purity in the tray reaches its maximum (this tray is selected for withdrawal), and the composition remains unchanged. The operation is then said to have achieved a steady state.

Step 8: When the operation has reached a steady state, the column operation is changed from the total reflux to the production phase, where the side withdrawal stream is removed from the selected tray.

Step 9. Terminate the simulation if the specified average composition of all products is met.

4.3 Selection of side withdrawal tray. At steady-state, a tray at which a middle component composition maximum is selected as a side withdrawal tray. Therefore, the side withdrawal tray position depends on the intermediate component composition at steady-state condition (at total reflux of start-up phase).

4.4 Single-stage vapor recompression in batch distillation with a side withdrawal

The principle operation of a VRC with a single-stage compression in BDS (SiVRBDS) is conceptually shown in Figure 4.2. The primary goal of SiVRBDS is to recover heat from top vapor, resulting in lowering hot and cold utility consumption. SiVRBDS comprises an extra compressor that is separate from the standard CMBD. Therefore, the SiVRBDS simulation approach consists of additional steps compared to CBDS, i.e., manipulation scenarios compared to CBDS. A detailed systematic design, operation and simulation approach of Si VRBDS is given in Table 4.2.

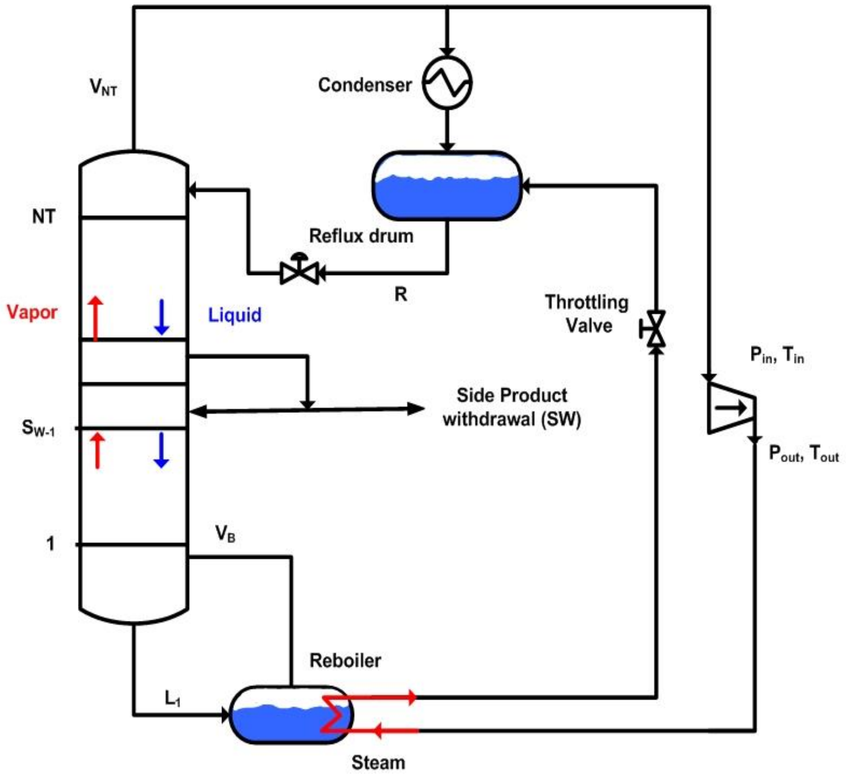

Figure 4.2. Single-stage vapor recompression in batch distillation with a side withdrawal

Table 4.2. Approach 2: A systematic design, operation and simulation approach

Step 1: Given input data: The column specifications, feed specifications (composition, flow rate, and temperature), tray efficiency, number of trays, initial conditions such as liquid compositions, liquid holdup on all trays, etc.,) are defined

Step 2: The feed mixture is introduced into the reboiler, trays, and reflux drum

Step 3: constant heat input is provided to the reboiler (fixed reboiler heat duty), and the vapor rises.

Step 4: The compressor receives the vapor from the top tray (V_{NT}). The vapor is then compressed to the required temperature (T_{out}). The latent heat (λ) released by the compressed vapor is utilized as heat input to the reboiler and thus condensed against the reboiler liquid.

Two manipulation criteria are employed to maintain (i) temperature difference, i.e., $\Delta T_T = T_{out} - T_B$ of atleast $15^0 C$ for total condensation in reboiler and (ii) same reboiler heat duty. (i) The vapor is compressed so that the compressed overhead vapor and bottom liquid (T_B) have a reasonable temperature difference ($\Delta T_T = 15^0 C$) for total condensation in the reboiler. To maintain $\Delta T_T = 15^0 C$ for total condensation of overhead vapor in the reboiler-condenser and to avoid compressor operation at maximum CR throughout the batch operation, a variable speed compressor (manipulation of CR) has been used.

Manipulation of CR:

We know the ΔT_T,T_B and T_{in} (top tray temperature), then calculate the compressor outlet temperature (T_{out}) from Equation (4.2), and then find out the corresponding CR to maintain constant value which means CR is variable (Equation 4.3).

$$\Delta T_T = T_{out} - T_B \tag{4.1}$$

$$T_{out} = \Delta T_T + T_B \tag{4.2}$$

$$CR = \frac{P_{out}}{P_{in}} = \left[\frac{T_{out}}{T_{in}}\right]^{\left(\frac{\mu}{\mu-1}\right)} \tag{4.3}$$

Step 5: The energy is released by the compressor vapour (Q_{CV}) is calculated from the following equation.

$$Q_{CV} = V_{NT} \times \lambda \tag{4.4}$$

(ii) An iterative vapor flow rate manipulation has been employed to accomplish the same dynamical performance between the standard CBDS and SiVRBDS, i.e., maintaining the same reboiler heat duty (Q_R).

Manipulation of vapor flow rate:

If the energy released by the compressed vapour (Q_{CV}) exceeds the required reboiler heat duty, split the top vapor into two parts: One part (V_{Comp}) goes to the compressor to raise the temperature and the rest (V_{Cond}) to the condenser.

If the compressed vapor latent heat (Q_{CV}) is less than the required reboiler heat duty, external heat (Q_E) must be supplied to the reboiler. Compressor work (W_{comp}) is calculated by Equation (4.8). The mathematical manipulation scenarios are presented below.

$$V_{NT} = V_{Comp} + V_{Cond} \tag{4.5}$$

If $Q_{CV} > Q_R$

$$V_{Comp} = Q_R/\lambda \tag{4.6}$$

$$V_{Cond} = V_{NT} - V_{Comp} \tag{4.7}$$

$$W_{comp} = \frac{(3.03\times10^{-5})\mu\, V_{comp}\, T_{in}}{\mu-1}\left[\left(\frac{P_{out}}{P_{in}}\right)^{\frac{\mu-1}{n\mu}} - 1\right] \tag{4.8}$$

$$Q_E = 0 \tag{4.9}$$

else

$$Q_E = Q_R - Q_{CV} \tag{4.10}$$

end

Where, $\mu -$ polytropic coefficient, P_{in} & P_{out} – inlet and outlet pressures of compressor, respectively

Step 6: The complete condensed liquid at high pressure is then throttled to top tray pressure and reaches the reflux drum. Finally, a throttling valve depressurizes the high-pressure condensed liquid before returning to the reflux drum.

Step 7: Total reflux starts, meaning that all condensed liquid from the reflux drum is sent back to the top tray.

Step 8: The liquid starts to drop from the downcomers, implying that the plates have good liquid holdup.

Step 9: Operational *Steps 5–8* are repeated until the middle component (octanol) purity in the tray reaches its maximum (this tray is selected for withdrawal), and the composition remains unchanged. The operation is then said to have achieved a steady state.

Step 10: When the operation has reached a steady state, the column operation is changed from the total reflux to the production phase, where the side withdrawal stream is removed from the selected tray.

Step 11. Terminate the simulation if the specified average composition of all products is met.

4.5 Multi-stage vapor recompressed multicomponent batch distillation

Single-stage vapor recompressed batch distillation requires a significant compression ratio (CR) in high-temperature lifts, which causes wear and tear problems in the compressor. As a result, the approach implementation has functional limitations. This work proposed multi-stage vapor recompression if the pressure ratio of SiVRMBD exceeds 3.5 at any time step throughout the batch operation. The proposed multi-stage vapor recompression in BDS improves the performance while also lowering the CR. However, if many compressors are demanded, it may not be superior because capital costs can increase too high. Therefore, the goal is to select the optimal number of compressors to achieve maximum TAC and CO_2 emission savings.

Figure 4.3 shows a schematic diagram of a multi-stage vapor recompression BDS with intercoolers. It comprises two or more compressors with intercoolers. For example, in a two-stage VRC configuration (consisting of two compressors in series with an intercooler), the overhead vapor mixture (V_{NT}) at atmospheric pressure P_{in} carried into the compressor 1 (C_1) and is compressed to a high pressure P_1. This pressure is intermediate between inlet pressure (P_{in}) and delivery pressure P_2. Hence this is known as intermediate pressure ($P_1 = P_{opt}$). The compressed vapor mixture is then passed into an intercooler1((I_1). In the intercooler1, the mixture is cooled to the initial temperature (T_{in}) at constant pressure by cold water. The cooled mixture from the intercooler1 is then passed into the compressor 2 (C_2). In the compressor 2, the vapor mixture is further compressed to a required high pressure (P_2). The optimal pressure of the first compressor becomes the intake pressure of the second compressor, the second compressor's optimized pressure is the inlet pressure of the third compressor, and so on for multiple compressors.

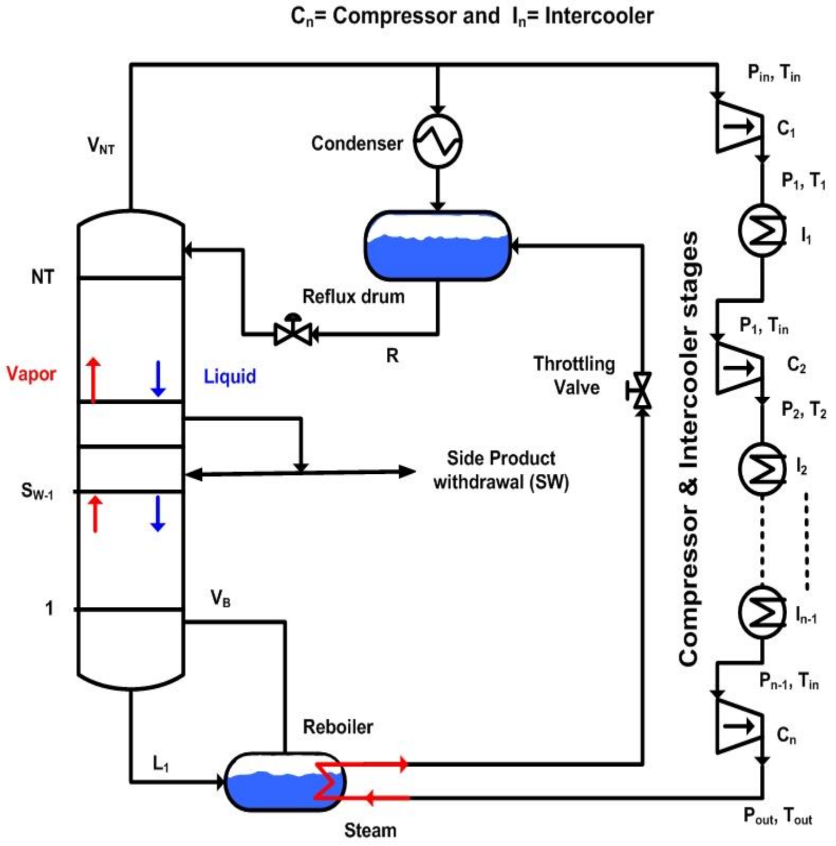

Figure 4.3. Multi-stage vapour recompression in batch distillation with a side withdrawal

In multi-stage compression, assumptions are made to calculate the compressors' work: the pressure remains constant during suction and delivery, intercooling takes place at a steady pressure at each stage, compression method is the same for each stage, and no pressure drop between the two stages.

