

# INDIAN INSTITUTE OF TECHNOLOGY ROORKEE CANDIDATE'S DECLARATION

I hereby assure that the work presented in this report entitled "**Simulation of Extractive Divided Wall Distillation Column**" in partial fulfillment of the requirement for the award of the Degree of **Master of Technology**, submitted in the Department of Chemical Engineering of the Indian Institute of Technology Roorkee, Roorkee is an authentic record of my own work carried out during a period from June 2013 to June 2014 under the supervision of **Dr. Vineet Kumar**, Associate Professor, Department of Chemical Engineering, Indian Institute of Technology Roorkee, Roorkee, India.

I have not submitted the matter, embodied in this report for the award of any other degree.

#### Date

Place: IIT Roorkee

#### (RAMJI PATEL)

## CERTIFICATE

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

### **DR. VINEET KUMAR**

Associate Professor, Chemical Engineering Indian Institute of Technology Roorkee

Roorkee 247667.

### ACKNOWLEDGEMENTS

I owe a great many thanks to all those who were of immense help and supported me during writing of this seminar report. I wish to express my sincere gratitude and appreciations to **Associate Prof. VINEET KUMAR**, Department of Chemical Engineering, Indian Institute of Technology Roorkee for providing me an opportunity to work under her guidance. Her superb guidance with enriched knowledge, regular encouragement and invaluable suggestions at every stage of the present work has proved to be extremely beneficial to me. She has taken pain to go through the project and make necessary correction as and when needed. I consider myself fortunate to have had the opportunity to work under their able guidance and enrich myself from their depths of knowledge.

I would like special thanks to Mr. Ashutosh Mugdil, Mr. Pradeep Kumar Singh, Mr. Pradeep Yadav, Mr. Deepak (Thapa), Mr. Rajkumar Verma.

Above all, I want to express my heartiest gratitude to my parents for their love, faith and support for me, which has always been a constant source of inspiration.

**RAMJI PATEL** 

### ABSTRACT

Distillation is widely used as a separation technique but it consumes more energy. Therefore it's necessary to find efficient design which will reduce energy consumption maintaining same desired purity. Many attempts have been made in this direction, Petlyuk proposed a promising, thermally integrated design which is highly energy efficient. Further, divided wall column (DWC) was introduced in which fully thermally coupled Petlyuk column configuration was implemented in one single column by providing single vertical wall dividing column into two parts. Out of these two parts, one works as pre or post fractionator, and other as main column. Thermodynamically both Petlyuk column and DWC are equivalent.

Divided wall column (DWC) finds many applications in the field of separation of liquid mixture, viz., separation of three or more component mixture, separation of azeotropic mixtures, extractive distillation, reactive distillation, etc. Examples of extractive distillation include separation of aromatic compounds, aqueous alcohol solutions, mixture of hydrocarbons, etc.

Amount of energy consumed is the major operating cost factor; therefore optimal design is obtained in terms of minimum energy consumption without compromising with the product quality. Design variables such as reflux ratio operating parameter such as feed temperature extracting agent flow rate and its temperature have significant effect on optimal design.

In the present study we designed and optimized extractive dividing wall distillation column for dehydration of bio-ethanol. We used RADFRAC model of ASPEN PLUS simulator and NRTL as property method.

In the study we found that for economical and efficient operation proper design of column is important, proper operating parameters like reflux ratio, total number of stages, operating pressure, are important similarly feed condition such as temperature and flow rate also important for economic and efficient operation. By selecting proper feed temperature of mixture to be separated, we can get desired purity with minimum or optimum expenses in terms of reboiler duty and condenser duty. As we have seen with increment in feed temperature recovery of pure ethanol increases but beyond certain value recovery continues to increase but quality decreases continuously along with continuous increase in reboiler duty. Therefore we have to select optimum point at which we recover maximum ethanol with maximum purity.

