A
Project Report
On

"STEADY STATE SIMULATION OF AN AZEOTROPIC DISTILLATION USING ASPEN PLUS"

Submitted by

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In the fulfilment of Bachelor of Technology in Chemical Engineering at Dr. Babasaheb Ambedkar Technological University, Lonere, Raigad in the academic year 2021-22.

Department of Chemical Engineering

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CERTIFICATE

This is to certify that the Project report entitled "**STEADY STATE SIMULATION OF AN AZEOTROPIC DISTILLATION USING ASPEN PLUS**" is the bonafide work carried out by Mr. Rohit Vinayak Vidhate (10303320181150710065) and Mr. Pratik Mukund Paralikar (10303320181150710047) of Final year Bachelor of Technology in Chemical Engineering. It is approved for fulfillment of requirement of Degree of Bachelor of Technology in Chemical Engineering of **Dr. BABASAHEB AMBEDKAR TECHNOLOGICAL UNIVERSITY, Lonere, Raigad 402103**.

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It gives us immense pleasure to present our report for seminar on "**STEADY STATE SIMULATION OF AN AZEOTROPIC DISTILLATION USING ASPEN PLUS**". The able guidance of all teaching staff of this department made the project possible. They have been a constant source of encouragement throughout the completion of this project. We would like to express our grateful thanks to **Dr. A. R. Chavan sir** who guided properly for this Project. We would also like to express our sincere thanks to **Department of Chemical Engineering** for giving us an opportunity to explore the subject by conducting this Project.

ABSTRACT

Ethanol – water forms homogenous minimum boiling azeotrope and it has most common occurrence in many industrial processes. There are various techniques exist to separate azeotropic compositions. In this work we studied the basic features of azeotropic distillation using aspen plus simulation. Since entrainers are indispensable in separating constant boiling mixture, in this work, performance of three different entrainers were studied viz. benzene, n-pentane and cyclohexane. The effect of various process variables viz. reflux-ratio and distillate rate on the purity of final products was studied in detail. Radfrac simulator was used to study the distillation process and NRTL method was used as the base thermodynamic method.

It has been found out after simulation that cyclohexane performed best where maximum purity of ethanol (99.91%) was obtained closely followed by n-pentane (99.84%) and benzene (99.77%) as entrainers. The optimum number of stages were 25 (for n-pentane), 45 each for benzene and cyclohexane respectively.

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NOMENCLATURE

τ^{12} and τ^{21} = dimensionless interaction parameter

Δg^{12} and Δg^{21} = interaction energy parameters

R = gas constant

T = Absolute Temperature

U_{ij} = Energy between surfaces i and j

CHAPTER 1

INTRODUCTION

This chapter highlights the basics on azeotropic distillation system. It mainly focusses on the ethanol water azeotropic distillation to get high purity ethanol as product. The background of the present thesis work is suitably explained. The objectives are also properly highlighted.

1.1. INTRODUCTION

Production of fuel grade bioethanol has recently got attention because of two important things. First, it is frequently being used as a fuel oxygenate in place of methyl t-butyl ether (MTBE). The second reason that it is used as alternate fuel. Sugar fermentation process gives rise to production of bioethanol; it can also be prepared by chemical process or as a by-product of some chemical processes [1]. Sources of ethanol are mainly sugar and crops. Crops are grown specifically for energy use this includes maize, wheat and corn crops, waste straw. There is also ongoing research into the use of municipal solid waste to produce ethanol fuel.

Ethanol or ethyl alcohol (C_2H_5OH) is a clear colourless liquid, which is degrade biologically, less toxic and causes minor ecological contamination if split. Ethanol gives carbon dioxide and water when it burns. Pollution caused by the petroleum product are being reduce by this such as SOx and NOx. Octane number of ethanol is high and has replaced as an octane enhancer in petrol [1]. Bioethanol and biodiesel the alternatives of the fuels which we use. The production of alternate type of fuel is due to the realization that crude oil stocks are limited, hence shifted towards more renewable sources of energy. As alternative fuels this bioethanol and biodiesels have got newly attention and have virtually limitless potential for growth However; separation of bioethanol is the main problem in production of bioethanol, because water present in it. The separation of ethanol from water is difficult because azeotrope present in the mixture. There are several methods of high purity ethanol separation such as [2]:

1. Azeotropic distillation
2. Extractive distillation
3. Pressure swing method
4. Chemical action separation
5. Distillation using dissolved salt

1.2 AZEOTROPIC DISTILLATION:

Azeotropic distillation is different kind of technique which is used to break the azeotrope of the mixture in separate distillation column. In chemical engineering azeotropic distillation normally refers the addition of another compound to produce another azeotrope which is heterogeneous lower boiling azeotrope and it produces two immiscible liquid phases e.g benzene is added is ethanol water mixture to break the azeotrope and to separate ethanol. A separation technique for azeotropic mixture has very crucial importance in industry and distillation is the major process used for such separations. Most liquid mixtures of organic components form non ideal systems. The presence of some specific groups, mainly polar groups such as oxygen, nitrogen, chlorine and fluorine, often results in the formation of azeotropes. However, separation becomes complicated because presence of non-ideality and azeotrope in mixture [3].

An azeotrope cannot be separated the constituents of an azeotrope by simple distillation process since at this point no enrichment of vapor phase presence. Therefore, the separation of this mixture involves the other techniques by putting an additional component, called entrainer. Entrainer affects the volatility of one of the azeotrope constituents more than another and form ternary azeotrope. But, after using entrainer it may cause some unwanted impurities and side streams because large energy consumed by this process.

Azeotropic mixtures may often be effectively separated by distillation by adjusting the reflux ratio of the system

1.3 Background of present work:

Alcohol-water mixture has a very common occurrence in many industrially important processes. However, apart from methanol-water, most of the combinations form nonideal solutions. Vapor-liquid equilibrium study generally shows that non-ideal solutions form homogeneous or heterogeneous minimum or maximum boiling azeotropes.

Separation of azeotropes using conventional distillation processes doesn't yield the desired degree of purity in terms of distillate or bottom products and hence we need to resort to alternative routes or techniques. Addition of a third component as an entrainer which changes the relative volatility of the constituting species is the most common method in azeotropic process. In our understanding, studying the azeotropic characteristics and separation mechanism of ethanol-water solution is very fundamental before venturing into separation of higher order alcohol-water systems and this was the reason and motivation behind taking up this project.

CHAPTER 2

LITERATURE REVIEW

In this chapter a brief review on, to understand the difficulties in the separation with the ethanol water system, it is important to study the phenomenon that applicable to non-ideal VLE behaviour, and how to overcome from the non-ideal VLE. How azeotrope formed and how to break it is briefly discussed in this chapter. In this chapter a review is presented on:

2.1 Introduction

- Azeotrope and formation of this azeotrope in ethanol water mixture.
- Brief descriptions of methods to overcome from the azeotropic behaviour of the mixture.
- Review of the list of chemicals use as entrainer in azeotropic distillation for high purity of ethanol production.

2.2 Azeotrope:

An azeotrope is a mixture of chemical components that has identical compositions of the liquid &vapor phases in equilibrium with each other. This is occurring because of molecular interactions between different chemical components [4]. If the element contains similar structures and elementary molecules then the molecular interaction will be very much less hence azeotrope do not occur examples: mixture of hydrocarbons (propane, butane, pentane etc.) this has phase equilibrium system. But if components are dissimilar then molecule can exhibit either repulsive or attractive forces at the given temperature vapor pressure increases by the repulsion or may give rise to minimum boiling point azeotrope. Activity coefficients become greater than one because of the repulsion. If the repulsion sufficiently greater, the repulsive forces give rise to formation of heterogeneous minimum-boiling point azeotropes

(two liquid phases). The azeotrope boils at a temperature that is lesser than the boiling point of the lighter component [4].

The methanol–water mixture is an example of mixture which has modest non ideality. The OH end of the molecule of CH_2OH and H_2O molecule are similar, but the hydrocarbon CH_3 end of the CH_2OH molecule is different from water. So, the system shows a minor amount of non-ideality, change in relative volatility as liquid composition changes represents this. If addition of extra more CH_2 group and when get the ethanol–water system, then it causes large repulsion because the CH_3–CH_2 end of the ethanol molecule is somewhat different than the OH end of the molecules of water. The system shows more non ideality and a minimum boiling azeotrope occurs. This is homogeneous azeotrope (only one liquid phase is in equilibrium with a vapor phase). If addition of two more CH_2 groups and when we move towards the n-butanol–water system, then the repulsion is more than the ethanol water [4]. The result is the formation of a heterogeneous minimum boiling azeotrope with two liquid phases in equilibrium with a vapor phase. In some chemicals compounds molecules attract instead of repulsion then this attraction reduces the effective vapor pressure of the compound and then it forms maximum boiling azeotrope due to molecular attraction. An example of maximum boiling azeotrope is nitric acid- water, chloroform- acetone, water and formic acid mixture etc.

2.3 Types of azeotropes:

2.3.1. Positive azeotrope or Minimum boiling azeotrope:

The azeotrope boils at a temperature that is lower than the boiling point of the lighter component, positive azeotrope also called minimum boiling azeotrope

The Fig.2.1 represents the T_{XY} diagram that is temperature vs composition curve of the mixture diagram. It shows a minimum boiling azeotrope of different constituent; provide the boiling temperature of various compositions. The upper curve represents the vapor compositions while lower curve represents the liquid compositions in the mixture. While boiling it has a mixture of both liquid and vapor phase. The point at which both curve touches each other that point shows the azeotropic point of the mixture. At this point liquid composition in the mixture and vapor

composition in the mixture are equal in amount [2], let X and Y has a mixture and it produces azeotrope and X has lower boiling point than Y then leftmost point where the vapor composition of X is zero that is at leftmost point is purely Y so leftmost point shows boiling point of Y and at rightmost point where X is 100% it shows the boiling point of X and point at which both curve touches that is azeotropic point of the XY mixture. So, when boiling point of azeotrope is lower than the boiling point of both the constituents then it shows the minimum boiling azeotrope.
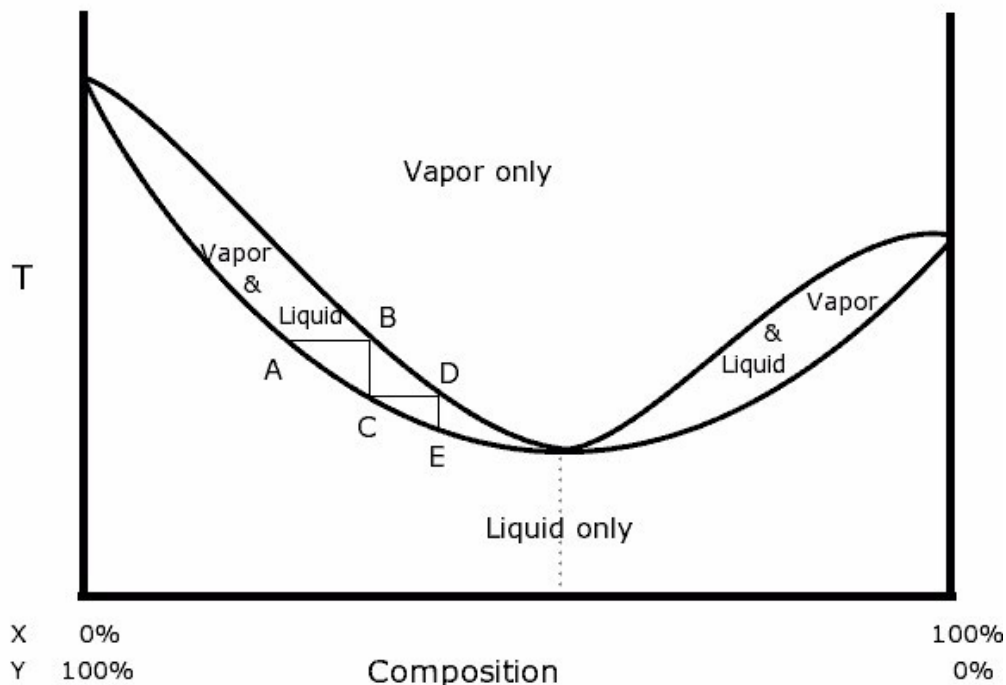
Fig.2.1 Phase diagram of a positive azeotrope [5]

2.3.2 Negative azeotrope or Maximum boiling point azeotrope:

The boiling point of this azeotrope is higher compared to the boiling points of its components. Negative azeotrope also called maximum boiling point azeotrope.