The sum of the work for each stage in an n-stage compression process is the total work, and estimated horse power (hp) for an isentropic centrifugal compressor is computed by the following Equation

$$W_{comp,n} = \frac{(3.03\times10^{-5})\,\mu\,V_{comp}\,T_{in}}{\mu-1}\left[\left(\frac{P_{opt}}{P_1}\right)^{\frac{\mu-1}{\mu}} - 1\right] + \frac{3.03\times10^{-5}\mu\,V_{comp}\,T_{in}}{\mu-1}\left[\left(\frac{P_2}{P_{opt}}\right)^{\frac{\mu-1}{\mu}} - 1\right] + \ldots \quad (4.11)$$

n – number of compressors, $W_{comp,n}$ – total work of compressors

Differentiating the above equation with regard to P_{opt} and setting the resulting expression equal to zero yields the optimum value (P_{opt}). The optimal pressure therefore becomes:

$$P_{opt} = (P_{in}\,P_{out})^{1/n} \quad (4.12)$$

As a result, the pressure ratio of each stage (Eqn 4.11) of the compressor is the same ($CR_1 = CR_2 = \cdots$) and compression work at each stage (Eqn 4.12) is the same $\left(W_{comp,1} = W_{comp,2} = \cdots\right)$, and energy conservation is maximized.

$$CR_n = \left(\frac{P_{out}}{P_{in}}\right)^{\frac{1}{n}}\ ;\ n = 1,2,3,4,5\ldots\ldots \quad (4.13)$$

$$W_{comp,n} = \frac{n\,(3.03\times10^{-5})\mu\,V_{comp}\,T_{in}}{\mu-1}\left[\left(\frac{P_{out}}{P_{in}}\right)^{\frac{\mu-1}{n\mu}} - 1\right] \quad (4.14)$$

In this equation, the pressure (inlet pressure, P_{in} and outlet pressure, P_{out} is in lb_f/ft^2, and the vapor inflow rate to the compressor (V_{comp}) ft^3/min, n is the number of compressors. The procedure to determine the optimal number of compressors so that each compressor CR is less than 3.5 ($CR_{spec} < 3.5$) throughout the batch operation is shown in Table 4.3.

Table 4.3. Approach 3: Proposed approach for identification of optimum number of compressors

Step 1: If SiVRBDS operates at CR >3.5 at any point during the batch operation (Approach 2).
Step 2. Assume two-stage vapor recompression (DoVRMBD; n=2), which means that system consist of two compressors and a single intercooler between compressors. Run the simulation Approach 2

Step 3. Throughout the batch operation

If $\mathrm{CR_n} > \mathrm{CR_{spec}}$ at any point throughout the batch operation. Assume three-stage vapor recompression (TrVRMBD; n=3), and repeat the Approach 2

Step 4. Stop the procedure, if minimum TAC is achieved and $\mathrm{CR_n} < \mathrm{CR_{spec}}$

4.6 Results and discussion

In this study, two ternary zeotropic wide boiling mixtures were investigated: hexanol/octanol/decanol (This system has a high wide boiling/zeotropic property because of the significant temperature difference between the lightest and heaviest components) and methanol/ethanol/1-propanol (This system has a moderate wide boiling/zeotropic property because of the moderate temperature difference between the lightest and heaviest components). The computer program is developed and simulated in MATLAB.

4.6.1. Illustrative example 1: Hexanol/octanol/decanol

4.6.1.1 Conventional batch distillation column with a side withdrawal

Conventional batch distillation column with a side withdrawal (CBDS) dynamics is constructed up of algebraic-differential equations that are applied in (Jana et al., 2009). In addition, this model was developed based on the following assumptions: The liquid and vapor are perfectly mixed on each plate; The molar vapor holdup is little compared to the molar liquid holdup; the delay time between plates is omitted; operates at atmospheric pressure; the nonlinear Francis–Weir formula for tray hydraulics; NRTL thermodynamic model for vapor-liquid compositions are all taken into account. The design and column conditions are shown in Table 4.4.

4.6.1.2 Selection of side withdrawal tray

In the total reflux period (start-up phase), the lightest (Hexanol) and heaviest (Decanol) components accumulate in the reflux drum and reboiler vessels, respectively. In the same period, the intermediate component (Octanol) concentration gradually rises in a region of middle trays. Finally, the BDS column reaches a steady-state in 892.5 min (start-up period). Figure 4.4 shows the composition at 892.5 min at each tray. It clearly indicates that a maximum intermediate octanol component purity, i.e., 99.9%, is attained in tray 16. Therefore, tray 16 was selected as a withdrawal tray for the intermediate product.

Table 4.4. System and column specifications

System	Hexanol/octanol/decanol
Total feed, kmol	13
Feed composition	0.15/0.75/0.15
Initial holdup (reboiler/condenser/trays),kmol	6/6/1
Tray efficiency, %	80
Number of trays	25
Heat input to the reboiler, kJ/min	2690
Composition at the steady state (at 892.5 min)	
Distillate vessel, mole fraction	$0.311/0.6802/6.92\times10^{-4}$
Sidewithdrawal vessel, mole fraction	$5.113\times10^{-5}/0.999/9.48\times10^{-4}$
Reboiler, mole fraction	$1.04\times10^{-11}/0.5184/0.4816$
Composition at the end of production phase (at 1064.1 min)	
Distillate vessel, mole fraction	$0.319/0.6808/2.43\times10^{-4}$
Sidewithdrawal vessel, mole fraction	$3.827\times10^{-5}/0.85/0.1539$
Reboiler, mole fraction	$8.11\times10^{-19}/0.0115/0.9885$

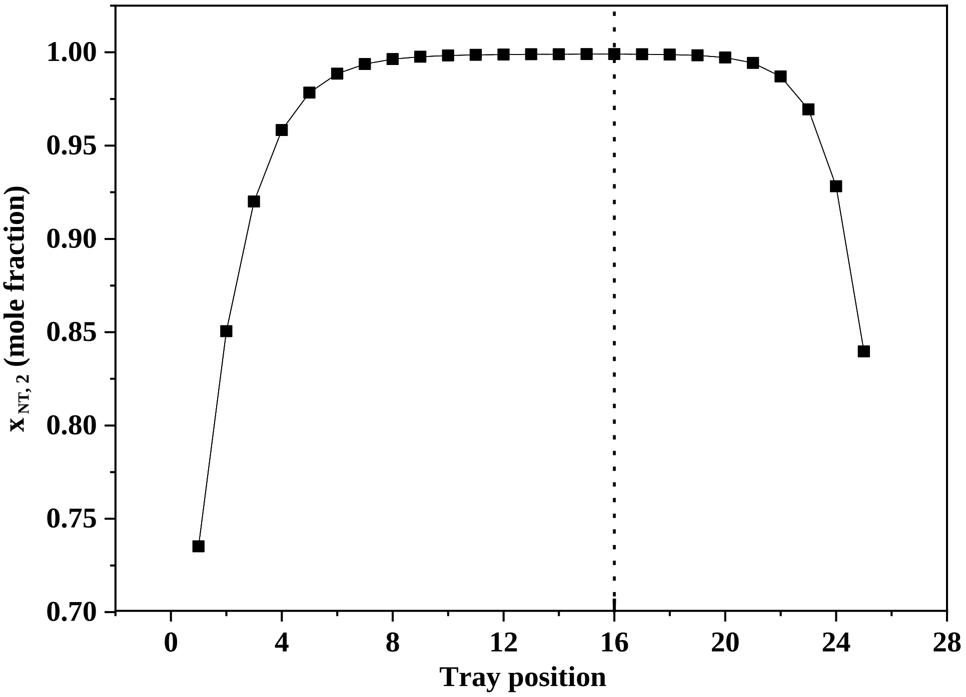

Figure 4.4. Composition profile of each tray at the end of the steady state

4.6.1.3 Dynamic composition profile

Steady-state results show the maximum achievable octanol purity in tray 16 at 892.5 min. After 892.5 min, the production phase begins, where the intermediate component is withdrawn at a constant flow rate of 0.021 kmol/min until the average composition in the product tank reaches 0.99. The total batch distillation process takes 1064.1 min. Figure 4.5 (Reflux drum), Figure 4.6 (side withdrawal tray), and Figure 4.7 (Reboiler) show the dynamic composition profiles throughout the batch operation.

4.6.1.4. Single-stage vapor recompression in batch distillation with a side withdrawal

Now, to operate the SiVRBDS at fixed reboiler duty of 2690 kJ/min, the variable manipulation policies as explained earlier (Approach 2) need to be followed. From simulation approach, we produced the manipulated variables profiles for the SiVRBDS. Figures 4.8 and 4.9 illustrate the adjustment of external energy from the source and vapor flow rate, respectively. It is evident that in the first few minutes (~20 min), the energy released by the compressed vapor is greater than the required reboiler heat duty; hence vapor flow rate manipulation is needed, and no external energy from the source is required.

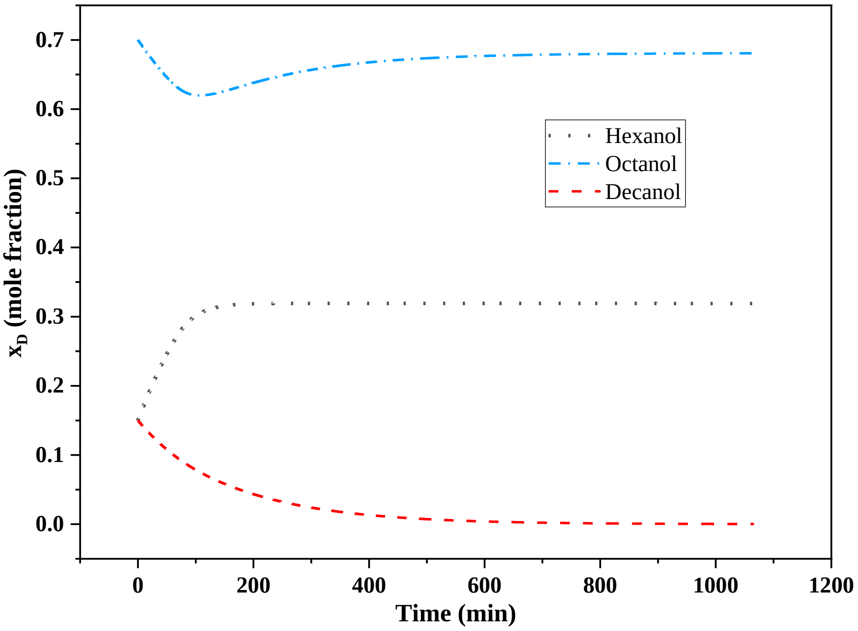

Figure 4.5. Reflux drum composition profile throughout the batch operation

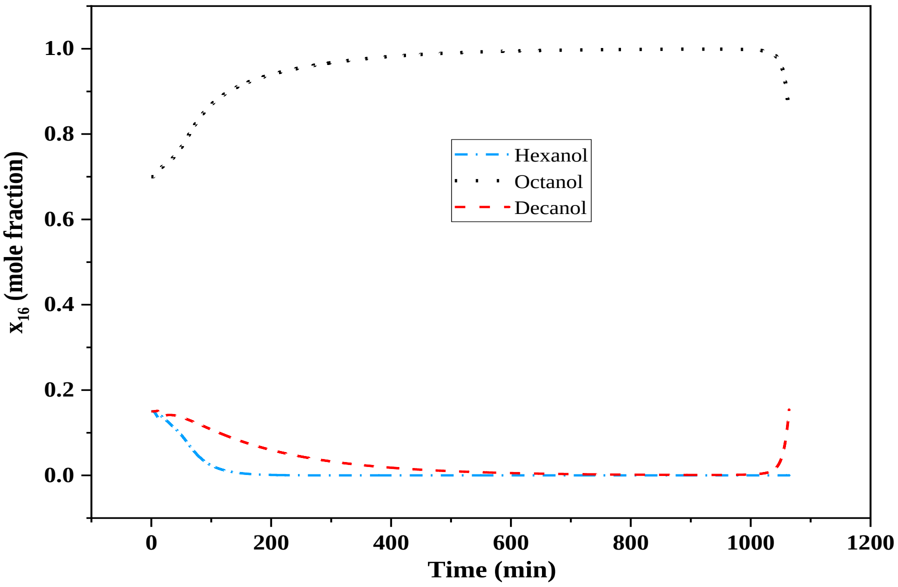

Figure 4.6. Composition profile throughout the batch operation at 16th tray

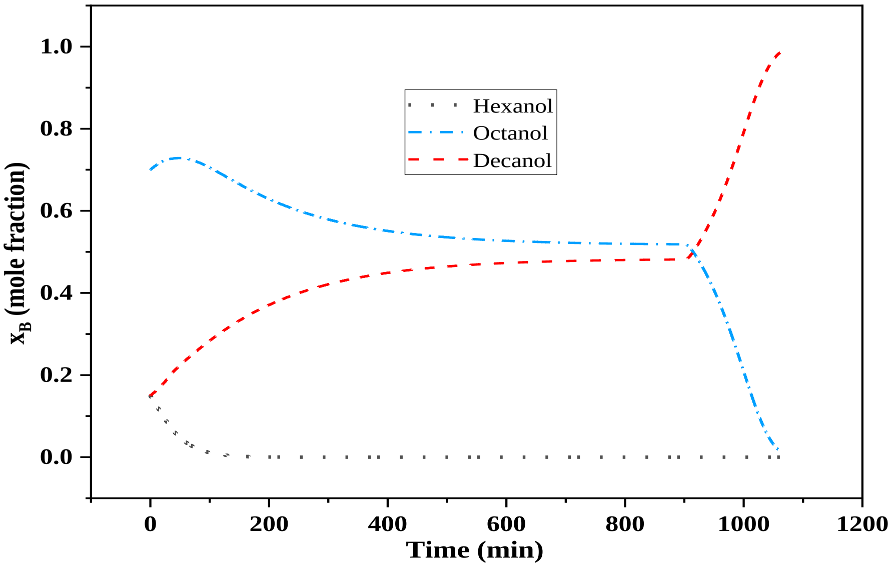

Figure 4.7. Reboiler composition profile throughout the batch operation

After that, energy released by the compressed vapor is lower than the necessary reboiler heat duty throughout the operation. Therefore, vapor flow rate manipulation is unnecessary, and energy from the external source is required. Figure 4.10 shows the manipulation of CR to maintain ΔT_T of atleast 15 °C. Furthermore, the temperature difference of the column throughout the batch operation is shown in Figure 4.11. It is clear that as CR increases with ΔT_T.

