

# MCPI Private Limited Haldia

**Project Report on** 

# **Distillation Analysis & Plant Overview**

**Duration: 17/05/2022 - 15/06/2022** 

**Prepared By** 

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3<sup>rd</sup> year (Bachelor of Technology)

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### **DECLARATION**

I declare that this written submission of project represents the processes and techniques used by MCPI Private limited (formerly MCC PTA India Corp. Private Limited and Materials Chemicals and Performance Intermediaries Private Limited). In my words and where philosophy related to these that have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in my submission. I understand that any violation of the above will be cause for disciplinary action by the Institute and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been taken when needed.

Name of the student:

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Date: 13:06:2022

Signature of student: Rajneesh Anand

## Acknowledgement

It's a great and pleasant opportunity for me to gain experience as well as knowledge in the industry. The time period that I have given at MCPI has truly motivated me and has helped me a lot by giving me the opportunity to develop both academically and professionally.

I would like to thank Mr. Ambar Baran Bhowmik (Vice President Production & Planning) for his guidance through the MCPI plant; as well his explanations of specific fields were truly helpful.

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# APPROVAL SHEET

THIS PROJECT REPORT ENTITLED "

# AZEOTROPIC DISTILATTION:

THEORITICAL STAGE CALCULATION BY GRAPHICAL METHOD AND VERIFICATION BY FUG EQUATION.

## BINARY DISTILLATION:

THEORITICAL STAGE CALCULATION BY GRAPHICAL METHOD AND VERIFICATION BY FUG EUATION.

BY 'MR. RAJNEESH ANAND' IS APPROVED BY-

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HR & A (s)

Signature & Date:

Name: Aronab Kanto Gin

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### **CERTIFICATE**

This is to certify that the work contained in the project report titled 'Distillation (Binary and Azeotropic )Optimisation' has been carried out by Rajneesh Anand under my supervision and can be presented in his institute for academic purposes.

Signature of Supervisor(s) 15. 6.22

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MCPI, Haldia

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### **Overview**

MCPI Private Limited (previously MCC PTA India Corp. Private Limited and Materials Chemicals and Performance Intermediaries Private Limited) was laid out in the year 1997, committed to the production and distribution of PTA.

Initially a partner of the Mitsubishi Chemical Corporation (MCC), MCC offered its larger part value to The Chatterjee Group in November 2016. TCG as of now holds a 99.4% stake in MCPI, notwithstanding a few different organizations inside the group. Different organizations where they are the significant partners (through its group organization MCPI Holdings Ltd. Mauritius) incorporate Haldia Petrochemicals Ltd., TCG Real Estate, TCG Life sciences, and so on.

Since the interest for polyester items filled in the mid 2000s, MCPI participated in an extension project in 2006, where a 800 000 ton PTA plant was built to work with expanded creation. Both the old and the recently extended plants are partitioned into two free stages, which are situated in a stretch of land evaluating to 324 acres of land, 104 of which is a green belt. The plant is constructed and works sticking to the principles set both in India, as well as universally. It additionally consents to somewhere safe orders and ecological regulations. The unrefined substance, Para-xylene, is provided by a 13 km pipeline, which starts at Haldia Port.



### **Product**

Purified Terephthalic Acid (PTA) is the main product produced by MCPI.

PTA: Molecular weight 166.13.

PTA is used in the manufacture of polyester PET, plastic bottles and clothing.

## Application of PTA:

- Terephthalic acid is used in paint as a carrier.
- Terephthalic acid is used as a raw material to make terephthalate plasticizers such as dioctyl terephthalate and dibutyl terephthalate.
- It is used in the pharmaceutical industry as a raw material for certain drugs.
- In addition to these end uses, Terephthalic acid based polyesters and polyamides are also used in hot melt adhesives.
- PTA is an important raw material for lower molecular weight saturated polyesters for powder and water-soluble coatings.
- In the research laboratory, terephthalic acid has been popularized as a component for the synthesis of metal-organic frameworks.
- The analgesic drug oxycodone occasionally comes as a terephthalate salt; however, the more usual salt of oxycodone is the hydrochloride. Pharmacologically, one milligram of *terephthalas oxycodonae* is equivalent to 1.13 mg of *hydrochloridum oxycodonae*.
- Terephthalic acid is used as a filler in some military smoke grenades, most notably the American M83 smoke grenade and M90 vehicle-employed smoke grenade, producing a thick white smoke that acts as an obscurant in the visual and near-infrared spectrum when burned.

## **Safety**

## "GO ANZEN NI!"

### **5S Principles**

- Sort (**Seiri**): All unneeded items, tools, parts and supplies are removed from the area.
- Systematic Arrangements (**Seiton**): A place where everything and anything is its proper designated area.
- Shine (Seisou): The area is clean as the work is performed.
- Standardize (Seiketsu): Cleaning and identification methods are consistently applied.
- Sustain/Self-discipline (**Shitsuke**): 5S is a habit that is continually improved.

## **Necessity of 5S**

- To secure the workplace and safe passage to make sure that there are no obstacles.
- To prevent everyone from any unsafe condition on site.
- To find the sign of plant abnormality at early stages.

## Non-violable safety rules of MCPI

- 1. **Prohibited Activities:** Smoking, Chewing of tobacco & carrying lighter/match box is strictly prohibited inside Factory Premises except designated places.
- 2. **Entry to Plant:** Unauthorized entry inside plant battery limit is strictly prohibited.

- 3. **Vehicle Safety:** Maintain speed limit up to 20 km/hr and use of spark arrester is mandatory in vehicles inside plant battery limit.
- 4. **Information Reporting**: All accidents/incidents must be reported to plant management.
- 5. **Communication:** Mobile Phones must be kept in switched off condition inside plant battery limit.
- 6. **General PPE:** Use of helmet, Safety shoe, Full sleeve dress & Safety spectacles inside plant battery limit is mandatory.
- 7. **Height Work:** Full body harness & proper access (ladder etc.) is mandatory.
- 8. **Confined space work**: Spotter, Gas testing and full body harness are mandatory.
- 9. **Hot Work**: Isolation of combustible/inflammable materials are mandatory and hot work to be confined.