Fig.2.2 represents the Txy diagram for negative azeotrope that is temperature vs composition curve. In the figure upper curve is the vapor composition while lower curve is the liquid composition in the two-phase mixture. When the mixture is heated then its azeotrope has the boiling point more than the boiling point of the individual components then this mixture shows the negative azeotrope or maximum boiling azeotrope. In this figure leftmost point is the boiling point of the Y whereas the rightmost point shows the boiling point of the Y and point at which both curve touches each other, then temperature at this point is the temperature of the azeotrope. And it is clearly shows that azeotrope has higher boiling temperature than the temperature of both X and Y.
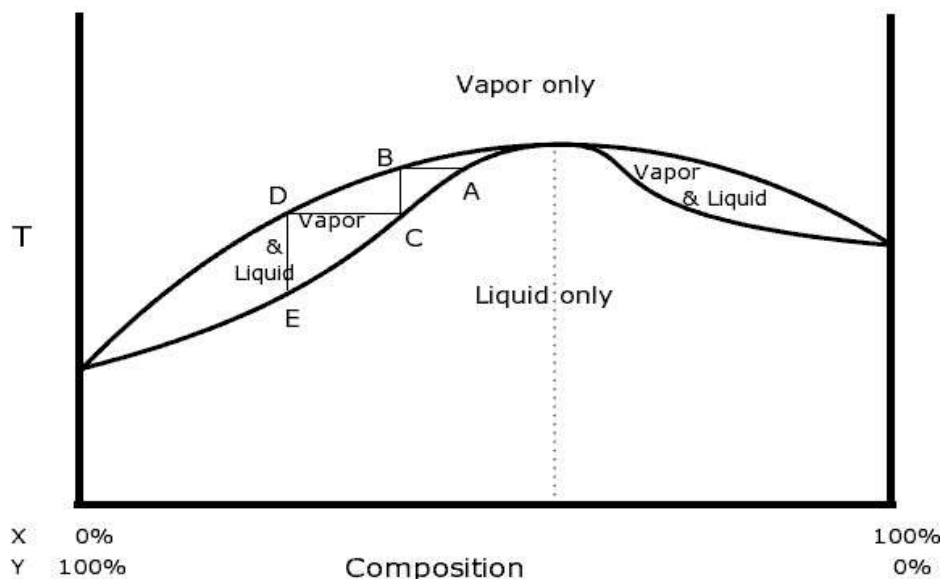
Fig.2.2 Phase diagram of negative azeotrope [5]

2.4 Ethanol water azeotropic mixture:

Ethanol also called ethyl alcohol which is a colourless, volatile and can catch fire type of liquid. Molecular formula of ethanol is C_2H_5OH which is straight chain compound. It is a constitutional isomer of dimethyl ether. Ethanol is often abbreviated as EtOH, using the common organic chemistry notation of representing the ethyl group (C_2H_5). Ethanol boils at 78.4°C while water boils at 100°C. Mixtures of ethanol and water boils at 78.2°C mixture of ethanol and water forms an azeotrope at 96 volume% ethanol and 4% water at normal pressure and temperature (351 K). Fig 2.3 represents the ethanol water composition curve/ y-x diagram. This azeotropic composition is strongly depends on temperature and pressure [4]. Fig.2.4 VLE diagram for ethanol-water system showing composition curve for ethanol and water
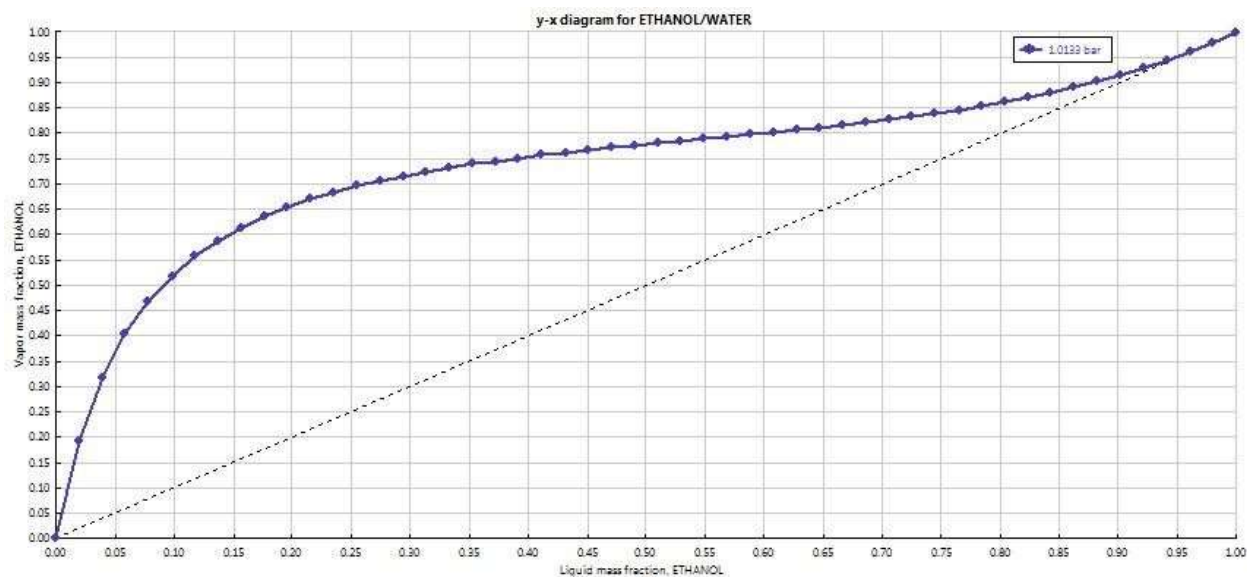
Fig. 2.3 Ethanol water composition curve/ y-x diagram
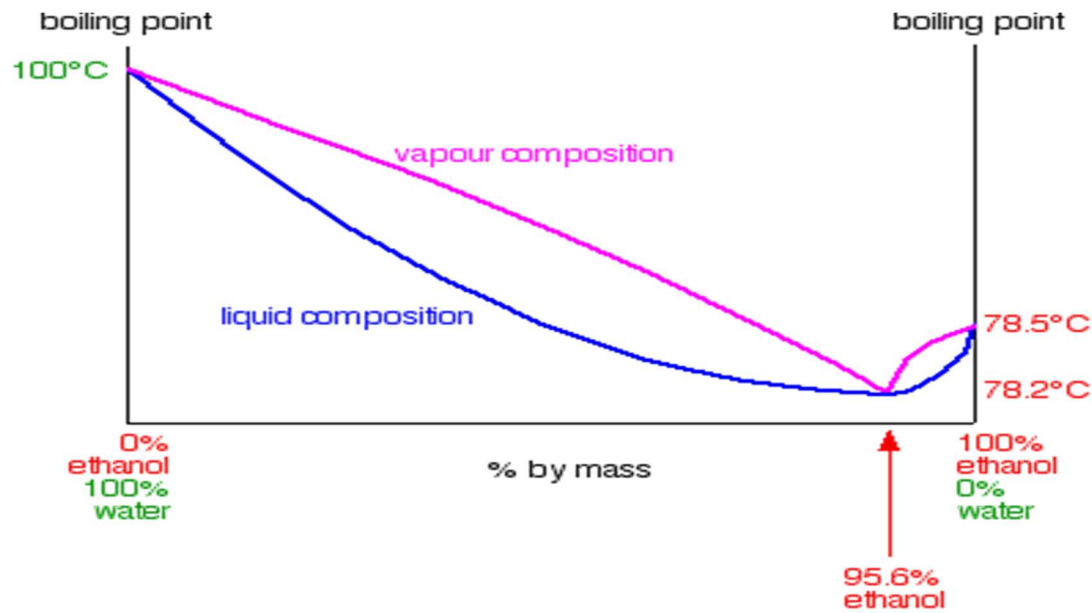
Fig.2.4 VLE diagram for ethanol-water system showing composition curve for ethanol and water [6]

2.5 Types of distillation process for azeotropic mixtures.

The separation of azeotropic systems can be achieved by the addition of a suitable solvent, which influences the activity coefficient and relative volatility. Methods for separating azeotropic mixtures as follow:

➢ **Extractive distillation** –At the top of the column solvent which has high boiling point is added to similarly change the relative volatility in order to make separation. Bottom is separated by the second column to recycle the solvent [7]
.

➢ **Azeotropic Distillation** – where another compound which is entrainer is added to the mixture, this entrainer affects the relative volatility of the one compound and separation becomes easier.

- ➢ **Distillation using salts** – the salts dissolves in the mixtures of liquid and changes the relative volatilities such that separation becomes possible.

- ➢ **Pressure swing distillation** – To separate binary azeotropes two columns which are operated at different pressures are used which change appreciably in composition over a moderate pressure range or where a separating agent which forms a pressure-sensitive azeotrope is added to separate a pressure-insensitive azeotrope.

- ➢ **Reactive distillation** – the separating agent reacts very good and reversibly with one of the azeotropic constituents in the mixture. To recover the initial component reaction is reversed by the distillation of the product from the non-reacting components [8].

So, this project focuses on azeotropic distillation hence it was reviewed.

2.6 Ethanol-water azeotropic distillation separation:

When the process requires high purity of ethanol as final product. Ethanol is mainly produced in process of fermentation. A typical mixture from a fermentation process has very low ethanol concentrations (4–6 mol %). In the column operating at atmospheric pressure mixture is fed into it from bottom high purity of water is taken out but purity of the ethanol cannot exceed more than 90% [5].

Some brilliant engineers gave the idea of running the fermentation liquid through a conventional "preconcentrator" distillation column which takes water as bottom product, and in distillate it produces 84% ethanol and 16% water. This binary mixture is further fed into a second distillation column. Also fed to the top of this column as reflux is a stream that contains a high concentration of benzene [9]. The benzene acts as a "light entrainer" that goes to top and take water with it because the large dissimilarity between water and benzene becomes the water very volatile. The

ethanol goes out from the bottom acts that water is the "heavier" component (normal boiling point of ethanol is 351.5 K, while that of water is 373.2 K). The Fig.2.5 given below shows Azeotropic distillation diagram for ethanol water azeotropic distillation. The overhead vapor is a ternary mixture of all three components. And bottom product of the second column will get the high purity ethanol as product. The overhead vapor in second column further can be treated to recycle the components.
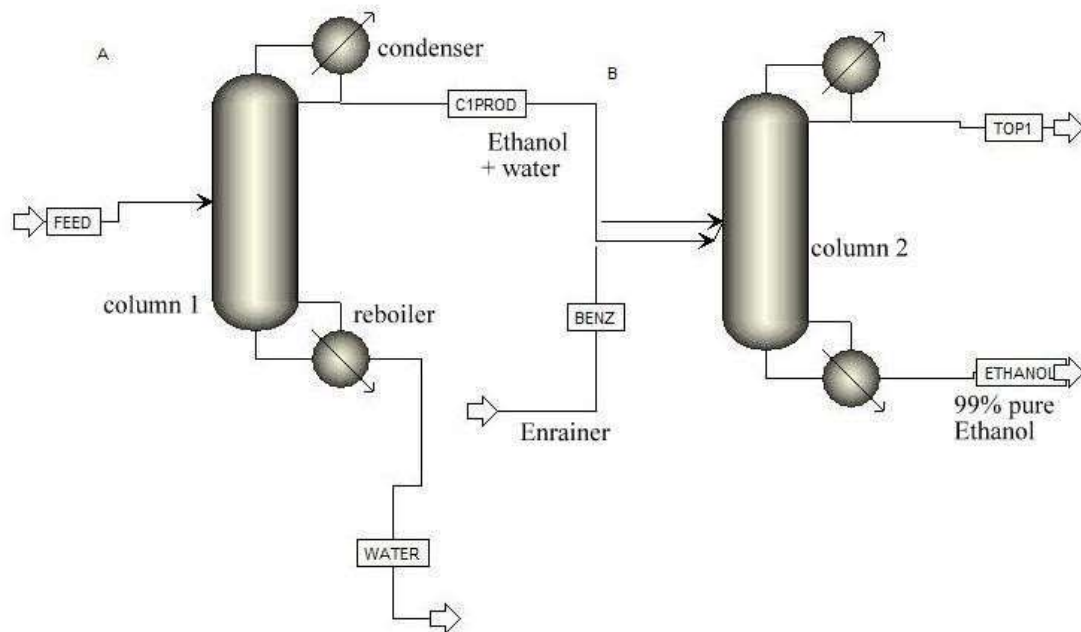
Fig.2.5 Azeotropic distillation diagram for ethanol water azeotropic distillation

Given below are the experimental data for the components this project is based on- ethanol,

water, and the ethanol- water system. We can conduct the simulation using this experimental

data or using the data given in aspen database.

