PRODUCTION OF 40 TONS PER DAY OF PURE TEREPHTHALIC ACID (PTA) FROM CRUDE TEREPHTHALIC ACID (CTA) VIA CRYSTALLIZATION

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CERTIFICATE

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DEDICATION

This FYP Thesis is dedicated to, foremost, our parents, who supported us and had our back throughout our life and our friends and teachers here at SCME, who have been so helpful.

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All thanks to Allah, the Generous and Almighty for His countless blessings and rewards, who granted us the strength and ability to complete this thesis.

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We are also thankful to our parents and special thanks to our friends and seniors; without their support and encouragement, it would not have been possible to complete the project successfully.

Authors

Abstract

This thesis designs and operates a smaller purified terephthalic acid (PTA) plant in tandem with an existing facility to increase production capacity and eliminate bottlenecks. The study begins by emphasizing PTA production and the problem. A comprehensive literature review covers PTA basics, methods for converting crude terephthalic acid (CTA) to PTA, and PTA crystallization processes. This literature evaluation lays the groundwork for the planned study and future research. Thesis then presents a process flow diagram and details PTA production procedures. CTA, hydrogen, water/steam, and nitrogen are important basic ingredients for high-quality PTA. The specifications and design criteria for crystallization equipment such feed tanks, centrifugal pumps, pre-heaters, packed bed reactors, crystallizers, centrifuges, and dryers are carefully studied. Feed tanks, pumps, pre-heaters, reactors, crystallizers, centrifuges, and dryers undergo material and energy balances. Thesis also covers critical equipment component design and specifications. The design, function, and design considerations of shell-and-tube heat exchangers are examined. Discussed include packed-bed reactor design and reactions. The crystallizer's design and operation are analyzed. Aspen Hysys and Aspen Plus software simulate process dynamics and behavior, providing a complete picture. Component lists, fluid packages, parameters, and reactor and crystallizer requirements are simulated. Simulations reveal process performance and enable optimization. Operation and process monitoring require instrumentation and control. Ratio and cascade control solutions are described for process stability and production optimization. This thesis assesses primary equipment costs, total investment, direct production expenses, income generation, and payback period for any industrial operation. These financial studies reveal the parallel PTA plant's economic viability. Hazard and Operability (HAZOP) research identifies process hazards and recommends risk assessment and reduction. Explaining HAZOP and identifying process hazards ensures safety and regulatory compliance. This final-year thesis suggests designing and operating a smaller PTA factory alongside an existing facility to boost production capacity and eliminate bottlenecks. Process overview, material and energy balance calculations, equipment design, simulation, instrumentation and control strategies, cost estimation, and HAZOP studies are examined in detail to inform future research.

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CHAPTER 1

INTRODUCTION

1.1 Project Description

This section gives a general description of the project with an emphasis on the crystallization-based purification of terephthalic acid (PTA). By creating a smaller PTA facility that can run in parallel with an existing plant and accommodate an additional 40 tons of feed per day, the project seeks to alleviate a production bottleneck. The project is a chemical engineers' senior thesis that focuses on improving the PTA production process.

Application in Industry of Terephthalic Acid: The manufacturing of polyethylene terephthalate (PET), a widely used polymer with multiple industrial applications, requires terephthalic acid as a crucial raw material. The production of bottles, fibers, films, and other packaging products uses PET predominantly. It is widely used in the beverage sector to make bottles for soft drinks and water, as well as in the textile industry to make polyester fabrics and fibers. Various household and personal care items, including food containers, cosmetics, and detergents, are also made from PET.

A typical purification process is given below:

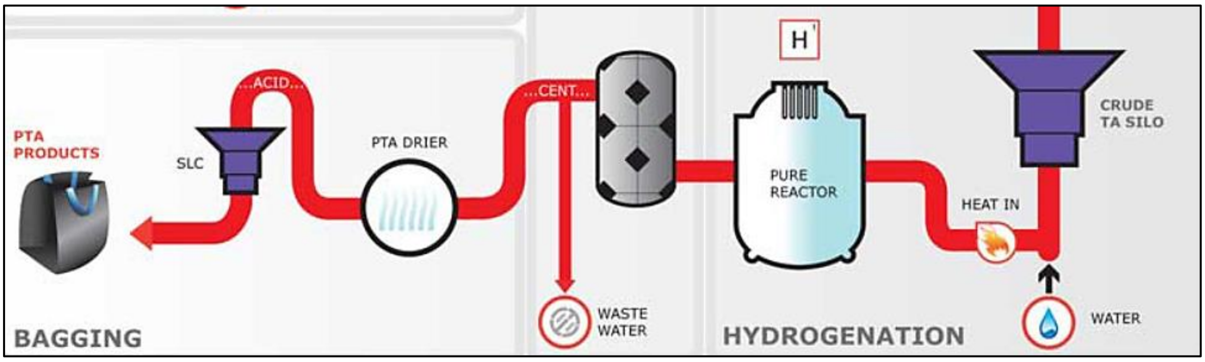


Figure 1: General CTA Purification Process

1.2 Importance of Improving PTA Production Purity

This thesis's problem statement centers on the requirement to raise PTA's purity from 99.7% to 99.98%. The performance and characteristics of PET-based goods can be significantly impacted by even a small increase in purity. PET is suitable for demanding applications because higher purity PTA offers increased mechanical strength, thermal stability, and chemical resistance. Higher purity levels are also necessary to meet the exact quality standards demanded by sectors like food and beverage packaging. Moreover, the presence of 4-CBA acts as a terminating agent in the polymerization of PET and hence needs to be removed.

The primary goal of this final-year thesis is to create a smaller PTA plant that runs concurrently with the current plant and has the capacity to accept an extra 40 tons of feed per day. The project's goal is to boost overall PTA production capacity and satisfy the expanding market demand for high-quality PTA by tackling the production bottleneck that is currently hindering progress. To achieve the requisite purity level of 99.98%, the thesis focuses on streamlining the crystallization process used to convert CTA to PTA.

The PTA industry has grown significantly during the past few years. This increase might be ascribed to the rising packaging and consumer products sectors, notably in places like Asia-Pacific. PTA is a primary constituent in many PET-based products, which are in high demand due to reasons like population expansion, urbanization, and shifting consumer lifestyles. It is crucial to streamline the PTA production process and deal with any bottlenecks preventing increasing production to satisfy the growing demand.

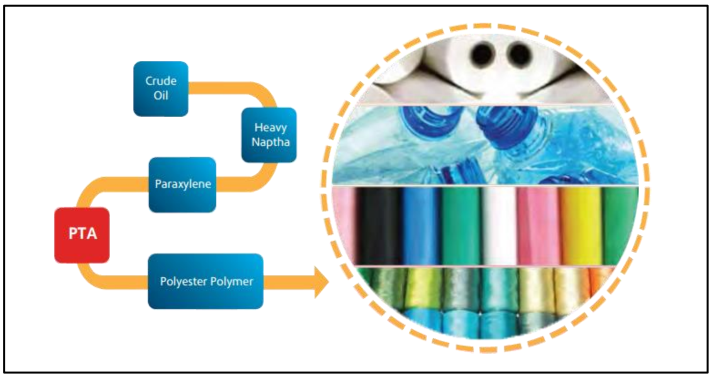


Figure 2: PTA Applications Downstream

CHAPTER 2

LITERATURE OVERVIEW

2.1 Overview of Purified Terephthalic Acid (PTA):

Purified Terephthalic Acid (PTA) is a crucial intermediate in the production of polyethylene terephthalate (PET), a widely used polymer with various industrial applications. PTA is obtained through the purification of Crude Terephthalic Acid (CTA), which is derived from the oxidation of para-xylene. PTA possesses superior chemical and physical properties, making it suitable for manufacturing high-quality PET products. The chemical formula of Terephthalic Acid is $C_8H_6O_4$.

PTA serves as a key building block for PET production, contributing to its mechanical strength, thermal stability, and chemical resistance. The quality and purity of PTA significantly impacts the performance and properties of PET-based materials. Achieving a high level of PTA purity, such as 99.98%, ensures the production of PET with enhanced mechanical properties and reduced defects.

2.2 CTA to PTA Conversion Method:

Crude Terephthalic acid is converted Pure Terephthalic via Hydrogenation of the impurities present in the impurities present in the crude variant. These impurities include polyaromatic compounds which are responsible for discoloration of the Crude Terephthalic, and their presence is deemed undesirable in the final product. Moreover 4-Carboxybenzaldehyde is a terminating group in the polymerization of PET for which purified terephthalic acid is used as a monomer. Hydrogenation of polyaromatic colored impurities converts them into colorless forms while 4-CBA is converted into para-toluic acid which can be separated via crystallization to yield Purified Terephthalic Acid.

The Reactions for the conversion of the conversion of CTA to PTA is given below:

$$\text{4-CBA} + H_2 \longrightarrow \text{4-HMBA} + H_2 \longrightarrow \text{p-TA} + H_2O$$

$$\text{Colored impurities + Hydrogen} \longrightarrow \begin{array}{l}\text{Non-colored impurities or}\\ \text{Soluble colored Impurities}\end{array}$$

2.3 Crystallization Techniques in PTA Production:

Crystallization plays a vital role in the production of high-purity Purified Terephthalic Acid (PTA). Various crystallization techniques have been explored to optimize the process and improve the purity of the final product. These techniques include cooling crystallization, antisolvent crystallization, and reactive crystallization.

Cooling crystallization involves controlled cooling of the CTA solution to induce the formation of PTA crystals, which are then separated and washed to remove impurities. Antisolvent crystallization involves the addition of a suitable antisolvent to the CTA solution, promoting the precipitation of PTA crystals. Reactive crystallization combines chemical reactions and crystallization, where an additional reactant is introduced to enhance the purity and yield of PTA crystals.

2.3.1 Cooling Crystallization:

Crude Terephthalic Acid (CTA) dissolved in a suitable solvent (such as water) is subjected to controlled cooling. As the solution cools, Purified Terephthalic Acid (PTA) crystals start to form, which can be separated from the solvent through filtration or other separation techniques.

2.3.2 Antisolvent Crystallization:

In antisolvent crystallization, CTA is dissolved in a solvent (such as water), and an antisolvent (e.g., a polar organic solvent) is added to the solution. The addition of the antisolvent reduces the solubility of PTA, leading to the precipitation of PTA crystals. The PTA crystals can then be separated from the solvent-antisolvent mixture.

2.3.3 Reactive Crystallization:

Reactive crystallization involves the use of a reactive additive or reactant during the crystallization process. The reactant reacts with CTA to form PTA crystals and generates a byproduct. This process helps in enhancing the purity and yield of the PTA crystals.

2.3.4 Evaporative Crystallization:

The target component is first dissolved in a solution before the evaporative crystallization process can begin. The temperature of the solution is raised above the solvent's boiling point. The concentration of the solute rises as the solvent evaporates. The solute will start to crystallize when its concentration reaches a critical level. The temperature of the solution, the solute's concentration, and the presence of contaminants are only a few of the variables that affect how quickly crystals form. The temperature of the mixture needs to be high enough for the solvent to evaporate quickly while not being too high to cause the solute to break down. The solute concentration must be sufficient to encourage crystallization without growing crystals to unacceptably large sizes. It's crucial to utilize a pure solvent and solution because impurities can hinder crystallization. Once the crystals have developed, the mother liquor (the remaining solution) is removed from the crystals. Filtration, centrifugation, or sedimentation can be used to accomplish this. The next step is to wash away any remaining contaminants from the crystals. After being cleaned, the crystals are dried and packaged. A flexible and effective crystallization technique is evaporative crystallization. It is a crucial procedure in the food, chemical, and pharmaceutical sectors and is used to make a wide range of products.

2.3.5 Flash Crystallization:

A phenomenon called flash crystallization occurs when a solvent evaporates, rapidly cooling a solution. The solute precipitates out of solution as tiny, homogeneous crystals as a result of the rapid cooling. A variety of crystals, including those for pharmaceuticals, food additives, and industrial chemicals, can be created using the flexible technique of flash crystallization. The required component is first dissolved in a solution before flash crystallization can take place. The liquid is poured into a flash crystallizer, which sprays it into a lower-pressure chamber. The temperature of the solution rapidly decreases as the solvent evaporates. The solute will precipitate out of solution when the solution's temperature hits its saturation point. The temperature of the solution, the solute's concentration, and the size of the droplets are only a few of the variables that affect how quickly crystals form in a flash crystallizer. The solution's temperature needs to be low enough to encourage crystallization while not being too low to cause the solute to crystallize too slowly. The solute concentration must be sufficient to encourage crystallization without growing crystals to unacceptably large sizes. The size of the droplets should be just right to encourage nucleation while preventing them from dissipating too quickly and preventing crystallization.

CHAPTER 3

PROCESS OVERVIEW

3.1 Process Flow Diagram

The PFD, which shows the order of unit operations and the flow of materials, provides a visual picture of the PTA production process. The PFD describes the primary machinery and significant processes in the conversion of CTA to pure terephthalic acid.

The CTA feed enters the system at a specific flow rate to start the PFD. The water serves as the solvent as it is combined with the CTA feed in a feed tank. Centrifugal pumps are then used to transfer the mixture to the packed bed reactor (PBR), where CTA is transformed into PTA. The creation of PTA crystals occurs in the PBR as a result of the required reactions being facilitated by the palladium-coated carbon catalyst.

Following PBR, the reaction mixture proceeds through flash evaporation in the crystallizers. This lowers the pressure and temperature of the reaction mixture, which encourages the growth of PTA crystals. A centrifuge is then used to separate the PTA crystals from the liquid phase, and the waste filtrate is sent to a recycle tank as mother liquor. To be processed further, the mother liquor is recycled back into the feed tank.

After being extracted from the centrifuge, the PTA crystals are passed to a steam tube rotary drier, where extra moisture is drained away to produce dried PTA crystals. The PTA crystals are recovered after drying and may then be packaged or processed further.

The process flow diagram offers a thorough breakdown of the PTA manufacturing process, enabling a better understanding of the machinery used and the order of steps.

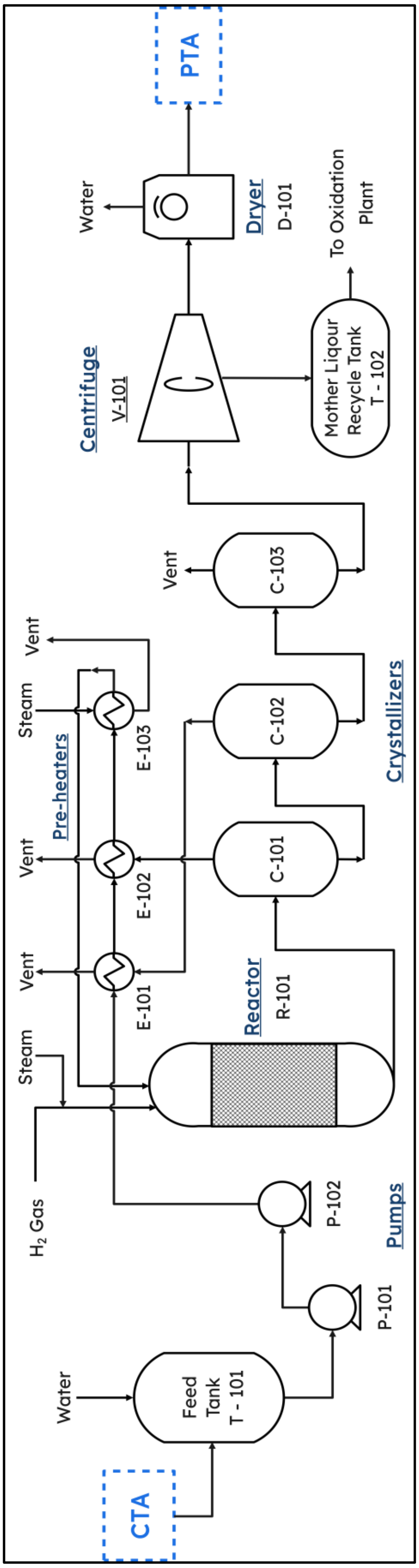


Figure 3: Process Flow Diagram

3.1.1 Process Description

The synthesis of high-quality Purified Terephthalic Acid (PTA), with a target purity of 99.98%, was the goal of the experimental design, which attempted to optimize the crystallization process. The impacts of various process factors on the crystallization process were examined through a series of laboratory-scale studies.

Three crystallizers and a packed bed reactor comprised the experimental setup. Crude terephthalic acid (CTA), which was fed into the process at a rate of 1667 kg per hour and contained impurities like 4-Carboxybenzaldehyde (4-CBA) at a rate of 4.18 kg per hour, was the initial feed. A water feed rate of 3889 kg/hr and a hydrogen feed rate of 0.15 kg/hr were added to the CTA feed. For the intended chemical reactions to occur, the packed bed reactor created the requisite conditions.

The conversion of CTA to PTA and the elimination of impurities were made possible by operating the reactor under a particular temperature and pressure settings. Flash evaporation took place in the packed bed reactor, which resulted in a drop in temperature and pressure. This phenomenon contributed to the development of PTA crystals in a suitable environment.

The effluent stream was sent to the heat exchanger after the reaction in the packed bed reactor. With a flow rate of 722 kg/hr of steam, the heat exchanger enabled cooling and heat transfer. The system's temperature could be controlled, and its energy could be recovered thanks to the usage of steam.

The stream was then routed toward the three crystallizers after the heat exchange procedure. To cause PTA to crystallize, the crystallizers used cooling methods such flash evaporations. The temperature dropped as a result of the flash evaporations, which involved lowering the pressure in the crystallizers. The production of PTA crystals was aided by the drop in temperature while impurities were left behind. The residence duration, temperature, and flash evaporation procedures were all optimized in the design of the crystallizers for quick crystal formation and separation.