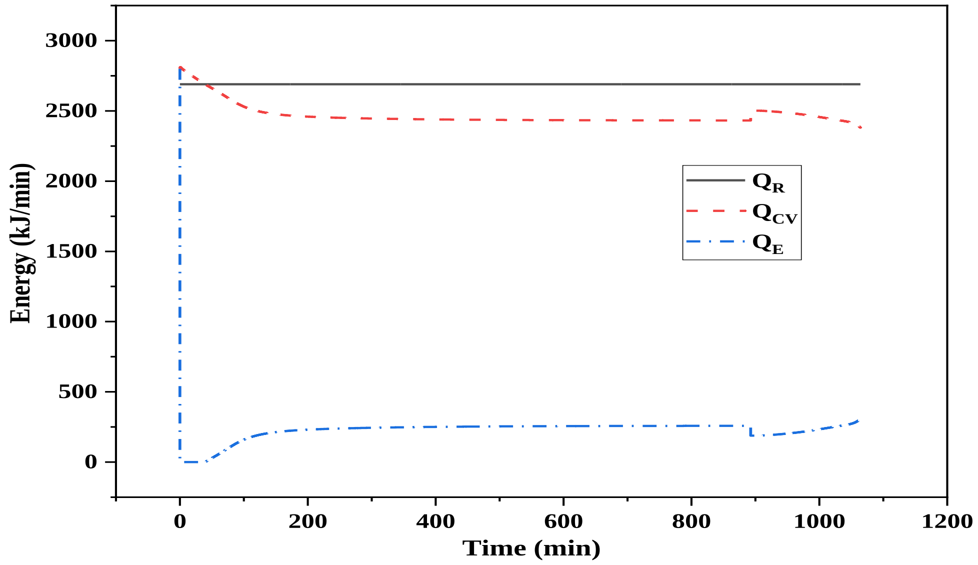

Figure 4.8. Energy profile throughout the batch operation

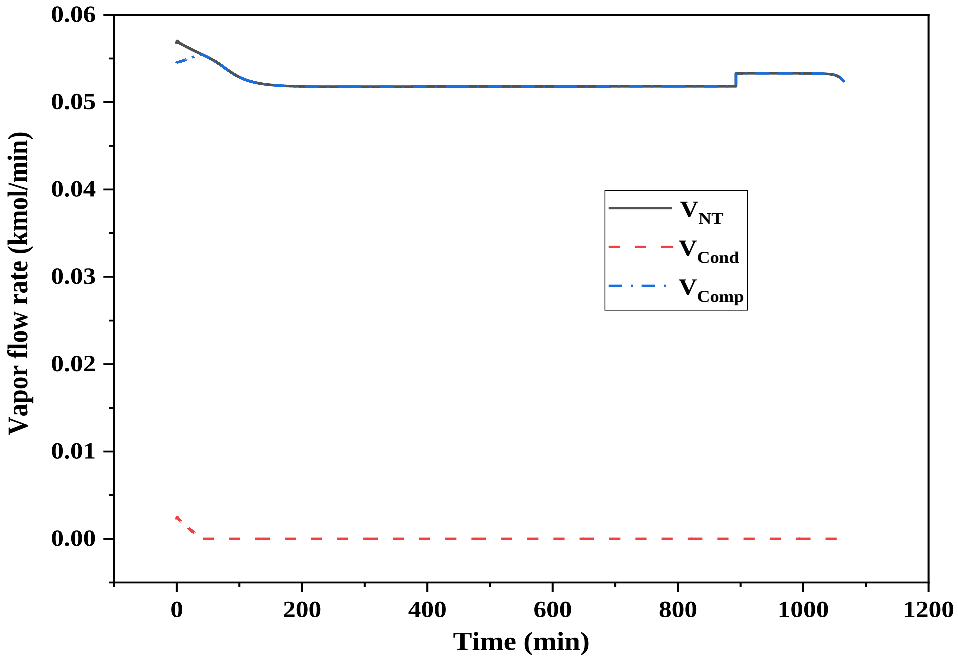

Figure 4.9. Vapor flow rate manipulation profile throughout the batch operation

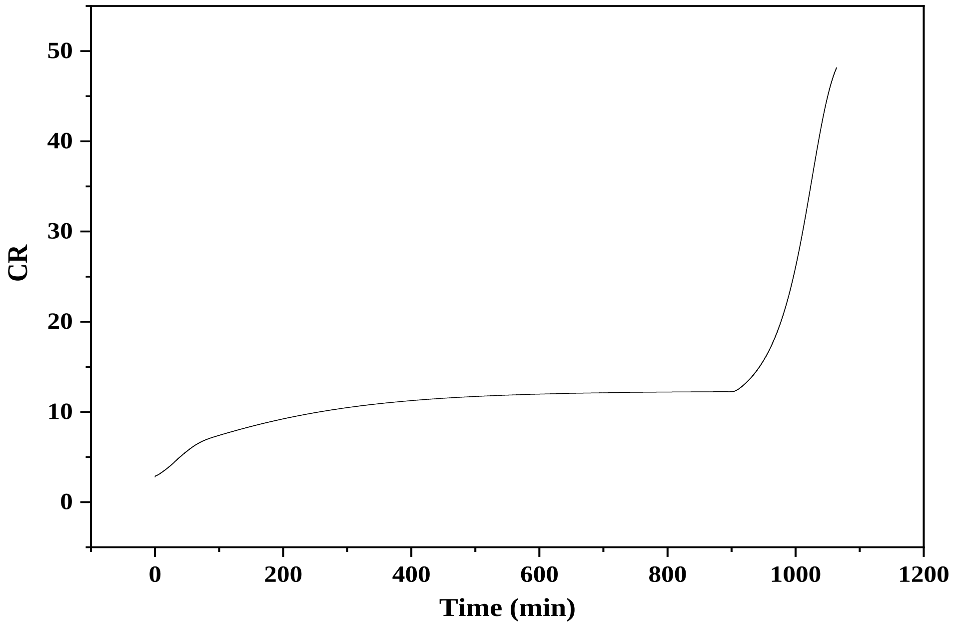

Figure 4.10. CR manipulation profile throughout the batch operation

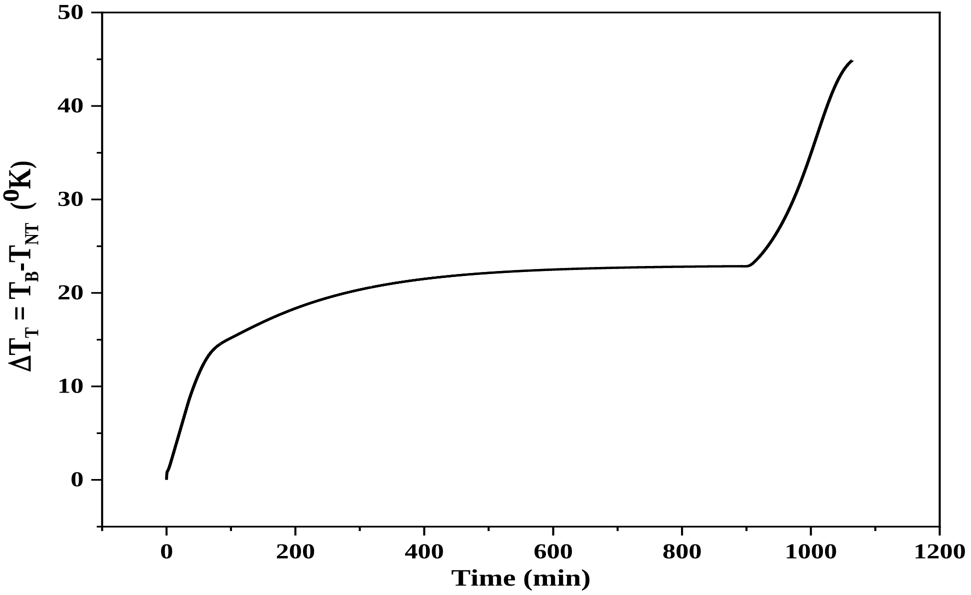

Figure 4.11. Temperature difference of the column throughout the batch operation

4.6.1.5. Multistage-vapor recompression in batch distillation with aside withdrawal

It is evident from Figure 4.10 that SiVRBDS operates at a high compression ratio during the batch operation, which is not feasible in the batch process industry. Therefore, the compressor must drive below specified CR (< 3.5) throughout the operation to restrict compressor workload and evade wear and tear problems. The proposed multi-stage VRBDS approach identifies a sufficient number of compressors to ensure that the compressors run at a below-specified CR and minimum TAC during the batch operation. Hence, achieves minimum process cost and CO_2 emissions compared to SiVRBDS. However, it is noticeable from Figure 4.12 that at least three compressors are required during the batch operation to maintain CR below 3.5.

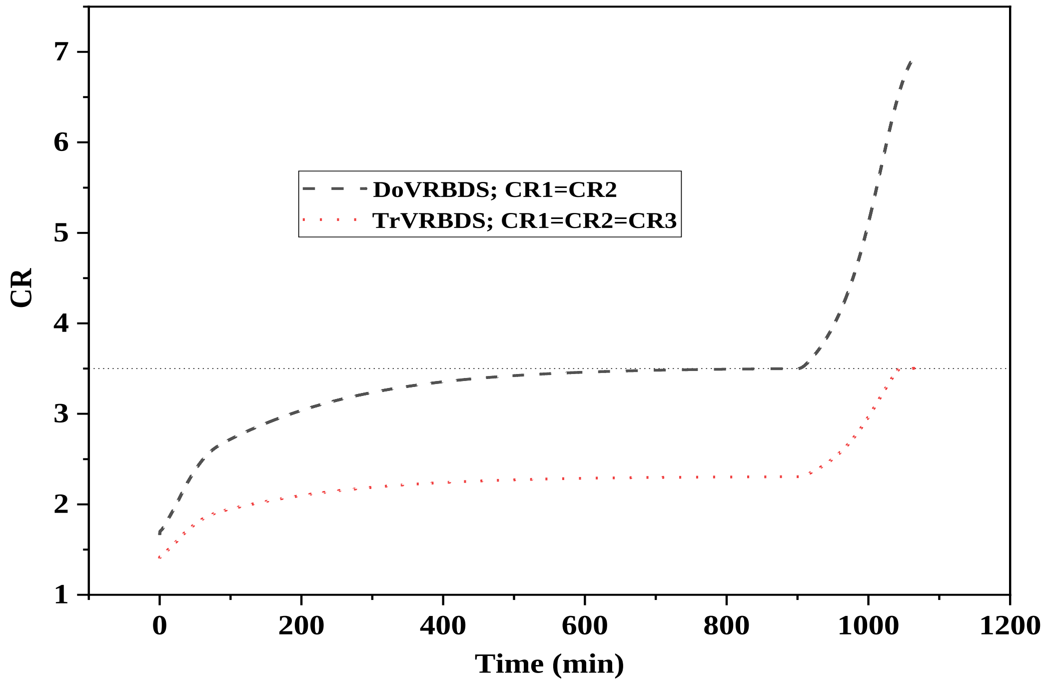

Figure 4.12. Compression ratio (CR) profile throughout the batch operation: multi-stage vapour recompression

4.6.1.6. Economic and environmental performance analysis

Table 4.5 compares the economic performance of the CBDS, SiVRBDS, and multi-stage VRBDS, respectively. It is clear from Table 4.5 that the minimum TAC clearly appears at three-stage compression. Furthermore, it is evident that the SiVRMBD system reduces process cost by 54.78% compared to CMBDS, while TrVRBDS reduces process cost (operating costs) by 55% compared to CBDS, and also it ensures that that the optimum number of compressors are three to ensure that the compressors are handled below 3.5 CR. Table 4.6 shows the environmental comparative performance analysis between CBDS, SiVRBDS, and multi-stage VRBDS, respectively. The results show that compared to CMBD, the proposed TrVRMBD process reduces CO_2 emissions by 83.54% globally. Therefore, we confined that TrVRBDS is the best alternative in terms of economic, environmental, and practical feasibility compared to SiVRBDS and DoVRBDS.