### **Importance of Safety in Industries**

- Human suffering can be reduced if proper precautions and safety measures are undertaken.
- Accidents, undesired events giving rise to death, ill health, injury, and damage can be reduced with proper safety.
- Unsafe acts (not wearing proper PPE etc.) and unsafe conditions (slippery floor, chemicals spill, poor condition of electrical equipment etc.) must be avoided at all cost.
- Assembly Points- during any emergency occurring in the plant all personnel should follow standard emergency procedure. All employees are supposed to reach the nearest assembly point for their own safety.

## Type of hazards

- Chemical hazards- fumes and smoke, mists/aerosols, liquids etc.
- Mechanical hazards- unguarded/inadequate moving parts, defective tools.
- Physical hazards- combustible liquids, flammable fluids, oxidizer etc.

• Health hazards- toxicity, corrosiveness, carcinogen, reproductive toxins etc.

### **Corrosive Chemicals**

- Contact may irritate or burn the skin, eyes and respiratory tract.
- The Severity of the damage caused by exposure of corrosive chemicals depends on duration of exposure, types of corrosive chemicals, concentration, contact location, time of first aid.
- Most injury accidents with corrosive chemicals involve skin contact and thereby tissue burning.

### **Material Safety Data Sheet (MSDS)**

- Provides detailed information on chemical properties, hazards and protective measures
- Required for all hazardous chemicals (flammable, toxic, corrosive, reactive and explosive)
- Must be easily available to employees

### **MSDS** Contents

- Product identity
- Physical and chemical characteristic
- Physical and health hazards of the chemicals
- Exposure limits
- Carcinogenic effect of the chemicals
- Precautions for safe handling and use
- Applicable control measures including PPE and first aid procedure

## **Equipment:**

There are some equipment which are to be seen in every industry.

### 4.1. Pumps

Any Fluid Machine that adds energy to a fluid known as Pump. The purpose of a pump is to add energy to fluid, resulting in an increase in fluid pressure, not necessarily an increase of fluid speed across the pump.

A hydraulic machine which converts mechanical energy into hydraulic energy.

It works on the principle of forced vortex flow.

Type of pumps:

Positive displacement pump

- i. Reciprocating pump
  - a. Piston
  - b. Plunger
  - c. Diaphragm
- ii. Rotary pump
  - a. Screw
  - b. Gear

Dynamic pump

- i. Centrifugal pump
- ii. Special type (like jet, ejector)

## Reciprocating pump:

- i. Used for generating high head and low flow.
- ii. Pulsation is observed here, to minimize pulsation one extra suction and discharge valve added.
- iii. Also known as constant flow pump. Discharge rate is nearly the same.
- iv. Wall openings do not affect discharge.
- v. PSV is important here.

### Gear pump:

i. Used for high viscous and less flow service.

### Centrifugal pump:

- i. Used for generating high volume.
- ii. It may need priming.
- iii. Constant head pump.
- iv. Used for generally low viscous, high delivery.

### Screw pump:

- i. High tolerance against water vapor and particles/dust.
- ii. Very high pumping speeds.
- iii. Highly efficient due to internal compression.

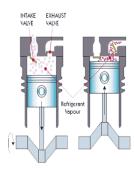
### 4.2. Compressors:

Compressor is a mechanical device that increases the pressure of a gas by reducing its volume. An air compressor is a specific type of gas compressor. Compressors are similar to pumps: both increase the pressure on a fluid and both can transport the fluid through a pipe. As gasses are compressible, the compressor also reduces the volume of a gas. Liquids are relatively incompressible; while some can be compressed, the main action of a pump is to pressurize and transport liquids. Many compressors can be staged, that is, the fluid is compressed several times in steps or stages, to increase discharge pressure. Often, the second stage is physically smaller than the primary stage, to accommodate the already compressed gas without reducing its pressure. Each stage further compresses the gas and increases its pressure and also temperature (if inter cooling between stages is not used).

### **Positive Displacement Compressor:**

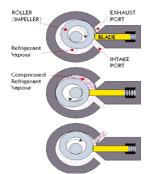
A positive displacement air compressor works by drawing air into a compression chamber through an intake and mechanically reducing the volume of the chamber through motion until a set pressure is reached. The compressed air is then discharged through a valve at the rated pressure, providing a flow of air. There are two main types of the PD compressor that are as follows:

• Reciprocating Compressor: Reciprocating compressors also widely known as piston compressors are mainly used to move air/gas at high pressure to be stored and used for different purposes. The main elements of the compressor are one or more cylinders and pistons which move within them. Automobile engines work almost the same way as reciprocating



compressors do through letting the air in from one chamber, mixing it with fuel and letting the fume out from another chamber with pressure.

• Rotary Screw Compressor: A rotary-screw compressor is a type of gas compressor, such as an air compressor, that uses a rotary-type positive-displacement mechanism. These compressors are common in industrial applications and replace more traditional piston compressors where larger volumes of compressed gas are needed,



e.g. for large refrigeration cycles such as chillers, or for compressed air systems to operate air-driven tools such as jackhammers and impact wrenches.

## **Dynamic Compressor:**

Dynamic compressors are rotary continuous-flow machines in which the rapidly rotating element accelerates the air as it passes through the element, converting the velocity head into pressure, partially in the rotating element and partially in stationary diffusers or blades. The velocity head is converted into pressure, partially in the rotating element and partially in stationary diffusers or blades range and then fluid flow is restricted so that the reduction in velocity causes pressure to increase. There are two main types of the dynamic compressor that are as follows:

|                        | Advantages   | Disadvantages  |
|------------------------|--|--|
| Dynamic<br>Compressors |  |  |
| Centrifugal            | <ul> <li>Wide operating range</li> <li>High reliability</li> <li>Low Maintenance</li> </ul>                            | <ul> <li>Instability at reduced flow</li> <li>Sensitive to gas composition change</li> </ul> |
| Axial                  | <ul> <li>High Capacity for given size</li> <li>High efficiency</li> <li>Heavy duty</li> <li>Low maintenance</li> </ul> | <ul><li> Low Compression ratios</li><li> Limited turndown</li></ul>                          |

## **Multistage compression:**

Multi-stage compressors feature two or more piston cylinders, each of a different diameter. After the first compression stage, air passes through a heat exchanger, where it is cooled before arriving at the second cylinder. Cooling the air reduces the work needed to compress it during the second or third stages.