The PTA crystals were moved from the crystallizers to a centrifuge for filtration. The mother liquor, which contained undesired contaminants and leftover solvents, was successfully removed from the crystals by the centrifuge. The PTA crystals were separated, and after that they were transferred to a steam tube rotary dryer to finish drying and get rid of any remaining moisture.

The mother liquor, or undesirable filtrate, was simultaneously gathered and sent to a recycle tank. This mother liquor, which contained contaminants that had been dissolved, was recycled back to the feed tank and then put back into the system. This recycling procedure reduced waste and improved workflow.

3.2 Raw Material

3.2.1 Crude Terephthalic Acid (CTA)

With the molecular formula $C_8H_6O_4$, crude terephthalic acid (CTA), also known as CTA, was used as the initial substance for the crystallization procedure. Impurities such 4-Carboxybenzaldehyde (4-CBA) and colored impurities, which were known to impair the purity of the final PTA product, were present in the CTA utilized in the experiment. To get the desired level of PTA purity, it was essential to get rid of these contaminants.
Terephthalic acid (PTA), a crucial ingredient in the manufacturing of polyester, is produced from crude terephthalic acid (CTA), a byproduct of the process. CTA is typically created by oxidizing p-xylene and includes a variety of impurities, such as:

- 4-carboxybenzaldehyde (4-CBA)
- p-toluic acid (p-TOL)
- Benzoic acid (BZ)
- Colored polyaromatics

Depending on the manufacturing method, the impurity levels in CTA can vary, although they are normally in the range of 2000–5000 ppm for 4-CBA and 100–200 ppm for p-TOL and BZ. Although the colored polyaromatics are frequently found at smaller concentrations, they can give CTA a yellow or brown hue.

The contaminants in CTA may cause a variety of detrimental outcomes, such as:

- Lowering PTA's purity
- Increasing PTA's viscosity
- Resulting in PTA's discoloration
- Lowering the polyester yield

For these reasons, it's crucial to clean the CTA of contaminants before converting it to PTA. The most popular technique for purging contaminants from CTA is hydropurification, which transforms the pollutants into innocuous molecules using a catalyst.

The colorless liquid 4-carboxybenzaldehyde (4-CBA) has a pungent odor. It is a monocarboxylic acid that is created when p-xylene is oxidized. Although 4-CBA is a relatively insignificant contaminant in CTA, it can significantly affect the PTA's color and purity.

The colorless liquid p-toluic acid (p-TOL) has a pungent odor. It is a monocarboxylic acid that is created when toluene is oxidized. Although p-TOL is a relatively insignificant contaminant in CTA, it can significantly affect PTA's color and purity.

White solid benzoic acid (BZ) has a faint odor. It is a monocarboxylic acid that is created when benzene is oxidized. Although BZ is a relatively insignificant contaminant in CTA, it can significantly affect the PTA's color and purity.

A class of chemicals known as colored polyaromatics are created when organic matter burns partially. They usually exist in little amounts in CTA, but they can give it a yellow or brown hue. The yield of polyester might also be adversely affected by colored polyaromatics.

3.2.2 Hydrogen

The hydropurification technique creates PTA from CTA using hydrogen as a basic ingredient. In this procedure, the contaminants in CTA are converted to safe chemicals by passing hydrogen across a catalyst like palladium on carbon. These steps often involve the reduction of 4-carboxybenzaldehyde (4-CBA), p-toluic acid (p-TOL), and benzoic acid (BZ) impurities.

As it eliminates contaminants that could have a detrimental effect on PTA quality, the hydropurification process is a crucial stage in the production of PTA. Because the impurities are changed into chemicals that may be recycled or sold, the hydropurification procedure also boosts the yield of PTA.

It is only recently that hydrogen has been used in the hydropurification procedure. In the past, a procedure known as distillation was used to get rid of the contaminants in CTA. However, distillation consumes a lot of energy and can result in PTA that is tainted with minute amounts of contaminants. The hydropurification procedure creates PTA that is free of contaminants while using less energy.

There are several advantages to using hydrogen in the hydropurification process. First off, compared to distillation, it is a more energy-efficient process. Second, it generates PTA that is impurity-free. Third, since there are no hazardous emissions produced, it is a more environmentally friendly procedure than distillation.

A crucial component in the creation of high-quality PTA is the use of hydrogen in the hydropurification procedure. In comparison to conventional procedures, the hydropurification procedure effectively removes contaminants from CTA while using less energy and harming the environment. As a result, the hydropurification method that uses hydrogen to produce PTA is becoming more widely used.

3.2.3 Water/Steam

When PTA is made from CTA, water is not a raw ingredient. Nevertheless, it serves as a solvent throughout the purifying process. After being crystallized, crude terephthalic acid (CTA) is then dissolved in water. In order to turn contaminants into para-toluic acid, hydrogen is added to the solution, and it is then passed through a hydrogenation reactor. After being cleaned, PTA is crystallized and dried.

There are various benefits to using water in the purifying process. It is a relatively cheap solvent, to start with. Second, it is safe for the environment and non-toxic. Thirdly, it aids in the removal of contaminants that are challenging to eliminate through mechanical means, such as 4-carboxybenzaldehyde (4-CBA).

There are certain drawbacks to using water in the purification phase as well. First off, it might raise the price of production. Second, it might make the process use more energy. Third, it can make the equipment more susceptible to rust.

3.2.4 Nitrogen

Nitrogen is applied in a variety of different methods to facilitate the enhancement of the purification process when separating PTA from CTA. In the first place, it is utilized in nitrogen blanketing, which involves the formation of a defensive layer all the way around the PTA distillation column. This serves the purpose of preventing the oxidation of PTA, which can lead to polymerization as well as the generation of byproducts that are not acceptable. Additionally, in the PTA crystallization unit, nitrogen sparging is used to prevent the production of air bubbles within the PTA. This is done by removing excess nitrogen from the PTA. Cloudiness in the PTA can be caused by air bubbles, which can also lead to the production of undesirable byproducts. In addition, nitrogen is used in the process known as nitrogen drying, which removes any trace of moisture that may have been present in the PTA product. PTA is susceptible to breakdown over time when exposed to moisture, which also promotes the production of undesirable byproducts. Because nitrogen is an inert gas, it does not react with other compounds. As a result, it is an excellent candidate for the purification of PTA. In addition, nitrogen is available at a price that is not prohibitively expensive, making it a viable option for use in purifying procedures. Nitrogen's applications in the PTA purification process give many benefits to the process as a whole, including the prevention of oxidation, the prevention of the production of air bubbles, and the effective drying of the PTA product.

3.3 Equipment

3.3.1 Feed Tank

To store, regulate the flow of raw materials, and their mixing, into the process, feed tanks are employed. In this instance, water and crude terephthalic acid (CTA) are stored in two feed tanks. The feed tanks make sure that the raw ingredients enter the process in a steady and controlled flow, enabling precise dosing and mixing.

3.3.2 Centrifugal Pumps

Slurry pumps and centrifugal pumps are crucial tools in the process of turning crude terephthalic acid (CTA) into purified terephthalic acid (PTA). Slurry pumps are used to move slurries of CTA and other chemicals throughout the purification process, whereas centrifugal pumps are used to move CTA from storage tanks to the purifying process.

Fluids are moved by centrifugal force in centrifugal pumps. High-speed impeller rotation of the pump causes centrifugal force, which forces the fluid outward. Following that, the fluid exits the discharge outlet after passing through the pump casing. Applications requiring large volume, low pressure often call for centrifugal pumps.

Slurry pumps are a particular kind of centrifugal pump made for handling fluids with solid particles. The solids may be heavier than the fluid or they may be floating in them. Slurry pumps are made to handle higher pressures and often have a larger impeller than centrifugal pumps.

Centrifugal pumps are utilized to move CTA from storage tanks to the purifying process in the manufacturing of PTA. The PTA is created by the reaction of the CTA with oxygen after the CTA has been heated to a high temperature and injected into a reactor. After cooling, the PTA is poured into a distillation column to be filtered.

During the purification procedure, slurries of CTA and other chemicals are moved using slurry pumps. The slurries are treated in a number of tanks and reactors after being pumped there. The high temperatures and pressures of the purifying process must be handled by the slurry pumps.

Slurry pumps and centrifugal pumps are crucial tools in the manufacture of PTA. These pumps are employed throughout the industrial process to move liquids and slurries. High temperatures, high pressures, and caustic chemicals must all be handled by the pumps in order to produce PTA.

- Increased efficiency: Centrifugal pumps and slurry pumps can move fluids and slurries more efficiently than other types of pumps. This can lead to reduced energy costs and improved production rates.
- Improved safety: Centrifugal pumps and slurry pumps are designed to handle hazardous chemicals. This can help to reduce the risk of accidents and injuries.
- Increased reliability: Centrifugal pumps and slurry pumps are built to last. This can help to reduce maintenance costs and improve uptime.

To make it easier to transfer the CTA and water from the feed tanks to the packed bed reactor, two centrifugal pumps are used. Due to their capacity for handling large flow rates and supplying enough pressure to move the fluids through the process, centrifugal pumps are used. They provide dependable and effective functioning, guaranteeing a constant and stable supply of raw resources.

3.3.3 Pre-Heater

Preheaters are used to heat the crude terephthalic acid (CTA) before it enters the hydropurification reactor in the purification of PTA from CTA.

There are numerous various kinds of preheaters, but the shell and tube heat exchangers are the most typical ones used in the manufacturing of PTA. A shell and tube heat exchangers are made up of a number of tubes encased in a shell. The heat source, which is steam from the crystallizers, is cycled through the shell while the CTA is pumped through the tubes.

Preheaters are used in the manufacturing of PTA because they have many advantages. First, preheaters can aid in increasing the industrial process' efficiency. The reaction can happen faster and at a lower temperature by heating the CTA before it enters the hydropurification reactor. The TPA is soluble in water at higher temperatures, which speeds up the chemical process. Due to the high temperature and pressure requirements in the industrial facility, shell and tube heat exchangers are used. Three heat exchangers are used in the final design to help with heat transfer and temperature regulation.

3.3.4 Packed Bed Reactor

Chemical reactions that use solid catalysts in tubes or vessels are known as packed bed reactors. The catalyst encourages the chemical interaction between the reactants as they flow through the bed. Packed bed reactors are frequently utilized while converting CTA into PTA.

Terephthalic acid (TPA) is initially produced by oxidizing crude terephthalic acid (CTA), which is then used to make PTA. After that, the TPA is hydrogenated to create PTA. A palladium catalyst that is crammed into a packed bed reactor catalyzes the hydrogenation reaction.

High pressure and temperature TPA is poured through the bed. The hydrogenation reaction is accelerated by the palladium catalyst, resulting in the conversion of TPA to PTA. After cooling, the PTA is collected.

A sort of packed bed reactor called a trickle bed reactor has the catalyst in the form of a solid and the reactants in the form of a liquid or slurry. In order to produce PTA from CTA, trickle bed reactors are frequently employed since they offer a good balance between efficiency and cost. The hydrogenation of TPA to PTA, the second phase in the production process, is carried out in trickle bed reactors. The trickling bed reactor offers a good approach to evenly distribute the catalyst throughout the liquid phase of the hydrogenation reaction, which is a liquid-phase reaction.

Benefits of producing PTA in trickle bed reactors include:

- High efficiency: Trickle bed reactors can achieve high conversion rates, which can lead to reduced energy costs and improved production rates.
- Low cost: Trickle bed reactors are relatively inexpensive to construct and operate.
- Versatile: Trickle bed reactors can be used for a variety of reactions, which can make them a valuable asset in a chemical plant.

However, employing trickle bed reactors has significant drawbacks as well:

- Pressure drop: Because of the packed bed's potential to significantly lower pressure, maintaining the appropriate flow rate may involve the usage of pumps.
- Deactivation of the catalyst: As the catalyst ages, the reactor's efficiency may be lowered.
- Clogging: The packed bed may become clogged over time, which may lower the reactor's flow rate and efficiency.

Chemical reactors known as trickling bed reactors are adaptable and effective for a range of processes, including the generation of PTA. The possible downsides of employing trickle bed reactors, such as pressure decrease, catalyst deactivation, and clogging, must be understood, though.

3.3.5 Crystallizer

The process of turning a liquid or supersaturated solution into a solid crystalline form is known as crystallization. Crystallizers are used to create pure PTA crystals from the crude PTA solution for making PTA from CTA.

There are several different kinds of crystallizers, but the flash crystallizer is the one used most frequently in the manufacture of PTA. A flash crystallizer is a particular kind of continuous crystallizer where crystallization is induced by rapidly cooling or concentrating the supersaturated solution.

There are various advantages to using flash crystallizers in the manufacture of PTA. First, PTA crystals with a restricted size distribution can be produced in high-quality using flash crystallizers. This is significant because the PTA crystals' size and form can have an impact on the characteristics of the polyester resin that is made from them. Second, flash crystallizers have great throughput capabilities. Given that PTA manufacture is a high-volume process, this is significant.

Thirdly, flash crystallizers use a fair amount of energy. This is significant since the process of producing PTA consumes a lot of energy.

Three crystallizers are used in the procedure to encourage the crystallization of PTA from the reaction mixture. In order to establish ideal circumstances for PTA crystal formation, these crystallizers use cooling techniques such flash evaporation. The temperature dropped as a result of the flash evaporations, which involved lowering the pressure in the crystallizers. The production of PTA crystals was aided by the drop in temperature while impurities were left behind. The residence duration, temperature, and flash evaporation procedures were all optimized in the design of the crystallizers for quick crystal formation and separation.

3.3.6 Centrifuge

A disk centrifuge is a type of centrifuge that separates solids from liquids using a revolving disk. The creation of PTA from CTA can be accomplished using the disk centrifuge, which is a flexible and effective piece of machinery.

Disk centrifuges are used to separate the raw PTA crystals from the mother liquor during the manufacturing of PTA from CTA. To create high-purity PTA, the raw PTA crystals are then cleaned and dried.

The disk centrifuge comprises of a revolving disk that has been punctured with numerous holes. A bowl that holds the separated solids surrounds the disk. Centrifugal force drives the liquid through the pores in the disk. The liquid is released from the centrifuge while the solids are collected in a dish.

A dependable and effective piece of machinery with numerous applications is the disk centrifuge. Because it can effectively separate the raw PTA crystals from the mother liquor, the disk centrifuge is especially well-suited for producing PTA from CTA.

3.3.7 Dryer

An industrial dryer called a rotating steam tube drier uses steam to evaporate moisture from a solid substance. A flexible and effective piece of machinery, the rotary steam tube dryer can be utilized for a variety of tasks, including the creation of PTA from CTA.

The raw PTA crystals are dried in rotary steam tube dryers during the process of producing PTA from CTA. Using a disk centrifuge, the mother liquor is first removed from the crude PTA crystals. The rotary steam tube drier is then fed with the raw PTA crystals.

A spinning drum that is heated by steam is the main component of the rotary steam tube dryer. The interior of the drum is covered in unprocessed PTA crystals. The steam warms the unprocessed PTA crystals while the drum turns. A vacuum device is then used to evaporate and remove the moisture from the raw PTA crystals in the drum.

A dependable and effective piece of machinery with numerous applications is the rotary steam tube dryer. The rotary steam tube dryer's great effectiveness at drying the raw PTA crystals makes it especially well-suited for the production of PTA from CTA.

3.4 Catalyst

Catalysts are essential for improving the selectivity and conversion efficiency of the conversion processes in the synthesis of purified terephthalic acid (PTA) from crude terephthalic acid (CTA). Palladium-coated carbon is one of the catalysts that is frequently employed in the process. In many chemical processes, including the oxidation of aromatic compounds, palladium catalysts have demonstrated exceptional performance.

Palladium coated carbon was selected as a catalyst in the packed bed reactor (PBR) because of its distinct qualities. High catalytic activity, stability, and selectivity are characteristics of palladium. To provide a broad surface area and increase the exposure of active areas, the metal is supported on carbon. Palladium on the carbon surface accelerates the desired conversion processes by facilitating the adsorption of reactant molecules.

The CTA to PTA conversion process benefits greatly from the palladium-coated carbon catalyst. First of all, it offers excellent conversion efficiency, enabling a full conversion of CTA into PTA. The catalyst guarantees that impurities are eliminated and that only pure PTA crystals are produced. It also has good selectivity, allowing for the required reactions while limiting unfavorable side effects. Higher product yields and better product quality result from this.

Palladium-coated carbon catalysts are also renowned for their durability and resistance to deactivation. By ensuring consistent performance during protracted periods of operation, this lowers the requirement for frequent catalyst replacement and raises the process' overall effectiveness. The catalyst is ideal for the particular conditions in the PTA manufacturing since it has a high tolerance to process factors like temperature and pressure.

Numerous studies and pieces of research support the choice of the palladium coated carbon catalyst for the packed bed reactor (PBR). For instance, a study by Zhang et al. (2018) showed the value of palladium catalysts in aromatic chemical hydropurification processes.