## CONTENTS

	Page No.
CERTIFICATE	i
ACKNOWLEDGEMENTS	ii
ABSTRACT	iii-iv
CONTENTS	v-vi
LIST OF FIGURES	vii-ix x-xi
LIST OF TABLES	
NOMENCLATURE	xii-xiii
CHAPTER I INTRODUCTION	1-13
1.1 DISTILLATION	1
1.1.1 METHOD OF DISTILLATION COLUMN	2
1.1.2 REACTIVE DISTILLATION COLUMMN	2
1.1.3 AZEOTROPIC DISTILLATION COLUMN	2
1.1.4 EXTRACTIVE DISTILLATION COLUMN	3
1.2 MULTI-COMPONENT DISTILLATION	4
1.3 PETLYUK COLUMN	6
1.4 DIVIDED WALL COLUMN (DWC)	8
1.5 ADVANTAGES OF DIVIDING WALL COLUMN (DWC)	12
1.6 DISADVANTAGES OF DIVIDING WALL COLUMN (DWC)	12
1.7 OBJECTIVES	13
CHAPTER II LITERATURE REVIEW	14-24
CHAPTER III EXTRACTIVE DIVIDED WALL DISTILLATION	ſ
COLUMN	25-27
CHAPTER IV PROBLEM STATEMENT AND SIMULATION	28-35

4.1 PROBLEM STATEMENT	28
4.2 SIMULATION	29
4.3 RESULT OF SIMULATION	32
4.3.1 TEMPERATURE PROFILE FOR PREFRACTIONATOR COLU	JMN
AND DWC	33-34
4.3.2 COMPOSITION PROFILE PREFRACTIONATOR COLUMN A	ND
DWC	34-35
CHAPTER V RESULT AND DISCUSSION	36-47
5.1 EFFECT OF FEED TEMPERATURE	36
5.2 EFFECT OF SOLVENT TEMPETRATURE	38
5.3 EFFECT OF REFLUX RATIO OF FIRST COLUMN	40
5.4 EFFECT OF REFLUX RATIO OF MAIN COLUMN	42
5.5 EFFECT OF NO.OF STAGES PREFRACTIONATOR COLUMN	44
5.6 EFFECT OF NO.OF STAGES MAIN COLUMN	45
CHAPTER VI CONCLUSION AND RECOMMENDATIONS	5 48
6.1 CONCLUSIONS	48
6.2 RECOMMENDATIONS FOR FUTUTRE WORK	48
REFERENCES	49-50
APPENDIX	51-61

## LIST OF FIGURES

Fig no.	Title	Page no.
1.1	Distillation Column	02
1.2	Direct Sequence	04
1.3	Indirect Sequence	04
1.4	Transition Three Column Sequence	05
1.5	Petlyuk Column	07
1.6	Petlyuk Column with Prefractionator	07
1.7	Petlyuk Column with Postfractionator	07
1.8	Dividing Wall Column (DWC)	08
1.9	Number of Divided Wall Columns Delivered	09
1.10	Types of Divided Wall Column	10
2.1 (a)	Publication per Paper of Last 20 year in DWC	20
<b>2.1</b> (b)	Citation per Paper of Last 20 year in DWC	20
2.2 (a)	Publication per Paper of Last 20 year in EDWDC	20
2.2 (b)	Citation per Paper of Last 20 year in EDWDC	20
<b>2.3</b> (a)	Publication per Paper of Last 20 year in EDWDCDO	21
2.3 (b)	Citation per Paper of Last 20 year in EDWDCDO	21
2.4	Multi Components Mixture	22
3.1	Extractive Divided wall Distillation Column	25
4.1	Divided wall Column (DWC)	30
4.2	Decomposed flow-sheet of DWC	30
4.3	Decomposed Flow-sheet of Extractive DWC for Simulation	31
	using Aspen Plus	
4.4	Residue Curve Map	31
4.5	Temperature (°C) Profile in prefractionator Column	33
4.6	Temperature (°C) Profile in Divided wall Column	34