## **Benefits of Multi-stage Compression**

- o Improved efficiency. Two-stage compressors perform less work to compress air to a given pressure, which means your operating costs are lower.
- o Better reliability. ...
- o Less moisture buildup.

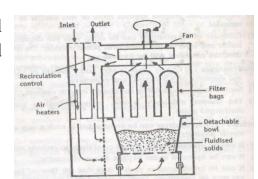
### **4.3. Blower:**

Generally, a fan is an electrical device that moves air, whereas a blower is a mechanical device that consists of a fan, and which channels the air from the fan and directs it to a specific location or point. Also, a fan circulates the air around an entire room or a large area, while a blower is only positioned to a specific direction or point.

|                | Fan  | Blower   |
|----------------|--|--|
| Definition     | A fan circulates air around an entire room, or space.                                  | A blower circulates the air only on the specific or pointed area.                    |
| Pressure       | It is uses less pressure to produce large amounts of gas.                              | It is uses high pressure to produce large amounts of gas.                            |
| Pressure ratio | The ratio of pressure is below 1.1.  | The ratio of pressure is from 1.1 to 1.2.  |
| Air area       | It provides air in the complete area.  | It provides air in a specific location or point.                                     |
| Types          | <ul><li>Axial flow fans.</li><li>Centrifugal fans.</li><li>Cross- flow fans.</li></ul> | <ul> <li>Centrifugal blowers.</li> <li>Positive-displacement<br/>blowers.</li> </ul> |
| Consists of    | It consists of a motor and blades, which run of electricity.                           | It consists of a fan, outer cover, inlet, out-let.                                   |

## 4.4. Fluidized Bed Dryer

Fluidized bed dryer (also called fluid bed dryer) is a kind of equipment used



extensively in the pharmaceutical industries to reduce the moisture content of pharmaceutical powder and granules. The equipment works on a principle of fluidization of the feed materials.

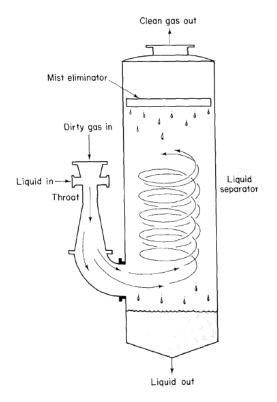
In the fluidization process, hot air is introduced at high pressure through a perforated bed of moist solid particulate. The wet solids are lifted from the bottom and suspended in a stream of air (fluidized state). Heat transfer is accomplished by direct contact between the wet solid and hot gases. The vaporized liquid is carried away by the drying gases. Sometimes to save energy, the exit gas is partially recycled.

### 4.5. Scrubber

A scrubber or scrubber system is a system that is used to remove harmful materials from industrial exhaust gases before they are released into the environment. There are two main ways to scrub pollutants out of exhaust, and they are:

### Wet Scrubbing:

In wet scrubbing processes, liquid or solid particles are removed from a gas stream by transferring them to a liquid. The liquid most commonly used is water. A wet scrubber's particulate collection efficiency is directly related to the amount of energy expended in contacting the gas stream with the scrubber liquid. Most wet scrubbing systems operate with particulate collection



efficiencies over 95 percent. Wet scrubbers can also be used to remove acid gas; however, this section addresses only wet scrubbers for control of particulate matter.

There are three energy usage levels for wet scrubbers. A low energy wet scrubber utilizes pressure drops less than 5 inches of water column and are capable of efficiently removing particles greater than about 5-10 micrometers in diameter. A medium energy scrubber has a pressure drop from 5 to 25 inches of water column and is capable of removing

micrometer-sized particles, but is not very efficient on sub-micrometer particles. A high energy scrubber expends the most energy and has a pressure drop of 25 to over 100 inches of water column, which is necessary to remove sub-micrometer particles.

A spray tower scrubber is a low energy scrubber and is the simplest wet scrubber used for particulate control. It consists of an open vessel with one or more sets of spray nozzles to distribute the scrubbing liquid. Typically, the gas stream enters at the bottom and passes upward through the sprays. The particles are collected when they impact the droplets. This is referred to as a counter-current operation. Spray towers can also be operated in a cross-current arrangement. In cross-current scrubbers, the gas flow is horizontal and the liquid sprays flow downward. Cross-current spray towers are not usually as efficient as counter-current units.

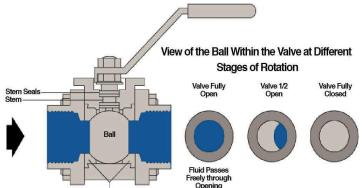
### **Dry Scrubbing**:

A dry scrubber or dry scrubber system is one type of scrubber that is used to remove harmful materials from industrial exhaust gases before they are released into the environment. Dry scrubbers are the type most commonly used in plants today, and they utilize a collection of dry substances to remove acidic gases that contribute to acid rain.<sup>[2]</sup>

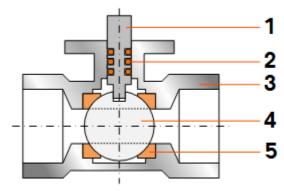
Dry scrubbers work similarly to other scrubbers. The system sprays a collection of dry reagents into an exhaust stream. These chemicals can react differently depending on which material they are specifically targeting for removal. Some of these materials neutralize harmful pollutants in the stream through a chemical reaction, while others cause a material to react and turn into a different substance. That substance then falls out of the gas stream or is caught in a particle screen.