CHAPTER 4

MATERIAL AND ENERGY BALANCE

4.1 Material Balance

The material balance involves tracking the quantities of CTA, water, and other raw materials entering the system and comparing them with the quantities of PTA crystals and byproducts produced. The material balance equations are typically based on the principle of conservation of mass and can be expressed as follows:

Basis: 1-hour steady state operation on an open system

General Material Balance Equation:

Units are (kg/hr)

$$Mass\ In - Mass\ Out + Generation - Consumption = Accumulation$$

$$Incoming\ Mass = Outgoing\ Mass$$

4.1.1 Feed Tank

- $Mass\ In = Mass\ Out$
- 4-CBA 2500ppm
- Water for 30% slurry

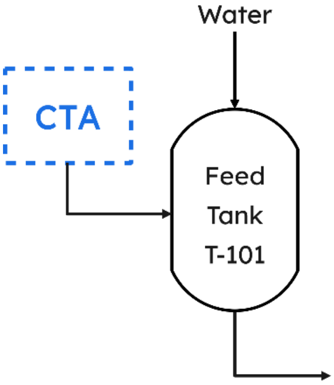


Figure 4: Feed Tank

Table 1: Feed Tank Material Balance

Components	CTA To Feed Tank	Water To Feed Tank	Feed Tank Outlet
TA (kg/hr)	1666.67	-	1666.67
Water (kg/hr)	-	3888.89	3888.89
4-CBA (kg/hr)	4.18	-	4.18
P-TA (kg/hr)	-	-	-
Hydrogen (kg/hr)	-	-	-
Total (kg/hr)	1670.84	3888.89	5559.73

4.1.2 Pumps

- $Mass\ In\ =\ Mass\ Out$

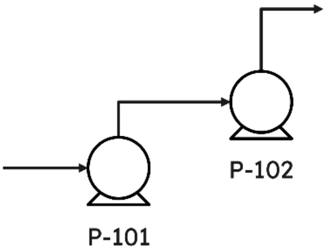


Figure 5: Pumps

Table 2: P-101 Parameters

P–101	
Flowrate (kg/hr)	5559.7
Pressure inlet (bar)	1.5
Pressure outlet (bar)	10.5
Specific volume (m^3/kg)	0.00112
Work in (kJ/hr)	7102.98

Table 3: P-102 Parameters

P–102	
Flowrate (kg/hr)	5559.7
Pressure at inlet (bar)	10.5
Pressure at outlet (bar)	80
Specific volume (m^3/kg)	0.00113
Work in (kJ/hr)	54575.16

4.1.3 Pre-heater

- $Mass\ In\ =\ Mass\ Out$

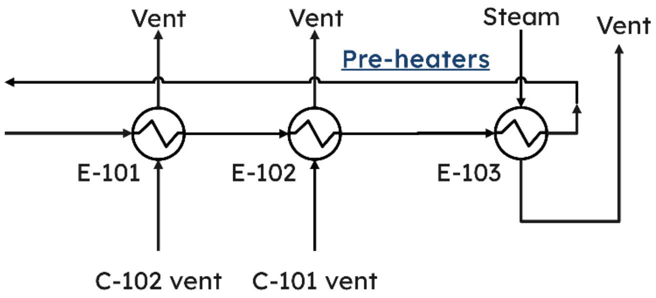


Figure 6: Heat Exchangers

Table 4: Heat Exchanger E-101 Material Balance

E-101	
Flowrate	5559.6
T$_{In}$	109
T$_{out}$	160
Delta T	51
Cp	3.3
Heat Duty	935680.7
Latent Heat of Steam from C-2	1985
Steam Required	471.3757

Table 5: Heat Exchanger E-102 Material Balance

E-102	
Flowrate	5559.6
T$_{In}$	160
T$_{out}$	220
Delta T	60
Cp	3.4
Heat duty	1134158.4
Latent Heat of Steam from C-1	1748
Steam Required	648.83

Table 6: Heat Exchanger E-103 Material Balance

E-103	
Flowrate	5559.6
T$_{in}$	220
T$_{out}$	280
Delta T	60
Cp	3.7
Heat duty	1234231

Latent Heat of HP Steam	1372.97
Steam Required	722

4.1.4 Reactor

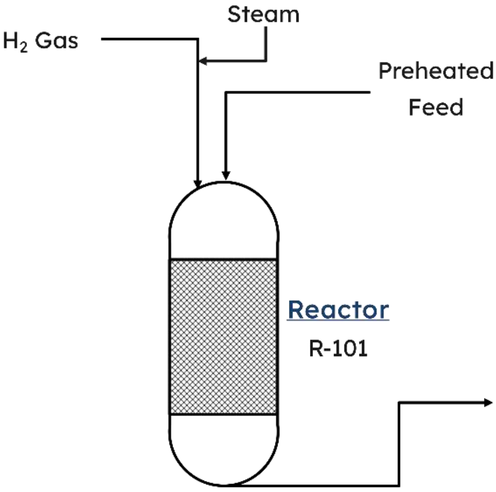


Figure 7: Reactor R-101

Material Balance:

Steady state operation

Mass Out = Mass in + Generation – Consumption

Reaction:

$4\text{-CBA} + 2H_2 \rightarrow \text{P-Toluic Acid} + H_2O$

4-CBA conc. in feed: 2500ppm

4-CBA conc. required at outlet: 25ppm

HP steam is used to heat H_2 gas.

Table 7: Reactor R-101 Material Balance

Components	Feed	H_2 Feed	HP Steam	Mass Consumed	Mass Generated	Reactor Outlet
TA (kg/hr)	1666.67	-	-	-		1666.67
Water (kg/hr)	3888.89	-	4.40	-	0.50	3893.78
4-CBA (kg/hr)	4.18	-	-	4.14	-	0.04
P-Toluic Acid (kg/hr)	-	-	-	-	3.75	3.75
Hydrogen (kg/hr)	-	0.11	-	0.11	-	-
Total (kg/hr)	5559.73	0.11	4.40	4.25	4.25	5564.24
Temperature (°C)	280	280	303	-	-	280
Pressure (bar)	80	80	90	-	-	80

4.1.5 Crystallizer

- $Mass\ In\ =\ Mass\ of\ water\ ejected\ as\ vapor\ +\ Mass\ of\ liquid\ at\ outlet$
- T = T_{sat} at the pressure of crystallizer

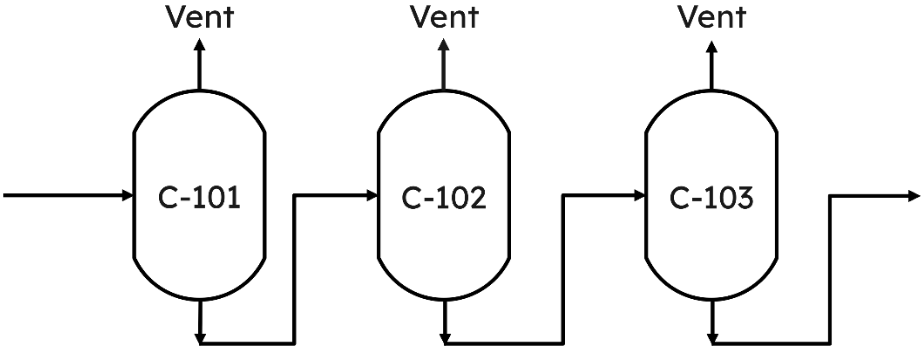


Figure 8: Crystallizers

Table 8: Crystallizers Material Balance

Components	To C1	C1-V	C1-L	C2-V	C2-L	C3-V	C3-L
TA (kg/hr)	1666.67	-	328.67	-	30.53	-	6.72
Water (kg/hr)	3893.78	766.83	3126.95	469.67	2657.28	205.05	2452.23
4-CBA (kg/hr)	0.0417	-	0.0417	-	0.0417	-	0.0417
P-TA (kg/hr)	3.75	-	3.75	-	3.75	-	3.75
TA solid (kg/hr)	-	-	1337.99	-	1636.14	-	1659.95
H2 (kg/hr)	-	-	-	-	-	-	-
Total (kg/hr)	5564.24	-	4797.41	-	4327.74	-	4122.69
Temperature (°C)	280	243.4	243.4	188.2	188.2	151.9	151.9
Pressure (bar)	80	34.5	34.5	11	11	4	4

4.1.6 Centrifuge

- $Mass\ In\ =\ Mass\ Out$

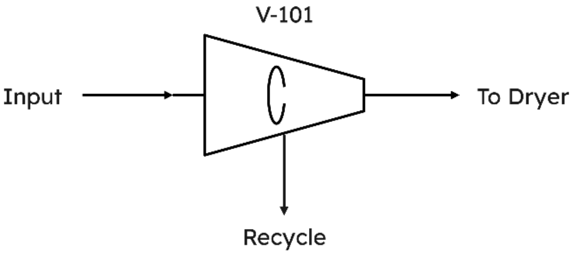


Figure 9: Centrifuge V-101

Table 9: Centrifuge V-101 Material Balance

Components	To Centrifuge	Recycle	To Dryer
TA (kg/hr)	6.72	6.72	-
Water (kg/hr)	2452.23	2207.01	245.22
4-CBA (kg/hr)	0.0417	0.0208	0.0208
P-TA (kg/hr)	3.75	3.375	0.375
TA solid (kg/hr)	1659.95	-	1659.95
Total (kg/hr)	4122.69	2217.13	1905.57
Temperature (°C)	151.9	99.6	99.6
Pressure (bar)	4	1	1

4.1.7 Dryer

- $Mass\ in\ =\ Mass\ out$

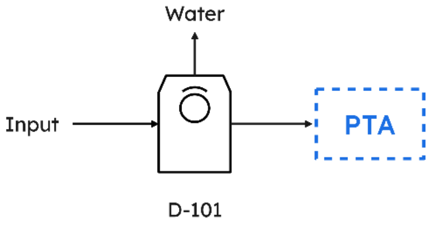


Figure 10: Dryer D-101

Table 10: Dryer D-101 Material Balance

Components	To Dryer	Moisture Removed	Product
TA (kg/hr)	-	-	-
Water (kg/hr)	245.22	244.98	0.245

4-CBA (kg/hr)	0.0208	-	0.0208
P-TA (kg/hr)	0.375	-	0.375
TA solid (kg/hr)	1659.95	-	1659.95
Total (kg/hr)	1905.57	244.98	1660.59
Temperature (°C)	99.6	105	105
Pressure (bar)	1	1	1

4.2 Energy Balance

Quantifying the energy inputs, such as heat and steam, and the energy outputs, such as waste heat, constitutes the energy balance. Based on the idea of energy conservation, the energy balance equations have the following form:

$$Energy\ In\ =\ Energy\ Out$$

The heat transfer within different unit operations, such as heat exchangers and steam generation, is considered by the energy balance equations. The energy balance equations also consider the energy content of the feed and products as well as the energy needed for chemical processes.

Depending on the unit operations and overall process design, the specific energy balance equations employed in the PTA production process may vary. The heat transfer equations, enthalpy calculations, and reaction energy calculations relevant to the chemical reactions involved in PTA manufacturing are some of the equations that are frequently utilized.

It is feasible to evaluate the general process efficiency, identify potential for improvement, and maximize resource use in the PTA production process by carrying out a thorough material and energy balancing.

4.2.1 Feed Tank

- $\Delta H = 0$
- $Enthalpy\ In = Enthalpy\ Out$

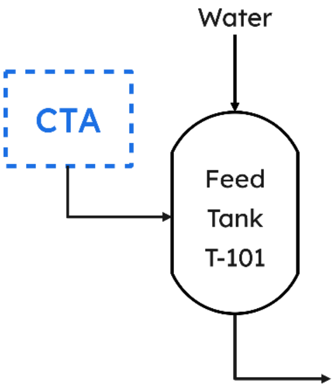


Figure 11: Feed Tank

This equation represents the calculation of the final temperature (T_f) resulting from the mixing of two fluid streams with different temperatures. The equation considers the mass flow rates and specific heat capacities of the fluids to determine the final temperature.

$$T_f = (m_1 c_{p1} t_1 + m_2 c_{p2} t_2)/(m_1 c_{p1} + m_2 c_{p2})$$ (Smith, Van Ness, & Abbott, 2005)

T_f: Final temperature of the system
m_1, m_2: Mass flow rates of the fluids
c_{p1}, c_{p2}: Specific heat capacities of the fluids
t_1, t_2: Initial temperatures of the fluids (Smith, Van Ness, & Abbott, 2005)

Table 11: Feed Tank Energy Balance

Feed Tank	CTA To Feed Tank	Water To Feed Tank	Feed Tank Outlet
Flowrate (kg/hr)	1670.80	3888.80	5559.60
Cp (kJ/kg°C)	1.34	4.18	3.33
Temperature (°C)	80.00	110.00	103.20
Pressure (bar)	1.00	1.50	1.50

4.2.2 Pumps

This equation represents the calculation of the work or power output (W) based on the mass flow rate, volume, and pressure difference of the fluid. The efficiency (η) accounts for any losses or inefficiencies in the system and is given as a percentage.

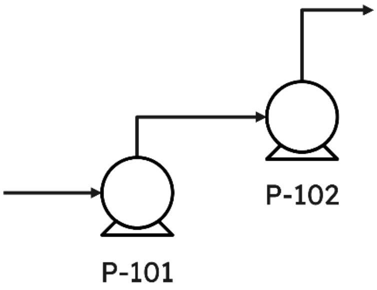


Figure 12: Pumps

$$W = \frac{mV(P_2 - P_1)}{\eta} \; ; \qquad \eta = 80\%$$

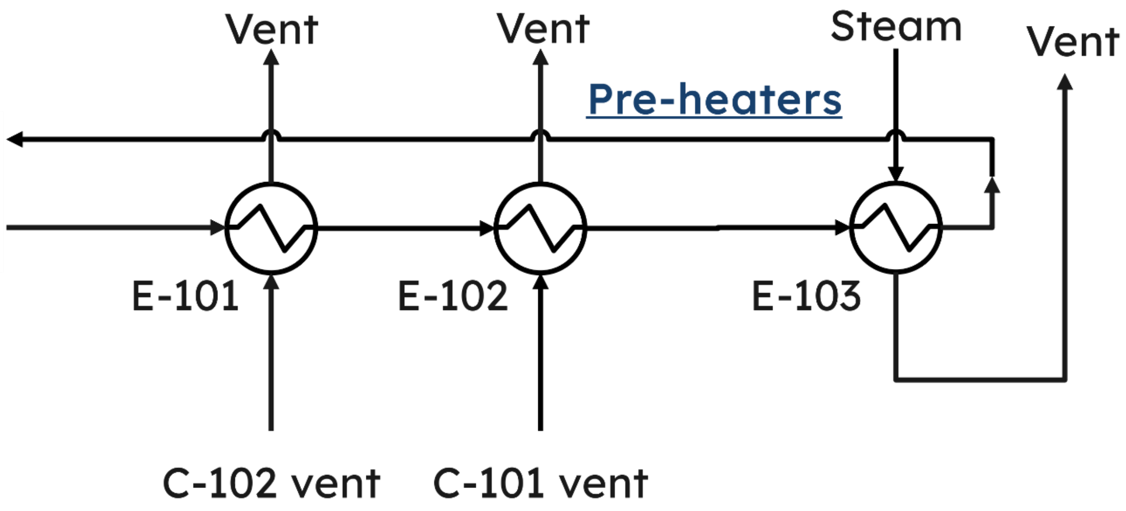
W: Work or power output
m: Mass flow rate of the fluid
V: Volume of the fluid
P_2, P_1: Pressure at the outlet and inlet, respectively
η: Efficiency of the system (given as 80% in the example)

Table 12: Pumps P-101 and P-102 Energy Balance

P-101	
Flowrate (kg/hr)	5559.7
Pressure inlet (bar)	1.5
Pressure outlet (bar)	10.5
Specific volume (m^3/kg)	0.00112
Work in (kJ/hr)	7102.98

P-102	
Flowrate (kg/hr)	5559.7
Pressure at inlet (bar)	10.5
Pressure at outlet (bar)	80
Specific volume (m^3/kg)	0.00113
Work in (kJ/hr)	54575.16

4.2.3 Pre Heaters



- $Q = mCp(T_f - T_i)$
- $Steam\ Required = Q/\boldsymbol{\lambda}$

Q: Heat transfer or energy transferred to or from the system

m: Mass flow rate of the fluid

C_p: Specific heat capacity of the fluid

T_f: Final temperature of the fluid or system

T_i: Initial temperature of the fluid or system

The subscripts "f" and "i" denote the final and initial conditions or temperatures, respectively. For example, Tf represents the final temperature, and Ti represents the initial temperature.

The equation $Q = mC_p (T_f - T_i)$ represents the energy balance equation, which calculates the amount of heat transferred to or from the system based on the mass flow rate, specific heat capacity, and the temperature difference between the final and initial states.

The equation Steam Required = Q/λ calculates the amount of steam required based on the heat transfer value (Q) divided by the latent heat of vaporization (λ) of the steam.

This equation determines the steam quantity needed to supply the required heat for a specific process.