Table 4.5. Economic comparative analysis

Cost	**CBDS**	**Multi-stage VRBDS**		
		SiVRBDS	**DoVRBDS**	**TrVRBDS**
Capital costs				
Column shell	199240	199240	199240	**199240**
Column tray	22067	22067	22067	**22067**
Reboiler	42023	42023	42023	**42023**
Condenser	7246	69.32	69.32	**69.32**
Intercooler's	**-**	-	1485.8	**2258**
Compressor	**-**	71720	70548	**70160**
Total	270580	335120	335430	**335820**
Operating costs				
Steam	14260	1190.6	1190.6	**1190.6**
Cooling water	930	0.727	0.727	**0.727**
Intercooler cooling	**-**	-	88.24	**115.66**
Electricity	**-**	5677	5564	**5527**
Total	15190	6868	6843	**6833**
TAC,$/10yr	42247	40380	40380.7	**40416**
TAC savings, %		4.42	4.42	**4.33**

Table 4.6. Environmental comparative analysis

CO_2 Emissions (ton/yr)	**CMBD**	**SiVRMBD**	**multi-stage VRC**	
			DoVRMBD	**TrVRMBD**
Steam boiler	132.52	11.064	11.064	11.064
gas turbine	-	76.747	75.219	74.716
Total Local Emissions (TLE)	132.52	87.811	86.283	85.78
TLE savings, %		33.737	34.89	35.27
Emissions saved at PS		-54.34	-53.255	-52.899
Total global emissions (TGE)	132.52	22.41	21.964	21.817
%, TGE savings		83.09	83.46	83.54

4.6.2 Illustrative example 2: Methanol/ethanol/1-propanol

4.6.2.1. Conventional batch distillation column with a side withdrawal

Conventional batch distillation column with a side withdrawal (CBDS) dynamics is constructed up of algebraic-differential equations that are applied in [25]. In addition, this model was developed based on the following assumptions: The liquid and vapor are perfectly mixed on each plate; The molar vapor holdup is little compared to the molar liquid holdup; the delay time between plates is omitted; operates at atmospheric pressure; the nonlinear Francis–Weir formula for tray hydraulics; NRTL thermodynamic model for vapor-liquid compositions are all taken into account. The design and column conditions are shown in Table 4.7.

Table 4.7. Operating conditions and column specifications

System	Methanol/ethanol/1-propanol
Feed composition	0.15/0.7/0.15
Heat input to the reboiler, kJ/min	4000
Tray efficiency, %	80
Number of trays	50
Total feed, kmol	5.385
Initial holdup on trays, kmol	0.1
Initial condenser holdup, kmol	0.035
Initial reboiler holdup, kmol	5.25
Side withdrawal rate, kmol/min	0.0175
Composition at the steady state (at 143.99 min)	
Distillate vessel, mol%	$0.9716/0.0284/1.107\times10^{-13}$
Side withdrawal vessel, %	$3.214\times10^{-5}/0.999/9.677\times10^{-5}$
Reboiler, %	$5.234\times10^{-7}/0.365/0.6347$
Composition at the end of the production phase (at 195.923 min)	
Distillate vessel, mol%	$0.9621/0.03/1.897\times10^{-13}$
Side withdrawal vessel, %	$1.01\times10^{-5}/0.97/0.03$
Reboiler, %	$1.31\times10^{-12}/3.35\times10^{-4}/0.999$

4.6.2.2 Selection of side withdrawal tray

In the total reflux period (start-up phase), the lightest (methanol) and heaviest (1-propanol) components accumulate in the reflux drum and reboiler vessels, respectively. In the same period, the intermediate component (ethanol) concentration gradually rises in a region of middle trays. Finally, the BDS column reaches a steady-state in 143.99 min (start-up period). Figure 4.13 shows the composition at end of stratup-pahse at each tray. It clearly indicates that a maximum intermediate ethanol component purity, i.e., 99.9%, is attained in tray 16. Therefore, tray 16 was selected as a withdrawal tray for the intermediate product.

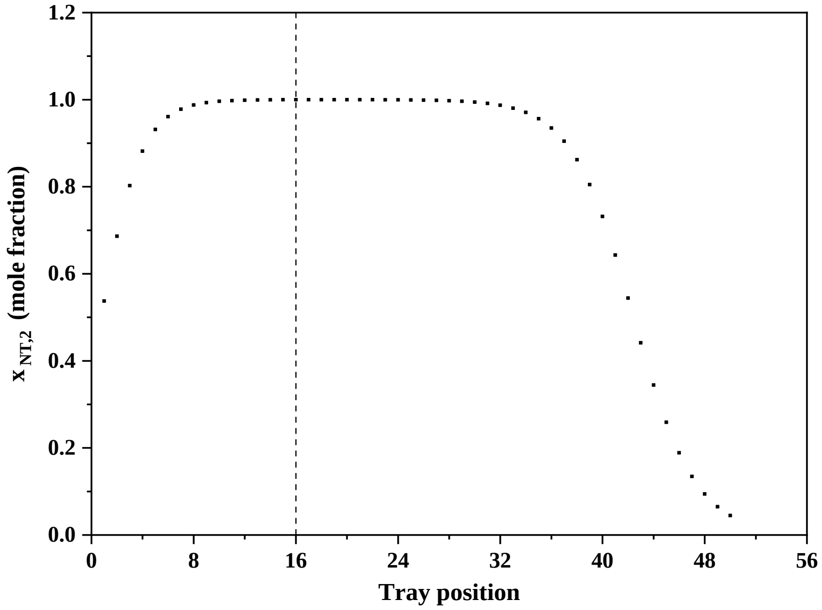

Figure 4.13. Composition profile of each tray at the end of the steady state

4.6.2.3 Dynamic composition profile

Steady-state results show the maximum achievable octanol purity in tray 16. After startup-pahse, the production phase begins, where the intermediate component is withdrawn at a constant flow rate of 0.0175 kmol/min until the average composition in the product tank reaches 0.97. The total batch distillation process takes 195.923 min. Figure 4.14 (Reflux drum), Figure 4.15 (side withdrawal tray), and Figure 4.16 (Reboiler) show the dynamic composition profiles throughout the batch operation.

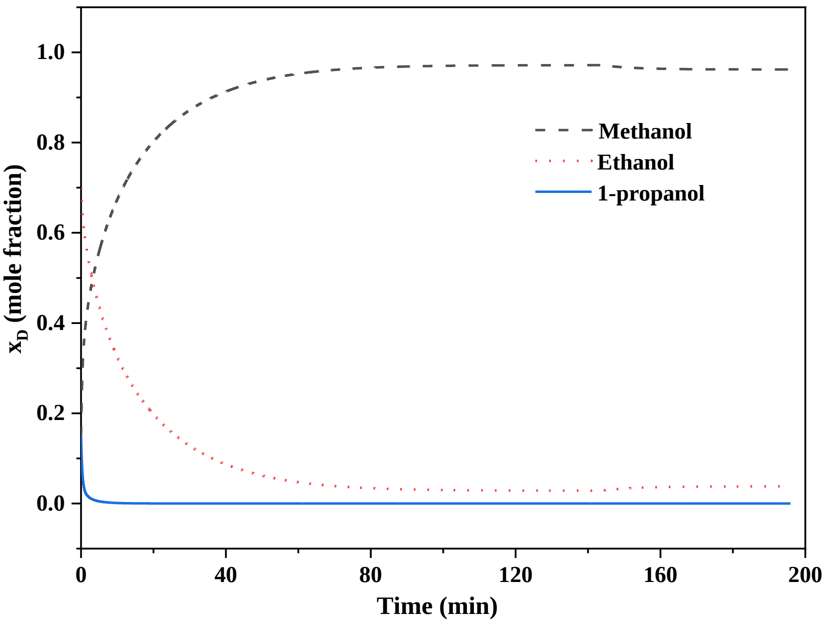

Figure 4.14. Reflux drum composition profile throughout the batch operation

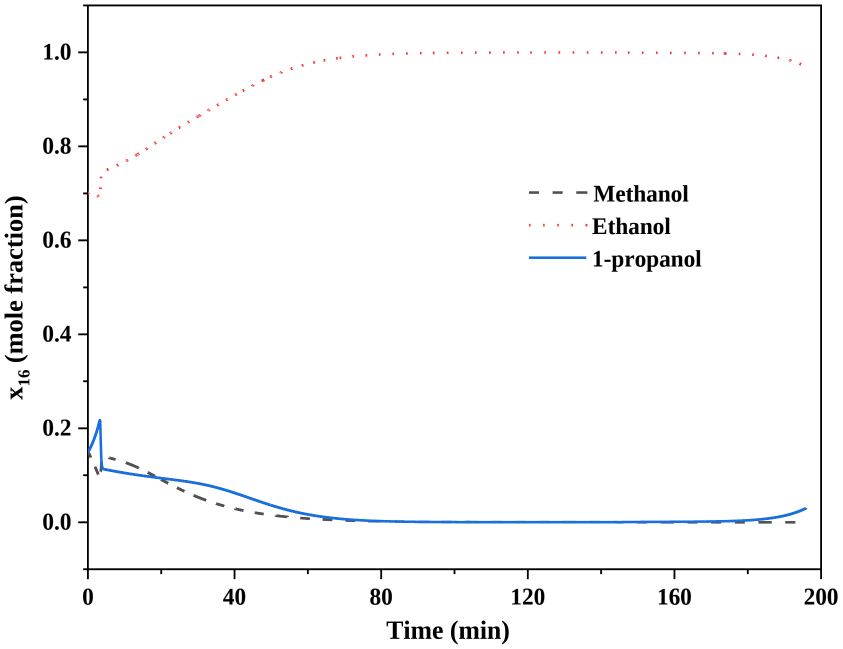

Figure 4.15. Composition profile throughout the batch operation at 16th tray

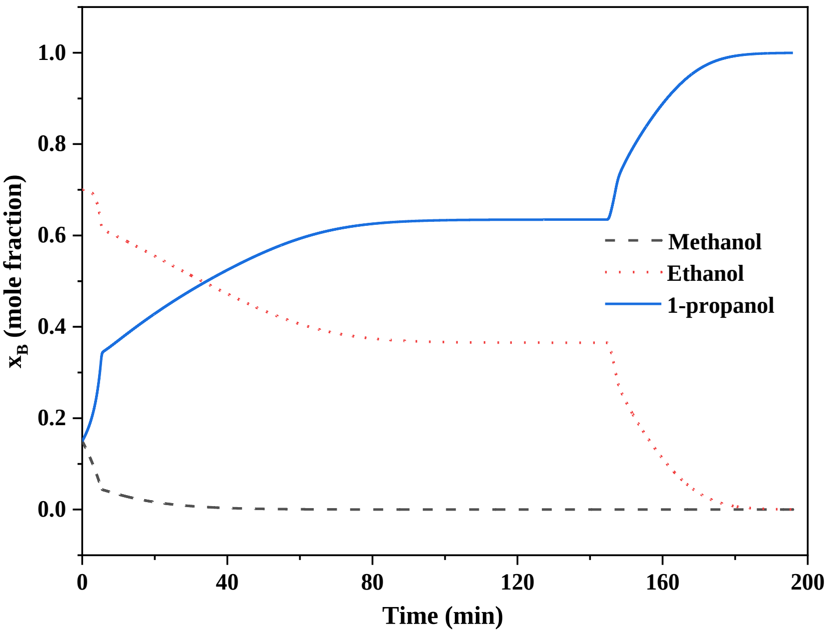

Figure 4.16. Reboiler composition profile throughout the batch operation

4.6.2.4 Single-stage vapor recompression in batch distillation with a side withdrawal.

Now, to operate the SiVRBDS at fixed reboiler duty of 4000 kJ/min, the variable manipulation policies as explained earlier (Approach 2) need to be followed. From simulation approach, we produced the manipulated variables profiles for the SiVRBDS. Figures 4.17 and 4.18 illustrate the adjustment of external energy from the source and vapor flow rate, respectively. It is evident that throughout the startup-pahse, the energy released by the compressed vapor is less than the required reboiler heat duty; hence vapor flow rate manipulation is not needed, and external energy from the source is required. After that, in production phase, energy released by the compressed vapor is greater than the necessary reboiler heat duty. Therefore, vapor flow rate manipulation is necessary, and energy from the external source is not required.