# 4.6. Valves Ball Valve

To understand the working principle of a ball valve, it is important to know the 5 main ball



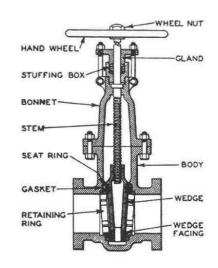
valve parts and 2 different operation types. The 5 main components can be seen in the ball valve diagram in Figure 2. The valve stem (1) is connected to the ball (4) and is either manually operated or automatically operated (electrically or pneumatically). The ball is supported and sealed by the ball valve seat (5) and there are o-rings (2) around the valve stem. All are inside the valve housing (3). The ball has a bore through it, as seen in the sectional view in Figure 1. When the valve stem is turned a quarter-turn the bore is either open to the flow allowing media to flow through or closed to prevent media flow.



### Gate Valve

Gate valve functions by the reciprocating action of a disc in its body. It can have a single disc or double disc. It is used for shut off operation. A double disc valve shut off is good.

Gate valves can have a rising or non-rising stem. Gate valves are available in various sizes ranging from 12 mm to 300 mm and even more. Gate valve is light in weight, economical and offers low pressure drop. Conduit type gate valve is common in chemical process industries because it gives accurate flow control. Gate valve can be used upto 20 kg/cm2 and upto 675°C. Material of construction for gate valve is cast iron, carbon steel, stainless steel, ductile iron, bronze, nickel



Gate (or sluice) valve

alloys etc. Rotating disc type gate valve with quick action lever actuator.

### **Utilities**

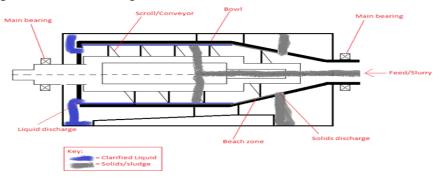
### 5.1. Decanter

A decanter is equipment used for the separation of suspended solids from a liquid. This separation is based on buoyancy, where the component with the higher density would fall to the bottom, and the component with the lower density would be suspended above it. By continuous rotation, the decanter enables the increase in the rate of settling.

The action of the centrifuge multiplies the g-force considerably, since it is the only variable that can be altered, provided that inlet conditions remain constant. The increase in the g-force results in an increase in the settling speed, which further results in the decrease of the time required for settling.

In order to further increase the efficiency, a bowl is included within the decanter's construction. This bowl contains both a cylindrical and a conical section, along with a screw conveyor, which continuously scrapes the solids from the sides of the bowl.

The slurry is fed to the centrifuge via a pipe. The conveyor then transports the slurry to a nozzle, and is transferred through it to the bowl area. The bowl rotates at high speeds to induce gravitational forces, thus separating the two components. The conveyor then passes the solids, which are discharged through the nozzle. The purified liquid exits via a different pathway.



## 5.2. Cooling Water

Water cooling is a method used to eliminate heat from equipment.

In the MCPI plant, the cooling water flow exists in a closed system, and is used in primarily two areas: Process and Utility. Following heat collection, the cooling water returns to the heat exchanger in order to be cooled in the tower. This return is facilitated by the closed system, and the temperature rise usually lies within the range of 7°C-9°C.

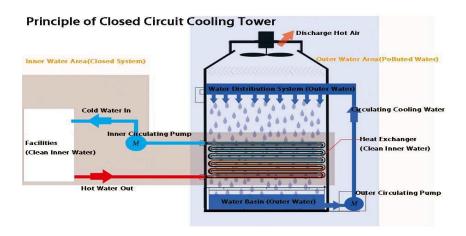
The Crude Terephthalic Acid (CTA) production process is the largest user of the cooling water, accounting for nearly 78% of the cooling water consumption. This is in sharp contrast with the Pure Terephthalic Acid (PTA) production, which utilises a mere fraction of the consumption.

Several additional components/equipments have been added, primarily in the CTA section, to aid the water cooling process.

A make-up tank is installed in the CTA section's highest point in order to facilitate air venting, and provide the pump inlet with a higher suction head, as well as keep the cooling water line (which is where the makeup water is added) flooded. The pipelines of the cooling water line are constructed of carbon steel.

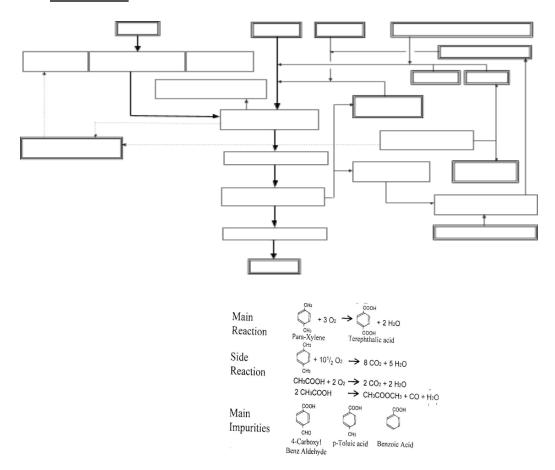
A set of 3 pumps are installed to fulfil the rather large cooling water requirement, 2 of which run at a time. The third remains on standby and is automated, working under specified pressure restrictions (when the header pressure falls below a certain required pressure).

A chemical dosing unit protects the closed system from the infiltration of unwanted particles, both foreign and native to the system. The rate is decided by the cooling water quality, as well as the makeup water quantity.



## **Process**

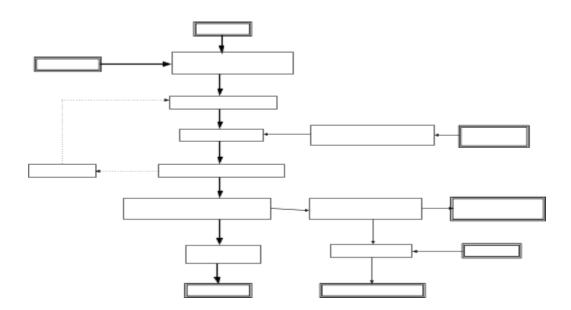
## 6.1. <u>CTA Unit</u>



In the production of terapthalic acid preparation, raw material is p-xylene and compressed air. Air is compressed by compressor using the energy in some amount

of steam generated from gas expander where flue gas exchanges their heat with water. HBR is added as promoter, Co(ac)2, Mn(ac)2 these two are used as catalyst. Acetic acid is also provided here. Oxidation Reaction is carried out in the CSTR reactor at the temperature around 200 degree Celsius and 20 bar pressure. Air is supplied through multiple ways so that channeling, bypassing and dead zone will not form as well as mass transfer coefficient will increase. After the reaction carried out, a small oxidation reactor is connected to minimize the vigorousity of the first reactor. It is actually exothermic reaction heat is released and used as steam. Further it is taken into crystallization section where it agglomerates and solid crystal will form, there are 4 crystallizer from here separation is done and after drying we get terapthalic acid which has impurities and not scalable so it is known as crude terapthalic acid. In the process of separation there are some important chemical like acetic acid, methyl acetate, water which has need in further process. So for that Distillation is required, in azeotropic distillation water and acetic acid is separated, and further it is supplied to reactor line. Further the water is impure some part of the water is used as process water (DM Water) in PTA section and remaining is gone to waste water treatment.