Table 13: Heat Exchanger E-101 Energy Balance

E-101	
Flowrate	5559.6
T_{In}	109
T_{out}	160
Delta T	51
Cp	3.3
Heat Duty	935680.7
Latent Heat of Steam from C-2	1985
Steam Required	471.3757

Table 14: Heat Exchanger E-102 Energy Balance

E-102	
Flowrate	5559.6
T_{In}	160
T_{out}	220
Delta T	60
Cp	3.4
Heat duty	1134158.4
Latent Heat of Steam from C-1	1748
Steam Required	648.83

Table 15: Heat Exchanger E-103 Energy Balance

E-103	
Flowrate	5559.6
T_{in}	220
T_{out}	280
Delta T	60
Cp	3.7
Heat duty	1234231
Latent Heat of HP Steam	1372.97
Steam Required	722

4.2.4 Reactor

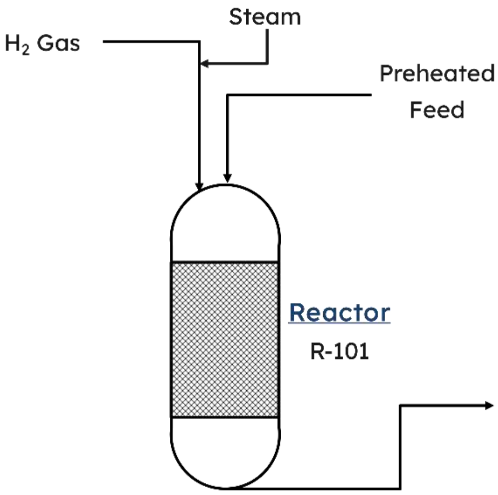


Figure 13: Reactor R-101

$\boldsymbol{Energy\ Balance}$:

- $H_{out} = H_{in} + m_{4\text{-}cba}.H_{rxn}$

  $Since\ m_{4\text{-}cba} <<<< m_{feed}$
- $H_{in} \approx H_{out}$
- $T_{in} \approx T_{out} \approx 280°C$

4.2.5 Crystallizers

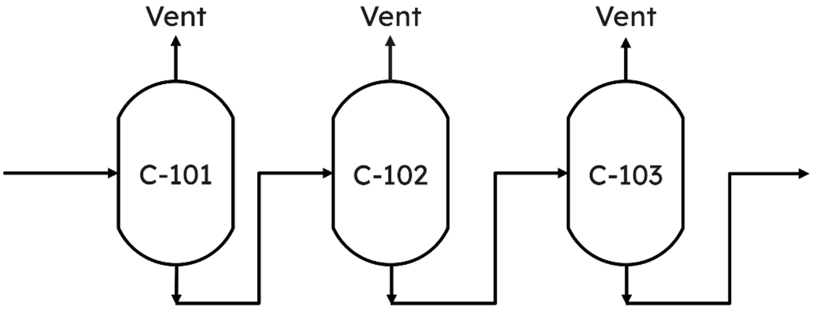


Figure 14: Crystallizers

Simultaneous Material & Energy Balance:

- Mass of Vapor Vented through:
- Flashing at saturation conditions
- Mass of water * Quality
- Quality = (H inlet – H at sat. conditions)/Latent Heat
- Heat evolved from Crystallization.
- (Mass of crystals formed * Heat of crystallization)/Latent Heat
- Mass of Crystals Formed:
- Using changes in solubilities.
- (Change in solubility) * (mass of solvent)

Table 16: Crystallizer C-101 Energy Balance

Data	Feed	C1
Solubility at T_1 (kg/kg)	-	0.44
Solubility at T_2 (kg/kg)	-	0.10
Mass crystalized (kg)	-	1305.20
Heat of crystallization (kJ/kg)	472	-
Heat evolved (kJ)	-	616052.83
Vapour formed from crystallization (kg)	-	356.77
Vapour from flashing (kg)	-	409.67
Latent heat of steam (kJ/kg)	-	1748.81
Enthalpy of liquid at upstream Temperature, Pressure (kJ/kg)	1236	-
Enthalpy of liquid at downstream T_{sat} (kJ/kg)	-	1053.8
Temperature (°C)	280	243.4
Pressure (bar)	80	34.5
Quality (x)	-	0.104
Total Vapour formed (kg)	-	766.83

4.2.6 Centrifuge

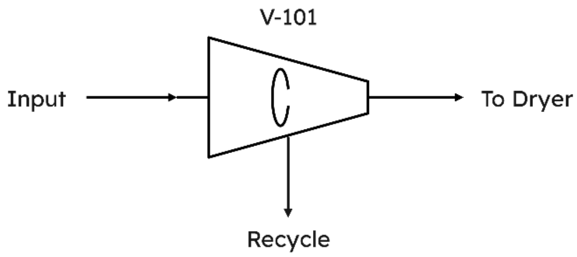


Figure 15: Centrifuge V-101

- Energy Consumption is taken as **25kW**. (From Literature).

4.2.7 Dryer

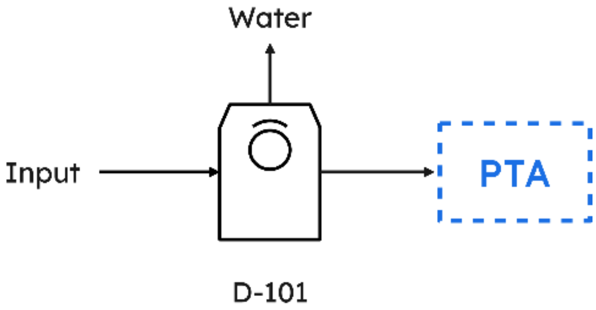


Figure 16: Dryer D-101

- Q = Sensible + Latent heat
- Steam Required = Q/λ
- λ = Latent heat of steam used for heating

Table 17; Dryer D-101 Energy Balance

Component	Moisture removed
Water (kg)	244.98
Water vapor (kg)	24.01
Temperature of feed (°C)	99.6
Temperature of dryer (°C)	105
Heat required for vaporization (kJ)	501275.25
Sensible heat for feed (kJ)	10560
Latent heat of steam (kJ/kg)	2107.61
Steam required (kg)	237.84

CHAPTER 5

EQUIPMENT DESIGN

5.1 Composition List

5.1.1 Shell and Tube geometrical design Considerations

1. Tube Layout and Size: The tube layout and size must be meticulously chosen to provide sufficient heat transfer area and to accommodate the desired fluid flow rates. Tube diameter, length, and pitch have an effect on heat transfer performance and pressure decrease.
2. Baffles and passes: Baffles are used to improve heat transfer by directing fluid flow and generating turbulence. Overall heat transfer coefficient and pressure drop are affected by the number and design of baffles, as well as the configuration of multiple tube passes.
3. Shell side: The design should take into account the fluid properties, flow rates, and pressure decrease on the shell side. Heat transfer efficacy is affected by the selection of shell-side baffles, inlet and outlet configurations, and flow arrangements (such as parallel or counter-flow).
4. Material Selection: Appropriate material selection is necessary to assure compatibility with the process fluids, temperature, and pressure conditions, and to prevent corrosion, erosion, and fouling.

5.1.2 Kern’s Method

Shell and tube heat exchanger design using Kern's method follows a systematic approach to determine the required heat transfer area and size of the heat exchanger appropriately. The method involves several steps:

1) Determine the Overall Heat Transfer Coefficient (U):

- Calculate the individual heat transfer coefficients for the hot and cold fluids inside the shell and tubes based on their properties, flow conditions, and heat transfer mechanisms (convection, conduction).
- Consider fouling factors to account for any surface fouling that may reduce the heat transfer coefficients.
- Calculate the overall heat transfer coefficient (U) using the appropriate equation, such as the Kern method equation for shell and tube heat exchangers.

2) Determine the Logarithmic Mean Temperature Difference (LMTD):

- Identify the temperature profiles of the hot and cold fluids at the inlet and outlet of the heat exchanger.
- Calculate the temperature differences between the fluids at each end and determine the logarithmic mean temperature difference (LMTD) using the appropriate equation based on the flow arrangement (parallel, counter-flow, or crossflow) and the number of shell and tube passes.

3) Determine the Required Heat Transfer Area:

- Calculate the required heat transfer area (A) using the equation: $Q = U \times A \times LMTD$
  where Q is the heat duty or the desired heat transfer rate.
- Rearrange the equation to solve for the required heat transfer area (A):
  $A = Q \div (U \times LMTD)$

4) Perform Iterative Sizing Calculations:

- Estimate the initial heat exchanger dimensions and configuration based on the required heat transfer area.
- Calculate dimensions, considering tube diameter, tube length, number of tubes, and overall dimensions.
- Identifying and calculating the baffle type and spacing.
- Calculate and verify that the pressure decrease across the heat exchanger meets the design specifications.
- If necessary, iterate the sizing calculations to obtain an optimal design.
- The tube side fluid is the CTA mixed to be heated.
- The shell side fluid is the saturated steam (at 35.5 bar) from the second crystallizer C-102.

Stream Properties:

- E-102

Table 18: Stream Properties in E-102

Cold Fluid - Tube Side		Hot Fluid - Shell Side	
Parameters	**Value**	**Parameters**	**Value**
Inlet Temperature (°C)	170	Inlet T (°C)	243
Outlet T (°C)	230	Outlet T (°C)	243
Mass flowrate (kg/hr)	5560	Mass Flowrate (kg/hr)	674
Avg. Cp (kJ/kg.K)	3.6	Cp (kJ/kg.K) vapor	4.145
Avg. density (kg/m^3)	814.5	Density (kg/m^3) vapor	14.9
Avg. viscosity (Cp)	0.24	Viscosity (Cp) vapor	0.0172
Thermal conductivity (W/mK)	0.62	Thermal Conductivity (W/mK)	0.0376

Calculated Parameters:

- E-102

Table 19: E-102 Design Parameters

Parameters	Tube Side	Shell Side
Duty (kW)	329.5	
LMTD (°C)	34.8	
Reynold number	5407	9682
Prandtl number	1.345	-
h (film coefficient)	797	14577
Uc (W/m^{2}K)	690	
Ud (W/m^{2}K)	526	
Rd (m^{2}K/W)	0.0002	
Area (m^2)	18.5	
Pressure Drop (kPa)	2.47	1.88

- The exchanger geometry was chosen based on Aspen Exchanger Design and Rating (EDR) recommendation.
- The design was modified considering operation warnings provided by EDR.

Tube and Shell Side Geometries

Table 20: E-102 Tube-Side Geometry

Tube Side	
Number	108
Length (m)	2.96
OD (m)	0.019
ID (m)	0.015
Pitch	Rotated Sq. Pitch
Passes	1
Material	Stainless Steel

Table 21: E-102 Shell-Side Geometry

Shell Side	
OD (m)	0.319
ID (m)	0.3048
Baffle spacing (m)	0.25
Passes	1
Material	Stainless Steel

The heat exchanger layout as generated EDR is given below based on the above design.

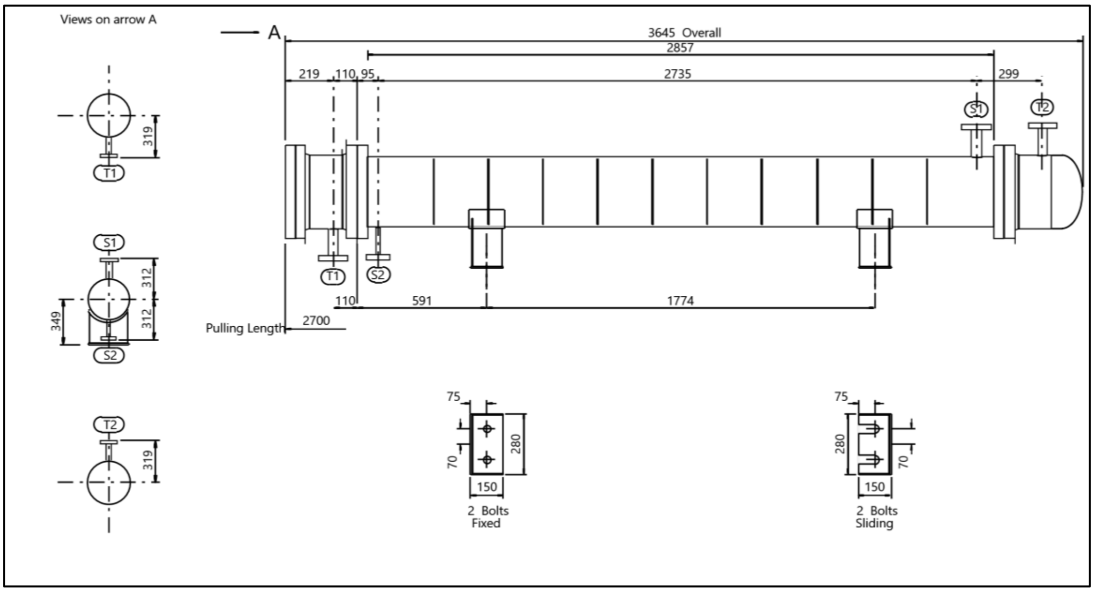


Figure 17: EDR Generated E-102 Layout

5.2 Packed-Bed Reactor Design

A typical Packed bed Reactor is shown and the some of the design considerations are described:

1. Catalyst Bed Design: The design should take into account the selection of an appropriate catalyst and its injection density within the reactor. The height, diameter, and void fraction of the bed influence the reaction kinetics, pressure decrease, and heat transfer efficiency.
2. Configuration of the Reactor: The configuration of the reactor, such as fixed-bed, fluidized-bed, or trickle-bed, is determined by the specific reaction requirements. Limitations on catalyst retention, heat transmission, and mass transfer are specific to each configuration.
3. Flow Distribution: Appropriate distribution of reactants and uniform flow across the catalyst bed are essential for efficient reaction and prevention of hot areas. The design must integrate appropriate distributor plates or devices to guarantee uniform flow distribution.

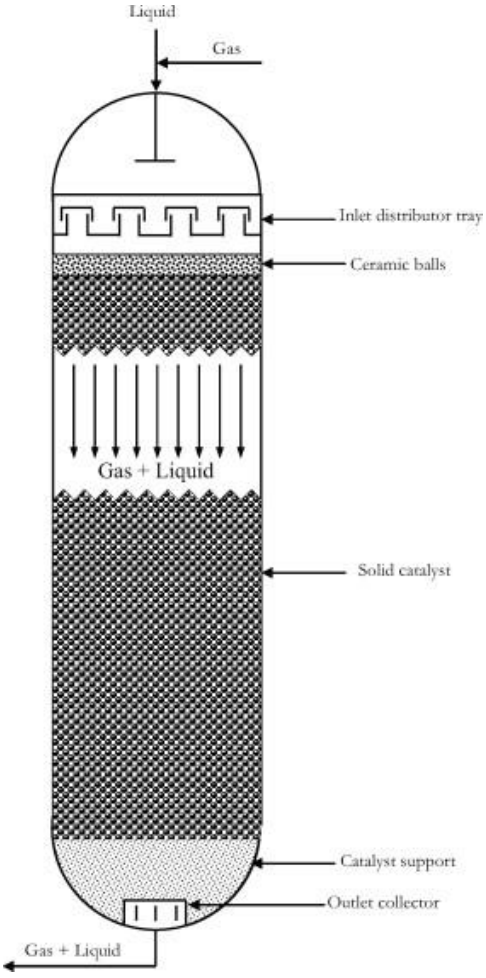


Figure 18: PBR Reactor R-101

4. Temperature and Pressure Control: The design of the reactor should accommodate for the use of heat transfer fluids, cooling jackets, or external heating systems for temperature control. Also, to be considered are pressure control measures, such as safety valves and pressure relief systems.

5.2.1 Procedure for Packed-bed Reactor Design Calculations:

Following are the PBR calculation steps:

1. Reaction Kinetics and Rate Equation:

- Identify the desired reaction and derive the rate equation representing the relationship between reactant concentrations, catalyst concentrations, and reaction rate.
- If necessary, conduct experimental studies or consult scientific literature to determine kinetic parameters such as reaction rate constants and reaction orders.

2. Mass balance:
   - Develop the overall mass balance equation for the PBR, taking into account the reaction stoichiometry, input flow rates, and reactant conversions.
   - Take into account any possible adverse or competing reactions.
3. Calculation of Residence Time:
   - Determine the weight of catalyst necessary in the PBR to accomplish the desired level of conversion.
   - Determine the residence time when employing a PBR model based on the reaction kinetics and desired conversion.
4. Pressure drop calculation:
   - Estimate the pressure drop across the PBR by considering the flow rates, bed height, particle size distribution, and bed void fraction.
   - Utilize the Ergun Equation to calculate the pressure drop.
5. Considerations for Heat Transfer:
   - Evaluate the heat transfer requirements, particularly in exothermic reactions, to prevent temperature rise and preserve desired reaction conditions.
   - Determine whether additional heat transfer surfaces, such as cooling coats or internal coils, are required to regulate the temperature of the reactor.
6. Reactor Sizing:
   - Determine the bed height and diameter of the PBR based on the desired conversion, reaction kinetics, pressure drop limitations, and heat transfer considerations.
   - Include in the sizing calculations variables such as catalyst loading, particle size, and bed vacancy fraction.

- As more impure TA is refined, the effectiveness of the catalyst decreases. The end of the catalyst's useful life is determined by the quality of the Pure TA product and the required reactor pressure.
- Utilize Aspen to obtain concentration profiles.

7. Safety Precautions:

- Conduct hazard and risk assessments to evaluate safety aspects, including potential temperature and pressure excursions.
- Implement safety measures to ensure secure operation, such as relief systems or interlocks.

5.2.2 Reactions:

The following reactions occur in series to convert 4-CBA to p-toluic acid by reacting with hydrogen gas.

$$C_8H_6O_3 + H_2 \longrightarrow C_8H_8O_3$$

$$C_8H_8O_3 + H_2 \longrightarrow C_8H_8O_2 + H_2O$$

Reaction kinetics:

$$;r_1 = 0.047 \exp\left(-\frac{16976.4}{RT}\right) C_{4-CBA}^{0.96} C_{H_2}^{0.24}$$

$$;r_2 = 0.153 \exp\left(-\frac{23438.6}{RT}\right) C_{4-HMBA}^{0.61} C_{H_2}^{0.75}$$

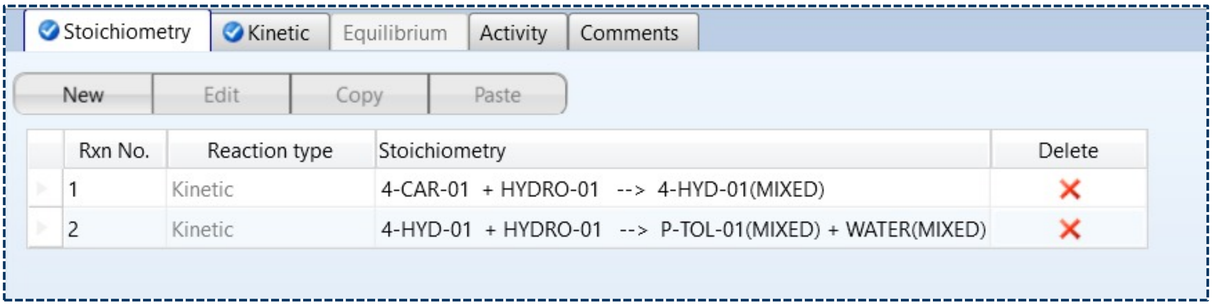
- Based on the reaction kinetics and operating conditions, weight of catalyst is calculated.
- Aspen Plus was used to find the amount of catalyst mass required for the desired conversion.