Table 2.1: Experimental vapour pressure data for ethanol.

Pressure (kPa)	Temperature (K)		ΔT
	Experimental	Literature	
10	302.351	302.428	0.077
20	315.309	315.391	0.082
30	323.518	323.592	0.074
40	329.729	329.717	0.012
50	334.677	334.655	0.022
60	338.771	338.817	0.046
70	342.385	342.430	0.045
80	345.662	345.632	0.030
90	348.543	348.514	0.029
100	351.041	351.138	0.097
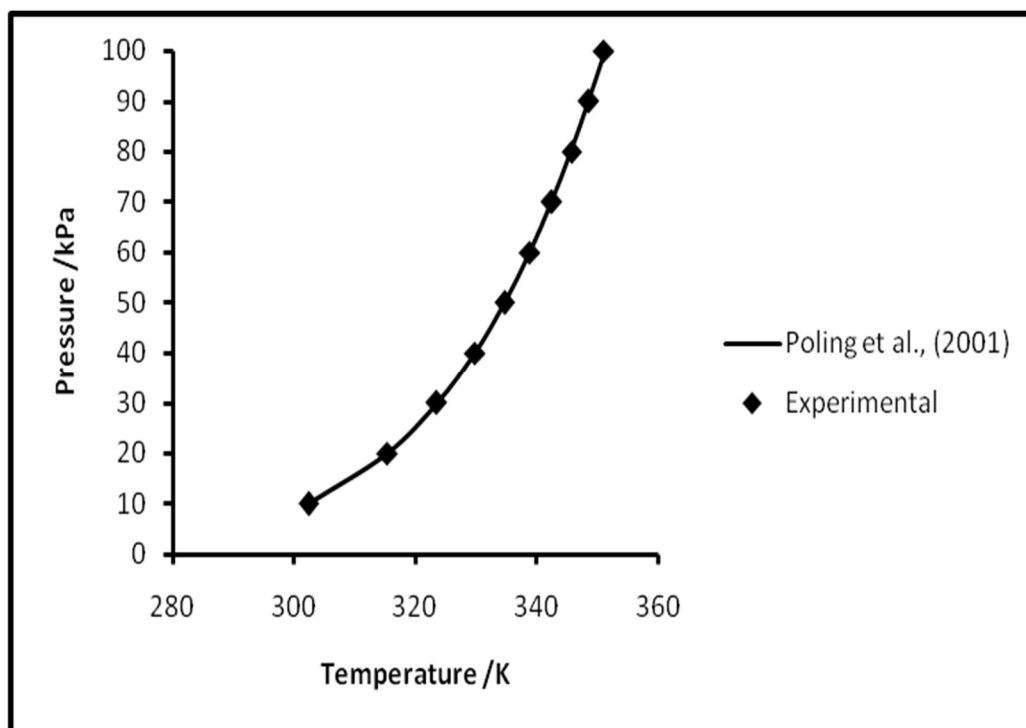
Figure 2.6: Vapour pressure data for ethanol.

Table 2.2: Experimental vapour pressure data for water.

Pressure (kPa)	Temperature (K)		ΔT
	Experimental	Literature	
10	318.130	318.195	0.065
20	332.482	332.589	0.107
30	341.733	341.718	0.015
40	348.445	348.546	0.101
50	354.006	354.059	0.053
60	358.771	358.711	0.060
70	362.785	362.753	0.032
80	366.275	366.338	0.063
90	369.543	369.566	0.023
100	372.909	372.909	0.061

Literature values

(Poling *et al.,* 2001)
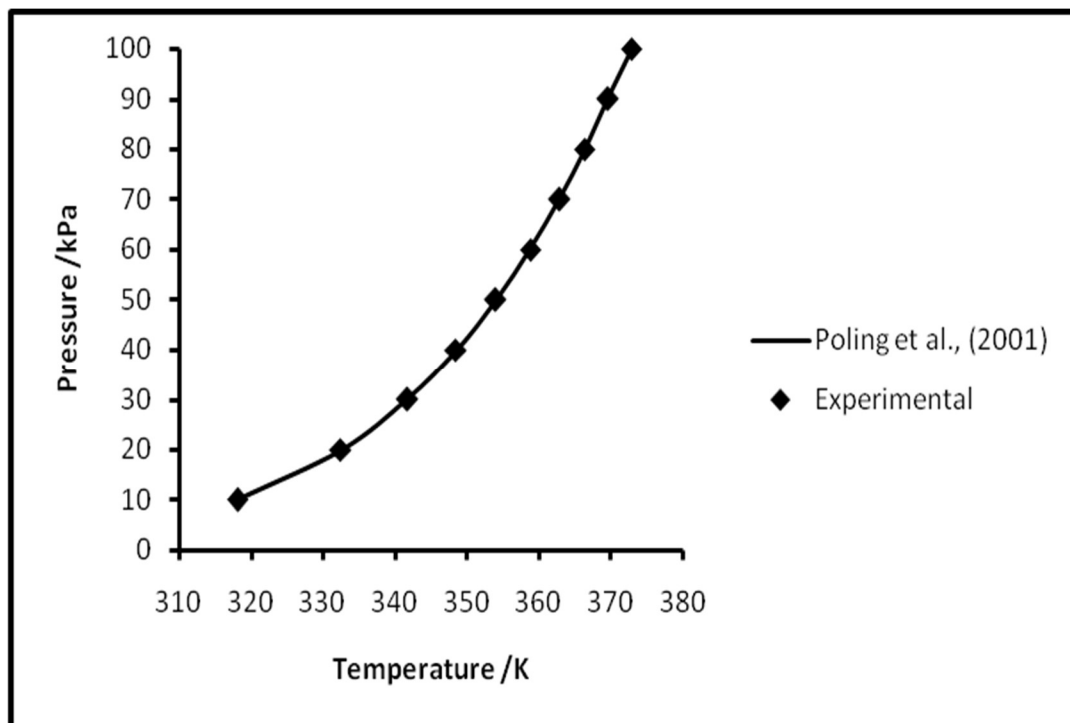
Figure 2.7: Vapour pressure data for water.

Table 2.3: Experimental VLE data for the ethanol (1) – water (2) system at 100 kPa (with no salt).

| T/K	x_1	y_1	T/K	x_1	y_1
372.97	0.000	0.000	355.13	0.337	0.590
368.64	0.024	0.241	354.88	0.374	0.606
367.09	0.051	0.339	354.08	0.417	0.624
364.32	0.071	0.386	353.95	0.489	0.624
363.35	0.088	0.413	353.10	0.564	0.645
361.57	0.109	0.457	352.67	0.605	0.666
359.89	0.126	0.478	352.17	0.649	0.709
358.98	0.152	0.506	351.98	0.712	0.742
358.13	0.182	0.522	351.28	0.789	0.785
357.47	0.209	0.530	351.18	0.832	0.831
357.07	0.232	0.536	351.12	0.870	0.872
356.66	0.256	0.545	351.12	1.000	1.000
355.95	0.288	0.556			
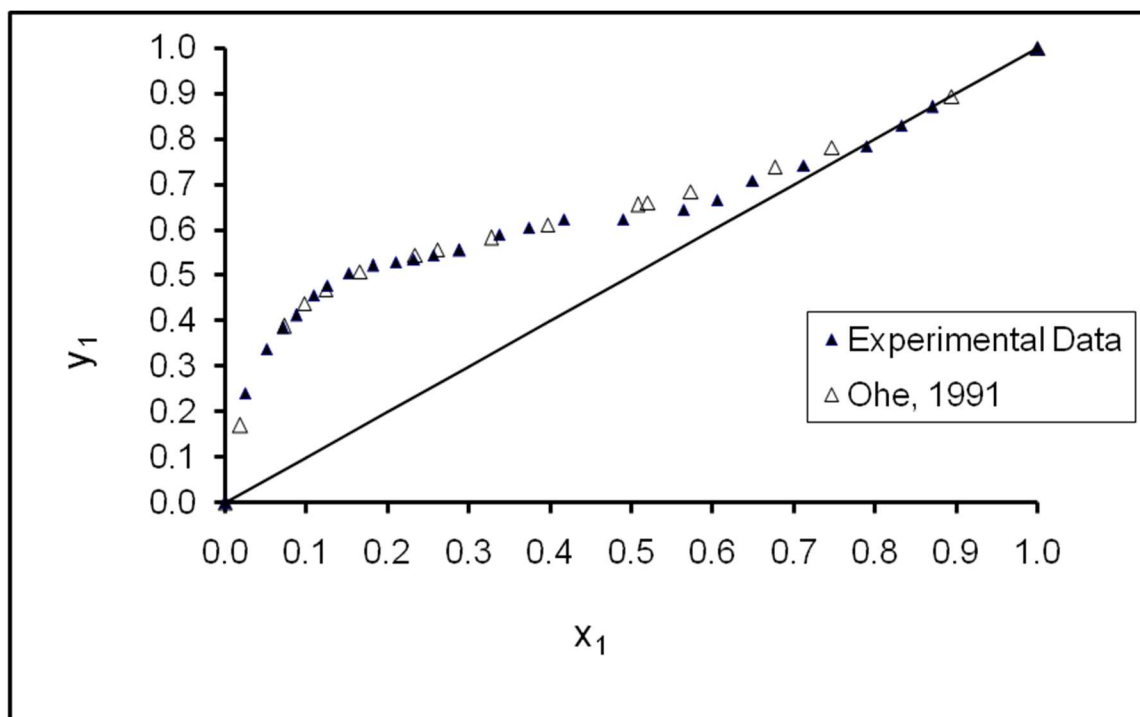
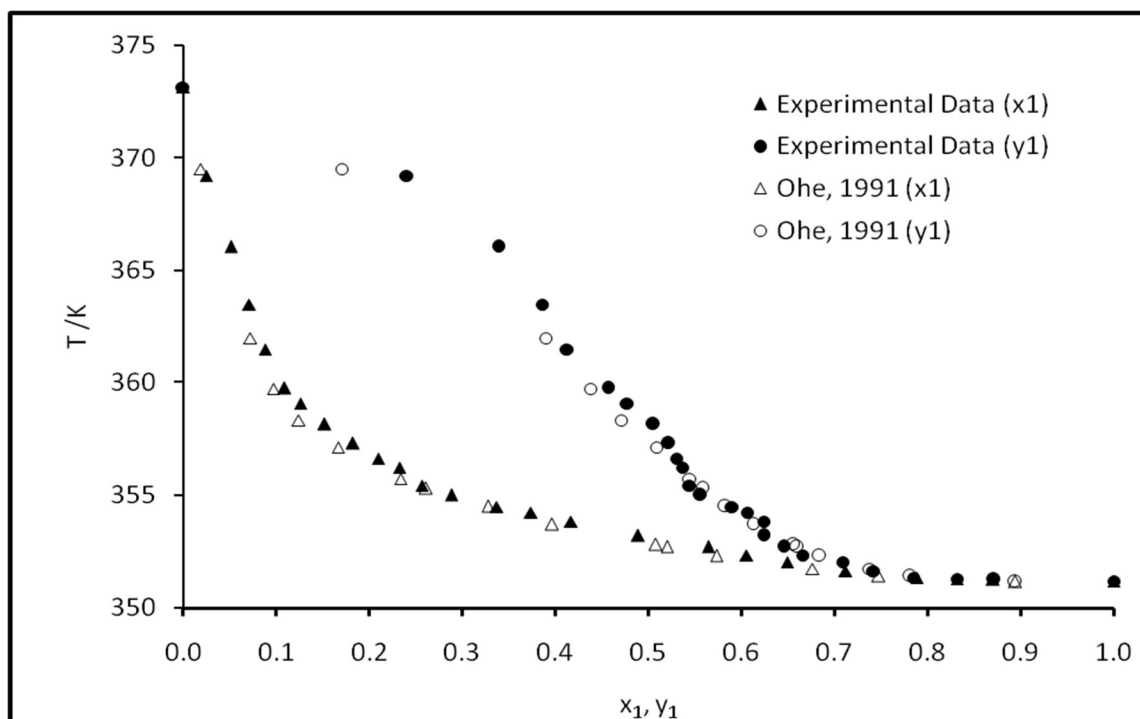Figure 2.8: Experimental data for the ethanol (1) – water (2) system at 100 kPa with no salt.

Figure 2.9: T-x-y diagram for ethanol (1) – water (2) system at 100 kPa with no salt.

CHAPTER 3

SIMULATION AND SOFTWARE WORK

In this chapter brief overview of the software and simulation working process is discussed. This chapter will lead to understand about the specific details about the software and minor thing about the simulation working technique which are going to use for the azeotropic distillation process.