Figure 4.19 shows the manipulation of CR to maintain ΔT_T of at least 15. Furthermore, the temperature difference of the column throughout the batch operation is shown in Figure 4.20. It is clear that as CR increases with delta. Also, it is evident that CR throughout the batch operation is within the specified i.e., 3.5. Hence, multi-stage vapor recompression is not recommended.

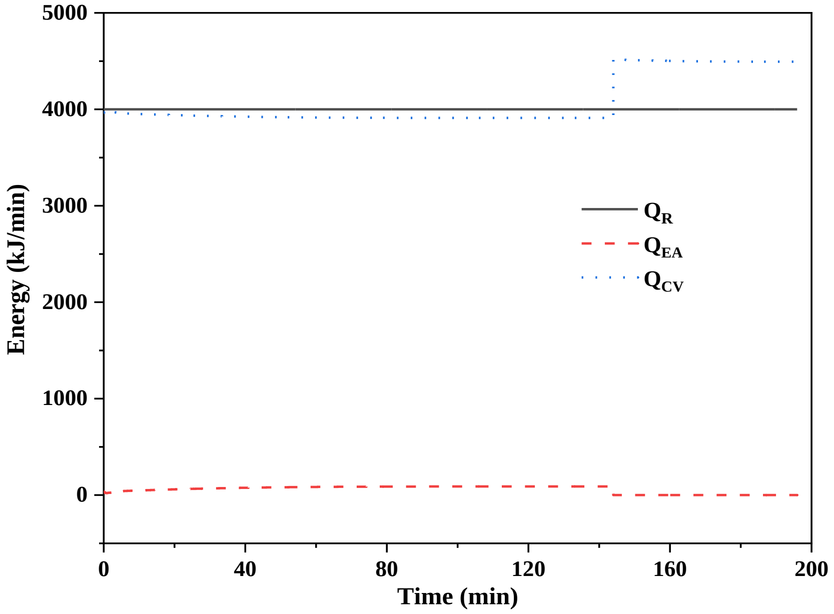

Figure 4.17. Energy profile throughout the batch operation

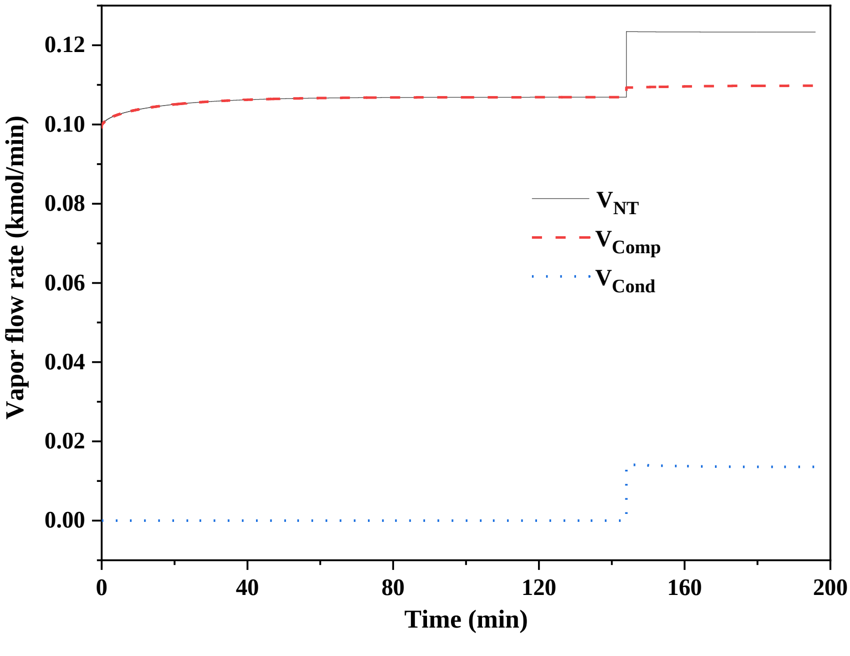

Figure 4.18. Vapor flow rate manipulation profile throughout the batch operation

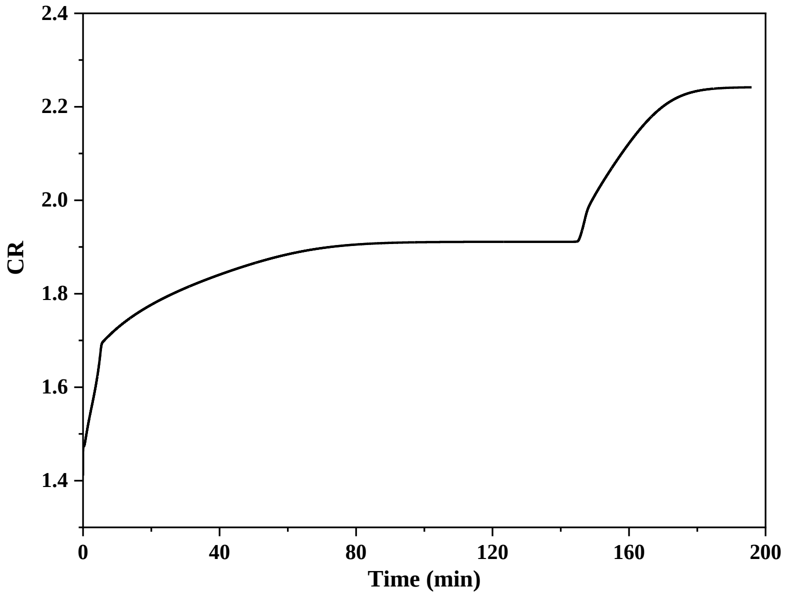

Figure 4.19. Compression ratio (CR) profile throughout the batch operation: multi-stage vapour recompression

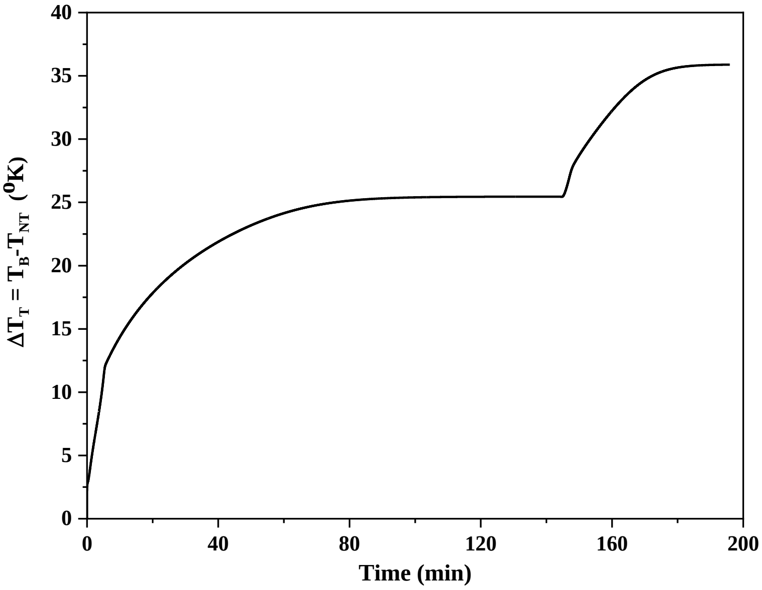

Figure 4.20. Temperature difference of the column throughout the batch operation

4.6.2.5 Economic and environmental performance analysis

Table 4.8 compares the economic performance of the CBDS and SiVRBDS. It is clear from Table 2 that the 4.54% TAC savings appears with SiVRBDS than CBDS. Furthermore, it is evident that the SiVRMBD system reduces process cost by 56.19% compared to CBDS. Table

4.9 shows the environmental comparative performance analysis between CBDS and SiVRBDS. The results show that compared to CBDS, the proposed SiVRBDS reduces CO_2 emissions by 92.2% globally. Therefore, we confined that SiVRBDS is the best alternative in terms of economic, environmental, and practical feasibility compared to CBDS.

Table 4.8. Economic comparative analysis

	CBDS	SiVRBDS
Capital cost		
Column shell	2.2547×10^5	2.2547×10^5
Column trays	2.3541×10^4	2.3541×10^4
Reboiler	5.4386×10^4	5.4386×10^4
Condenser	2.9369×10^4	3.1772×10^3
Compressor	-	3.8074×10^4
Total	3.3277×10^5	3.4465×10^5
Operating cost		
Steam	1.0956×10^4	158.9432
Water	1.4333×10^3	46.8183
Electricity	-	2.6227×10^3
Total	1.2390×10^4	2.8284×10^3
TAC	1.2331×10^5	1.1771×10^5
%, TAC savings		4.54

Table 4.9. Environmental comparative analysis

CO_2 Emissions (ton/yr)	CBDS	SiVRBDS
Steam boiler	119.8368	1.7385
gas turbine	-	31.9102
TLE	119.8368	42.085
TLE savings, %		
Emissions saved at PS	-	22.5924
Total global emissions (TGE)	119.8368	9.3178
%, TGE savings	-	92.2

4.7 Conclusion

Batch distillation systems are energy-intensive processes and contribute significantly to greenhouse gas emissions (e.g., carbon dioxide) than continuous distillation systems. Reducing carbon dioxide (CO_2) emissions is an absolute necessity and expensive challenge to the chemical process industries to meet the environmental targets as agreed in the Kyoto Protocol. Vapor recompression (VRC) can reduce process costs and, consequently, CO_2 emissions in batch distillation. Therefore, batch distillation with a side withdrawal has received intense attention in the industry and academia.

Previous research has focused on separating close-boiling ternary mixtures in batch distillation with a side withdrawal (BDS) using single-stage vapor recompression (SiVRBDS), which employs a single compressor to reduce energy usage. For the first time, the present research is focused on improving the performance of BDS for the separation of ternary zeotropic mixtures using VRC. In this study, two ternary zeotropic wide boiling mixtures were investigated: hexanol/octanol/decanol (This system has a high wide boiling/zeotropic property because of the significant temperature difference between the lightest and heaviest components) and methanol/ethanol/1-propanol (This system has a moderate wide boiling/zeotropic property because of the moderate temperature difference between the lightest and heaviest components). First, single-stage vapor recompression is applied, allowing fair comparison with the conventional BDS. We find that the minimum TAC and CO_2 emissions are achieved by SiVRBDS, but the compression ratio (CR) is operated at high during the operation due to the significant temperature difference of the column, which is not practically feasible. Secondly, we proposed a new optimal multi-stage VRC to operate the compressors at a minimum CR of 3.5 during the batch operation and further reduce process cost and CO_2 emissions. Finally, SiVRBDS and multi-stage VRC processes are evaluated concerning TAC, and CO_2 emissions.

The results show that in case of hexanol/octanol/decanol system, the TrVRBDS reduces process cost (operating costs) by 55% compared to CBDS, and also it ensures that that the optimum number of compressors are three to ensure that the compressors are handled below 3.5 CR. The proposed TrVRMBD process reduces CO_2 emissions by 83.54% globally compared to CBDS. However inc case of methanol/ethanol/1-propoanol systems, the SiVRMBD system reduces process cost by 56.19% and reduces CO_2 emissions by 92.2% globally compared to CBDS.

Chapter 5

Exploring the economic and environmental feasibility of vapor recompression in middle vessel batch distillation

Chapter 5

5. Exploring the economic and environmental feasibility of vapor recompression in middle vessel batch distillation *

Batch distillation systems are energy-intensive processes and contribute significantly to greenhouse gas emissions (e.g., carbon dioxide) than continuous distillation systems. Middle vessel batch distillation have received intense attention in the industry and academia. In this chapter, focused on improving the performance of middle vessel batch distillation for the separation of ternary zeotropic mixture, i.e., methanol/ethanol/1-propanol using VRC.

5.1 Introduction

Batch distillation is most widely used unit operation in which multi-component liquid mixtures can be separated in a single batch distillation column. This type of operation is commonly used when the priority is to achieve very high product purity from expensive and valuable resources used in pharmaceutical industries for fine chemical production with very high purity. Its flexibility makes it more preferable than continuous distillation as it can handle different feed compositions by switching a few operational parameters. When the batch mode of operation comes into the discussion, it means high purity throughout the operation; otherwise, the government may impose heavy fines to destroy the environment on industries. Greenhouse gas (i.e., Carbon dioxide (CO_2)) emissions from fossil fuel combustion are one of the numerous severe environmental problems of the current century. For example, distillation is the widely used unit operation in the chemical industry for separating liquid mixtures. It accounts for around 95% of separations in the chemical and allied sectors. But, distillation uses a significant amount of energy, increasing CO_2 emissions released into the atmosphere.

Multi-component liquid mixtures can be separated in a single batch distillation column. Also, multi-component batch distillation handles efficiently for various feed fractions, difficulty separations, easy separations, and various product specifications.