### 6.2. PTA Unit



In purification unit firstly DM water (process water) is supplied to the slurry preparation tank with the help of centrifugal pump and booster pump so that it can generate a head of more than 80 bar. After slurry preparation it is sent to preheating zone to make complete dissolution of solid powder, the preheating section there is 7 heat exchanger in series, in the shell and tube heat exchanger slurry is filled inside the tube and hot fluid in the shell side, in first 3 heat exchanger steam is brought from crystallizer unit the last three crystallizer has less temperature that steam is filled in first 3 heat exchanger in reverse order in 4th and 5th heat exchanger steam is brought from 1st crystallizer because it has the highest heat among them, but still in reduction reactor temperature is needed about 290 degree Celsius, but even steam from the 1st crystallizer has temperature of 250 degree Celsius and there is somehow fouling in each section so to get the required heat hot oil is filled in the last two section of heat exchanger in the shell side. Further it is sent into packed bed reactor where hydrogen is used for reduction, there is some side reaction in CTA unit and some compound 4-CBA, MA, p-toluic acid these all are formed in the reduction unit these all are converted into p-xylene and further it sent to CTA unit after the reduction it further sent for the centrifuge and drying section from there we get purified terapthalic acid.

## **Hydrogen Generation Unit**

Here for the production of hydrogen methanol scrubbing is done, methanol and water is passed in packed bed reactor in the presence of Zinc and Copper catalyst reaction is done, here for the supply of methanol reciprocating pump is used since it has less flow. After reaction Hydrogen and CO<sub>x</sub> is achieved along with water.

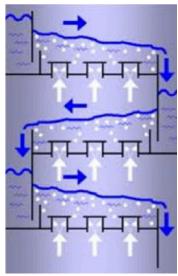
## **Major Project**

## **Analysis of Binary Distillation and Azeotropic Distillation:**

### **Distillation**

Distillation refers to the physical separation of a mixture into two or more fractions that have different boiling points. The general objective is to separate substances having different vapour pressures at any given temperature. If a liquid mixture of two volatile materials is heated, the vapour that comes out with a higher concentration of the lower boiling material than the liquid from which it was evolved. Conversely, if a warm vapour is cooled, the higher boiling material has a tendency to condense in a greater proportion than the lower boiling material. The early distillers of alcohol for beverages applied these fundamental ideas.

A distillation column consists of a series of plates (or trays). In normal operation, there is a certain amount of liquid on each plate, and some arrangement is made for ascending vapour to



pass through the liquid and make contact with it. The descending liquid flows down from the plate above through a downcomer, across the next plate, and then over a weir and into another downcomer to the next lower plate. Earlier on, bubble caps were used on the trays for the purpose of vapor and liquid contacting.

**Azeotropic distillation** is a process of distillation wherein you can add a certain component into the mixture to have a better separation process. Usually, the certain component added into the mixture is water or benzene, because these can help in increasing the volatility of a substance.

Azeotropic distillation is accomplished by adding to the liquid phase, a volatile third component (entrainer), which changes the volatility of one of the two components more than the other so that the components are separated by distillation.

Here we are analysis the binary and azeotropic distillation for the separation of acetic acid from water, and my aim is to calculate all the necessary data without using the graph.

A binary solution in a distillation column is used to separate water from acetic acid. The Feed is 14 wt.% water and 86wt% acetic acid mixture that is saturated mixture at atmospheric pressure. The feed rate is 80 Tons/hr. to provide a distillate containing 96 wt. % water and a residue containing 4 wt. % water. A reflux ratio is 1.2 times the minimum will be used. Relative volatility is 1.8 and tray efficiency is 0.4. Calculation of no. of theoretical and actual trays.

### **SOLUTION:**

Here, we are taking WATER as reference.

For the calculation of no. of theoretical and actual trays, we'll use McCabe-Thiele method.

we've to plot vapor-liquid equilibrium diagram, Relative volatility is 1.8 (given).

As, we know  $y = \alpha x / 1 + (\alpha - 1) *x$ 

Where, y= mole fraction of distillate of more volatile component in the feed,

X = mole fraction of residue of more volatile component in the feed.

| X   | Y        |
|-----|----------|
| 0   | 0        |
| 0.1 | 0.166667 |
| 0.2 | 0.310345 |
| 0.3 | 0.435484 |
| 0.4 | 0.545455 |
| 0.5 | 0.642857 |
| 0.6 | 0.72973  |
| 0.7 | 0.807692 |
| 0.8 | 0.878049 |
| 0.9 | 0.94186  |
| 1   | 1        |

Since, in the problem, feed is given in wt.% and we need feed in the term of mole fraction. We know Mol. Wt. of water is 18 and Mol. wt. of acetic acid is 60.

### Feed concentration:

| Water | Acetic Acid | X f                              |
|-------|-------------|----------------------------------|
| 0.14  | 0.86        | (0.14/18)/((0.14/18) + (0.86/60) |
|       |             | = 0.351759                       |

### Distillate concentration:

| Water | Acetic acid | X <sub>d</sub>                   |
|-------|-------------|----------------------------------|
| 0.96  | 0.04        | (0.96/18)/((0.96/18) + (0.04/60) |
|       |             | = 0.987654                       |

### Residue concentration:

| Water | Acetic acid | X w                              |
|-------|-------------|----------------------------------|
| 0.04  | 0.96        | (0.04/18)/((0.04/18) + (0.96/60) |
|       |             | = 0.121951                       |

Here, x <sub>f</sub>=feed water mole fraction

x<sub>d</sub>=Distillate water mole fraction,

 $x_{\rm w}$  = bottoms water mole fraction

Now, we've to find minimum reflux ratio.

