

# Simulation of Extractive Distillation Using CHEMCAD

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**Abstract:** *Isobutyl acetate forms the binary azeotrope with Isobutyl alcohol. Extractive distillation is the method of separating close boiling azeotrope in presence of some suitable solvent or the mixture of the solvents. In case of separation of Isobutyl acetate and isobutyl alcohol the solvents used can be n-butyl propionate, dimethyl acetamide, 1-hexanol, and Dimethyl formamide or dimethyl sulfoxide. The separation of Isobutyl Alcohol-Isobutyl Acetate azeotropic mixture, we have carried out the simulation by means of package using commercial software CHEMCAD. The simulation and economic evaluation for the extractive distillation using above solvents using CHEMCAD package shows that n-butyl propionate is the best solvent among these solvents. The Isobutyl alcohol-Isobutyl acetate mixture is also pressure sensitive. So, Vacuum distillation can also be used to recover pure components with a simple change in pressure. But the cost requires for the vacuum distillation for the same separation is comparatively large than the extractive distillation using n-butyl propionate as a solvent.*

**Keywords:** Alcohol, Azeotrope, Extractive distillation, CHEMCAD, Vacuum distillation

## 1. Introduction

Distillation is a method of separating mixtures based on differences in their volatilities in a boiling liquid mixture. But in case of the azeotropic components the separation by the process of simple distillation will not be beneficial. To separate the azeotropic components different distillation methods can be used such as, the extractive distillation, membrane distillation or Vacuum distillation.

For the separation of Isobutyl Alcohol-Isobutyl Acetate azeotropic mixture, we have carried out the simulation by means of package using commercial software CHEMCAD. We have studied, simulated and evaluated economically and concluded for the best technique suitable for the separation of IBA-IBAc azeotropic mixture.

Isobutyl acetate forms the binary azeotrope with Isobutyl alcohol. Extractive distillation is the method of separating close boiling azeotrope in presence of some suitable solvent or the mixture of the solvents. In case of separation of Isobutyl acetate and isobutyl alcohol the solvents used can be n-butyl propionate, dimethyl acetamide, 1-hexanol, and Dimethyl formamide or dimethyl sulfoxide. The simulation and economic evaluation for the extractive distillation using above solvents using CHEMCAD package shows that n-butyl propionate is the best solvent among these solvents.

The Isobutyl alcohol-Isobutyl acetate mixture is also pressure sensitive. So, Vacuum distillation can also be used to recover pure components with a simple change in pressure. But the cost requires for the vacuum distillation for the same separation is comparatively large than the extractive distillation using n-butyl propionate as a solvent.

## 2. Types of Distillation

There are several types of distillation depending on the procedure and the instrument setup. Each of the distillation type is used for the purification of compounds having

different properties. Following are the common types of distillation.

### A. Simple Distillation:

Simple distillation is practiced for a mixture in which the boiling point of the components differs by at least 70°C, or to separate liquids from in volatile solids or oils. For these cases, the vapor pressures of the components are usually sufficiently different that Raoult's law may be neglected due to the insignificant contribution of the less volatile component.

### B. Fractional Distillation:

Those mixtures, in which the volatility of the components is nearly similar or differs by 25°C (at 1 atmosphere pressure), cannot be separated by simple distillation. In such cases, fractional distillation is used whereby the constituents are separated by a fractionating column. In the fractionating column, the plates are arranged and the compound with the least boiling point is collected at the top while those with higher boiling point are present at the bottom. A series of compounds are separated simultaneously one after another. Fractional distillation is used for the alcohol purification and gasoline purification in petroleum refining industries.

### C. Steam Distillation:

Steam distillation is used for the purification of mixtures, in which the components are temperature or heat sensitive; for example, organic compounds. In the instrument setup, steam is introduced by heating water, which allows the compounds to boil at a lower temperature. This way, the temperature sensitive compounds are separated before decomposition. The vapors are collected and condensed in the same way as other distillation types. The resultant liquid consists of two phases, water and compound, which is then purified by using simple distillation. Steam distillation is practiced for the large scale separation of essential oils, perfumes.

## D. Vacuum Distillation:

Vacuum distillation is a special method of separating compounds at pressure lower than the standard atmospheric pressure. Under this condition, the compounds boil below their normal boiling temperature. Hence, vacuum distillation is best suited for separation of compounds with higher boiling points (more than 200°C), which tend to decompose at their boiling temperature. Vacuum distillation can be conducted without heating the mixture, as usually followed in other distillation types. For the separation of some aromatic compounds, vacuum distillation is used along with the steam distillation.