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* This work has been submitted to Energy (Under Review)

This drives batch distillation suitable where the products' demand and a lifetime can vary immensely with time and be tentative, such as fine, pharmaceutical, and specialty chemicals. Although it has some advantages over continuous distillation, batch distillation has several inherent disadvantages: long batch time, increased energy usage, a high temperature in the reboiler, and a complex operation (Demicoli et al., 2004). In addition, batch distillation is less energy efficient than continuous distillation operation. However, batch distillation has acquired revived interest because of the flexibility offered. To solve these issues, middle vessel batch distillation (MVBD) column configurations, have received intense attention both in the industry and academia (Demicoli and Stichlmair, 2004; Skouras and Skogestad, 2004; Gruetzmann et al., 2006). The MVBD strategy combines the batch rectifier and batch stripper in one column. For separating ternary mixtures, it is clear (Warter et al., 2002) that the MVBD operation is much easier than the regular column operation due to a more straightforward process to handle liquid fractions, i.e., distillate slop-cuts are not required compared to regular batch distillation operation. Hence, there is no production phase when separating ternary mixtures in an MVBD operation, lowering the batch operation period. Furthermore, as stated, the MVBD column has no slop-cuts, and therefore, the quantity left for reprocessing in the next batch run is reasonably low. However, there are intensive publications on regular batch distillation, and few works have been published in the middle vessel column for separating ternary mixtures. Therefore, the current work chose the MVBD operation as a forthcoming candidate for additional improvement.

Improving the performance of a MVBD, cost savings, and reducing CO_2 emissions is a significant and pricey challenge to satisfy the environmental marks as consented in the Kyoto Protocol. Therefore, to further enhance the performance of MVBD, we explore the feasibility of heat integration in BDS. Recently, numerous heat integration schemes have been formulated to enhance performance, especially continuous distillation. As a result, energy may be integrated internally and externally for distillation operations. The internally heat integrated distillation column (HIDiC) for continuous distillation (Naito et al., 2000; Gadalla et al., 2005; wakabe et al., 2003; Suphanit et al., 2010; Shenvi et al., 2011) is the most typical example of internal heat integration, whereas the vapor recompression (VRC) column is established on external heat integration (Jana, 2016; Maiti et al., 2011).

Few papers dealing with the external heat integration of batch distillation for separation of close-boiling point mixtures have emerged so far, so this research area is only commencing to be explored. A novel external energy integrated vapor recompression column (VRC) approach was proposed for a regular reactive batch column (Johri et al., 2012). Explored an adaptive VRC approach for separating ternary closed-boiling mixtures in a batch distillation with a side withdrawal and middle vessel batch columns without vapor bypass (Babu et al., 2013). The VRC is more feasible for separation of close boiling mixtures in batch distillation due to the low-temperature difference between reboiler temperature ($\Delta T_B = T_B - T_{NT}$), i.e., the difference between the reboiler temperature (T_B) and top tray temperature (T_{NT}). As a result, the compressors require low compression; this gives improved energy and TAC (Total annualized cost) savings. VRC approach can enhance the performance of batch distillation for many separation systems, including binary and multi-component mixtures. The majority of work on VRC was focused on the separation of close boiling mixtures (Babu et al., 2012; Vibhute et al., 2020; Parhi et al., 2019; Babu et al., 2013). The separation of multi-component zeotropic mixtures was easier in batch distillation. Still, it has two significant problems: high-temperature difference in column and increased relative volatility, which leads to high compressor power or CR.

Very few papers have appeared to deal with VRC in MVBD distillation to separate multi-component zeotropic wide boiling mixtures (Nair et al.,2020; Babu et al., 2013). There is no work reporting on VRC in MVBD for separating ternary zeotropic mixture i.e., methanol/ethanol/1-propanol. To the best of the author's knowledge, this is the first work on the vapor recompression in a middle vessel batch distillation for separating a ternary zeotropic wide-boiling mixture (methanol/ethanol/1-propanol).

5.2. Conventional middle vessel batch distillation (CMVBD)

Figure 5.1 shows the schematic representation of the CMVBD process without vapor bypass. It consists of three vessels: reboiler, middle vessel, reflux drum, and trays. The CMVBD column is divided into rectifying section and stripping section by a middle vessel. The CMVBD operation is different from the conventional batch distillation column. In CMVBD, the operation involves only the startup phase (total reflux condition), which means that no product withdrawal occurs. This process obtains light, intermediate, and heavy components simultaneously from the reflux drum, middle vessel, and reboiler. A systematic design, operation procedure for CMVBD is given in Table 5.1.

Table 5.1. *A systematic design, operation and simulation approach of CMVBD*

Step 1: Given input data: The column specifications, feed specifications (composition, flow rate, and temperature), tray efficiency, number of trays, initial conditions such as liquid compositions, liquid holdup on all trays, etc.,) are defined

Step 2: The feed mixture is introduced into the reboiler, middle vessel, trays, and reflux drum

Step 3: Constant heat input is provided to the reboiler (fixed reboiler heat duty), and the vapor rises.

Step 4: The vapor departs through the perforations in the plate and into the condenser. The vapor begins to condense, and the reflux drum fills up.

Step 5: Total reflux starts, meaning that all condensed liquid from the reflux drum is sent back to the top tray.

Step 6: The liquid starts to drop from the downcomers, implying that the plates have good liquid holdup.

Step 7: Operational *Steps 5–8* are repeated until the middle component (ethanol) purity in the middle vessel reaches its maximum, and the composition remains unchanged. The operation is then said to have achieved a steady state.

Step 8: When the operation has reached a steady state, terminate the simulation

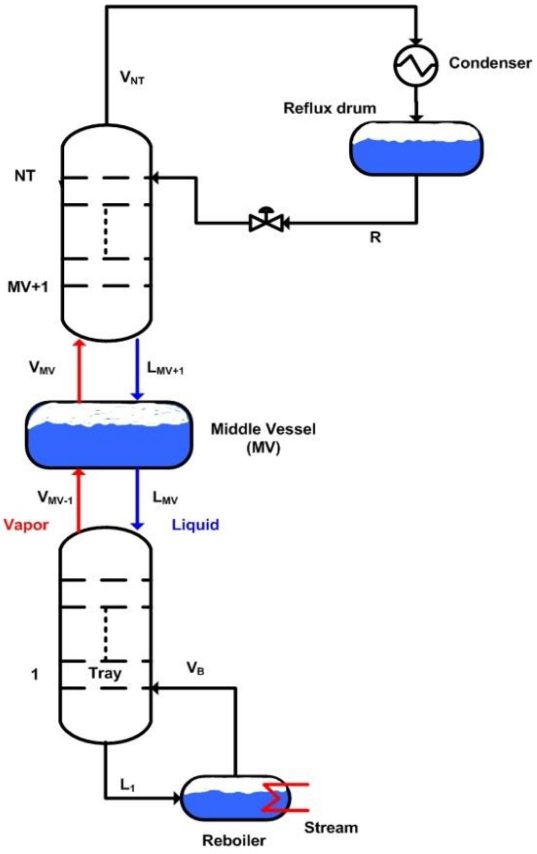

Figure 5.1. Schematic diagram of Middle vessel batch distillation

5.3. Single-stage vapor recompression in middle vessel batch distillation

The principle operation of a VRC with a single-stage compression (SiVRMVBD) in CMVBD is conceptually shown in Figure 5.2. The primary goal of SiVRMVBD is to recover heat from top vapor, resulting in lowering hot and cold utility consumption. SiVRMVBD comprises an extra compressor that is separate from the standard CMVBD. Therefore, the SiVRMVBD simulation approach consists of additional steps compared to CMVBD, i.e., manipulation scenarios. A systematic design, operation and simulation approach of SiVRMVBD is given Table 5.2.

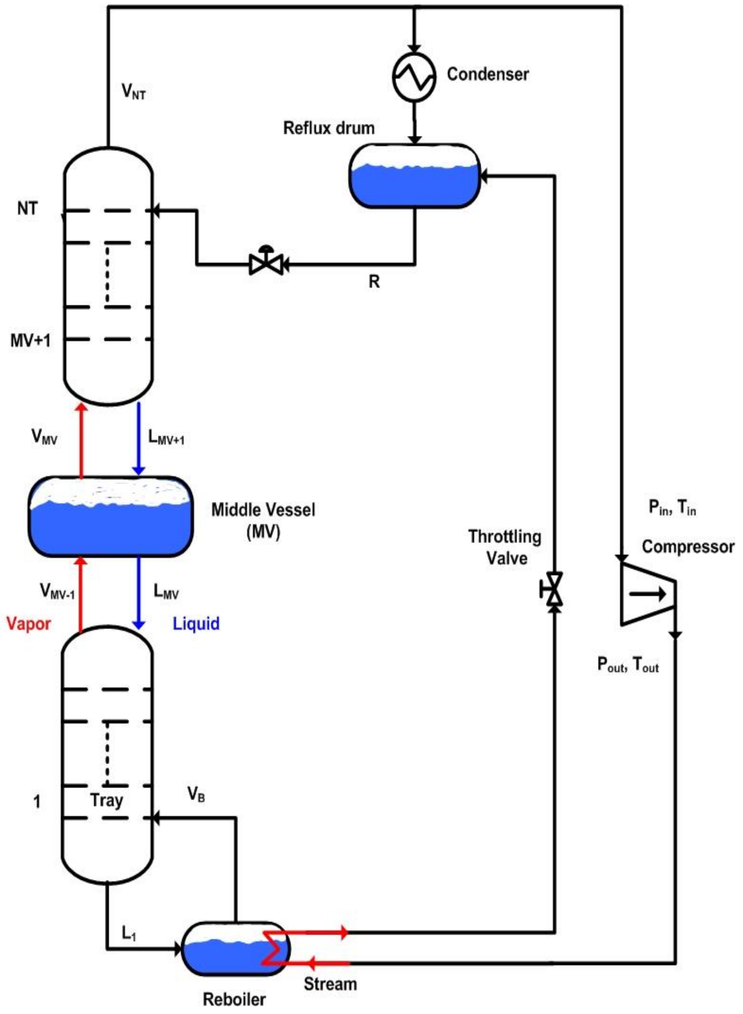

Figure 5.2. Schematic diagram of single-stage VRC in middle vessel batch distillation

Table 5.2. A systematic design, operation and simulation approach of SiVRMVBD

Step 1: Given input data: The column specifications, feed specifications (composition, flow rate, and temperature), tray efficiency, number of trays, initial conditions such as liquid compositions, liquid holdup on all trays, etc.,) are defined

Step 2: The feed mixture is introduced into the reboiler, middle vessel, trays, and reflux drum

Step 3: constant heat input is provided to the reboiler (fixed reboiler heat duty), and the vapor rises.

Step 4: The compressor receives the vapor from the top tray (V_{NT}). The vapor is then compressed to the required temperature (T_{out}). The latent heat (λ) released by the compressed vapor is utilized as heat input to the reboiler and thus condensed against the reboiler liquid.

Two manipulation criteria are employed to maintain (i) temperature difference, i.e., $\Delta T_T = T_{out} - T_B$ of atleast $15^0 C$ for total condensation in reboiler and (ii) same reboiler heat duty. (i) The vapor is compressed so that the compressed overhead vapor and bottom liquid (T_B) have a reasonable temperature difference ($\Delta T_T = 15^0 C$) for total condensation in the reboiler. To maintain $\Delta T_T = 15^0 C$ for total condensation of overhead vapor in the reboiler-condenser and to avoid compressor operation at maximum CR throughout the batch operation, a variable speed compressor (manipulation of CR) has been used.

Manipulation of CR:

We know the ΔT_T,T_B and T_{in} (top tray temperature), then calculate the compressor outlet temperature (T_{out}) from Equation (6), and then find out the corresponding CR to maintain constant value which means CR is variable (Equation 7).

$$\Delta T_T = T_{out} - T_B \tag{5.1}$$

$$T_{out} = \Delta T_T + T_B \tag{5.2}$$

$$CR = \frac{P_{out}}{P_{in}} = \left[\frac{T_{out}}{T_{in}}\right]^{\left(\frac{\mu}{\mu-1}\right)} \tag{5.3}$$

Step 5: The energy is released by the compressor vapour (Q_{CV}) is calculated from the following equation.

$$Q_{CV} = V_{NT} \times \lambda \tag{5.4}$$

(ii) An iterative vapor flow rate manipulation has been employed to accomplish the same dynamical performance between the standard CBDS and SiVRBDS, i.e., maintaining the same reboiler heat duty (Q_R).

Manipulation of vapor flow rate:

If the energy released by the compressed vapour (Q_{CV}) exceeds the required reboiler heat duty, split the top vapor into two parts: One part (V_{Comp}) goes to the compressor to raise the temperature and the rest (V_{Cond}) to the condenser.