We've 2 methods for the calculation of R  $_{min.}$ 

### First method:

From slope,

We know  $(x_d, x_d) = (0.987654, 0.987654)$ 

We know for saturated mixture q=1, hence slope(=q/q-1) = infinite.

$$x_f = 0.531759$$
,  $y_f = 0.351759*1.8/(1+0.351759*1.8) = 0.494117$ 

For R  $_{min}$ , the line will pass from  $(x_{d}, x_{d})$  and intersection of q=1 to equilibrium curve that is  $(x_{f}, x_{f})$ .

Hence,

Slope = 
$$(x_d - y_f)/(x_d - x_f) = (0.987654 - 0.494117)/(0.987654 - 0.351759) = 0.78$$

from McCabe-Thiele, 
$$y_n = (R/R+1) x_n + (x_d/R+1)$$

Hence, 
$$R_{min}/R_{min}+1 = 0.78$$

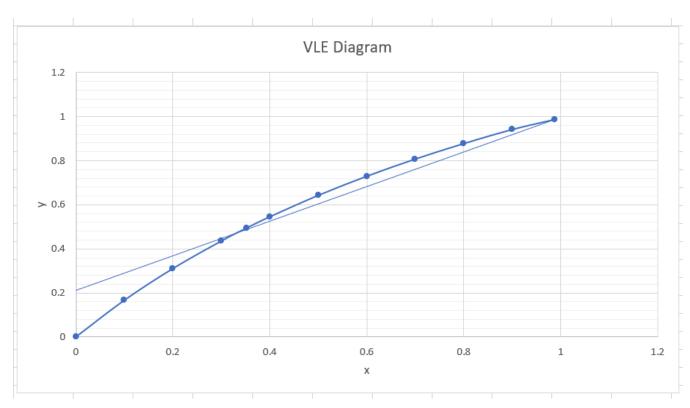
$$R_{min} = 3.54$$

Since, 
$$R = 1.2 * R_{min}$$
 (Given)

$$R = 1.2 * 3.54 = 4.25$$

## Second method:

## From graph,



Intercept is 0.215, 0.215 = 
$$x_d/R_{min}+1$$
;  $R_{min}=(0.98/0.215)-1=3.54$ 

From here Also, R = 4.25

Now new intercept =  $x_d/R + 1 = 0.987654/5.25 = 0.188$ 

Now, for the calculation of no. of theoretical plate, we've again two methods.

### First method:

From analytical approach,

Gilliland equation, 
$$N - N_{min}/N+1 = 0.75[1 - (R-R_{min}/R+1)^{0.567}]$$

For total condenser and partial reboiler, the minimum number of plates can be calculated by the Fenske Equation.

$$N_{min}+1 = \ln \left( \left( x_{d} \left( 1-x_{w} \right) / x_{w} \left( 1-x_{d} \right) \right) / \ln \alpha$$
 
$$\ln \left( \left( 0.987654 (1-0.121) / \left( 0.121 (1-0.987654) \right) / \ln \left( 1.8 \right) = 10.7 \right.$$
 
$$N_{min}=9.7$$

Now put this value in Gilliland equation,

N- 9.7/N+1 = 0.75[1- 
$$(4.25-3.54/4.25+1)^{0.567}$$
] = 0.5  
N = 20.4

For total condenser and partial reboiler, N = 19.4

Given tray efficiency = 0.4 = no. of ideal tray / no. of actual tray

No. of actual trays 
$$= 49$$

for feed tray location, just replace the value of x  $_{\rm w}$  with x  $_{\rm f}$  in the Fenske equation.

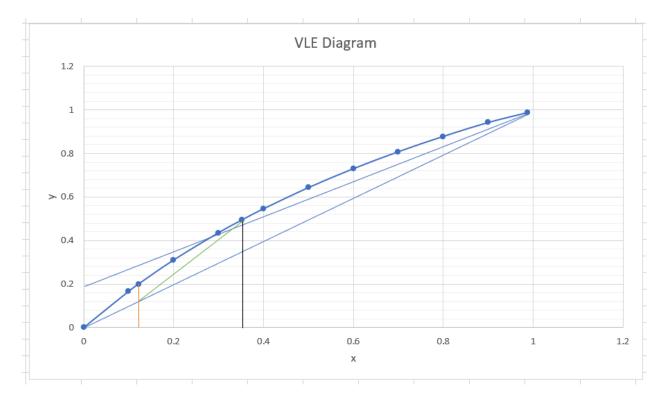
$$N_{min}=7.4$$

Now put this value in Gilliland equation,

N- 7.4/N+1 = 0.75[1- 
$$(4.25-3.54/4.25+1)^{0.567}$$
] = 0.5  
N = 15.8

For total condenser and partial reboiler, N = 14.8, so feed is entering the 15th **plate.** 

### Second method:



From Graph, we can calculate the no. of ideal stages.

From the plot we observe 20.2 no. of ideal trays. Feed is entering the column in the 15<sup>th</sup> tray.

Hence, actual no. of tray = (20.2-1)/0.4 = 49

### Mass balance:

Now, we've to find the amount of distillate and residue.

Given, feed rate: 80 Tons/hr. = 80000kg/hr.

Feed composition = 14 wt. % + 86 wt.% composition

Mass feed rate of water = 80000\*0.14 = 11200kg/hr.

Molar feed rate of water = 112000/18= 622.22 mol/hr.

Mass feed rate of acetic acid = 80000\*0.86 = 68800kg/hr.

Molar feed rate of acetic acid = 68800/60 = 1146.67 mol/hr.

Total molar feed rate = 1768.89 mol/hr.