Operating Conditions:

Table 22: Operating Conditions R-101

Parameter	Value
Temperature (°C)	280
Pressure (bar)	80
Feed Flowrate (kg/hr)	5560
H2 Flowrate (kg/hr)	0.15
4-CBA feed concentration (ppm)	2500
Catalyst	0.5% (Pd/C)
Bulk density (kg/m3)	475
Bed porosity	0.44
Particle diameter (mm)	3.53

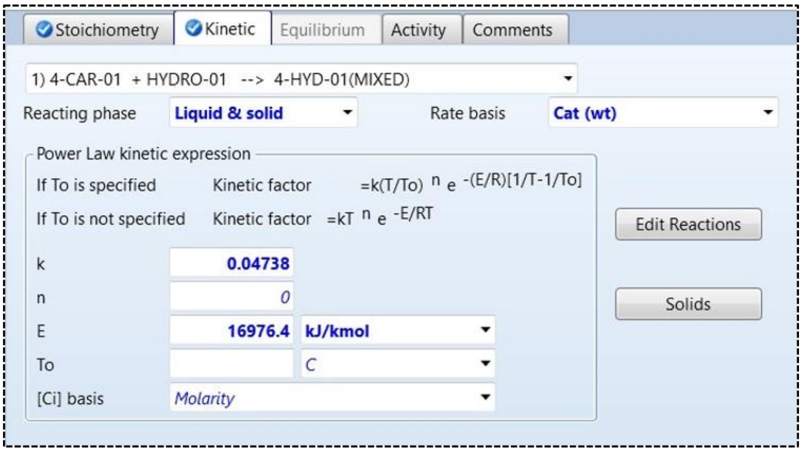
Reaction sets were defined in Aspen Plus based on reaction kinetics data.



Rxn No.	Reaction type	Stoichiometry	Delete
1	Kinetic	4-CAR-01 + HYDRO-01 --> 4-HYD-01(MIXED)	✕
2	Kinetic	4-HYD-01 + HYDRO-01 --> P-TOL-01(MIXED) + WATER(MIXED)	✕

Figure 19: Reaction Sets in Aspen Plus

Reaction 1:



1) 4-CAR-01 + HYDRO-01 --> 4-HYD-01(MIXED)

Reacting phase: **Liquid & solid**

Rate basis: **Cat (wt)**

Power Law kinetic expression

If To is specified Kinetic factor $=k(T/To)^n e^{-(E/R)[1/T-1/To]}$

If To is not specified Kinetic factor $=kT^n e^{-E/RT}$

k: **0.04738**

n: *0*

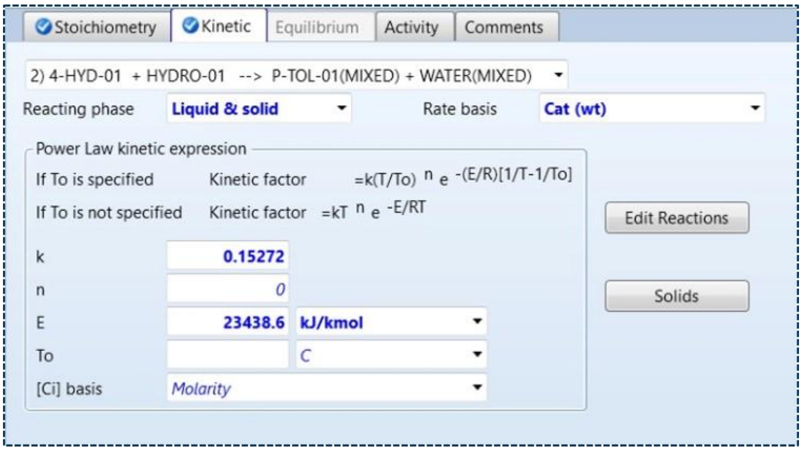
E: **16976.4** **kJ/kmol**

To: *C*

[Ci] basis: *Molarity*

Figure 20: Rxn-1

Reaction 2:



2) 4-HYD-01 + HYDRO-01 --> P-TOL-01(MIXED) + WATER(MIXED)

Reacting phase: **Liquid & solid**

Rate basis: **Cat (wt)**

Power Law kinetic expression

If To is specified Kinetic factor $=k(T/To)^n e^{-(E/R)[1/T-1/To]}$

If To is not specified Kinetic factor $=kT^n e^{-E/RT}$

k: **0.15272**

n: *0*

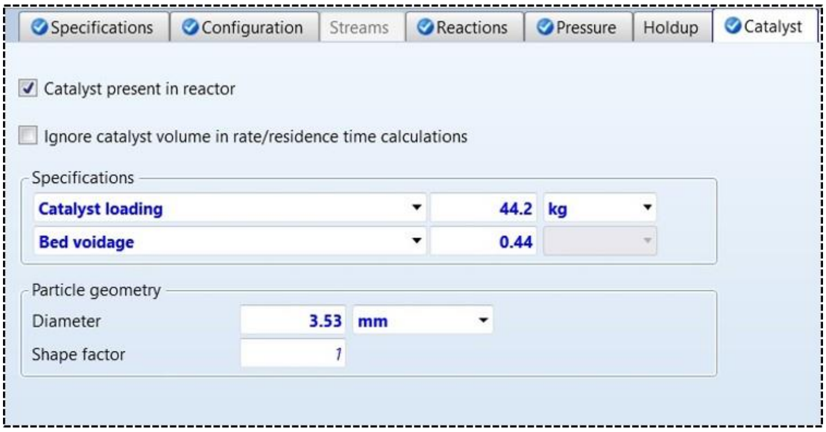
E: **23438.6** **kJ/kmol**

To: *C*

[Ci] basis: *Molarity*

Figure 21: Rxn-2

- Catalyst weight (W) was then determined based on conversion desired.



☑ Catalyst present in reactor

☐ Ignore catalyst volume in rate/residence time calculations

Specifications

Catalyst loading: **44.2** **kg**

Bed voidage: **0.44**

Particle geometry

Diameter: **3.53** **mm**

Shape factor: *1*

Figure 22: Catalyst Input in Reactor

- The following composition profile was obtained highlighting removal of impurities from the TA.
- Reaction Length = 0.8m

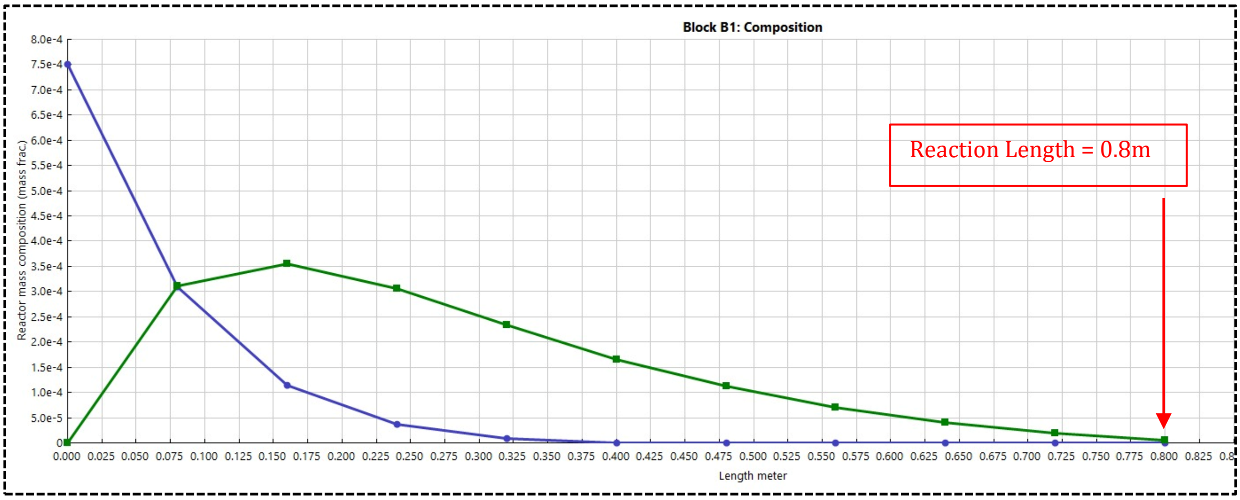


Figure 23: Reaction Results According to Reactor Length (R-101)

Equations used:

- $V_r = \frac{W}{\rho_b(1-\varepsilon)}$ (V_r is the reactor volume)
- $V_v = 1.2V_r$ (V_v is the vessel volume)
- $\frac{L}{D} = 2$ (Length to diameter ratio)
- $D = \sqrt[3]{\frac{2V_r}{\pi}}$ (D is the vessel diameter)

Calculations:

Table 23: R-101 Specifications

Parameter	Value
Weight of Catalyst (kg/m^3)	44.2
Vol. of Reaction section (m^3)	0.166
Volume of Vessel (m^3)	0.2
Diameter (m)	0.5
Length (m)	1

5.3 Crystallizer

5.3.1 Crystallizer design Considerations

A typical crystallizer is shown with some design considerations given below:

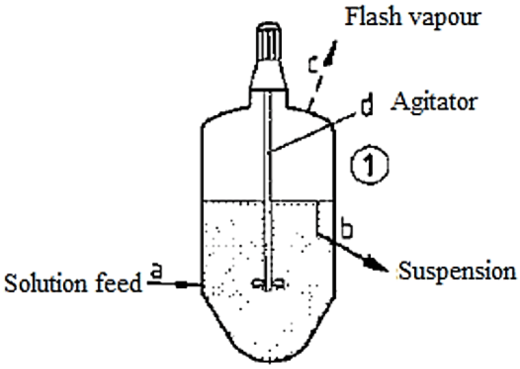


Figure 24: General Crystallizer Schematic

1. **Crystallization Method:** The selection of an appropriate crystallization method, such as cooling crystallization, evaporation crystallization, or anti-solvent crystallization, is contingent on the process requirements and desired crystal properties.
2. Mixing and agitation: Effective mingling and agitation ensure uniform solute distribution, regulate supersaturation, and promote crystal growth. The design should include suitable mixing mechanisms, such as impellers, to accomplish the desired levels of agitation.
3. Cooling or heating system: The design should take into account the refrigeration or heating system required to control the crystallization temperature. This may involve the use of heating coils, chilling water, or refrigerants.
4. Crystallizer configuration: various crystallizer configurations, such as draft tube, stirred tank, and crystallizer with internals, offer various advantages based on the desired crystal characteristics and process requirements.

5.3.2 Procedure for Crystallizer Design Calculations:

1. **Solubility Data:**
   - Gather solubility data for the solute in the solvent at different temperatures and concentrations.
   - The table used for solubility data is shown below.

Table 24: Solubility Data od Crystallizer C-101

Temperature (^0C)	Solids Strength (%)
200	1.77
220	3.85
240	8.25
250	11.75
260	16.67
261	17.35
262	17.83
263	18.37
264	19.03
265	19.68
266	20.32
267	20.95
268	21.57
269	22.18
270	23.08
271	23.66
272	24.24
273	25.09
274	25.92
275	26.74
276	27.54
277	28.32
278	29.08
279	30.07
280	30.08
281	31.74
282	32.43
283	33.34
284	34.43

285	35.28
286	36.1
287	37.1
288	38.27
289	39.02
290	40.12

2. **Crystal Growth Kinetics:**
   - Understand the crystal growth kinetics of the solute in the solvent, such as the growth rate and nucleation rate. This information can be obtained from literature or experimental data.
     - $G = k_g e^{-\frac{E_a}{RT}} \left(C - C_{eq}\right)^g$
3. **Residence Time:**
   - Based on the average crystal growth rate, the residence time is calculated which yields the required volume
     - $\tau = \frac{L_c}{6G}$
     - $V_{req} = Q\tau$
     - $V_{tank} = \frac{Vreq}{0.6}$

- Flash Evaporation is used to crystallize terephthalic acid.
- This leaves the liquid p-toluic acid in the mother liquor.
- The saturated flash steam is used as heating medium for the feed.

Operating Conditions:

Table 25: Operating Conditions of Crystallizer C-101

Parameters	Value
Temperature (°C)	244
Pressure (bar)	35.5
Feed flowrate (kg/hr)	5564
Vapor flowrate (kg/hr)	851
Liquid flowrate (kg/hr)	3043
Volumetric liquid flowrate (m^3/hr)	5.4

- The crystallizer residence time is determined by the average crystal growth rate (G) which is given by:

$$G = k_g e^{-\frac{E_a}{RT}} \left(C - C_{eq}\right)^g$$

- The values for the equation parameters are tabulate.

Crystallization Parameters:

Table 26: Process Crystallization Parameters

Parameters	Value
kg (um/s)	1.7251
Ea/R (K)	1763.7
g (constant)	0.984
Concentration	0.3
Equilibrium conc. (C_{eq})	0.00251
Dominant crystal size (mm)	0.2
Avg. Growth rate (m/s)	7.934E-09

- Based on the average crystal growth rate, the residence time is calculated which yields the required volume.
    - $\tau = \frac{L_c}{6G}$ (τ is the residence time & L_c crystal size)

- $V_{req} = Q\tau$ (V_{req} is the required volume)
- $V_{tank} = \frac{Vreq}{0.6}$ (V_{tank} is the tank volume)

Crystallizer Parameters:

Table 27: Crystallizer C-101 Parameters

Parameter	Value
Tank residence time (hr)	0.76
Volume required (m3)	4.1
Volume of vessel (m3)	7.07
Diameter (m)	1.5
Length (m)	4
Level (%)	60

CHAPTER 6

PROCESS SIMULATION

6.1 Simulation – HYSYS:

6.1.1 Process Simulation Overview

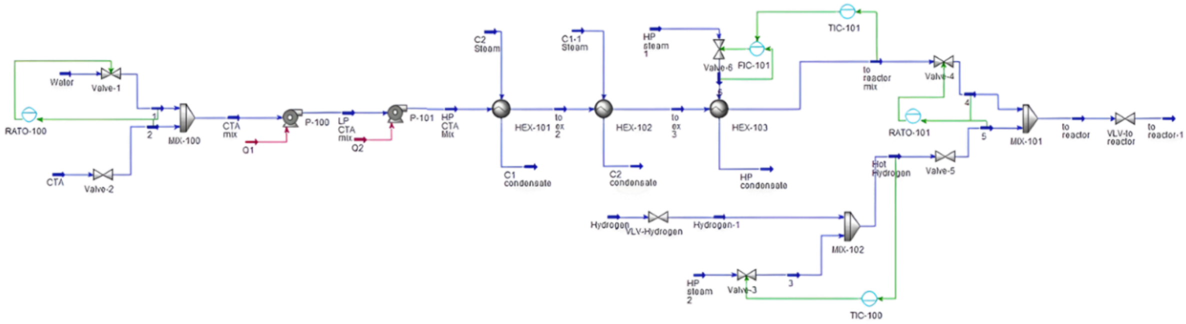
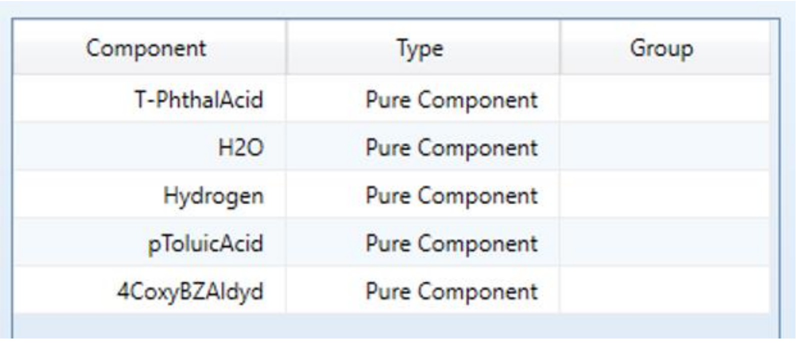


Figure 25: Aspen HYSYS Simulation Overview

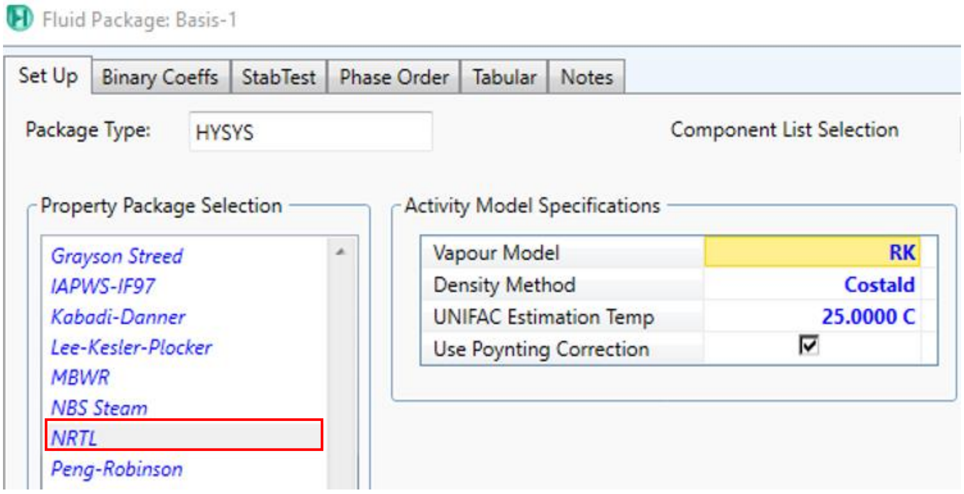
6.1.2 Component List



Component	Type	Group
T-PhthalAcid	Pure Component	
H2O	Pure Component	
Hydrogen	Pure Component	
pToluicAcid	Pure Component	
4CoxyBZAldyd	Pure Component	