## E. Short Path Distillation:

Thermal sensitive compounds can also be separated by following short path distillation. In this technique, the separated compounds are condensed immediately without traveling the condenser. The condenser is configured in a vertical manner between the heating flask and the collecting flask. Similar to vacuum type, the pressure is maintained below the atmospheric pressure. Short path distillation is used for the separation of organic compounds with high molecular weight especially in the pharmaceutical industries.

## F. Azeotropic Distillation:

Interactions between the components of the solution create properties unique to the solution, as most processes entail nonideal mixtures, where Raoult's law does not hold. Such interactions can result in a constant-boiling azeotrope which behaves as if it were a pure compound. There exist some techniques to break the azeotrope to give a pure distillate. This set of techniques is known as azeotropic distillation.

### 3. Feed Prproperties

## A. ISOBUTANOL

## a. Chemical nature:

1. MOLECULAR FORMULA - C<sub>4</sub>H<sub>10</sub>O.
2. MOLARMASS - 74, 12 gm/mol.

## b. Properties:

Isobutanol is a clear, mobile, neutral liquid with a characteristic odour. It is miscible with all common solvents, e.g. alcohols, ketones, aldehydes, ethers, glycols and aromatic and aliphatic hydrocarbons. Its miscibility with water, however, is limited.

## c. Storage:

Isobutanol can be stored in suitable containers at temperatures below 40 °C and the exclusion of humidity for at least 1 year.

## d. Safety:

When using this product, the information and advice given in our Safety Data Sheet should be observed. Due attention should also be given to the precautions necessary for handling chemicals.

## e. Applications:

1. Solvent for printing Inks.
2. Extractant in the production of drugs and natural substances such as antibiotics, hormones, vitamins, alkaloids and camphor.
3. Additive in polishes and cleaners, e. g. floor cleaners and stain removers.
4. Solubilizer in the textile industry, e. g. additive in spinning baths or Carrier.
5. Used for colouring plastics.
6. Used as an Additive in de-icing fluids.

## B. Isobutyl Acetate

## a. Chemical Nature:

1. Molecular formula – C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>.
2. Molecular mass - 116 gm/mol.

## b. Safety:

## 1. Eye Contact:

Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

## 2. Skin Contact:

After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cold water may be used. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

## 3. Serious Skin Contact:

Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.

## 4. Inhalation:

Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

## 5. Ingestion:

Do not induce vomiting. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

## c. Storage:

Flammable materials should be stored in a separate safety storage cabinet or room. Keep away from heat. Keep away from sources of ignition. Keep container tightly closed. Keep in a cool, well ventilated place. Ground all equipment containing material. A refrigerated room would be preferable for materials with a flash point lower than 37.8°C (100°F).

#### d. Physical and Chemical Properties:

1. Physical state and appearance: Liquid.
2. Odor: Fruity. (Slight.)
3. Molecular Weight: 116.16 g/mole
4. Color: Colorless..
5. Boiling Point: 117.2°C (243°F)
6. Melting Point: -98.6°C (-145.5°F)
7. Critical Temperature: 296°C (564.8°F).
8. Vapor Pressure: 13 mm of Hg (@ 20°C)
9. Vapor Density: 4 (Air = 1)
10. Odor Threshold: 0.64 ppm

#### e. Applications:

Used as a solvent blend in applications like coatings, inks, adhesives, industrial cleaners.

#### C. Dimethyl Acetamide (DMA)

##### a. Physical and Chemical Properties:

1. PHYSICAL STATE: Clear, colorless liquid
2. MELTING POINT: -20 C
3. BOILING POINT: 164 - 166 C
4. SPECIFIC GRAVITY: 0.94
5. SOLUBILITY IN WATER: Miscible
6. VAPOR DENSITY: 3.0
7. NFPA RATINGS: Health: 2; Flammability: 2; Reactivity: 0
8. REFRACTIVE INDEX: 1.4380
9. FLASH POINT: 66 C
10. STABILITY: Stable under ordinary conditions
11. MF: C<sub>4</sub>H<sub>9</sub>NO