3.1 PROCESS SIMULATION:

Process simulation is utilized for the configuration, advancement, investigation, and enhancement of specialized methodologies, for example, chemical plants, natural system, natural frameworks, power stations, complex assembling operations, organic methods, and similar kind of techniques.

3.2 ASPEN PLUS SOFTWARE:

For the simulation process we have used the software Aspen Tech's **ASPEN PLUS™ V 10** licensed version software. And all the simulation was performed in this ASPEN PLUS V 10. Given below in Fig 3.1 is the ASPEN PLUS software normal window. This ASPEN PLUS software permits you to make your own methodology model, beginning with the flow sheet, then indicating the chemical components and working conditions. ASPEN PLUSTM will take the majority of your details and, one click, **simulate the model**. The process simulation is the activity that executes every vital calculation expected to understand the result of the framework, thus anticipating its action. At the point when the computations are finished, ASPEN PLUSTM records the outcomes, stream by stream and unit by unit, so you can watch what happened to the synthetic types of your chemical process [5].

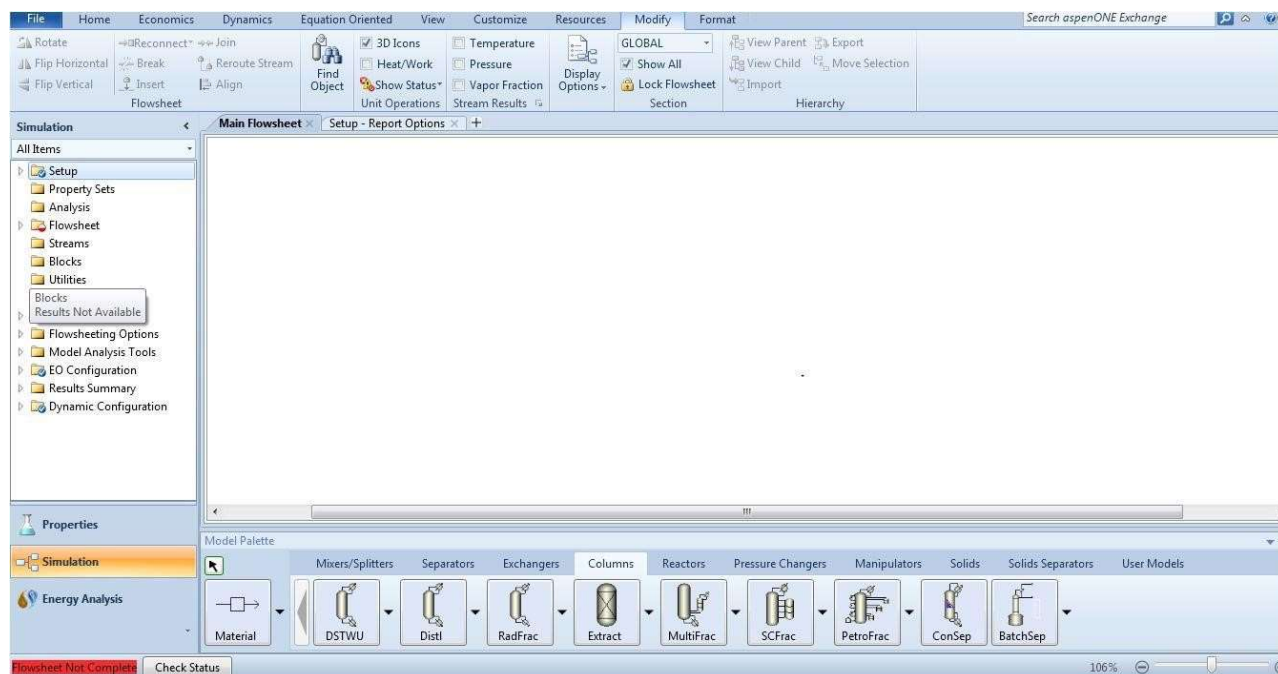Fig.3.1 ASPEN PLUS V 10 Normal window

3.3 DISTILLATION COLUMN MODEL USED:

The distillation column used were all are **RadFrac MODELS.** RadFrac model is widely used for the azeotropic distillation mixture. They are gives us to vary the process parameters like we can change the number of trays, reflux ratio, distillate rate easily. And from tray-to-tray variation of feed is possible in the RadFrac model.
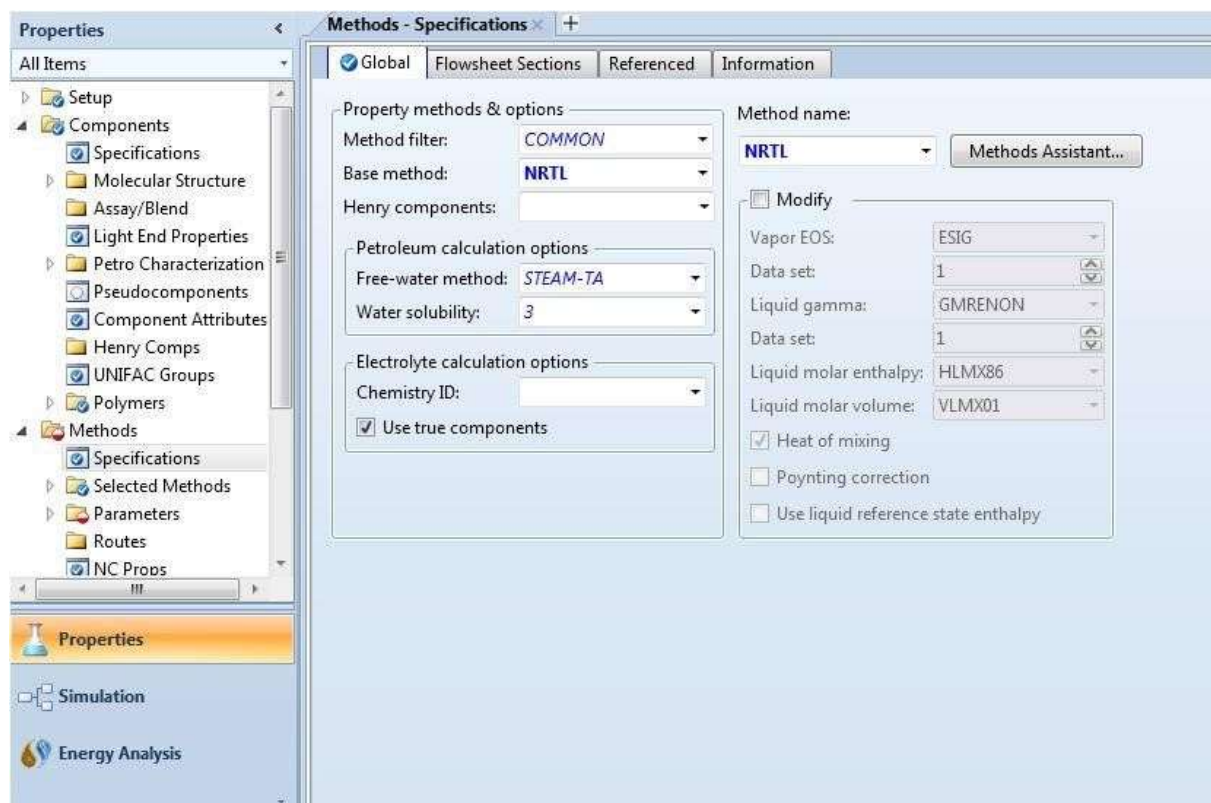
Fig.3.2 NRTL method input window

3.4 SIMULATION WORK:

We have successfully carried out the simulation work for the azeotropic distillation for the ethanol water azeotropic distillation system. For this simulation work we have taken the RadFrac column and process which is used in simulation was NRTL method, because NRTL method normally performs better for the azeotropic distillation system. Given above Fig. 3.2 represents NRTL method input window. Working pressure for the ethanol water azeotropic distillation simulation was taken 1 atm. And it was carried out for the 350ºK. After the simulation successfully completed steam result for the simulation process (given in Fig 3.2) was checked for the results and comparing the different results for the different working conditions of various process.

Fig.3.3 Successful status of simulation run and steam result display window

CHAPTER 4

RESULTS AND DISCUSSION

This chapter summarizes all the results. All simulation data for ethanol water distillation with and without entrainers to get high purity ethanol as product. Interesting observations are made and explained in detail. The results of different entrainers and behaviour of column various like reflux ratio, distillate rate and number of trays are plotted and explained in detail. The relative graphs and tables are plotted to explain process parameters. Comparisons of simulation data to get optimum data are explained.

4.1 Ethanol water azeotrope:
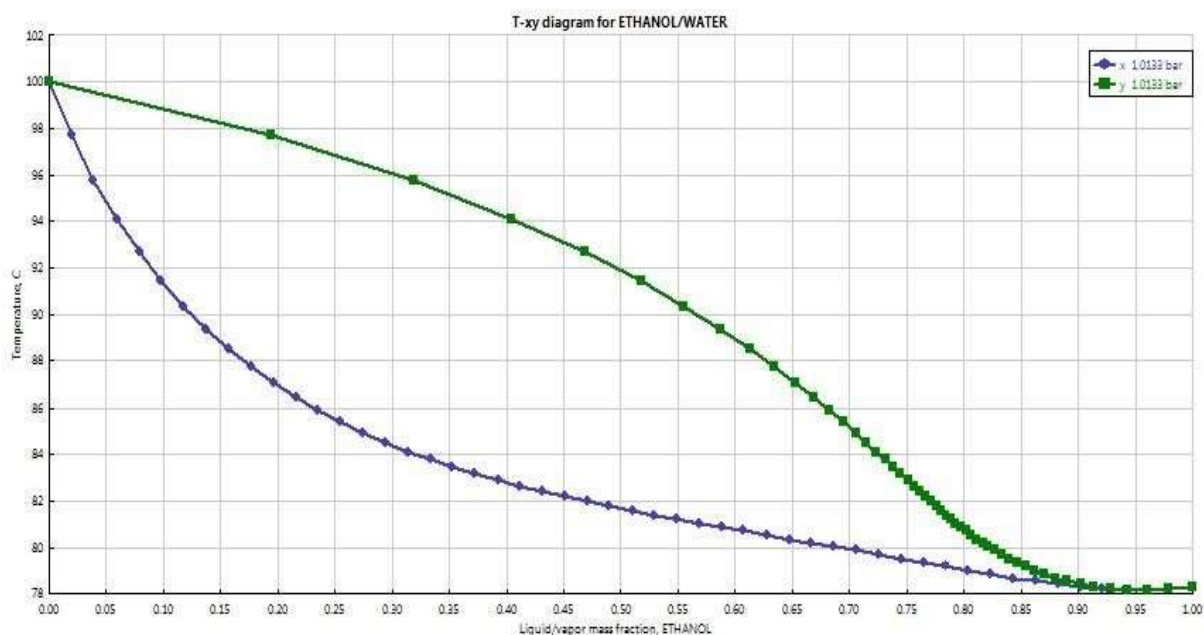
Fig.4.1 Txy diagram of ethanol water mixture
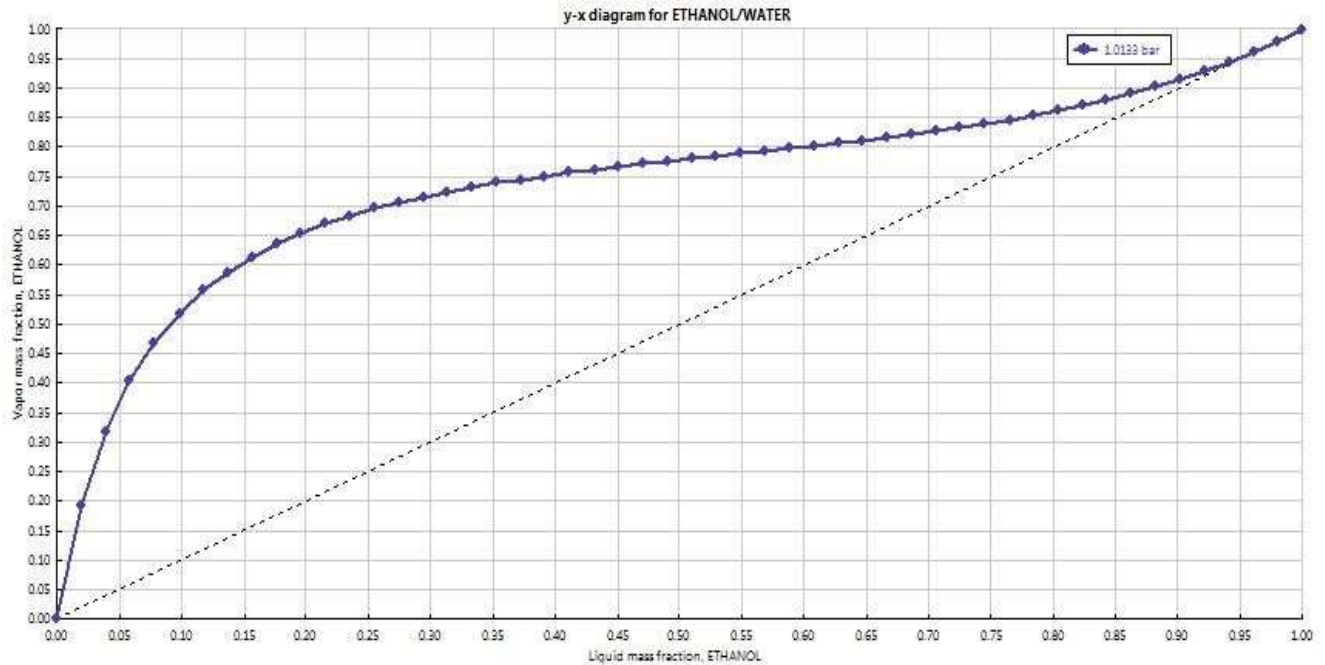
Fig.4.2   x-y curve/ concentration profile curve for ethanol water mixture