Figure 26: Components List in Aspen HYSYS Simulation

6.1.3 Fluid Package



Package Type: HYSYS

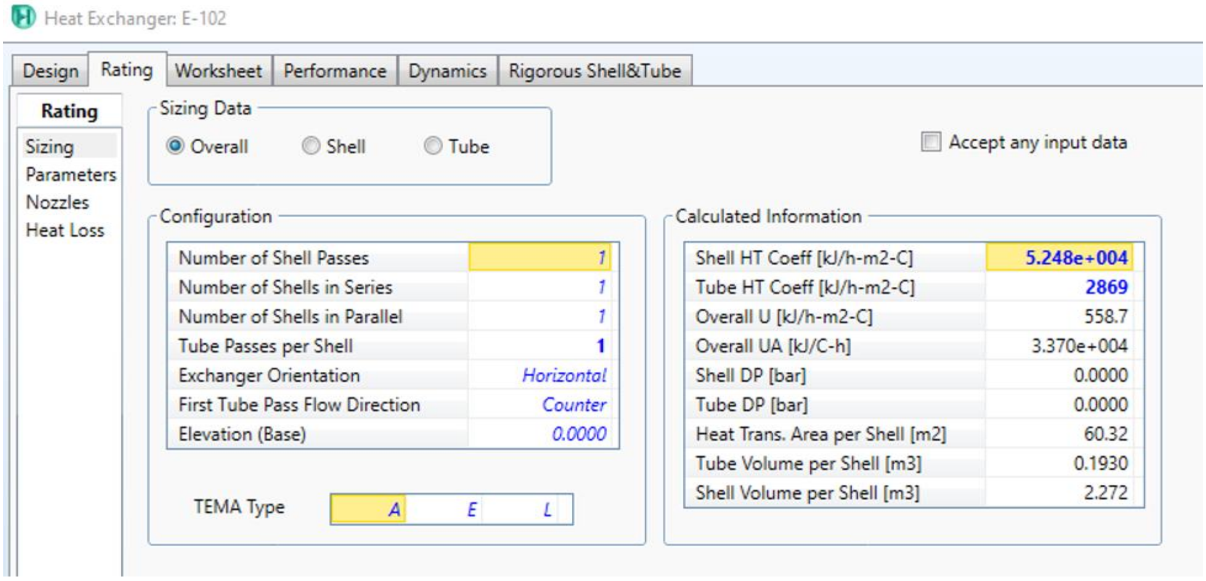
Property Package Selection:

- Grayson Streed
- IAPWS-IF97
- Kabadi-Danner
- Lee-Kesler-Plocker
- MBWR
- NBS Steam
- NRTL
- Peng-Robinson

Vapour Model	RK
Density Method	Costald
UNIFAC Estimation Temp	25.0000 C
Use Poynting Correction	☑

Figure 27: Fluid Package in Aspen HYSYS Simulation

6.1.4 E-102 Parameters



Sizing Data: ● Overall ○ Shell ○ Tube

☐ Accept any input data

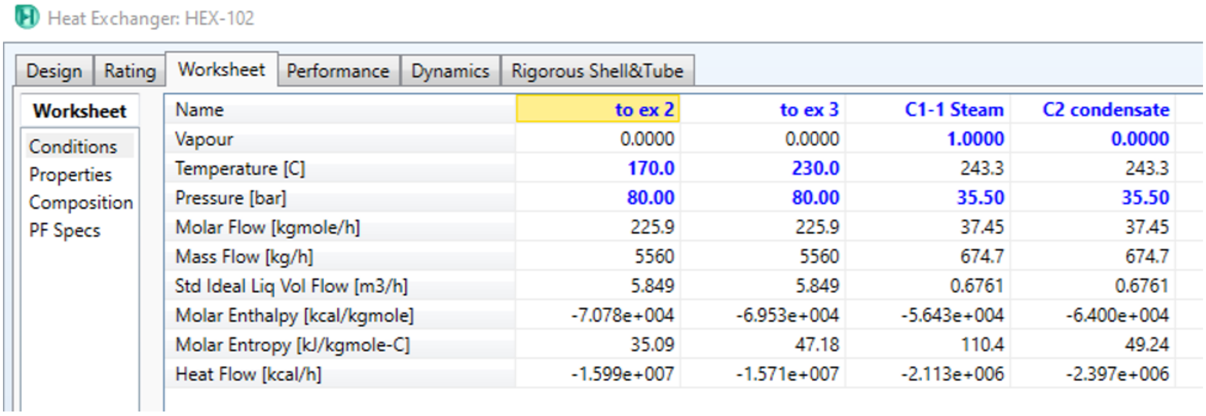
Number of Shell Passes	1
Number of Shells in Series	1
Number of Shells in Parallel	1
Tube Passes per Shell	1
Exchanger Orientation	Horizontal
First Tube Pass Flow Direction	Counter
Elevation (Base)	0.0000

TEMA Type: A E L

Shell HT Coeff [kJ/h-m2-C]	5.248e+004
Tube HT Coeff [kJ/h-m2-C]	2869
Overall U [kJ/h-m2-C]	558.7
Overall UA [kJ/C-h]	3.370e+004
Shell DP [bar]	0.0000
Tube DP [bar]	0.0000
Heat Trans. Area per Shell [m2]	60.32
Tube Volume per Shell [m3]	0.1930
Shell Volume per Shell [m3]	2.272

Figure 28: E-102 Parameters

6.1.5 E-102 Worksheet



Name	to ex 2	to ex 3	C1-1 Steam	C2 condensate
Vapour	0.0000	0.0000	1.0000	0.0000
Temperature [C]	170.0	230.0	243.3	243.3
Pressure [bar]	80.00	80.00	35.50	35.50
Molar Flow [kgmole/h]	225.9	225.9	37.45	37.45
Mass Flow [kg/h]	5560	5560	674.7	674.7
Std Ideal Liq Vol Flow [m3/h]	5.849	5.849	0.6761	0.6761
Molar Enthalpy [kcal/kgmole]	-7.078e+004	-6.953e+004	-5.643e+004	-6.400e+004
Molar Entropy [kJ/kgmole-C]	35.09	47.18	110.4	49.24
Heat Flow [kcal/h]	-1.599e+007	-1.571e+007	-2.113e+006	-2.397e+006

Figure 29: E-102 Worksheet

6.2 Simulation – Aspen Plus:

6.2.1 Process Simulation Overview

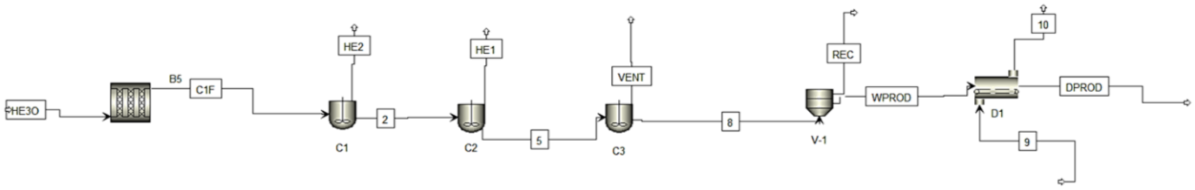
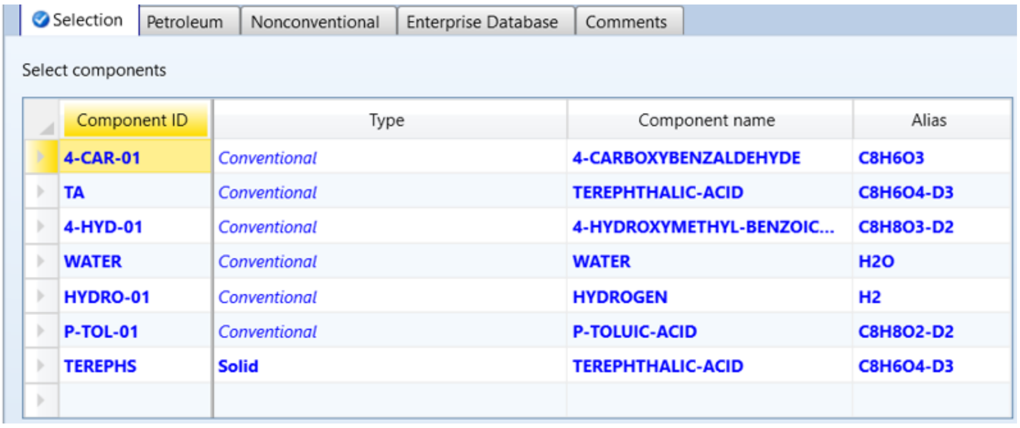


Figure 30: Aspen Plus Process Simulation Overview

6.2.2 Component List

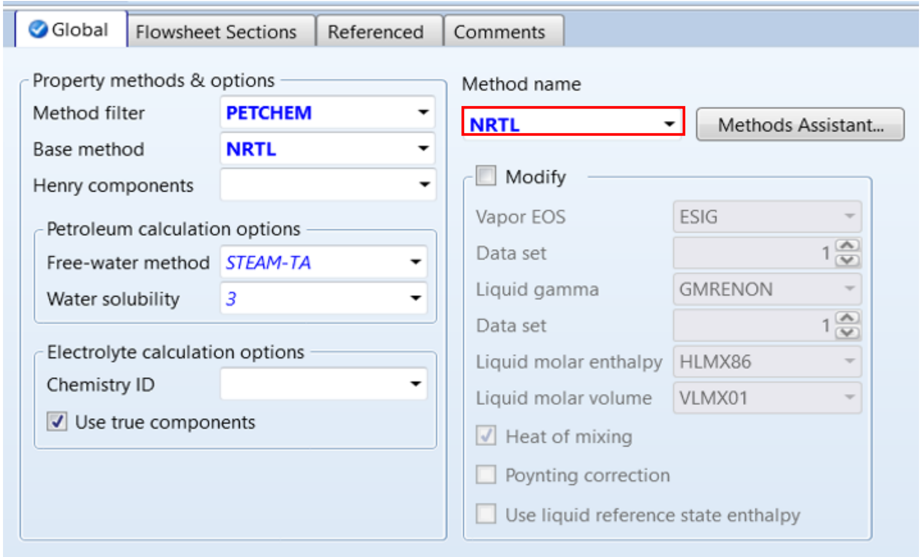


Select components

Component ID	Type	Component name	Alias
4-CAR-01	Conventional	4-CARBOXYBENZALDEHYDE	C8H6O3
TA	Conventional	TEREPHTHALIC-ACID	C8H6O4-D3
4-HYD-01	Conventional	4-HYDROXYMETHYL-BENZOIC...	C8H8O3-D2
WATER	Conventional	WATER	H2O
HYDRO-01	Conventional	HYDROGEN	H2
P-TOL-01	Conventional	P-TOLUIC-ACID	C8H8O2-D2
TEREPHS	Solid	TEREPHTHALIC-ACID	C8H6O4-D3

Figure 31: Components List in Aspen Plus Simulation

6.2.3 Fluid Package



Property methods & options

Method filter: PETCHEM

Base method: NRTL

Henry components:

Petroleum calculation options

Free-water method: STEAM-TA

Water solubility: 3

Electrolyte calculation options

Chemistry ID:

☑ Use true components

Method name: NRTL

☐ Modify

Vapor EOS: ESIG

Data set: 1

Liquid gamma: GMRENON

Data set: 1

Liquid molar enthalpy: HLMX86

Liquid molar volume: VLMX01

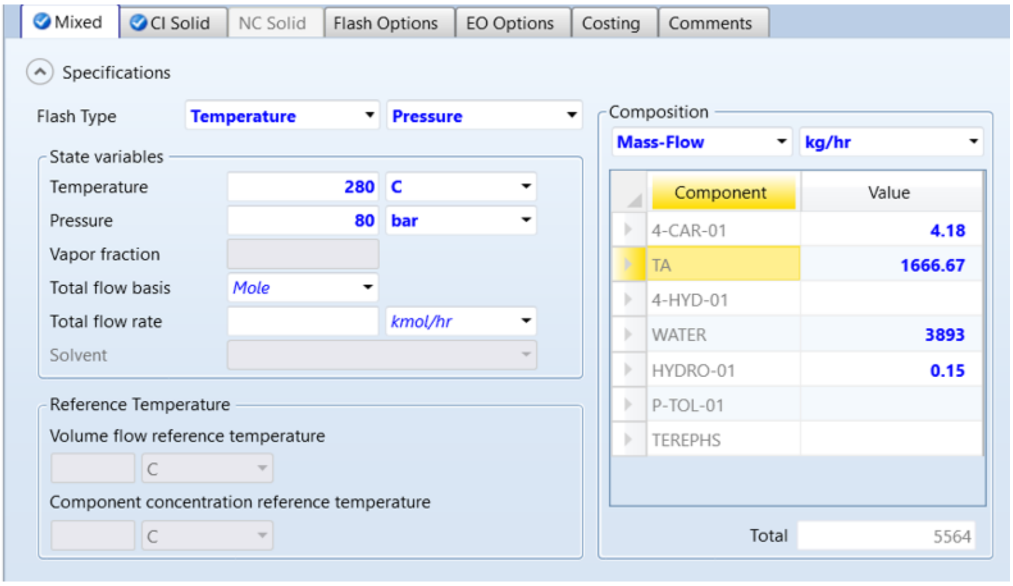
☑ Heat of mixing

☐ Poynting correction

☐ Use liquid reference state enthalpy

Figure 32: Fluid Package in Aspen Plus Simulation

6.2.4 Reactor Inlet Stream



Specifications

Flash Type: Temperature, Pressure

State variables

Temperature: 280 C

Pressure: 80 bar

Vapor fraction:

Total flow basis: Mole

Total flow rate: kmol/hr

Solvent:

Reference Temperature

Volume flow reference temperature: C

Component concentration reference temperature: C

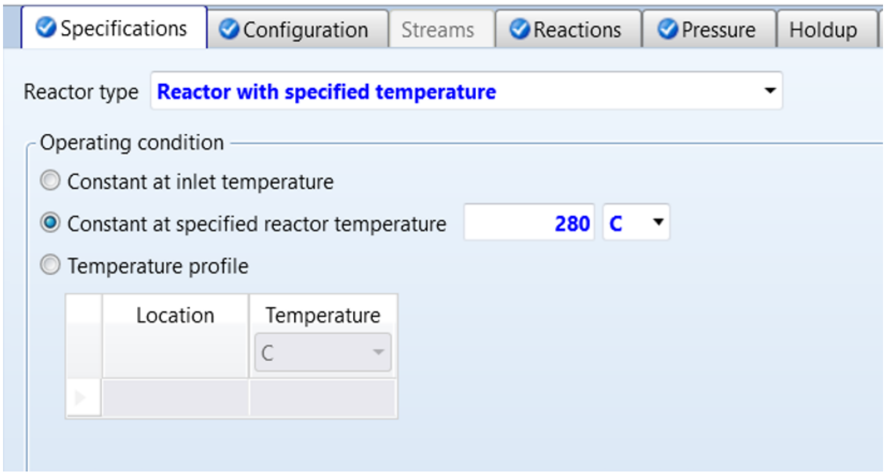
Composition: Mass-Flow, kg/hr

Component	Value
4-CAR-01	4.18
TA	1666.67
4-HYD-01	
WATER	3893
HYDRO-01	0.15
P-TOL-01	
TEREPHS	

Total: 5564

Figure 33: Reactor Inlet Stream in Aspen Plus Simulation

6.2.5 Reactor Specification



Reactor type: Reactor with specified temperature

Operating condition

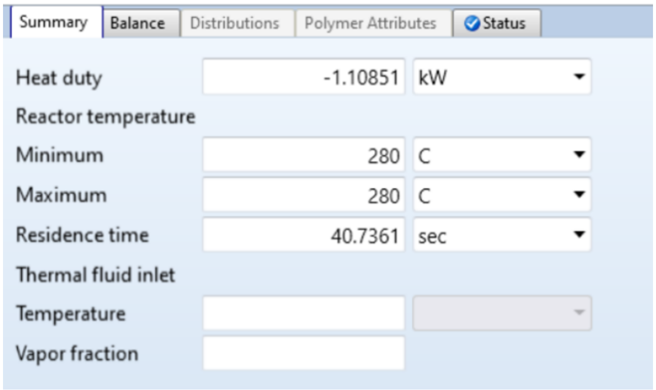
○ Constant at inlet temperature

● Constant at specified reactor temperature 280 C

○ Temperature profile

Figure 34: Reactor Specification in Aspen Plus Simulation

6.2.6 Reactor Results



Heat duty -1.10851 kW

Reactor temperature

Minimum 280 C

Maximum 280 C

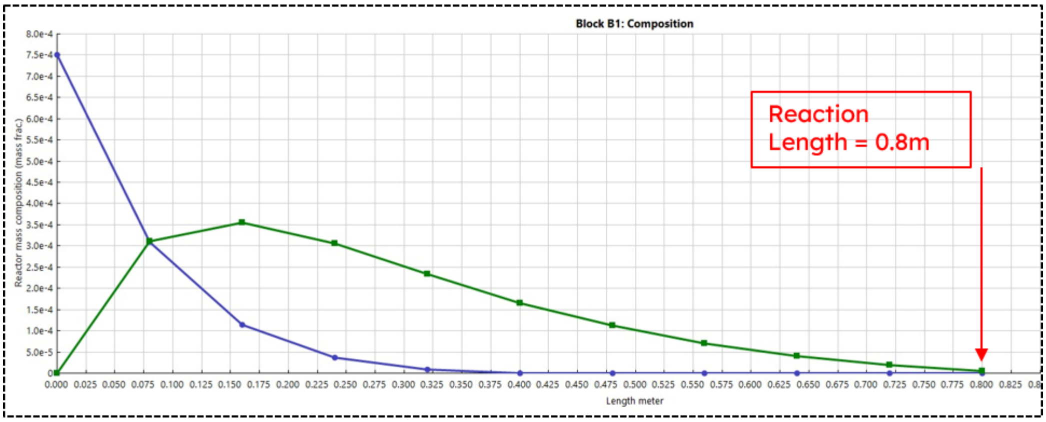
Residence time 40.7361 sec

Thermal fluid inlet

Temperature

Vapor fraction

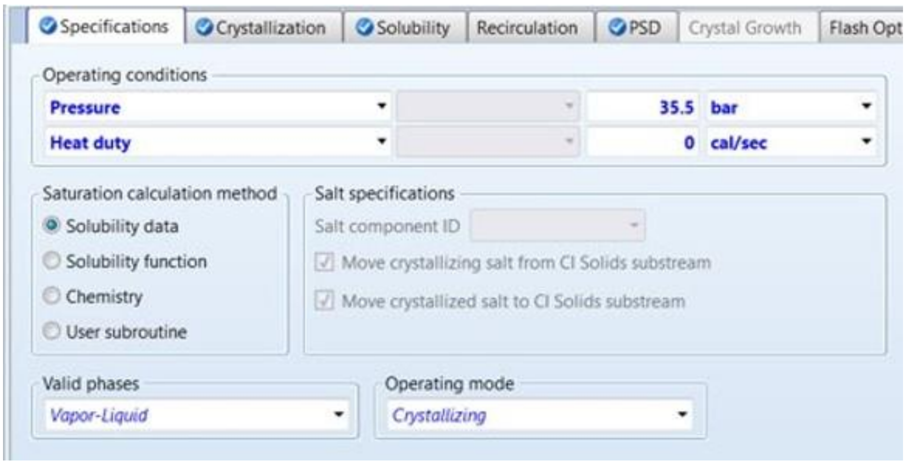
Figure 35: Reactor Results in Aspen Plus Simulation