From mass balance,

$$F = D + W$$
  
1768.89 = D + W ----(1)

; D is molar distillate flow rate, W is the residual flow rate

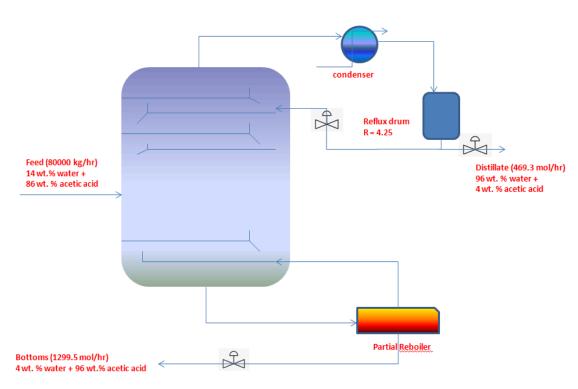
From component balance,

$$F x_f = D x_d + W x_w$$
....(2)

$$622.22 = D*(0.987654) + W*(0.121951)$$

On solving this equation,

D = 469.56 mol/hr. W = 1299.32 mol/hr.



Schematic view of distillation column of given problem

A mixture of a solution in a distillation column is used to separate water from acetic acid. NBA is added here as an entrainer. The feed is 14 wt. % water + NBA (9 wt. % water and 5 wt. % NBA) and 86% acetic acid mixture that is a saturated mixture at atmospheric pressure. The feed rate is 80 Tons/hr to provide a distillate containing 96 wt. % water +NBA and a residue containing 4 wt. % water +NBA. A reflux ratio of 1.2 times the minimum will be used. Relative volatility is 2.5 and tray efficiency is 0.4. Calculation no. of theoretical and actual trays.

### **SOLUTION:**

Here, we are taking WATER + NBA as reference.

For the calculation of no. of theoretical and actual trays, we'll use the McCabe-Thiele method.

We've to plot a vapor-liquid equilibrium diagram, Relative volatility is 2.5 (given).

As, we know 
$$y = \alpha * x / 1 + (\alpha - 1) * x$$

Where, y= mole fraction of distillate of more volatile component in the feed,

X = mole fraction of residue of more volatile component in the feed.

| X   | Y        |
|-----|----------|
| 0   | 0        |
| 0.1 | 0.217391 |
| 0.2 | 0.384615 |
| 0.3 | 0.517241 |
| 0.4 | 0.625    |
| 0.5 | 0.714286 |
| 0.6 | 0.789474 |
| 0.7 | 0.853659 |
| 0.8 | 0.909091 |
| 0.9 | 0.957447 |
| 1   | 1        |

Since, in the problem, feed is given in wt. % and we need feed in the term of mole fraction. We know Mol. Wt. of water is 18, Mol. Wt. of NBA(n butyl acetate) is 116 and Mol. wt. of acetic acid is 60.

Mole fraction of water = (0.09/18)/((0.09/18)+(0.05/116)) = 0.92

Mole fraction of NBA =0.08

Hence, mol. Wt. of Water + NBA = (0.92\*18) + (0.08\*116) = 25.84

### Feed concentration:

| Water + NBA | Acetic Acid | X f                                     |
|-------------|-------------|---|
| 0.14        | 0.86        | (0.14/25.84)/ ((0.14/25.84) + (0.86/60) |
|             |             | = 0.274309                              |

### Distillate concentration:

| Water + NBA | Acetic acid | X <sub>d</sub>                |
|-------------|-------------|-------------------------------|
| 0.96        | 0.04        | (0.96/25.84)/ ((0.96/25.84) + |
|             |             | (0.04/60)                     |
|             |             | = 0.9823718                   |

### Residue concentration:

| Water + NBA | Acetic acid | X w                           |
|-------------|-------------|-------------------------------|
| 0.04        | 0.96        | (0.04/25.84)/ ((0.04/25.84) + |
|             |             | (0.96/60)                     |
|             |             | = 0.0882145                   |

Here,  $x_f$ =feed water mole fraction,  $x_d$ =Distillate water mole fraction,

 $x_{w}$  = bottoms water mole fraction

We've to find the minimum reflux ratio. We've 2 methods for the calculation of R min.

### First method:

From slope,

We know  $(x_d, x_d) = (0.9823718, 0.9823718)$ 

We know for saturated mixture q=1, hence slope (=q/q-1) = infinite.

$$x_f = 0.274309$$
,  $y_f = 0.274309*2.5/(1+0.274309*1.5) = 0.485859$ 

For R  $_{min}$ , the line will pass from  $(x_d, x_d)$  and the intersection of q=1 to the equilibrium curve that is  $(x_f, x_f)$ .

Hence,

Slope = 
$$(x_d - y_f)/(x_d - x_f) = (0.9823718 - 0.485859)/(0.9823718 - 0.274309) = 0.70$$

From McCabe-Thiele, 
$$y_n = (R/R+1) x_n + (x_d/R+1)$$

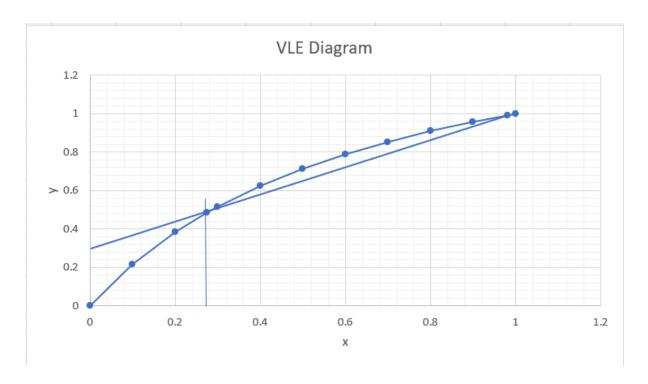
Hence, R 
$$_{min}/R _{min}+1 = 0.70$$
; R  $_{min}=2.33$ 

Since, 
$$R = 1.2 * R_{min}$$
 (Given);

$$R = 1.2 * 2.33 = 2.79$$

### Second method:

From graph,



Intercept is 0.295,  $0.295 = x_d/R_{min} + 1$ ;  $R_{min} = (0.9823718/0.295) - 1 = 2.33$ 

From here Also, R = 2.79; Now new intercept =  $x_d/R + 1 = 0.9823718/3.79 =$ **0.26** 

Now, for the calculation of no. of theoretical plates, we've again two methods.