Fig.4.1 and Fig4.2 x-y curve for the ethanol water mixture give us the idea about the azeotropic point of the mixture.it is shown that at concentration of nearly 95% ethanol and 4%water it follows the azeotropic point beyond that point further separation is not possible by simple distillation process. The Txy diagram for the ethanol/water system at a pressure of 1 atm. The abscissa shows the mole fraction of ethanol; the ordinate, temperature. The lower curve is the

"Saturated liquid" line, which gives the mole fraction of ethanol in the liquid phase x. The upper curve is the "saturated vapor" line, which gives the mole fraction of ethanol in the vapor phase y. Drawing a horizontal line at some temperature and reading off the intersection of this line with the two curves give the compositions of the two phases.

4.2 Ethanol water without adding entrainer:
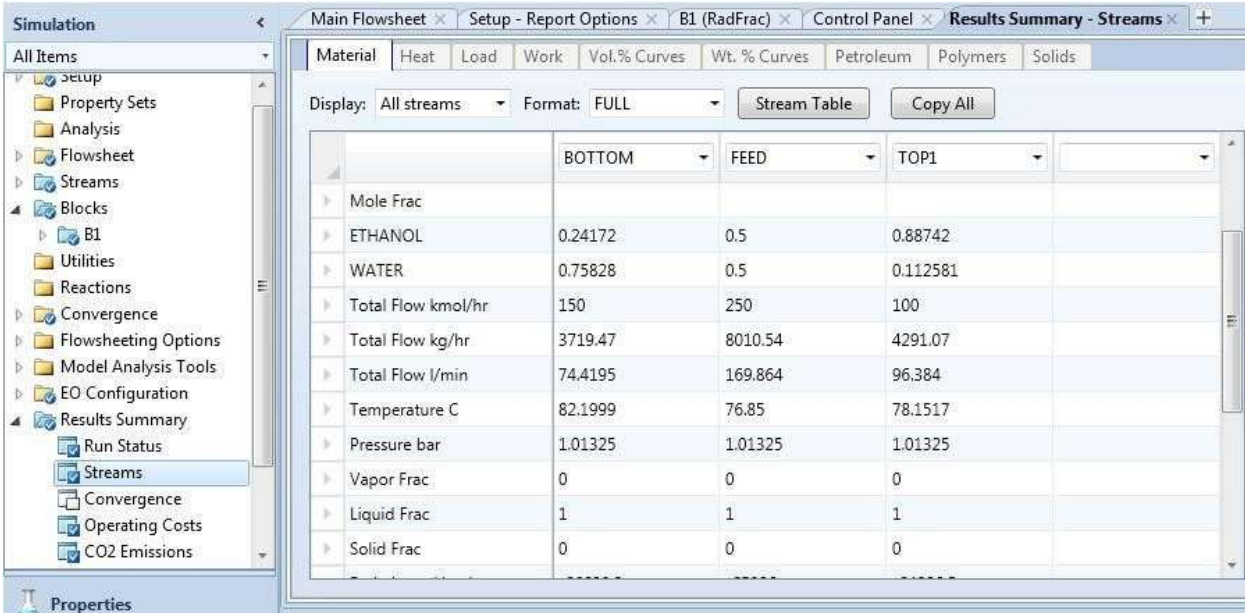
It is single column distillation, by simulation it is state that by normal distillation process ethanol and after mixture cannot get separated completely. In this simulation only up to 89% purity of ethanol is achieved, it is very important to get high purity of ethanol by further distillation with other technique i.e., azeotropic distillation using entrainer,

Details of simulation1:

Table: 4.1 Simulations detail without adding entrainer:

Parameters and conditions:	Value
Feed flow rate:	250 kmol/hr
Feed composition: ethanol	0.5 mole fraction
Water	0.5 mole fraction
Feed temperature	350 K
Pressure:	1atm
Number of trays	50
Feed location	35
Distillate rate	100 kmol/hr
Reflux ratio	10

Table: 4.2 Simulations result without adding entrainer:

	BOTTOM	FEED	TOP1	
Mole Frac				
ETHANOL	0.24172	0.5	0.88742	
WATER	0.75828	0.5	0.112581	
Total Flow kmol/hr	150	250	100	
Total Flow kg/hr	3719.47	8010.54	4291.07	
Total Flow l/min	74.4195	169.864	96.384	
Temperature C	82.1999	76.85	78.1517	
Pressure bar	1.01325	1.01325	1.01325	
Vapor Frac	0	0	0	
Liquid Frac	1	1	1	
Solid Frac	0	0	0	

4.3 Ethanol water distillation with entrainer:

Fig 4.3 shows the process diagram, now it is carried out by two column. In first column normal distillation of ethanol and water is performed to get around 84% ethanol and 16% water concentration. And then this mixture is feed to second column with entrainer to get high purity ethanol.

But, here we have taken only second column for study to simplify the process and to maily focus on second column where azeotropic distillation takes place. This is to get high purity ethanol, i.e feed inserted in this second column is 84% ethanol and 16% water and high concentration of benzene was added as entrainer and from bottom, pure ethanol is taken out as product, and from top mixture of ethanol water is taken out which can be used further.
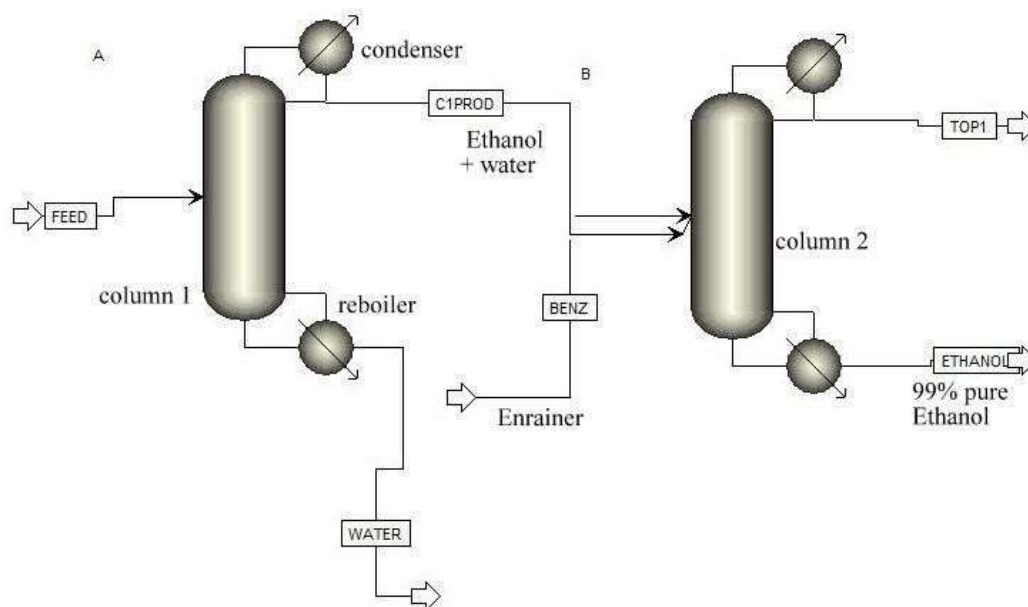
Fig.4.3 Two column ASPEN flow sheet for ethanol-water azeotropic distillation
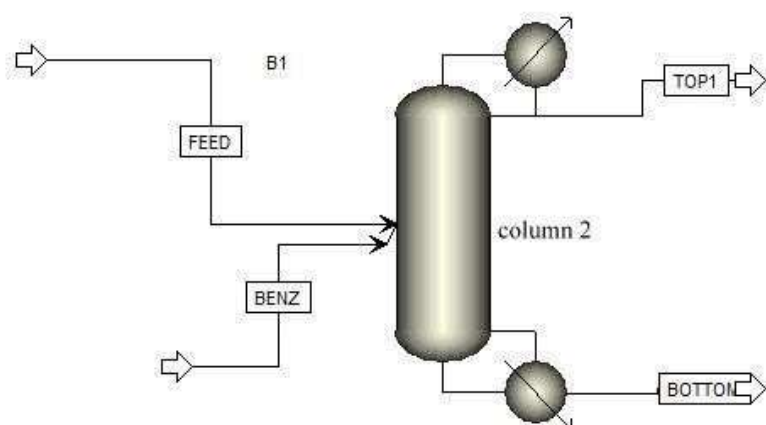
Fig.4.4 Simplified flow sheet of second column for azeotropic distillation

Details of simulation2:

Table 4.3 Details of simulation with benzene as entrainer:

Parameters and conditions:	Value
Feed flow rate:	250 kmol/hr
Feed composition: ethanol	0.84 mole fraction
Water	0.16 mole fraction
Feed temperature	350 K
Pressure:	1atm
Entrainer: Benzene	1.0 mole fraction
Entrainer flow rate	150 kmol/hr
Number of trays	45
Feed location	35
Benzene feed location	38
Distillate rate	380kmol/hr
Reflux ratio	0.5

Table: 4.4 Result of simulation 1: Steam table ethanol water distillation after addition of benzene as entrainer:

Heat and Material Balance Table					
Stream ID		BENZENE	BOTTOM	FEED	TOP1
From			B		B
To		B		B	
Phase		LIQUID	LIQUID	LIQUID	LIQUID
Substream: MIXED					
Mole Flow	kmol/hr				
ETHANOL		0.0	19.95452	210.0000	190.0455
WATER		0.0	.0454828	40.00000	39.95452
BENZENE		150.0000	2.6261E-17	0.0	150.0000
Mole Frac					
ETHANOL		0.0	.9977259	.8400000	.5001197
WATER		0.0	2.27414E-3	.1600000	.1051435
BENZENE		1.000000	1.3131E-18	0.0	.3947368
Total Flow	kmol/hr	150.0000	20.00000	250.0000	380.0000
Total Flow	kg/hr	11717.05	920.1048	10395.11	21192.05
Total Flow	l/min	238.7240	20.90353	231.7455	433.8823
Temperature	C	76.85000	78.30326	76.85000	64.61562
Pressure	bar	1.013250	1.013250	1.013250	1.013250
Vapor Frac		0.0	0.0	0.0	0.0
Liquid Frac		1.000000	1.000000	1.000000	1.000000
Solid Frac		0.0	0.0	0.0	0.0
Enthalpy	cal/mol	13527.88	-64596.92	-65054.73	-34219.26
Enthalpy	cal/gm	173.1820	-1404.121	-1564.551	-613.5941
Enthalpy	cal/sec	5.63662E+5	-3.5887E+5	-4.5177E+6	-3.6120E+6
Entropy	cal/mol-K	-54.87664	-77.53350	-70.47624	-64.00034
Entropy	cal/gm-K	-.7025232	-1.685319	-1.694937	-1.147606
Density	mol/cc	.0104723	.0159462	.0179794	.0145968
Density	gm/cc	.8180328	.7336120	.7475951	.8140476
Average MW		78.11364	46.00524	41.58044	55.76855
Liq Vol 60F	l/min	221.2728	19.36049	215.6378	417.5500

4.3.1 Effect of reflux ration on purity of ethanol:

For the purity reflux ratio play an important role. As ethanol is our bottom product it shows increasing purity of ethanol as the reflux ratio is decreasing continuously up to reflux ratio 0.5. At reflux ratio 0.5 it shows purity around 99.15% and further reduction in reflux ratio decreasing purity. As reflux ratio is decreased below 0.5 its purity goes on decreasing. Data at distillate rate 380kmol/hr and total number of trays are 45. The table 4.5 given below shows the comparison between Reflux ratio and the purity on addition of benzene.