Reaction Length = 0.8m

Figure 36: Reaction Results According to Reactor Length (R-101)

6.2.7 C-101 Specification



Operating conditions

Pressure 35.5 **bar**

Heat duty 0 **cal/sec**

Saturation calculation method

● Solubility data
○ Solubility function
○ Chemistry
○ User subroutine

Salt specifications

Salt component ID

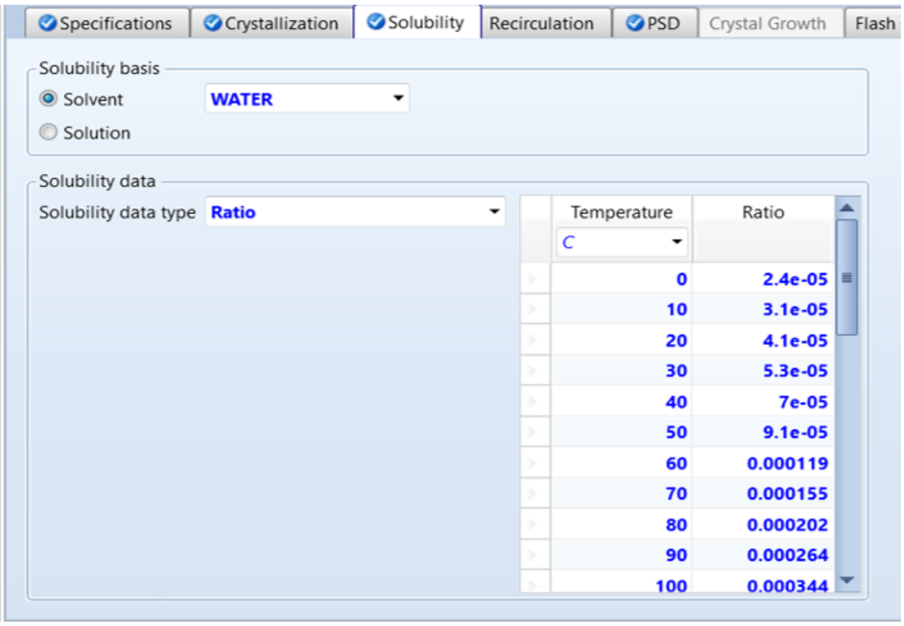
☑ Move crystallizing salt from CI Solids substream
☑ Move crystallized salt to CI Solids substream

Valid phases: *Vapor-Liquid*

Operating mode: *Crystallizing*

Figure 37: C-101 Specifications

6.2.8 C-101 Solubility Data



Solubility basis

● Solvent **WATER**
○ Solution

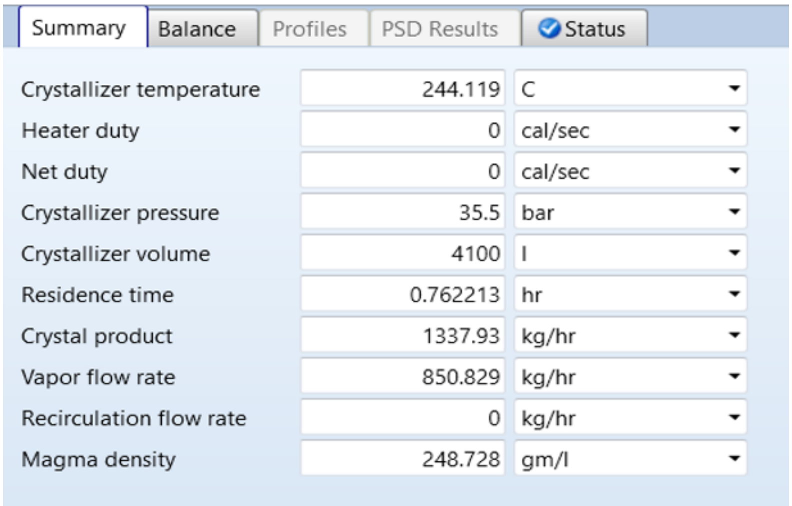
Solubility data

Solubility data type **Ratio**

Temperature C	Ratio
0	2.4e-05
10	3.1e-05
20	4.1e-05
30	5.3e-05
40	7e-05
50	9.1e-05
60	0.000119
70	0.000155
80	0.000202
90	0.000264
100	0.000344

Figure 38: C-101 Solubility Data

6.2.9 C-101 Results



Crystallizer temperature	244.119	C
Heater duty	0	cal/sec
Net duty	0	cal/sec
Crystallizer pressure	35.5	bar
Crystallizer volume	4100	l
Residence time	0.762213	hr
Crystal product	1337.93	kg/hr
Vapor flow rate	850.829	kg/hr
Recirculation flow rate	0	kg/hr
Magma density	248.728	gm/l

Figure 39: C-101 Design Results

CHAPTER 7

INSTRUMENTATION AND CONTROL

7.1 Control Strategies

To guarantee that the purification of PTA from CTA is proceeding in a manner that is both effective and efficient, control methods are implemented into the instrumentation and control phases of the process. The likes of temperature, pressure, flow rate, and concentration are just some of the characteristics that may be monitored and controlled with the help of control techniques.

The following is a list of the control methods that are utilized most frequently in the process of separating PTA from CTA:

Cascade control is a control approach that uses the output of one controller to influence the input of another controller. It works like this: the output of one controller controls the input of another controller. Cascade control is a technique that is frequently employed in complicated processes in which it is essential to exert control over a number of parameters in order to produce the desired result.

Feedforward control is a type of control technique that calculates the control output based on the measured value of a process variable. This type of control is also known as "feedforward" control. To enhance the overall performance of the control system, it is common practice to employ feedforward control in conjunction with PID control.

7.1.1 Ratio Control

In instrumentation and control, ratio control refers to a control strategy in which the flow rate or output of one process stream is adjusted relative to the flow rate or output of another process stream. Maintaining a specific ratio between two variables, typically in a continuous process, is the objective of ratio control.

The ratio control system constantly monitors and compares the controlled variable to the intended set point. The system adjusts the controlled variable based on the deviation between the actual value and the set point to maintain the desired ratio. Utilizing Proportional-Integral-Derivative (PID) controllers, the manipulated variable is adjusted in response to deviations in the controlled variable. This can be significant in processes that require precise proportions or concentrations of distinct components for optimal operation or product quality. On the feed tank is located the ratio control. The ratio between the CTA stream and the water stream must be managed. The disturbance variable is the CTA mass flowrate, the manipulated variable is the water mass flowrate, and the controlled variable is the valve-1 actuator.

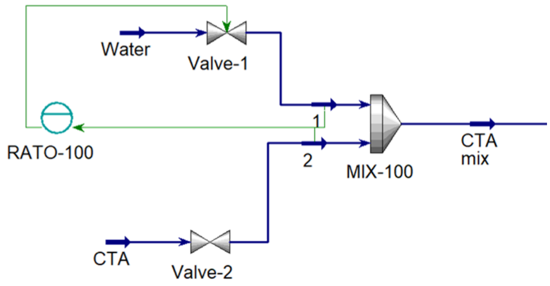
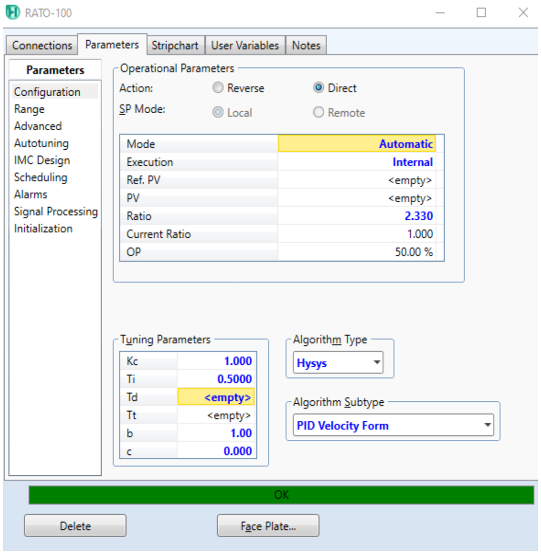


Figure 40: Ratio Controller Schematic in Process



Operational Parameters

Action: ○ Reverse ● Direct

SP Mode: ● Local ○ Remote

Mode	Automatic
Execution	Internal
Ref. PV	<empty>
PV	<empty>
Ratio	2.330
Current Ratio	1.000
OP	50.00 %

Tuning Parameters

Kc	1.000
Ti	0.5000
Td	<empty>
Tt	<empty>
b	1.00
c	0.000

Algorithm Type: Hysys

Algorithm Subtype: PID Velocity Form

Figure 41: Ratio Controller Parameters

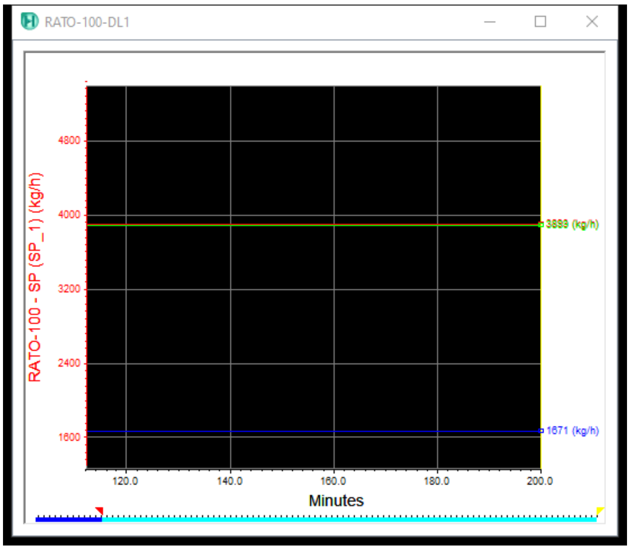


Figure 42: Ratio Controller Strip chart

7.1.2 Cascade Control

Cascade control is utilized for slow-changing output processes. By dividing the control effort between the outer and interior loops, cascade control provides faster response times. The outer loop, which typically controls a slower process variable, can respond at a delayed rate to changes in set points or disturbances. In the meantime, the inner loop, which is responsible for regulating a faster process variable, can respond to changes more quickly, resulting in a more responsive control system overall. Cascade control increases the system's stability by minimizing the effects of disturbances and variable interactions. Cascade control aids in isolating disturbances and preventing their propagation throughout the system by employing multiple control circuits, each of which regulates specific process variables. This results in more precise and reliable control.

In our process, the disturbance variable is the TIC-101 input, the manipulated variable is the mass flowrate of HP steam, and the controlled variable is the valve-6 actuator. Cascade control is located on the heat exchanger, where output variations are typically gradual. Master and slave controls comprise cascade control. Temperature is the master control in a heat exchanger, while flowrate is the slave control. The master controller monitors the temperature continuously and adjusts the manipulated variable to sustain the desired temperature set point. The slave controller receives the master controller's desired temperature signal and compares it to the actual flow rate. If there is a deviation, the slave controller adjusts the manipulated variable to accomplish the desired flow rate, regulating the temperature indirectly through flowrate regulation.

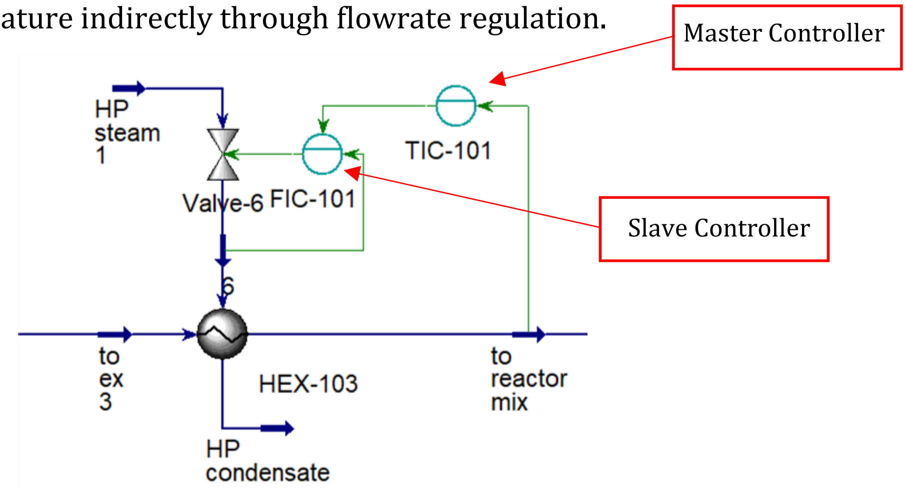
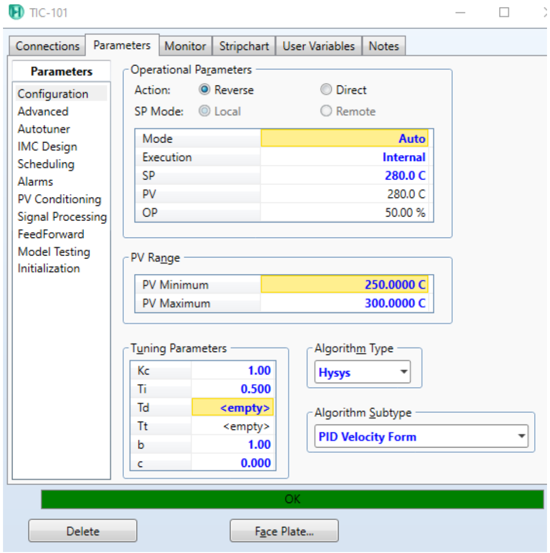


Figure 43: Cascade Controller Schematic



Operational Parameters

Action: ● Reverse ○ Direct

SP Mode: ○ Local ○ Remote

Mode	Auto
Execution	Internal
SP	280.0 C
PV	280.0 C
OP	50.00 %

PV Range

PV Minimum	250.0000 C
PV Maximum	300.0000 C

Tuning Parameters

Kc	1.00
Ti	0.500
Td	<empty>
Tt	<empty>
b	1.00
c	0.000

Algorithm Type: Hysys

Algorithm Subtype: PID Velocity Form

Figure 45: Master controller Parameters

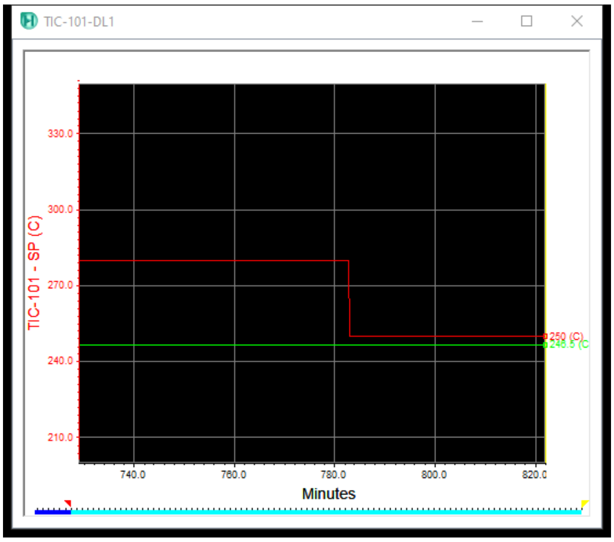
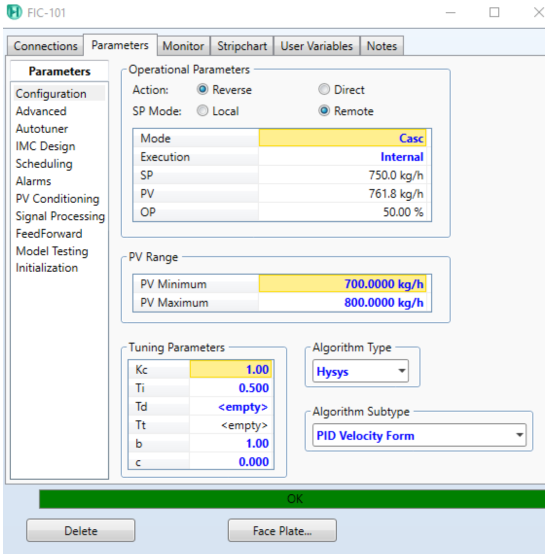


Figure 44: Master Controller Strip chart



Operational Parameters

Action: ● Reverse ○ Direct

SP Mode: ○ Local ● Remote

Mode	Casc
Execution	Internal
SP	750.0 kg/h
PV	761.8 kg/h
OP	50.00 %

PV Range

PV Minimum	700.0000 kg/h
PV Maximum	800.0000 kg/h

Tuning Parameters

Kc	1.00
Ti	0.500
Td	<empty>
Tt	<empty>
b	1.00
c	0.000

Algorithm Type: Hysys

Algorithm Subtype: PID Velocity Form

Figure 46: Slave Controller Parameters

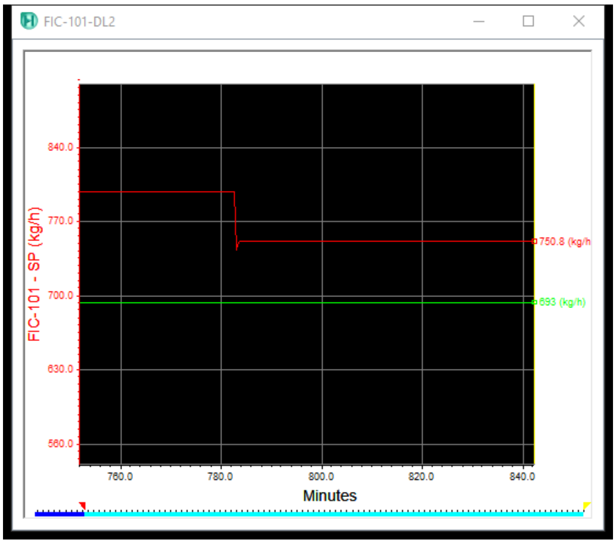


Figure 47: Slave Controller Strip Chart

7.2 Instrumentation Requirements

Valves, temperature sensors, pressure sensors, and flow sensors are vital instrumentation components in our process.