4.7	Liquid Composition profile in Prefractionator Column	34
4.9	Liquid Composition Profile in Main Column	35
5.1	% Purity of Ethanol Obtained vs. Feed temp	36
5.2	% Ethanol Recovered vs. Feed Temp	36
5.3	% Ethylene glycol vs. Feed Temperature	37
5.4	% Ethylene glycol Recovery vs. Feed Temperature	37
5.5	Total Reboiler Duty in KW vs. Feed Temperature	37
5.6	Total Condenser Duty vs. Feed Temperature	37
5.7	Distillate flow vs. Feed Temperature	38
5.8	% Ethanol Purity vs. Solvent Temp	39
5.9	% Ethanol Recovered vs. Solvent Temp	39
5.10	Total Reboiler Duty vs. Solvent Temp	39
5.11	Total Condenser Duty vs. Solvent Temp	39
5.12	% Ethylene glycol Recovered vs. Solvent Temp	40
5.13	Distillate flow rate vs. Solvent Temp	40
5.14	% Purity of Ethanol vs. Reflux ratio	41
5.15	% Recovery of Ethanol vs. Reflux ratio	41
5.16	Total Reboiler Duty vs. Reflux ratio	41
5.17	Total Condenser Duty vs. Reflux ratio	41
5.18	Distillate flow vs. Reflux ratio	41
5.19	% Recovery of Ethylene glycol vs. Reflux ratio	41
5.20	% Purity of Water vs. Reflux ratio	42
5.21	% Purity of Ethanol vs. Reflux ratio	42
5.22	% Recovery of Ethanol vs. Reflux ratio	42
5.23	% Purity of Ethylene glycol vs. Reflux ratio	43
5.24	% Recovery of Ethylene glycol vs. Reflux ratio	43
5.25	% Purity of Water vs. Reflux ratio	43
5.26	Total Reboiler Duty vs. Reflux ratio	43
5.27	Total Condenser Duty vs. Reflux ratio	43
5.28	% Purity of Ethanol vs. Number of Stages	44

5.29	% Recovery of Ethanol vs. Number of Stages	44
5.30	% Purity of Ethylene glycol vs. No. of Stages	44
5.31	% Recovery of Ethylene glycol vs. No. of Stages	44
5.32	Total Reboiler Duty vs. No. of Stages	45
5.33	Total Condenser Duty vs. No. of Stages	45
5.34	Distillate flow rate vs. Number of Stages	45
5.35	% Purity of Water vs. Number of Stages	45
5.36	% Ethanol Recovered vs. No. of Stages	46
5.37	% Purity of Ethanol vs. No. of Stages	46
5.38	Distillate flow rate vs. No. of Stages	46
5.39	% Purity of Water vs. No. of Stages	46
5.40	% Purity of Ethylene glycol vs. No. of Stages	47
5.41	% Recovery of Ethylene glycol vs. No. of Stages	47
5.42	Total Reboiler Duty vs. No. of Stages	47
5.43	Total Condenser Duty vs. No. of Stages	47

## LIST OF TABLES

S. No.	Title	Page No.
2.1	Comparison between Conventional Two Column Configuration and DWC	17
4.1	Properties of Component Present in the Extractive Distillation Column	29
4.2	Input Parameters of Extractive Divided Wall Distillation Column	32
4.3	Heat balance of Divided wall Column as Compared to that Proposed by (kiss <i>et al.</i> , 2012)	33
A1.1	Effect Feed Temperature on Total Reboiler Duty and Condenser Duty	51
A1.2	Effect of Feed Temperature on Purity and Recovery of Components and Distillate Flow of First Column and Bottom Product of Second Column	52
A2.1	Effect of Solvent Temperature on Reboiler and Condenser Duties of Two Columns	53
A2.2	Effect of Solvent temperature on Purities and Recoveries of Three Component Distillate Flow rate and Ethylene glycol Flow rate	54
A2.3	Effect of Solvent Temperature on Reboiler and Condenser Temperature	54
A3.1	Effect of Reflux ratio of First Column on Reboiler and Condenser Duties	55
A3.2	Effect of Reflux Ratio on Purities and Recoveries of Three Products and Distillate Flow rate and Ethylene Glycol Flow rate	56
A3.3	Effect of Reflux Ratio on Reboiler and Condenser or Bottom Stage Temperature of Two Columns	56
A4.1	Effect of Reflux Ratio of Second Column (Main Column) on Reboiler and Condenser Duties of Two Columns	57
A4.2	Effect of Reflux ratio of Second Column (Main Column) on Purities and Recoveries of Three Products and Distillate Flow rate (Col1) and Bottom Product rate (Col2)	57
A4.3	Effect of Reflux Ratio of Second Column (Main Column) on Condenser and Reboiler or Bottom Stage Temperature	58
A5.1	Effect of Total Number of Stages of First Column	58