Table 4.5: Reflux ratio vs purity with addition of benzene

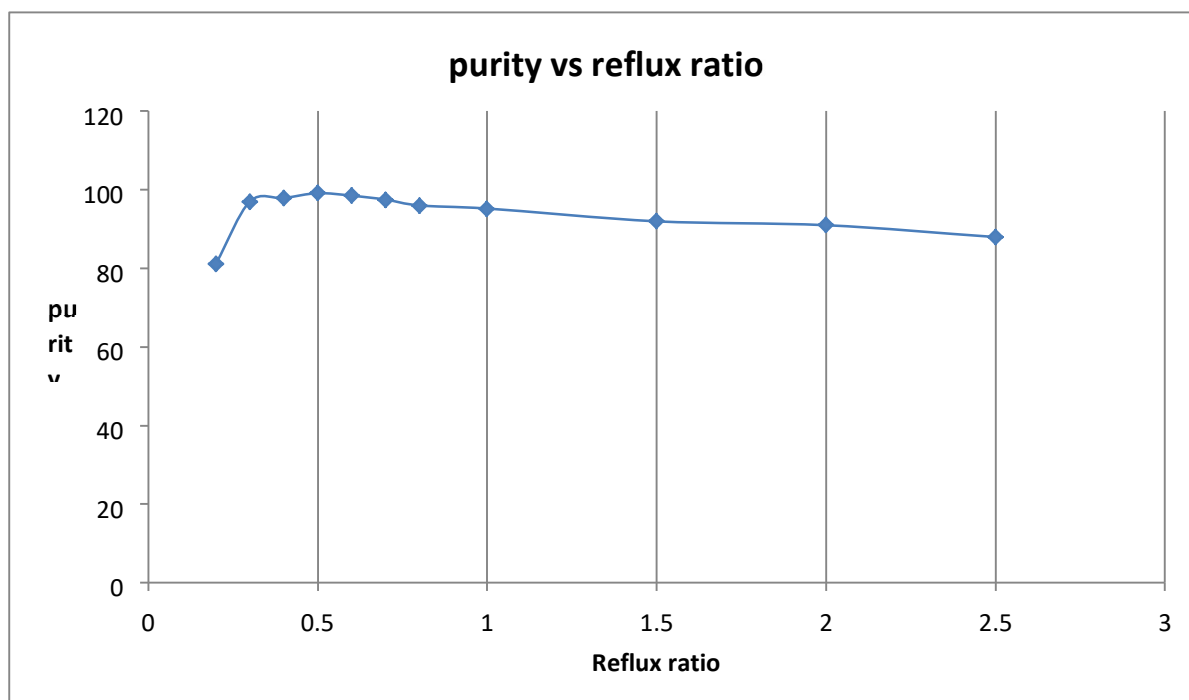| Reflux ratio	Purity of ethanol
0.2	81.2
0.3	97
0.4	97.9
0.5	99.77
0.6	98.5
0.7	97.5
0.8	96
1.0	95.2
1.5	92
2.0	91
2.5	88

Fig.4.5 Reflux ratio vs purity of ethanol

4.3.2 Effect of number of trays on purity of ethanol:

Total number of trays in distillation column is important factor in column properties. It determines the column height and diameter and total energy required for reboiler and condenser to condensate and heating. If number of trays are less then purity of the product will decrease and if there are large or infinite number of trays then purity will increase but reboiler heat duty will increase and effectively price will also increase. Hence trays to be keep at optimum level where beyond that purity will be nearly same. Hence here data was taken at reflux ratio 0.5 and distillate rate 350kmol/hr.

It is observed that above 42 trays 99% purity is achieved and then further increasing number of plates will increase very less purity. In graph i.e., in Fig 4.6 it is observed that above 45 trays graph is showing nearly straight line.

Table: 4.6 Number of trays vs purity of ethanol:

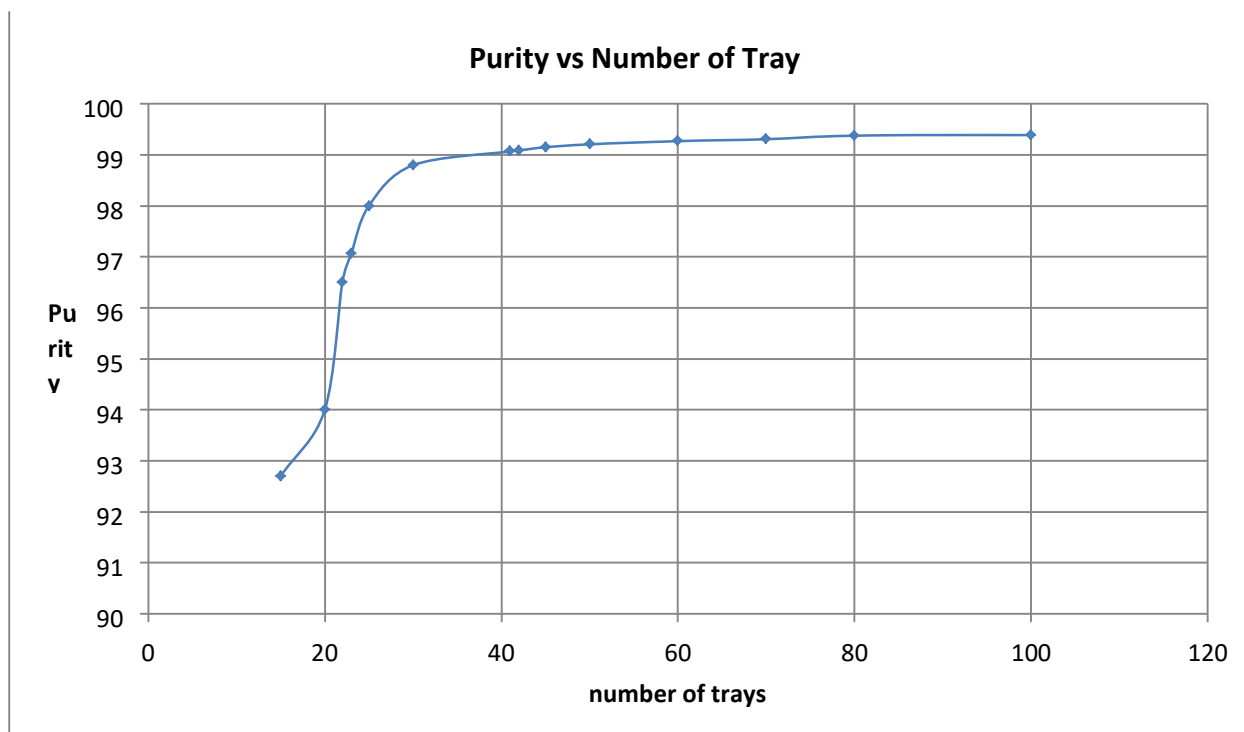| Number of trays	Purity of ethanol in %	Reboiler heat duty In x (10^6) cal/sec.
15	92.7	1.2256
20	94	1.2322
22	96.5	1.2325
23	97.07	1.2342
25	98	1.2349
30	98.8	1.23578
41	99.07	1.23529
42	99.09	1.23529
45	**99.15**	**1.23510**
50	99.21	1.2354
60	99.27	1.2368
70	99.31	1.238
80	99.38	1.2389
100	99.39	1.457

Fig.4.6 Number of trays vs purity of ethanol

4.3.3 Effect of distillate rate on purity of product (ethanol):

Distillate rate is also important parameter column for distillation. For ethanol water with benzene as entrainer it is shown in table 4.7 given below. As the distillate rate increases our product i.e., ethanol from bottom product purity increases. So, increasing distillate rate increases the purity of product of ethanol, but after certain value no more increase in purity for further increase in distillate rate. From graph it is shown here that the above 350kmol/hr purity becomes 99%. At 350kmol/hr 99.1% purity while at 380kmol/hr   99.77% purity and nearly straight line i.e., nearly same purity for further increase in distillate rate. Above 350kmol/hr purity become nearly same but reboiler heat duty increases i.e., more energy consumption.

Table 4.7 Distillate rate vs purity of ethanol for Benzene as entrainer:

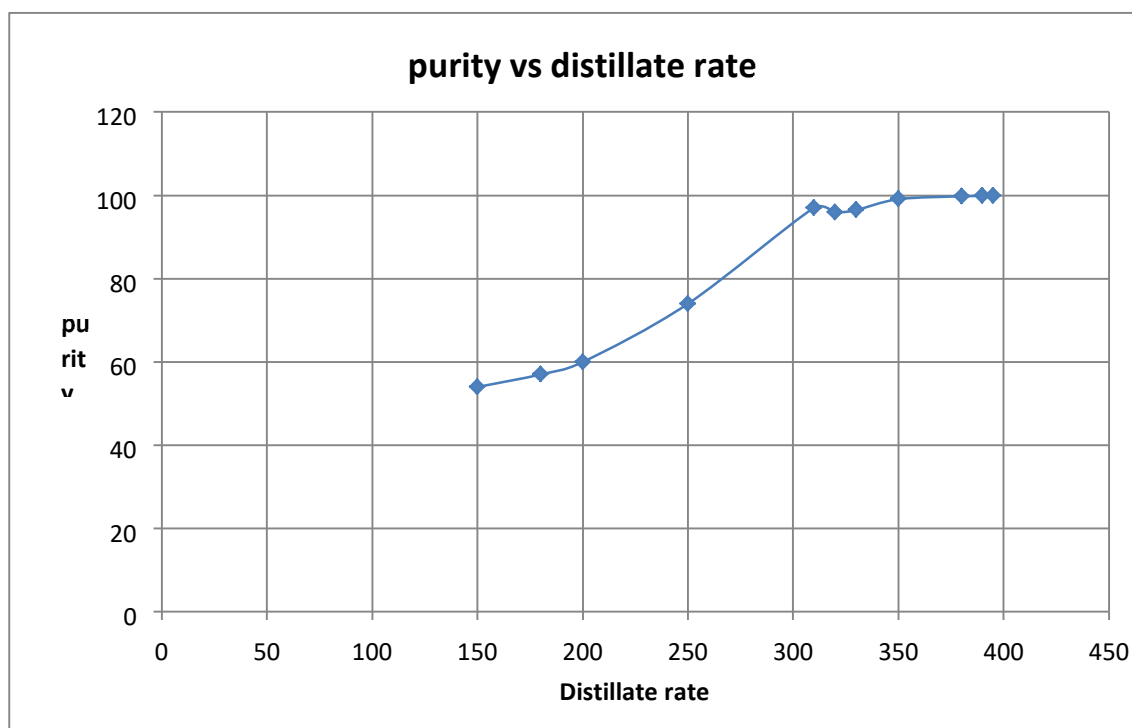| Distillate Rate	Purity
150	54
180	57
200	60
250	74
310	97
320	96
330	96.5
350	99.1
380	**99.77**
390	99.88
395	99.88

Fig 4.7 Purity vs distillate rate graph for benzene as entrainer

Entrainer2: N-PENTANE AS ENTRAINER

Details of simulation3:

Table 4.8 Simulation2 details N-pentane as entrainer:

Parameters and conditions:	Value
Feed flow rate:	250 kmol/hr
Feed composition: ethanol	0.84 mole fraction
Water	0.16 mole fraction
Feed temperature	350 K
Pressure:	1atm
Entrainer N-pentane	1.0 mole fraction
Entrainer flow rate	150 kmol/hr
Number of tray	25
Feed location	15
Benzene feed location	20
Distillate rate	360kmol/hr
Reflux ratio	0.15

Table 4.9 Distillation for N pentane as entrainer:

	BOTTOM	FEED	NPENTANE	TOP1	
Substream: MIXED					
Mole Flow kmol/hr					
ETHANOL	39.9364	210	0	170.064	
WATER	0.0509326	40	0	39.9491	
N-PEN-01	0.0126438	0	150	149.987	
Mole Frac					
ETHANOL	0.998411	0.84	0	0.472399	
WATER	0.00127332	0.16	0	0.11097	
N-PEN-01	0.000316097	0	1	0.416632	
Total Flow kmol/hr	40	250	150	360	
Total Flow kg/hr	1841.66	10395.1	10822.5	19376	
Total Flow l/min	41.8479	231.746	71799.1	473.203	
Temperature C	78.2864	76.85	76.85	48.5127	
Pressure bar	1.01325	1.01325	1.01325	1.01325	
Vapor Frac	0	0	1	0	
Liquid Frac	1	1	0	1	
Solid Frac	0	0	0	0	

Here for n-pentane as entrainer 99.84% purity of ethanol is achieved. When distillate rate is 360 kmol/hr. and reflux ratio is 0.15 and feed location is at 15th tray above the column. And for these 947825 cal/secs reboiler heat duty was required.