If the compressed vapor latent heat (Q_{CV}) is less than the required reboiler heat duty, external heat (Q_E) must be supplied to the reboiler. Compressor work (W_{comp}) is calculated by Equation (12). The mathematical manipulation scenarios are presented below.

$$V_{NT} = V_{Comp} + V_{Cond} \tag{5.5}$$

$$\text{If } Q_{CV} > Q_R$$

```
        $V_{\mathrm{Comp}} = Q_R/\lambda$                                        (5.6)
        $V_{\mathrm{Cond}} = V_{\mathrm{NT}} - V_{\mathrm{Comp}}$                        (5.7)
        $W_{\mathrm{comp}} = \frac{(3.03\times10^{-5})\mu\, V_{\mathrm{comp}}\, T_{\mathrm{in}}}{\mu-1}\left[\left(\frac{P_{\mathrm{out}}}{P_{\mathrm{in}}}\right)^{\frac{\mu-1}{n\mu}} - 1\right]$    (5.8)
        $Q_E = 0$                                                      (5.9)
    else
        $Q_E = Q_R - Q_{\mathrm{CV}}$                                        (5.10)
    end
Where, $\mu$ – polytropic coefficient, $P_{\mathrm{in}}$ & $P_{\mathrm{out}}$ – inlet and outlet pressures of compressor, respectively
Step 6: The complete condensed liquid at high pressure is then throttled to top tray pressure and reaches the reflux drum. Finally, a throttling valve depressurizes the high-pressure condensed liquid before returning to the reflux drum.
Step 7: Total reflux starts, meaning that all condensed liquid from the reflux drum is sent back to the top tray.
Step 8: The liquid starts to drop from the downcomers, implying that the plates have good liquid holdup.
Step 9: Operational Steps 5–8 are repeated until the middle component (ethanol) purity in the middle vessel reaches its maximum, and the composition remains unchanged. The operation is then said to have achieved a steady state.
Step 10: Terminate the simulation, when the operation has reached a steady state
```

5.4. Results and discussion

In this study, ternary zeotropic wide boiling mixture was investigated: Methanol/ethanol/1-propanol. The computer program is developed and simulated in MATLAB.

5.4.1. Conventional middle vessel batch distillation column

Conventional middle vessel batch distillation column dynamics is constructed up of algebraic-differential equations that are applied in (Babu et al., 2013). In addition, this model was developed based on the following assumptions: The liquid and vapor are perfectly mixed on each plate; The molar vapor holdup is little compared to the molar liquid holdup; the delay time between plates is omitted; operates at atmospheric pressure; the nonlinear Francis–Weir formula for tray hydraulics; NRTL thermodynamic model for vapor-liquid compositions are all taken into account. The design and column conditions are shown in Table 5.3.

Table 5.3. Operating conditions and column specifications

System	Methanol/ethanol/1-propanol
Feed composition	0.15/0.7/0.15
Heat input to the reboiler, kJ/min	4000
Tray efficiency, %	80
Number of trays	49
Total feed, kmol	5.385
Initial holdup on trays, kmol	0.098
Initial condenser holdup, kmol	0.035
Initial middle vessel holdup, kmol	0.25
Initial reboiler holdup, kmol	5.25
Steady state compositions	
Distillate vessel, mol%	$0.999/8.4159\times10^{-4}/4.95\times10^{-12}$
Middle vessel, %	0.03/0.97/0.004
Reboiler, %	$2.04\times10^{-4}/1.91\times10^{-5}/0.999$

5.4.2. Dynamic composition profile

Steady-state results show the maximum achievable ethanol purity in the middle vessel, methanol purity in the reflux drum, and 1-propanol purity in the reboiler at 160 min. There is no production phase in the middle vessel batch distillation operation. Therefore, The total batch distillation process takes 160 min. Figure 5.3 (Reflux drum), Figure 5.4 (Middle vessel), and Figure 5.5 (Reboiler) show the dynamic composition profiles throughout the batch operation. It is clear from the Figures that the compositions remain unchanged indicates that the process reaches a steady state. The compositions at the end of steady-state are shown in Table 5.3.

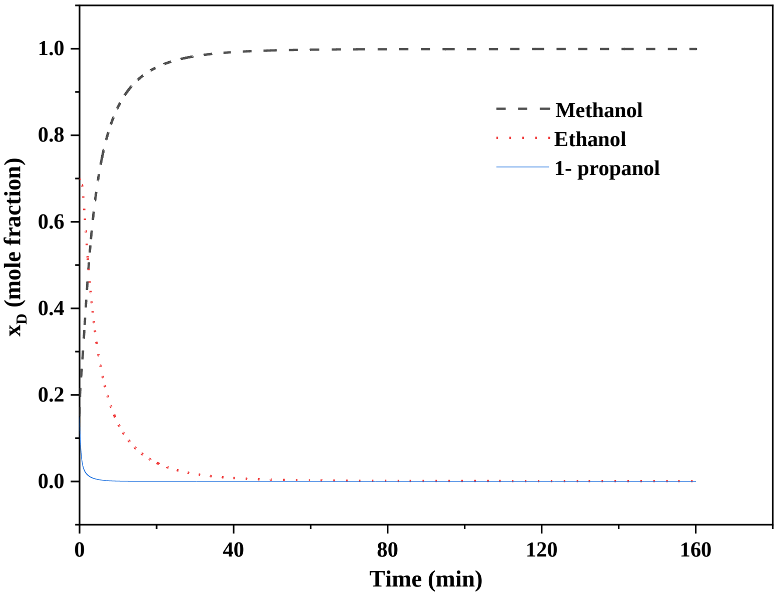

Figure 5.3. Distillate composition profile throughout the operation

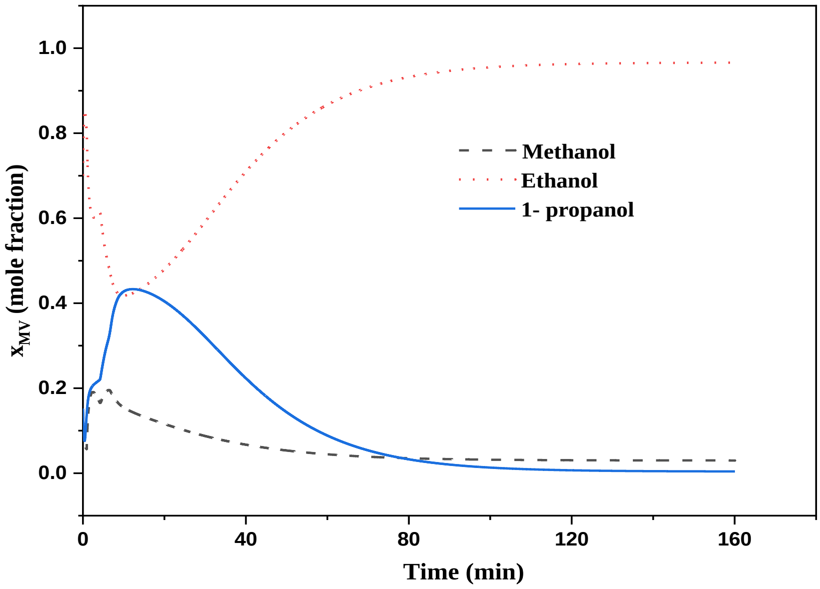

Figure 5.4. Middle vessel composition profile throughout the operation

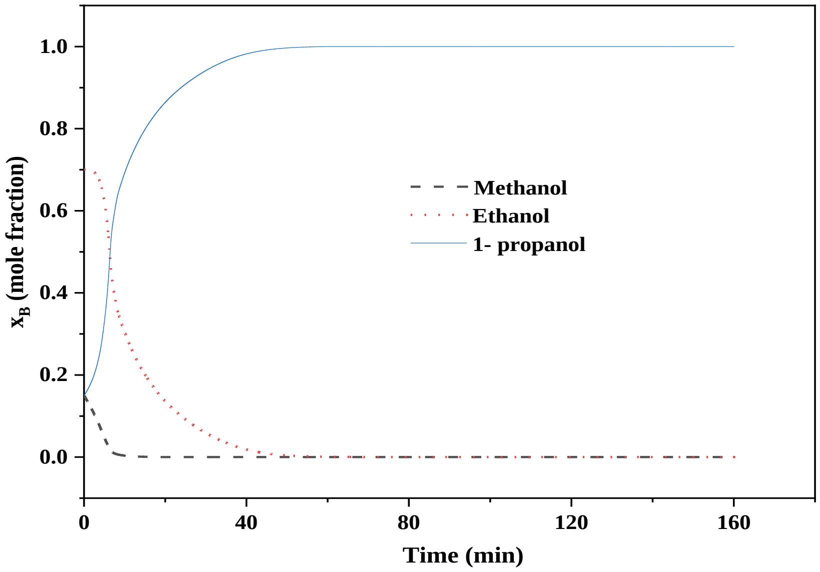

Figure 5.5. Reboiler composition profile throughout the operation

5.4.3. Single-stage vapor recompression in middle vessel batch distillation

Now, to operate the SiVRMVBD at fixed reboiler duty of 4000 kJ/min, the variable manipulation policies as explained earlier (Table 5.2) need to be applied. From simulation approach, we produced the manipulated variables profiles for the SiVRMVBD. Figures 5.6 and 5.7 illustrate the adjustment of external energy from the source and vapor flow rate, respectively. It is evident that throughout the operation, the energy released by the compressed vapor is less than the required reboiler heat duty; hence vapor flow rate manipulation is not needed. Figure 5.8 shows the manipulation of CR to maintain ΔT_T of at least 15. Furthermore, the temperature difference of the column throughout the batch operation is shown in Figure 5.9. It is clear that as CR increases with delta. Also, it is evident that CR throughout the batch operation is within the specified i.e., 3.5. Hence, multi-stage vapor recompression is not recommended.