##### B. Applications:

1. Major use is to make other chemicals which can be used in various applications.
2. Peptide Synthesis;
3. Solvents and Mixtures for Peptide Synthesis;
4. Specialty Synthesis;
5. Amber Glass Bottles; Solvent Bottles;
6. Spectrophotometric Grade Solvents;
7. Spectrophotometric Grade Solvents;

## 4. Optimization

We begin the procedure by specifying some design variables and using the total reboiler heat duty as an objective function. Design variables are those whose values are set by market demands or physical conditions. In our study we have chosen flow rate, temperature, pressure and composition of binary feed as design variables. The binary feed is a mixture of isobutyl acetate

and isobutyl alcohol, containing 50% by mole isobutyl acetate and 40% by mole isobutyl alcohol. The temperature of the feed is 300°K and pressure is 101.325 kpa. We also specified distillate purity of extractive column and top & bottoms purity of solvent recovery column. The component from the extractive column is isobutyl alcohol from the top with 98.5% purity and the component from the top of the solvent recovery column is isobutyl acetate with 99% purity. The bottoms of the solvent recovery column consist of n- butyl propionate with 99.5% purity.

Once design variables have been specified, their values remain constant throughout the optimization procedure. On the contrary, optimization variables are those that must arbitrarily be assigned a value. The values are subject to change as we proceed from the base to the optimal design. Here the no of trays of both the extractive column and the solvent recovery column, the recycled solvent flow rate, reflux ratio and the vapors to bottoms molar flow rate are the design variables.

The optimization procedure using CHEMCAD requires setting the number of trays in both columns with the help of CHEMCAD, we did a case study analyzing the variation of stage number and reboiler heat duty (RHD) as a function of the recycle solvent flow rate and finally a graph is plotted between these 3 things for both the distillation columns and optimum point is found out.

The following table shows the design variables in the process.

	Stream	Variables	Specifications
		Extractive Column.	Feed stream
Pressure	1 atm		
Molar composition	0.50 – IBA 0.50– IBAC		
Solvent stream	Temperature		300 K
	Pressure		1 atm
	Molar composition		DMA
Distillate	Molar composition	0.9995 –IBA	
Solvent Recovery Column	Distillate	Molar composition	0.9995 IBAC
	Bottoms	Molar composition	>0.9995 DMA

## 5. A Case Study

The feed is a mixture consisting of 50% by mole isobutyl acetate and 50% by mole isobutyl alcohol. The feed flow rate is 100 kmol/hr, which accounts to 9597.7 kg/hr. The required composition of isobutyl alcohol is 99.95% by mole from the extraction column and the desired composition of isobutyl acetate is 99.95% by mole from the solvent recovery column.

### Procedure

Consider the separation of a mixture containing 50 mol% IBA and 50 mol% IBAC to treat 100 kmol/hr of the original mixture. This needs to be separated to produce

pure IBA & IBAC. Unfortunately, there is an azeotrope between IBA and IBAC which boils at 107.4°C and contain 65.7 mol% IBA and 34.3mol% IBAC. This azeotrope can be broken up by using suitable solvent. The process configuration is shown in Fig.2, in which the solvent is added at the top trays of the extractive column. As this solvent flows down, it alters the relative volatility of IBAC with respect to IBA and we get 99.95% IBA as overhead product of the Extractive Column (EC). The bottom component containing IBAC and solvent are removed and fed to the Solvent Recovery Column (SRC) where the solvent is separated from IBAC by convention distillation method. IBAC is obtained at the top of SRC having 99.99% purity and bottoms product containing solvent having purity of greater than 99.95%. The solvent is then recycled back to the extractive column and the procedure continues.

So, keeping the feed flow rate as constant, we have varied the recycle solvent flow rate from 150 kmol/hr to 350 kmol/hr. this is carried out in five different parts.

1. The flow rate of the solvent is kept at 150 kmol/hr and desired composition of IBA and IBAC is obtained is found out.
2. The flow rate of the solvent is kept at 200 kmol/hr and desired composition of IBA and IBAC is obtained is found out.
3. The flow rate of the solvent is kept at 250 kmol/hr and desired composition of IBA and IBAC is obtained is found out
4. The flow rate of the solvent is kept at 300 kmol/hr and desired composition of IBA and IBAC is obtained is found out
5. The flow rate of the solvent is kept at 350 kmol/hr and desired composition of IBA and IBAC is obtained is found out

Then finally a graph is plotted between the reboiler heat duties, recycle solvent flow rate and number of plates for both the columns, and the optimum conditions are found out.