4.3.4 Effect of tray number, reflux ratio and effect of distillate rate:

Table: 4.10 Number of trays and purity for n-pentane as entrainer

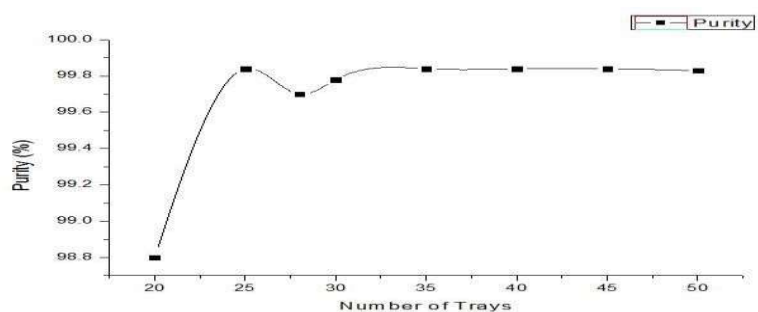| Number of Trays	Purity
20	98.8
25	99.84
28	99.7
30	99.78
35	99.84
40	99.839
45	99.84
50	99.83

Fig.4.8 Purity vs number of trays for n-pentane

Table: 4.11 Distillate rate and purity of n-pentane as entrainer

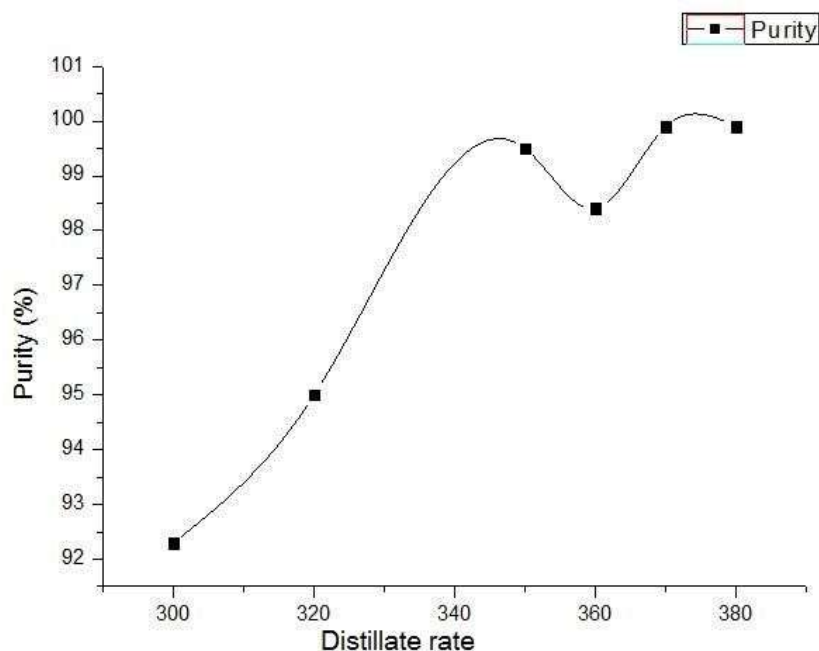| Distillate rate	Purity
300	92.30
320	95.00
350	99.50
360	98.40
370	99.90
380	99.90

Fig. 4.9 Purity vs distillate rate for n-pentane as entrainer

Table: 4.12 Reflux ratio and purity for n-pentane

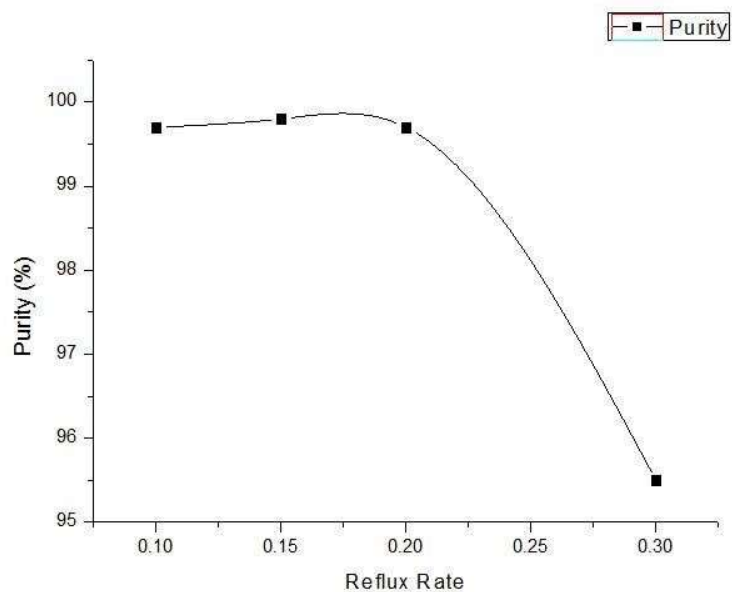| Reflux Rate	Purity
0.1	99.7
0.15	99.8
0.2	99.7
0.3	95.5

Fig. 4.10 Purity vs reflux ratio for n-pentane

Entrainer3: CYCLOHEXANE AS ENTRAINER:

Table 4.13 Simulation shows result for distillation for cyclohexane as entrainer:

Table: 4.14 Number of trays and purity for cyclohexane as entrainer:

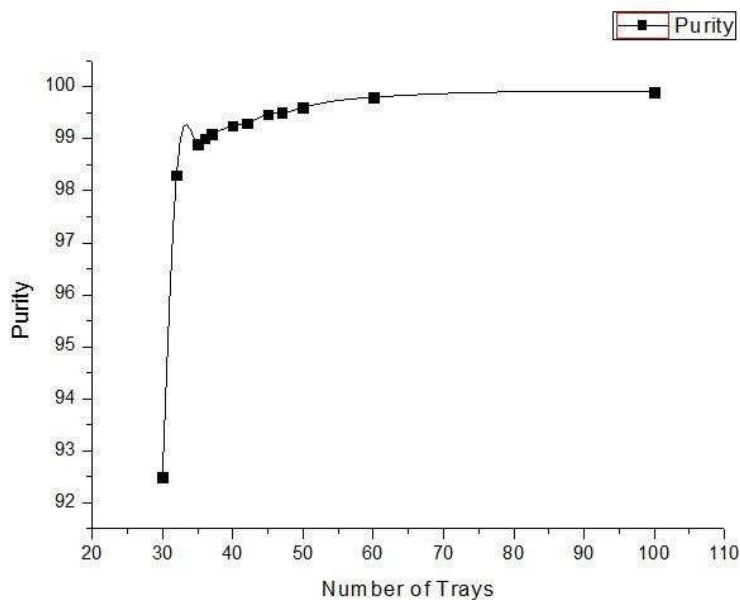| Number of Trays	Purity
30	92.50
32	98.30
35	98.90
36	99.01
37	99.09
40	99.25
42	99.30
45	99.91
47	99.91
50	99.92
60	99.92
100	99.92

Fig. 4.11 Purity vs number of trays for cyclohexane

Table: 4.15 Distillate rate and purity for cyclohexane as entrainer

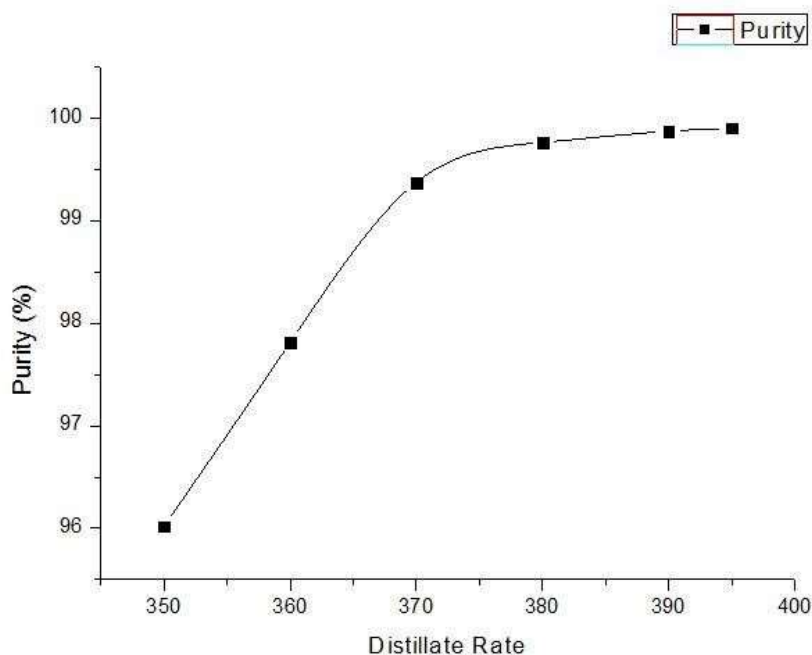| Distillate Rate	Purity
350	96.02
360	97.81
370	99.37
380	99.91
390	99.91
395	99.92

Fig. 4.12 Purity vs distillate rate for cyclohexane as entrainer

Table: 4.16 Reflux ratio and purity for cyclohexane as entrainer

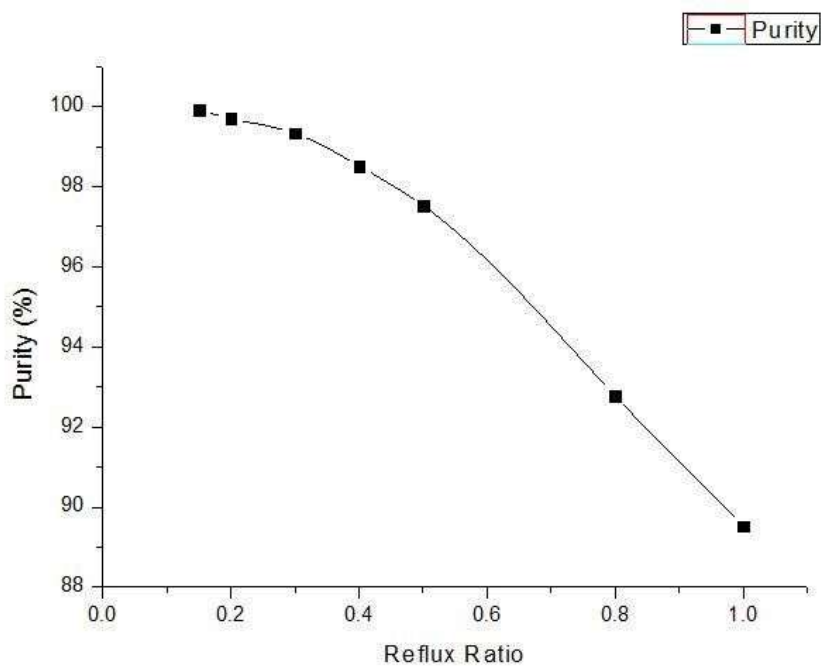| Reflux Ratio	Purity
0.15	99.91
0.2	99.70
0.3	99.33
0.4	98.52
0.5	97.53
0.8	92.78
1	89.52

Fig. 4.13 Purity vs reflux ratio for cyclohexane as entrainer

4.4 Optimization of Column Parameters:

4.4.1 Feed plate optimization:

For optimum feed plate location in most distillation columns the major working expense is reboiler heat duty. In the event that refrigeration was utilized as a part of the condenser, this heat reduction cost would likewise be expansive. For our propane/isobutane illustration, the pressure was intentionally set with the goal that cooling water could be utilized as a part of the condenser. Therefore, reboiler heat duty of the column should be kept low or minimum [10].