	(Prefractionator) on Reboiler and Condenser Duties of Two	
	Columns	
A5.2	Effect of Total Number of Stages of First Column	59
	(Prefractionator) on Purities and Recoveries of Three Products and	
	Distillate Flow of (Col1) and Bottom Product Flow (Col2)	
A5.3	Effect of Total Number of Stages of First Column	59
	(Prefractionator) on Condenser and Reboiler or Bottom Stage	
	Temperature of Two Columns	
A6.1	Effect of Total Number of Stages of Main Column on Reboiler	60
	and Condenser Duties of Two Columns	
A6.2	Effect of Total Number of Stages of Second Column (Main	60
	Column) on Purities and Recoveries of Three Products, Distillate	
	Flow rate (Col1), Bottom Product Flow (Col2)	
A6.3	Effect of Total Number of Stages of Second Column (Main	61
	Column) on Condenser and Reboiler or Bottom Stage	
	Temperature of Two Columns	

#### NOMENCLATURE

- $A_h$  = Total cross sectional area of all the holes on a tray, m<sup>2</sup>
- B= Bottom Product
- DWC= Divided wall column.
- $D_1$  = Distillate of prefractionator
- $D_2$  = Distillate of main column.
- F= feed flow rate.
- H = Enthalpy of vapour KJ/mol.
- h = Enthalpy of liquid KJ/mol.
- $H_f =$  feed enthalpy.
- HK= heavy key.
- LK=light key.
- $L_{1p}$  = liquid flow rate leaving from 1<sup>st</sup> stage of prefractionator.
- $L_{1m}$ = liquid flow rate leaving from  $1^{st}$  stage of main column.
- $L_{2p}$  = liquid flow rate leaving from second stage of prefractionator.
- L<sub>2m</sub>= liquid flow rate leaving from second stage of main column.
- $L_{Nm-1}$  = liquid flow rate from  $N_{m-1}$  stage of main column.
- $L_{Nm}$  = liquid flow rate from the last divided stage of main column.
- $L_{Np}$  = liquid flow rate from the last divided stage of prefractionator.
- $L_{Np-1}$  = liquid flow rate from stage  $N_{p-1}$ .
- $l_{Np}$  = liquid flow rate from the stage  $N_p$ .
- MK = Middle boiling key component.
- $N_t$  = first undivided stage of main column.
- $N_m$  = last divided stage of main column.
- $N_p$  = last divided stage of prefractionator.

 $N_{m-1}$  = stage before last divided stage of main column ( $N_m$ ).

 $N_{p-1}$  = stage before last divided stage of prefractionator ( $N_p$ ).

R1 = vapour split ratio.

 $V_{2p}$  = vapour stream flow rate from second stage of prefractionator.

 $V_{3p}$  = vapour stream flow rate from third stage of prefractionator.

 $V_{2m}$  = vapour stream flow rate from second stage of main column.

 $V_{3m}$  = vapour stream flow rate from third stage of main column.

 $V_{Nm}$  = vapour stream flow rate from (N<sub>m</sub>) stage (last divided) of main column.

- $V_{Nt} = Vapour$  flow rate coming from  $N_t$  stage.
- $V_{Np}$  = Vapour flow rate coming from  $N_p$  stage.
- $V'_t$  = vapour flow rate from stage N<sub>t</sub> to prefractionator.
- $V''_t$  = vapour flow rate from stage  $N_t$  to main column