## 6. Result and discussion

### D. Isobutanol and Isobutyl Acetate

XY data for Isobutyl Alcohol / Isobutyl Acetate;

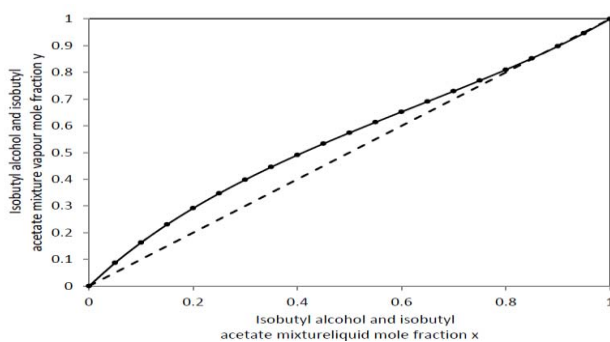


Figure 1

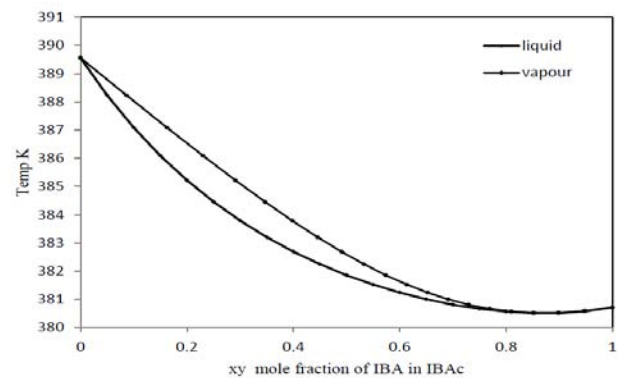


Figure 2

### E. Isobutanol and Dimethyl Acetamide

XY data for Isobutyl Alcohol / Dimethyl Acetamide;

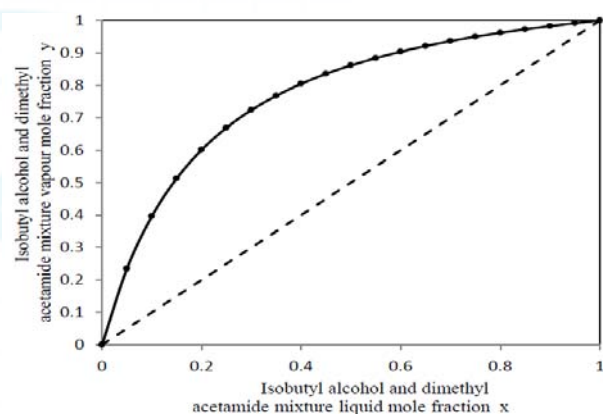


Figure 3

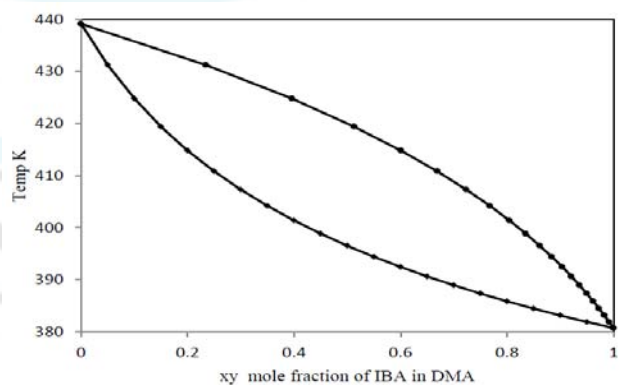


Figure 4

### F. Isobutyl Acetate and Dimethyl Acetamide

XY data for Isobutyl Acetate/ Dimethyl Acetamide;