4.4.1.1   Benzene as Entrainer:

Table: 4.17 Optimum feed plate location for benzene as entrainer

Feed Location	Heat Duty MW
25	5.74611
28	5.74608
30	5.74602
35	5.74493
38	5.74271
40	5.74232
42	5.74270
43	5.74291

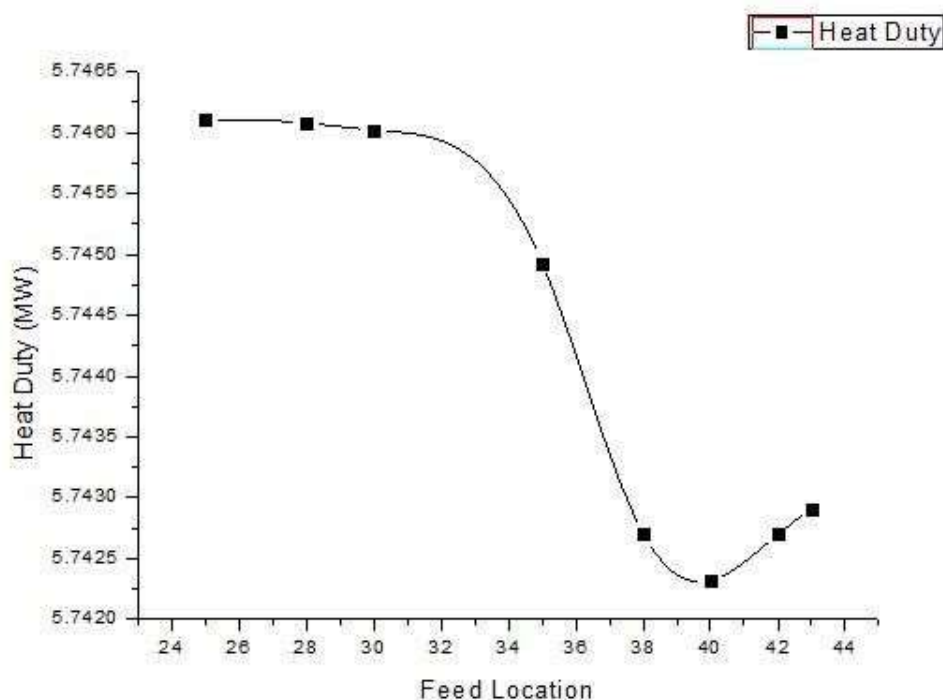Fig.4.14 Heat duty vs feed location for benzene

At feed location plate 40 it is showing minimum reboiler heat duty which is 5.74232 MW so at feed 40th tray is optimum feed tray location.

4.4.1.2   N-pentane as entrainer:

Table: 4.18 Optimum feed plate location for n-pentane as entrainer

Feed Location	Heat Duty MW
2	4.14855
5	4.14799
10	4.14515
12	4.14211
15	4.12991
17	4.12863
20	4.12924
22	4.13004
23	4.13072

Fig.4.15 Heat duty vs feed plate location for n-pentane as entrainer

At feed location 17th plate it is showing minimum reboiler heat duty which is 4.12863 MW so at 17th plate is optimum feed location.

4.4.1.3   Cyclohexane as entrainer:

Table: 4.19 Optimum feed plate location for cyclohexane as entrainer

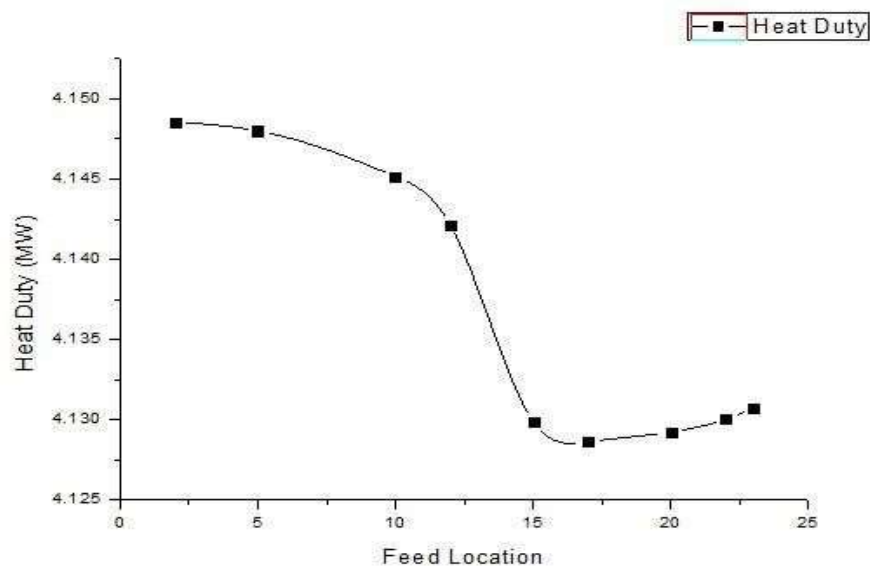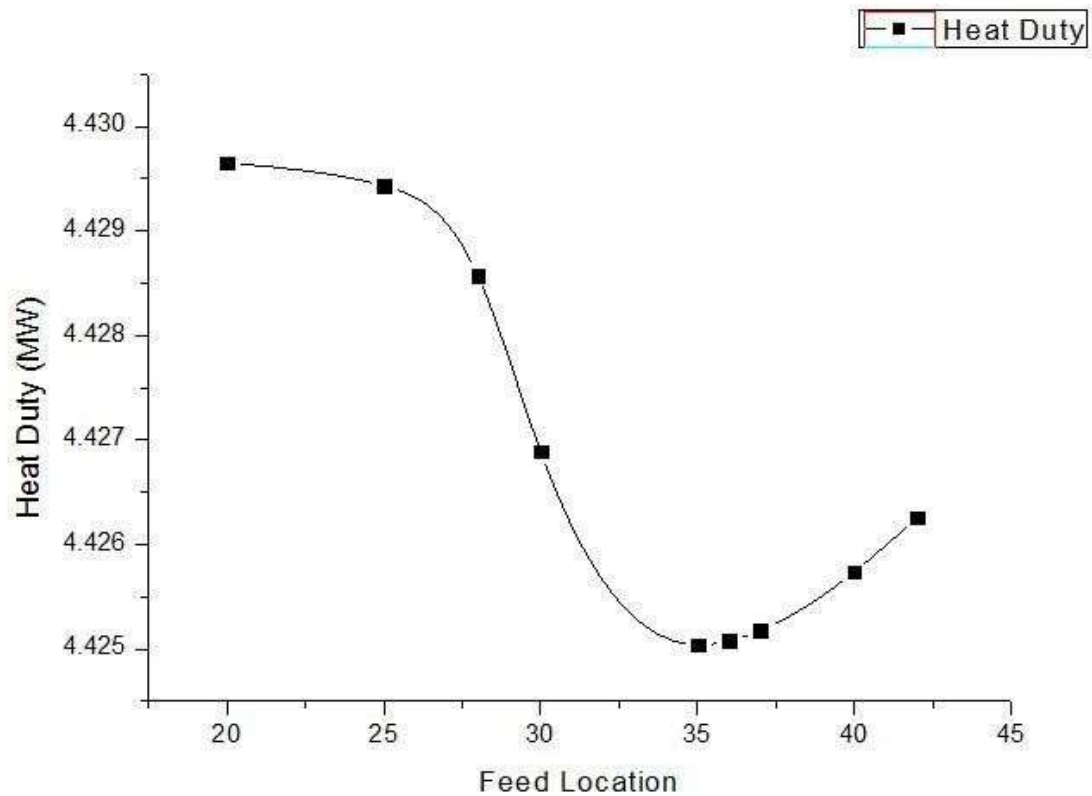| Feed Location	Heat Duty
20	4.42965
25	4.42943
28	4.42857
30	4.42689
35	4.42504
36	4.42508
37	4.42518
40	4.42574
42	4.42626

Fig.4.16 Heat duty vs feed location plate for n-pentane as entrainer

At feed location 35th plate it is showing minimum reboiler heat duty which is 4.42504 MW so at 35th plate is optimum feed location.

4.4.2 Column sizing:

Length:

Calculating the height of the column is very easy if we know the number of trays present in the column. The normal distance between adjacent trays (tray spacing) is 0.61 m (2 ft) [10]. If there are NT stages, the number of trays is NT -2 (one stage for the reflux drum and one for the reboiler).

$$L = 1.2(0.61)(N_T - 2)$$

4.4.2.1 Ethanol water with benzene as entrainer column:

Ethanol: 84%, water 16%mole fraction.

Reflux ratio: 0.5, distillate rate: 380, Number of stages: 45

Purity: 99.77

Length of column= 1.2 (0.61) (N_T -2)

Length of column= **31.476m** and diameter of column= **1.829m** (from ASPEN tray sizing result)

4.4.2.2 Ethanol water with cyclohexane as entrainer column:

Ethanol: 84%, water 16% mole fraction

Reflux ratio: 0.1, distillate rate = 380, number of stages= 45

Purity = 99.91%

Length of column=**37.476m** and column diameter= **1.764m**

4.4.2.3 Ethanol water with N-pentane as entrainer column:
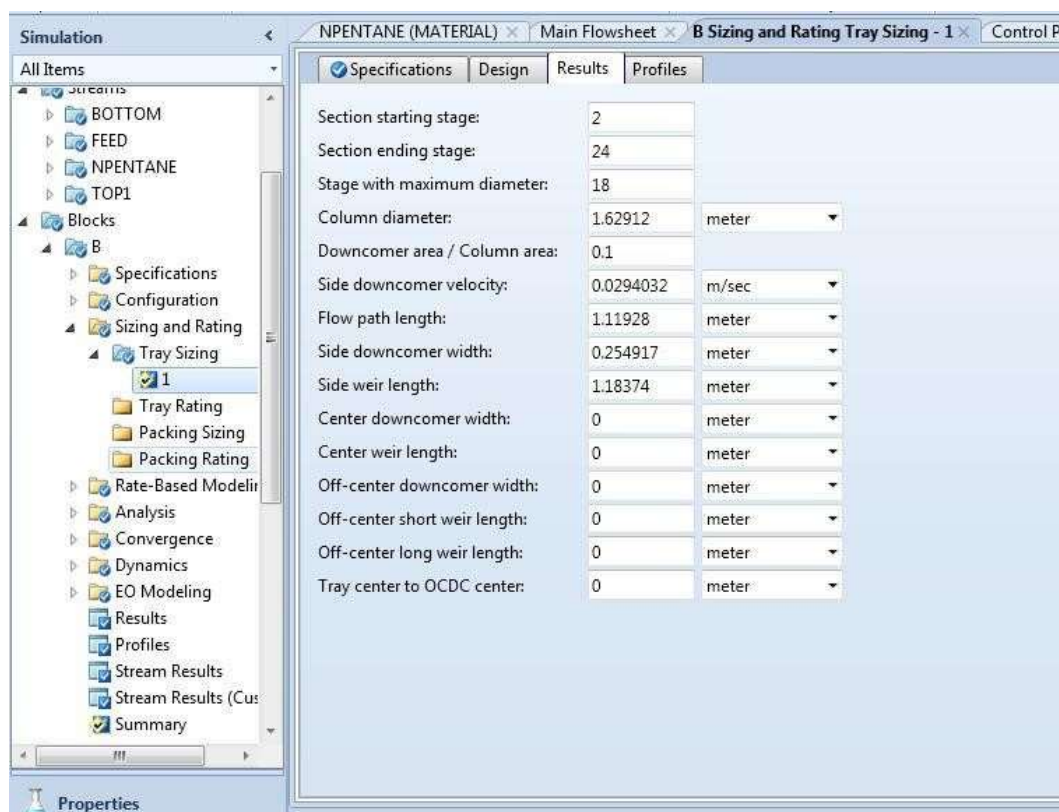
Ethanol: 84%, water 16%mole fraction.

Reflux ratio: 0.15, distillate rate: 360, Number of stages: 25

Purity: 99.84%

Length of column= 1.2 (0.61) (N_T -2)

Length of column= **16.36m** and diameter of column= **1.62m**

Table 4.20 Ethanol water with N-Pentane as entrainer column tray sizing

CHAPTER 6

CONCLUSIONS AND FUTURE WORKS

We have successfully simulated an azeotropic distillation system for separating ethanol water mixture using three different entrainers namely 1) benzene 2) n-pentane 3) cyclohexane it has been found out after simulation that cyclohexane performance is best where we get maximum purity of ethanol (99.91%) followed by n-pentane (99.84%) and benzene (99.77%) entrainer. In all the three cases after simulation we have found out the respective number of stages 25 for n-pentane, 45 for benzene and 45 for cyclohexane. This work is particularly important because alcohol water is abundantly form in various industrial important processes and their separation become critical and that's why understanding the distillation procedure become more and more crucial that's why azeotropic distillation is becoming important and in similar way extractive distillation and divided wall distillation are equally capable of separating azeotropic mixture so as in future work same work can be used in extractive distillation system as well as in divided wall distillation system as a future reference. As well as we have just carried out the steady state distillation system however for any industrial process dynamic distillation is more crucial because there would be sudden change in the system which needs to be controlled so controller mechanism has to put in to the place and that is the reason why dynamic simulation comes in to that place so as in extension of this project dynamic simulation can as well be done to find out controller parameters.

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