## First method:

From analytical approach,

Gilliland equation, 
$$N - N_{min}/N+1 = 0.75[1 - (R-R_{min}/R+1)^{0.567}]$$

For total condenser and partial reboiler, the minimum number of plates can be calculated by Fenske Equation.

$$N_{min}+1 = \ln \left( \left( x_{d} \left( 1-x_{w} \right) / x_{w} \left( 1-x_{d} \right) \right) / \ln \alpha$$
 
$$\ln \left( \left( 0.9823718 \left( 1-0.088 \right) / \left( 0.088 \left( 1-0.9823718 \right) \right) / \ln \left( 2.5 \right) = 6.91$$
 
$$N_{min}=5.91$$

Now put this value in Gilliland equation,

$$N-5.91/N+1 = 0.75[1-(2.79-2.33/2.79+1)^{0.567}] = 0.52$$

$$N = 13.4$$

For total condenser and partial reboiler, N = 12.4

Given tray efficiency = 0.4 = no. of ideal tray / no. of actual tray

No. of actual trays 
$$= 31$$

For feed tray location, just replace the value of x with x in the Fenske equation.

$$N_{min}=4.44$$

Now put this value in Gilliland equation,

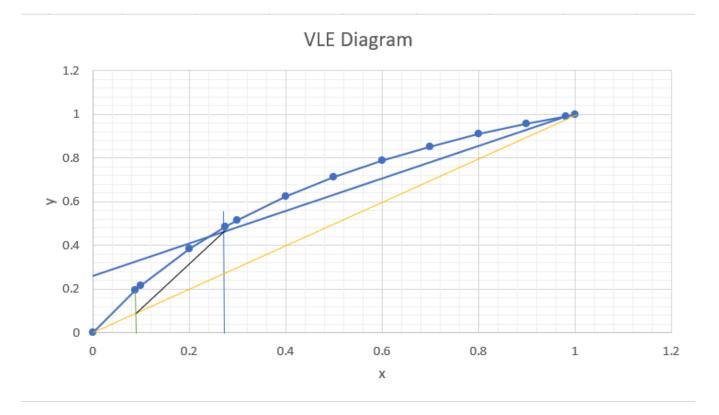
$$N-4.44/N+1 = 0.75[1-(2.79-2.33/2.79+1)^{0.567}] = 0.52$$

$$N = 10.33$$

For total condenser and partial reboiler, N = 10.33, so feed is entering in the 11th plate.

### Second method:

From Graph, we can calculate the no. of ideal stages.



From the plot we observe 13.4 no. of ideal trays. Feed is entering the column in the 11<sup>th</sup> tray.

Hence, actual no. of tray = (13.4-1-1)/0.4 = 31

### Mass balance:

Now, we've to find the amount of distillate and residue.

Given, Feed rate: 80 Tons/hr. = 80000 kg/hr.

Feed composition = 14 wt. % + 86 wt. % composition

Mass feed rate of water = 80000\*0.09 = 7200 kg/hr.

Molar feed rate of water  $(x_{fl}) = 7200 / 18 = 400 \text{ mol/hr}$ .

Mass feed rate of NBA = 80000\*0.05 = 4000 kg/hr.

Molar feed rate of NBA  $(x_{f2}) = 4000 / 116 = 34.48 \text{ mol/hr}.$ 

Mass feed rate of acetic acid = 80000\*0.8 = 68800kg/hr.

Molar feed rate of acetic acid  $(x_B) = 68800/60 = 1146.67 \text{ mol/hr}.$ 

Total molar feed rate  $(x_{f1}+x_{f2}+x_{f3}) = 1581.15 \text{ mol/hr}.$ 

From mass balance, F = D + W

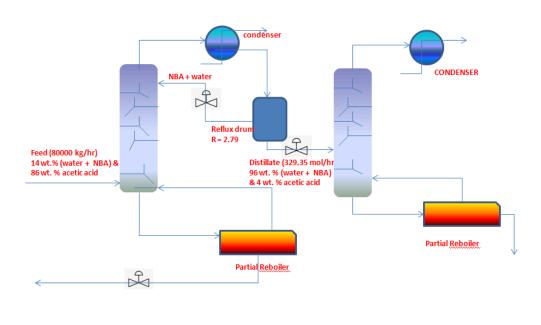
$$1581.15 = D + W ---- (1)$$

; D is molar distillate flow rate, W is the residual flow rate

From component balance,  $F x_f = D x_d + W x_w$  (2)

$$433.7 = D*(0.9823718) + W*(0.088)$$

On solving this equation, D = 329.35 mol/hr. W = 1251.80 mol/hr.



Bottoms (1251.8 mol/hr) 4 wt. % water + 96 wt.% acetic acid

Schematic view of Azeotropic distillation column of given problem

## **RESULTS AND ANALYSIS:**

The desired product being recovered from the distillation tower is Acetic acid and its mole fraction in the bottoms of the distillation tower is:

Binary Distillation : 0.878

Azeotropic Distillation: 0.912

Hence, we can hereby conclude that efficiency of Azeotropic Distillation Tower is better than Binary Distillation.

| Type of Distillation | No. of Theoretical<br>Tray | No. of Actual Tray | Molar flow rates |
|----------------------|----------------------------|--------------------|------------------|
|                      |                            |                    |                  |
| Binary Distillation  | 20.4                       | 49                 | F = 1768.89      |
|                      |                            |                    | D = 469.5        |
|                      |                            |                    | W = 1299.32      |
| Azeotropic           | 13.4                       | 31                 | F = 1581.15      |
| Distillation         |                            |                    | D = 329.35       |
|                      |                            |                    | W = 1251.80      |

## No. of Tray Required:

It is also concluded that no. of theoretical stages and actual trays required for the desired recovery of the solvent, Acetic acid requires less no. of trays in the **Azeotropic Distillation Tower**. Hence, we can conclude that Azeotropic Distillation Tower is lesser in height than Binary Distillation and efficiency is also good in Azeotropic distillation.