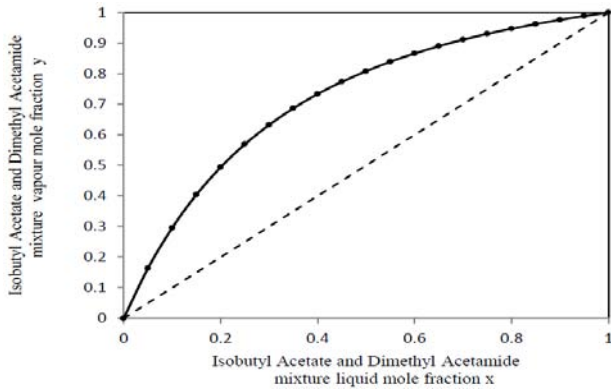


Figure 5

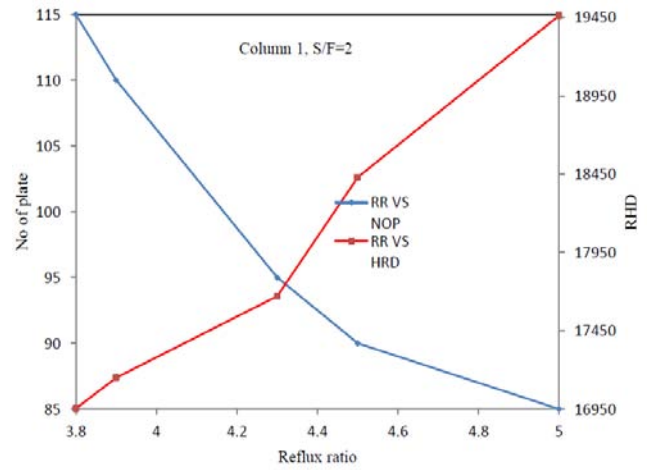


Figure 9

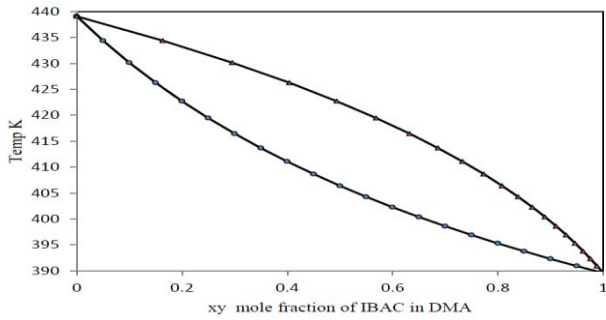


Figure 6

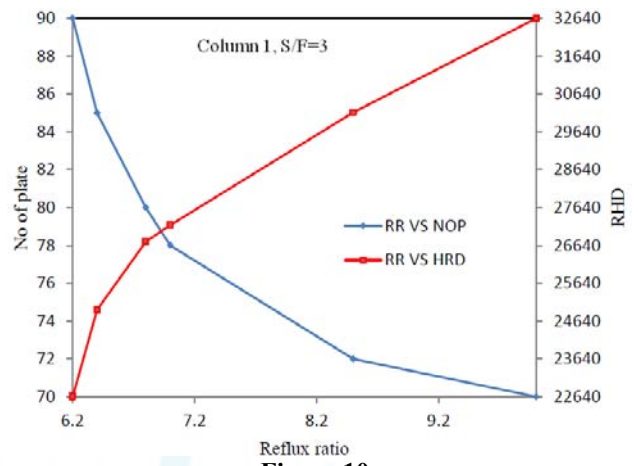


Figure 10

G. Reflux Ratio vs Number of Plate and Reflux Ratio vs Reboiler Heat Duty

1. For Extraction Column

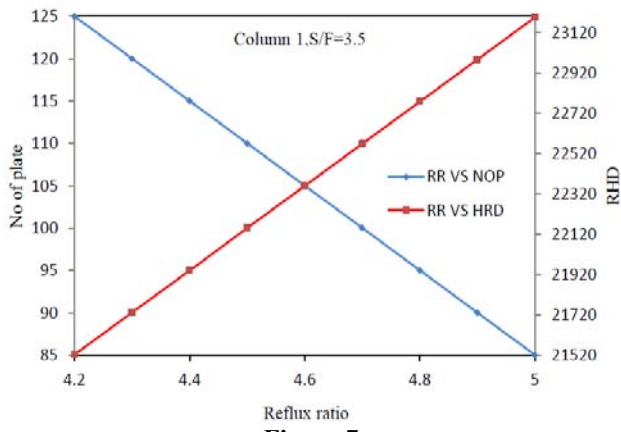


Figure 7

2. For Solvent Recovery Column

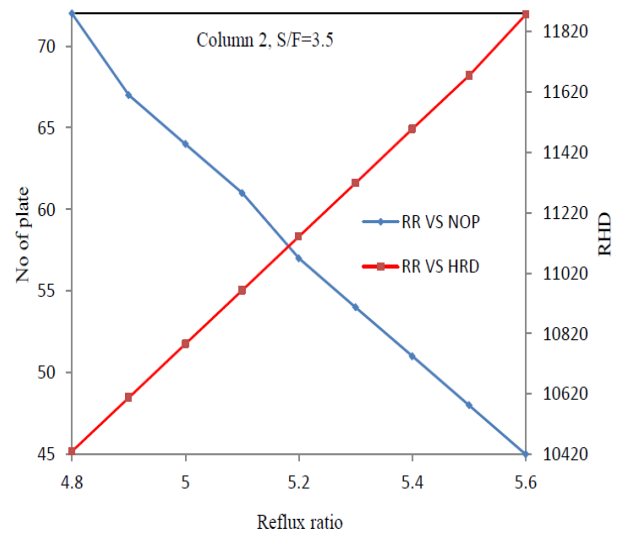


Figure 11

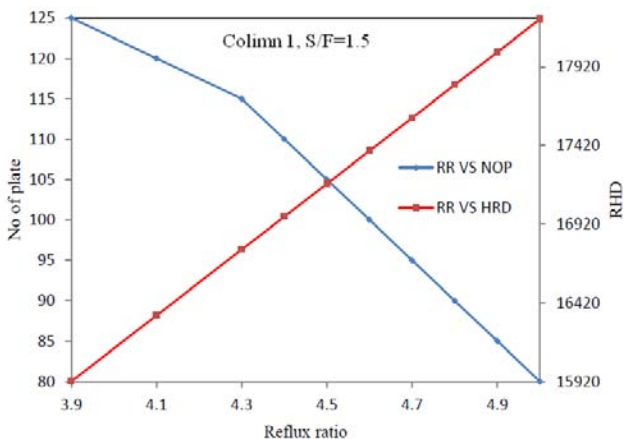


Figure 8

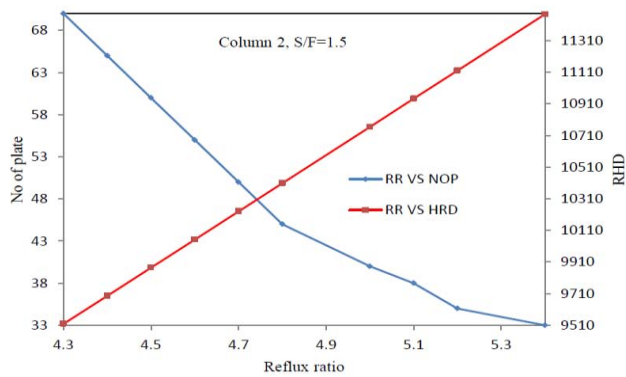


Figure 12

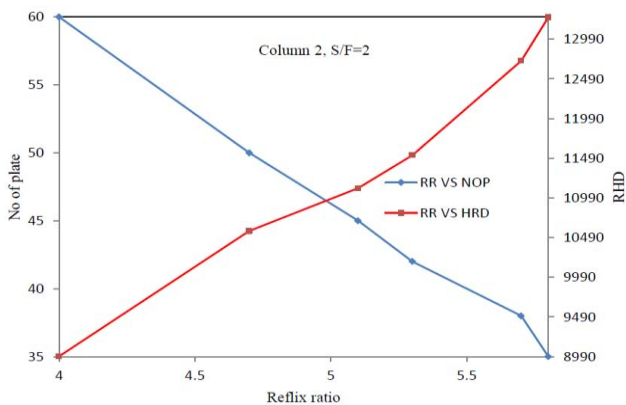


Figure 13

## 7. Conclusion

For the separation of IBA and IBAC we have studied simulated extractive distillation separation method.

From the simulated results, we can calculate the optimum conditions for separation of IBA and IBAC

1. Optimum solvent flow rate = 150 kmol/hr.

2. for extractive column,

a. Reboiler heat duty = 15713.06 MJ/hr.

b. Number of plates = 125.

3. for solvent recovery column,

a. Reboiler heat duty = 10408 MJ/hr.

b. Number of plates = 40.

From the above data we can also conclude that as the recycle flow rate increases, the reboiler duty increases and so is the condenser duty but the number of plate's decreases.

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