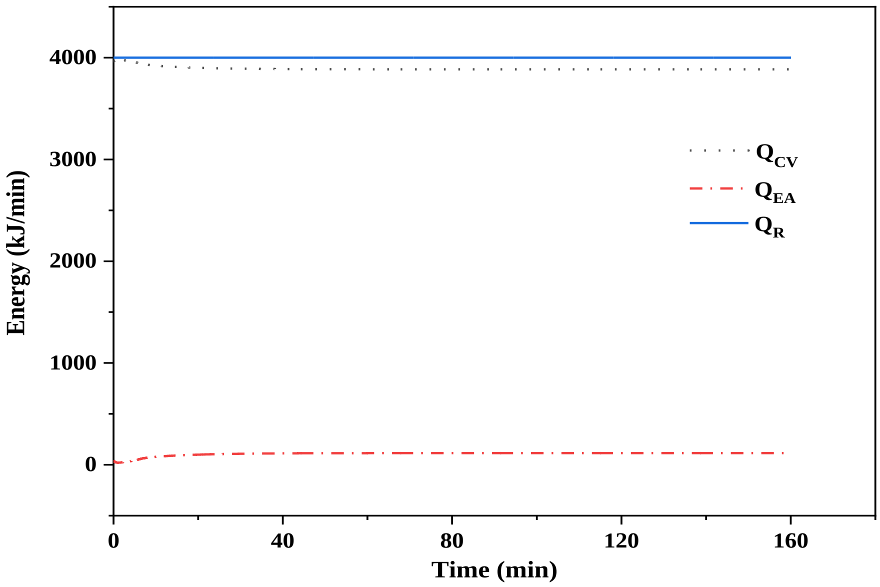

Figure 5.6. Energy profile throughout the batch operation

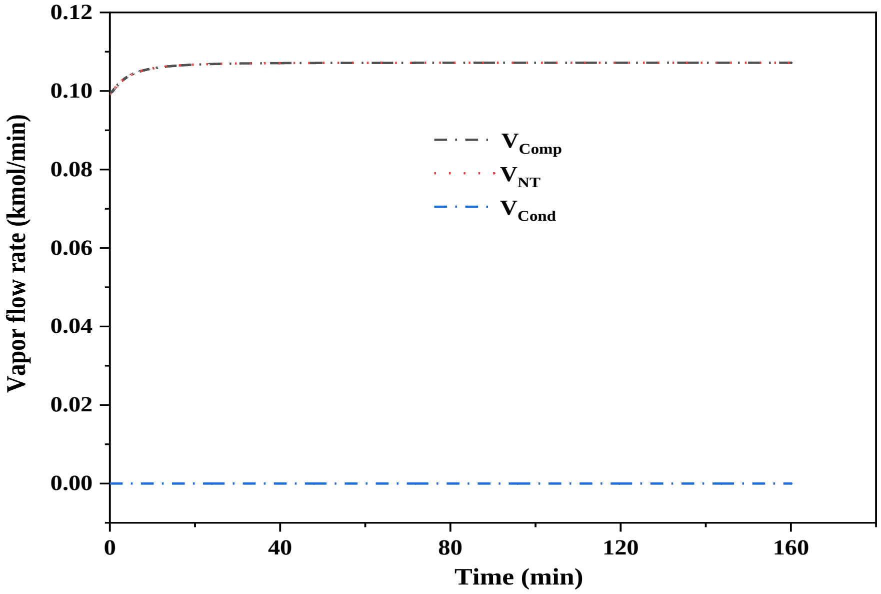

Figure 5.7. Vapor flow rate manipulation profile throughout the batch operation

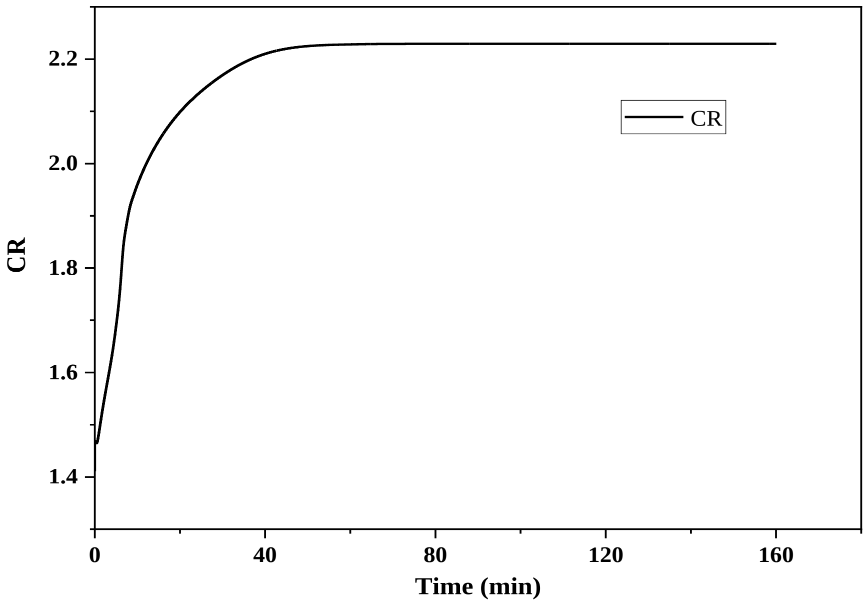

Figure 5.8. Compression ratio (CR) profile throughout the batch operation: multi-stage vapour recompression

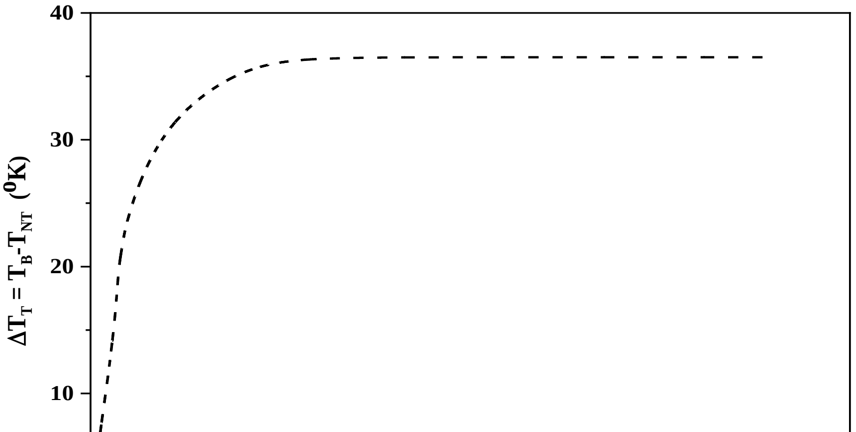

Figure 5.9. Temperature difference of the column throughout the batch operation

5.4.4. Economic and environmental performance analysis

Table 5.4 compares the economic performance between CMVBD and SiVRMVBD. It is clear from Table that the 4.295% TAC savings appears with SiVRMVBD than CMVBD. Furthermore, it is evident that the SiVRMVBD system reduces process cost by 54.7% compared to CMVBD. Table 5.5 shows the environmental comparative performance analysis between

CMVBD and SiVRMVBD. The results show that compared to CMVBD, the proposed SiVRMVBD reduces CO_2 emissions by 93.3% globally. Therefore, we confined that SiVRMVBD is the best alternative in terms of economic, environmental, and practical feasibility compared to CBDS.

Table 5.4. Economic comparative analysis

	CMVBD	**SiVRMVBD**
Capital cost		
Column shell	2.2185×10^5	2.2185×10^5
Column tray	2.3070×10^4	2.3070×10^4
Reboiler	5.4386×10^4	5.4386×10^4
Condenser	2.2618×10^4	-
Compressor	-	4.3770×10^4
Total	3.2192×10^5	3.4307×10^5
Operating cost		
Steam	1.4468×10^4	389.6185
Cooling water	1.3767×10^3	-
Electricity	-	3.1087×10^3
Total	1.5845×10^4	3.4983×10^3
TAC	1.2315×10^5	1.1786×10^5
%, TAC savings		4.295

Table 5.5. Environmental comparative analysis

	CMVBD	**SiVRMVBD**
CO_2 Emissions (ton/yr)		
steam boiler	158.2473	4.2615
gas turbine	-	37.8235
TLE	158.2473	42.085
TLE savings, %	-	73.405
Emissions saved at PS	-	26.7791
Total global emissions (TGE)	158.2473	11.0445
%, TGE savings	-	93.02

5.5. Conclusion

Batch distillation systems are energy-intensive processes and contribute significantly to greenhouse gas emissions (e.g., carbon dioxide) than continuous distillation systems. Reducing carbon dioxide (CO_2) emissions is an absolute necessity and expensive challenge to the chemical process industries to meet the environmental targets as agreed in the Kyoto Protocol.

Vapor recompression (VRC) can reduce process costs and, consequently, CO_2 emissions in batch distillation. Therefore, middle vessel batch distillation have received intense attention in the industry and academia. For the first time, the present research is focused on improving the performance of middle vessel batch distillation for the separation of ternary zeotropic mixture, i.e., methanol/ethanol/1-propanol using VRC. Overall, the results show that the single-stage vapor recompression in middle vessel batch distillation gives a 54.7% reduction in process cost and a 93.3% saving in CO_2 emissions compared to the conventional middle vessel batch distillation.

Chapter 6

Conclusions and future work

Chapter 6

General Conclusions

Apart from the specific conclusions made at the end of each chapter of this dissertation, the general conclusions are drawn in the following:

The present research work aims to improve the performance (minimize process cost and CO_2 emissions) for ternary zeotropic wide boiling mixtures in batch distillation columns using vapor recompression column (VRC). This work proposes a systematic design, operational, and simulation approaches for conventional batch distillation, single-stage vapor recompressed batch distillation, and multi-stage vapor recompressed batch distillation columns. The performance indices total annual cost, CO_2 emissions, and energy savings are used to evaluate the vapor recompressed batch distillation column performance.

We investigated tight composition control of high purity, multicomponent batch distillation for separating zeotropic mixture with and without vapor recompression scheme. Further, double-stage variable speed compressors are used in this study to improve the energy consumption under the limitation of compressors. The comparative analysis has been carried out between open-loop and closed-loop with and without vapor recompression schemes. For tight composition control with vapor recompression, the GSPI controller gives better control performance. Overall observation concludes that the system with GSPI controller integrated with double-stage VRC gives a higher product amount at very high purity than PI and energy savings than closed-loop single-stage VRC.

The batch distillation industry requires a minimum TAC and CO_2 for any investments in heat integrated systems, such as VRC. Consequently, the design conditions for implementing VRC should be chosen such that the energetic performance is maximum at minimum TAC. In this work, heat pump-assisted vapor recompression design approaches are proposed to separate ternary wide boiling mixtures in batch distillation to reduce TAC and CO_2 emissions. Hexanol–octanol–decanol ternary wide boiling mixture model system was selected in this paper. First, a systematic simulation approach was developed to determine the optimal trays based on the lowest energy and TAC reduction. Furthermore, it has been observed that in a SiVRMBD, a high CR is necessary throughout the batch operation, which is practically not feasible for the batch process industry resulting in compressor wear and tear. Therefore, a novel optimal multi-stage vapor recompression approach is proposed to identify the optimum number of compressors with the same design and operating conditions as SiVRMBD, so the batch distillation works at the lowest possible CR throughout while conserving the most TAC. The results suggest that the proposed method saves more TAC and CO_2 emissions than the literature and single-stage VRC scheme. It's worth mentioning that the practices described above can be used for any vapor recompressed batch distillation system.

Aiming to improve further the economic and environmental prospect, in this work, we investigate the feasibility of single-stage and multi-stage VRC schemes in unconventional batch distillation columns, namely, batch distillation with a side withdrawal and middle vessel batch distillation. For the first time, the present research is focused on improving the performance of BDS for the separation of ternary zeotropic mixtures using VRC. In this study, two ternary zeotropic wide boiling mixtures were investigated: hexanol/octanol/decanol (This system has a high wide boiling/zeotropic property because of the significant temperature difference between the lightest and heaviest components) and methanol/ethanol/1-propanol (This system has a moderate wide boiling/zeotropic property because of the moderate temperature difference between the lightest and heaviest components). The results show that in the case of hexanol/octanol/decanol system, the multi-stage VRC reduces process cost (operating costs) CO2 emissions compared to CBDS. However, in the case of methanol/ethanol/1-propoanol systems, the single-stage VRC saves process cost and reduces CO_2 emissions compared to CBDS within the limitations of CR. Also, this work considered middle vessel batch distillation for the separation of ternary zeotropic mixture, i.e., methanol/ethanol/1-propanol using VRC. It was found that the single-stage vapor recompression in middle vessel batch distillation reduces process cost saving in CO_2 emissions compared to the conventional middle vessel batch distillation with minimum specified CR.

We can conclude from this work that a multi-stage VRC scheme is recommended for batch distillation that involves ternary zeotropic wide boiling separations for performance improvement and practical feasibility. A single-stage VRC scheme is recommended for separation of moderate ternary zeotropic mixtures, i.e., methanol/ethanol/1-propanol.

The perspective of future work

The results obtained from the proposed approaches in Chapters 3, 4, and 5 are pretty significant. Hence, the proposed approaches can be extended to extractive batch distillation columns, inverted batch distillation columns, and reactive separations.

This work considered two ternary zeotropic mixtures, i.e., hexanol/octanol/decanol and methanol/ethanol1-propanol. Hence, different ternary zeotropic mixture separations can be regarded in the proposed batch distillation configurations.

Extent the work by applying different energy integration schemes for separating ternary zeotropic wide boiling mixtures: Bottom flashing, closed cycle, internally heat integrated, divided wall batch distillation, etc.

Controllability analysis with advanced model-based control schemes for the proposed schemes

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Zou X, Cui YH, Dong HG, Wang J, Grossmann IE. Optimal design of complex distillation system for multicomponent zeotropic separations. Chemical engineering science, (2012) Jun 18; 75:133-43.

List of Publications

1. **Radhika, Gandu**, Burolia, Akash Kumar, Ambati, Seshagiri Rao and Gara, Uday Bhaskar Babu. "Reducing total annual cost and CO_2 emissions in batch distillation for separating ternary wide boiling mixtures using vapor recompression heat pump" Chemical Product and Process Modeling, vol. , no. , 2022. https://doi.org/10.1515/cppm-2021-0057 (ESCI)

2. **Radhika, Gandu,** Burolia, Akash Kumar, Raghu Raja, Pandiyan Kuppusamy, Ambati, Seshagiri Rao, Patle, Dipesh S. and Gara, Uday Bhaskar Babu. "Energy saving in batch distillation for separation of ternary zeotropic mixture integrated with vapor recompression scheme: dynamics and control" Chemical Product and Process Modeling, vol. 16, no. 2, 2020, pp. 101-115. https://doi.org/10.1515/cppm-2020-0045 (ESCI)

3. **Radhika, Gandu**, Burolia, Akash Kumar, Patle, Dipesh S, Ambati, Seshagiri Rao and Gara, Uday Bhaskar Babu. “Improving energy effieciency and cost-effectiveness of middle vessel batch distillation for separation of ternary zeotropic mixtures via vapor recompression" Energy (Submitted) (SCI)

4. **Radhika, Gandu**, Burolia, Akash Kumar, Patle, Dipesh S, Ambati, Seshagiri Rao and Gara, Uday Bhaskar Babu. “Reducing total annual cost and CO_2 emissions in batch distillation with a side withdrawal for separating ternary zeotropic mixtures using vapor recompression heat pump “Periodica Polytechnica Chemical Engineering” (Under review) (SCI)

5. Burolia, Akash Kumar, **Radhika, Gandu**, Ambati, Patle, Dipesh S, Seshagiri Rao and Gara, Uday Bhaskar Babu. "Reducing CO_2 emissions and process cost in batch distillation with a side withdrawal for separating ternary zeotropic mixtures via multi-stage vapor recompression" Asia pacific Journal of Chemical Engineering (Under preparation) (SCI)

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