7.2.1 Valves

Valves are used in the process to regulate the passage of fluids or gases. Valves should be chosen according to the required flow rate, pressure range, and desired control precision. The instrumentation should be able to precisely measure and control the flow rate or valve position. There should be precise control over valve positioning.

Valves may require routine calibration and maintenance. Instrumentation may include fault detection and position monitoring to facilitate maintenance and troubleshooting. Relief valves, gate valves, and plug valves are all critical components that were used in the plant for the purification of PTA from CTA.

Relief valves are used to prevent the buildup of excessive pressure in a system. They are normally designed to open at a specified pressure, and when the pressure in the system reaches this level, the valve opens and allows the excess pressure to escape. This serves to protect the system from harm and to avoid the release of hazardous materials.

Gate valves are used to control the flow of fluids in a system. They are normally composed of metal and have a gate that moves up and down to open and close the valve. Gate valves are quite adaptable and can be utilized in a variety of applications.

Plug valves are used to accurately control the flow of fluids in a system. They are typically composed of metal and have a plug that spins to open and close the valve. Plug valves are widely employed in laboratory settings and in other applications where precise control of fluid flow is essential.

In the purification of PTA from CTA, relief valves are utilized to prevent the building of excessive pressure in the distillation column. Gate valves are used to control the flow of PTA and CTA into and out of the distillation column. Plug valves are used to accurately control the flow of PTA and CTA into the crystallization unit.

The usage of these valves helps to ensure that the purification process is safe and efficient.

7.2.2 Temperature Sensors

Temperature sensors are utilized for measuring and monitoring the temperature of a process. Temperature sensors must provide precise readings within the specified temperature range. The response time should be appropriate for the process's dynamics and control needs. The accuracy, temperature range, and response characteristics of various sensor types, such as thermocouples, resistance temperature detectors (RTDs), and thermistors, differ. The sensor type should be selected based on the specific requirements of the application. The calibration and placement of temperature sensors are essential for precise measurements. Sensors should be set in representative locations, away from sources of heat or cold, and with adequate insulation to minimize the effects of ambient conditions.

7.2.3 Pressure Sensors

Pressure sensors are utilized to measure and monitor the fluid or gas pressure in a process. Pressure sensors should be chosen according to the requisite pressure range and the desired measurement precision. The process medium and operating conditions must be compatible with the pressure sensor. Regular calibration is required to preserve precision. To ensure leak-free connections and prevent the sensor from damage caused by excessive pressure or vibrations, proper installation techniques, such as using the appropriate fittings and seals, should be followed.

7.2.4 Flow Sensors

Flow sensors are utilized to determine the fluid flow rate in a process. Flow sensors must be chosen based on the requisite flow range and the desired measurement precision. The flow range capabilities of various sensor types, such as magnetic, ultrasonic, and differential pressure-based, vary. Installation must be performed correctly to assure accurate flow measurement. To minimize disturbances and obtain accurate readings, it is necessary to take into account pipe diameter, straight pipelines, and orientation. Flow sensors must be calibrated on a regular basis to ensure accuracy. The sensor's efficacy can be validated using verification procedures and a known flow rate or reference standard. Cleaning and Maintenance are required.

CHAPTER 8

ECONOMIC ANALYSIS

8.1 Introduction

Estimating costs is both a specialized field of study and a distinct field of work in its own right. However, the design engineer needs to be capable of making quick, rough cost estimates to choose between various designs and for the purpose of project evaluation. Chemical plants are constructed with the intention of turning a profit, and in order to determine whether or not a certain project will be profitable, it is necessary to first produce an estimate of the amount of capital investment necessary as well as the total cost of production.

8.2 Purpose of Cost Estimation

The quantity of design detail that is readily available, the precision of the cost data that is readily available, and the amount of time spent preparing the estimate all contribute to the accuracy of the estimate. In the beginning phases of a project, only a rough estimate will be necessary, and this requirement will be justified by the amount of information that has been developed up to that point.

Estimates of the cost of capital can be broadly divided into three categories according to the degree of accuracy they provide and the function they serve:

1. Approximate preliminary estimates, also known as preliminary estimates, are utilized in preliminary feasibility studies and for making broad selections between several design alternatives. The accuracy of preliminary estimates is normally within a margin of error of 30 percent. They are derived from scant information regarding costs and design details.
2. Authorization (Budgeting) estimations, with an accuracy range that is normally between ten and fifteen percent. These are used for the authorization of money to continue with the design to the point where an accurate and more complete estimate can be generated. They are used for the purposes of the design. In order to prevent further

delays with the project, the authorization may also include monies to pay cancellation fees for any lengthy delivery equipment that has been ordered at this stage of the design. In an organization that does contracting, this kind of estimate might be utilized in conjunction with a significant contingency factor to produce a price for tendering. However, in the normal course of events, an accuracy of approximately 5% would be required if there was enough time, we would also provide a more comprehensive estimate. At the flow-sheet stage of the project, estimations of an appropriate level of accuracy can be established if the organization has sufficient expertise and is in possession of cost data from previously completed projects that are comparable. In addition to this, you will require a P and 1 diagram in rough form as well as an approximation of the sizes of the primary pieces of equipment.

3. Detailed estimates (Quotation), with an accuracy range of 5% to 10%, are utilized for project cost control as well as estimates for fixed price contracts. These are based on the finished (or almost finished) process design, firm quotations for the equipment, as well as a full breakdown and estimation of the cost of the construction.

The cost of preparing an estimate rises from around 0.1 percent of the overall cost of the project for an accuracy of 30 percent to approximately 2 percent of the whole cost of the project for a comprehensive estimate with an accuracy of 5 percent.

- The factorial method of cost estimation is used. A sample calculation is shown below with relevant factor to calculate cost of HX-103.

Table 28: Purchase cost of E-103

For E-103	
Heat Transfer Area (m^2)	18.5
Materials	Stainless Steel
Pressure Factor	1.6
Type Factor	1
Cost	**$ 49500**

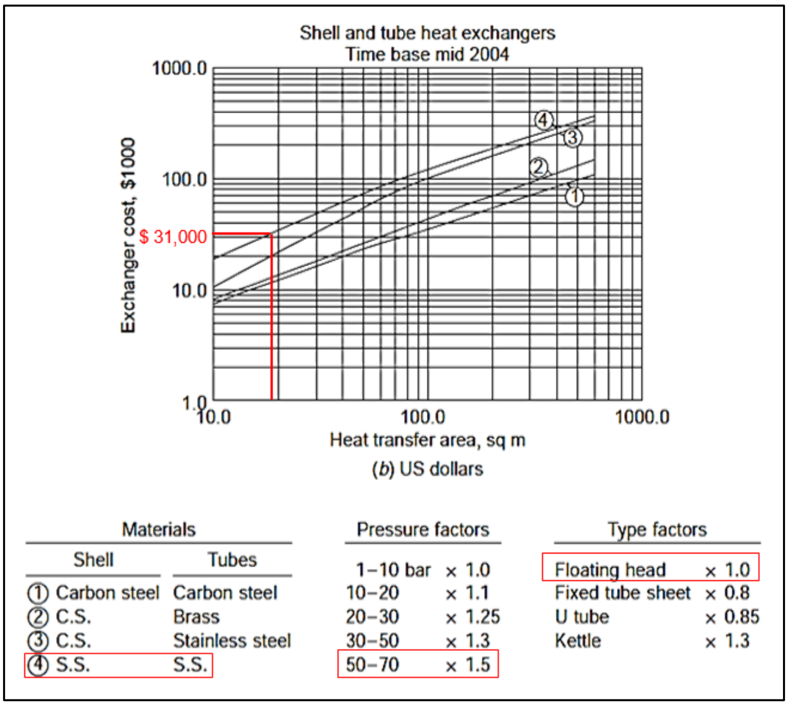


Figure 48: Purchase cost of Shell and Tube Heat Exchanger

8.3 Main Equipment Costs

Table 29: Purchase Cost of All Equipments

Specification	Equipment Cost ($)
Mixing Vessel	16,000
Reactor (R-101)	18,000
HEX-1	45,000
HEX-2	49,500
HEX-3	40,500
C-101	36,000
C-102	36,000
C-103	36,000
Centrifuge	40,000
Recycle Tank	16,000
Dryer	57,400
Total	**390,400**
PCE (after adjusting for Inflation) = $ 562,901.60	

8.4 Total Investment

Table 30: Factors Considered for overall process.

f1 (Equipment erection)	0.4
f2 (Piping)	0.7
f3 (Instrumentation)	0.2
f4 (Electrical)	0.1
f5 (Buildings, process)	0.15
*f6 (Utilities)	-
*f7 (Storages)	0.15
*f8 (Site development)	-
*f9 (Ancillary buildings)	-
f10 (Design and Engr.)	0.3
f11 (Contractor's fee)	0.05
f12 (Contingency)	0.1

Table 31: Total Fixed Cost

(Total Physical Plant Cost) PPC = PCE (1+f1+ ……. + f9)	$ 1.52M
(Fixed Capital) FC = PPC (1+f10+f11+f12)	$ 2.20M
Working Capital (15% of FC)	$ 0.33M
Total Investment Required (Fixed Capital + Working Capital)	**$ 2.53M**
Operating Time of Plant	347 days/year

8.5 Direct Production Cost

Table 32: Fixed operating Cost

Fixed Operating Cost	
Maintenance (3% of FC)	$ 66,112.79
Operating Labor (OL)	$ 43,244.68
Plant Overheads (40% of OL)	$ 17,297.87
Laboratory (20% of OL)	$ 8,648.94
Capital Charges (2% of FC)	$ 44,075.19
Insurance (1% of FC)	$ 22,037.60
Local Taxes (1% of FC)	$ 22,037.60
Total Fixed Operating Cost	**$ 223,454.67**

Table 33: Variable Operating Costs

Variable Operation Cost	
CTA Feed Rate	$40 ton/day
CTA Feed Price	$875/ton
CTA Feed Price	$35,000/day
CTA Feed Price	$12,145,000/yr
Water Requirement	$323112 ton/yr
Water Price	$1/yr
Water Price	$371,579 kg/yr
Catalyst (Pd/C) Quantity Required	$45 kg/yr
Catalyst (Pd/C) Price/ton	$1,800/kg
Catalyst (Pd/C) Price	$81,000.00/yr
Steam Quantity Required	$6,047.79/yr
Steam Price	$15.00/ton

Steam Cost	$90,717
H_2 cost	$1,200.00
Miscellaneous Operating Material (5% of Maintenance Cost)	$3,305.64
Electric Power Required	$3240 MJ/day
Electric Power Required	$1124280 MJ/day
Cost of electricity	$31,230.00/yr
Total Variable Cost (TVC)	$**12,720,725.70**

Table 34: Direct Production Cost

Direct Production Cost (FOC+TVC)	$ 12.94M

8.6 Revenue Generated

Table 35: Overall Revenue and Profits

Revenue			
Product	Production (ton/yr)	Price ($/ton)	Revenue Generated
Pure Terephthalic Acid	13880	1,000.00	$13.88M
		Total Revenue/yr	**$13.88M**
		Total Profit/yr	**$0.935M**

8.7 Payback Period

Table 36: Payback Period Summery

Payback Period				
No. of Years (k)	Cash Inflow ($)	Cash Outflow ($)	Net Cash Flow ($)	Net Present Worth ($) (10%, k)
0	-	2.53M	-2.53M	2.53M
1	13.88M	12.94M	0.935M	0.850M
2	13.88M	12.94M	0.935M	0.773M
3	13.88M	12.94M	0.935M	0.703M
4	13.88M	12.94M	0.935M	0.639M
5	13.88M	12.94M	0.935M	0.581M
6	13.88M	12.94M	0.935M	0.528M

Table 37: Payback Period in year

No. of Years (k)	Cumulative Present Worth ($)
0	- 2.53M
1	- 1.68M
2	- 0.910M
3	- 0.207M
4	0.432M
5	1.01M
Payback Period	**3 Years**

CHAPTER 8

HAZOP ANALYSIS

9.1 HAZOP Methodology

The HAZOP methodology, which stands for "Hazard and Operability," is a strategy that is structured and methodical, and it is used to detect and assess potential risks and concerns with operability in process systems. In the subject of process safety, it is a method that is well recognized and utilized, and it is frequently employed during the period of a process in which it is either designed or modified.

In order to carry out an analysis, the following phases are utilized from the HAZOP procedure:

1. Get started with a comprehensive flow sheet. The flow sheet should be broken up into a number of different process units. Therefore, the reactor area may be considered one unit, while the storage tank could be considered another. Choose a unit to focus your attention on.
2. Decide on a study node, which could be a vessel, a line, or an operational instruction.
3. Specify the goals you have in mind when designing the research node. As an illustration, the vessel V-1 is intended to be a repository for the benzene feedstock and to supply the reactor with it on demand.
4. Select a process parameter from the following options: flow, level, temperature, pressure, concentration, pH, viscosity, agitation, volume, reaction, sample, component, start, stop, stability, and inert.
5. Attach a guiding word to the process parameter so that possible deviations can be suggested.
6. If the deviation is relevant, find out what could have caused it and make a note of any safeguards that are in place.
7. Determine if there will be any repercussions as a result of the departure.
8. Suggest a course of action.
9. Make sure to record all of the information.

10. Repeat steps 5 through 9 until all applicable guide words have been applied to the selected process parameter.
11. Repeat steps 4 through 10 until all process parameters that are relevant to the specified research node have been taken into consideration.
12. Repeat steps 2 through 11 until all of the study nodes for the given segment have been taken into consideration, and then move on to the next area on the flow sheet.

9.2 HAZOP Analysis on Reactor

In the realm of process safety, a method known as HAZOP, which stands for Hazard and Operability Study, is a method that is both methodical and structured, and it is used to identify and evaluate potential risks and operability issues in a process plant or system. It is primarily utilized during the design or modification phase of a process to guarantee that dangers are identified and that adequate safeguards are put in place to prevent accidents. This is done to ensure that the process is safe. It analyzes every step of the process by making use of directional terms such as "No," "more," "less," "part of," "as well as" "other than" "sooner," "later," and "reverse." We explain the likely or potential reasons for these deviations as well as the repercussions associated with them, along with the relevant activities that can be taken to mitigate the risks that have been discovered.

Table 38: HAZOP Analysis of Reactor Flow

Item	Study node	Process parameters	Deviations (Guide words)	Possible Causes	Possible Consequences	Action Required
1A	Reactor	Flow	No	Failure of hydrogen supply valve	Incomplete reaction, reduced production	Check and repair/replace the hydrogen supply valve, implement redundant valves or backup systems, and implement an alarm system
				CTA supply failure	No reaction, loss of production	Investigate and address the CTA supply failure, check for blockages or leaks, and implement redundant CTA supply systems
1B			High	Malfunction of hydrogen flow control valve	Over pressurization, increased reaction rate	Repair or replace the malfunctioning hydrogen flow control valve, install pressure relief valves or safety valves, and implement an alarm system

				Malfunction of steam flow control valve	Excessive heat transfer, potential reactor overheating	Repair or replace the malfunctioning steam flow control valve, install additional temperature sensors, implement automated systems, and ensure proper insulation and cooling mechanisms
1C			Low	Steam supply failure	Inadequate heat transfer, reduced reaction rate	Investigate and address the steam supply failure, check for blockages or leaks, and implement redundancy in the steam supply system
1D			Part of	Partial blockage in the CTA line	Incomplete reaction, variable production	Identify and remove the blockage in the CTA line, install additional flow meters or sensors, and implement regular maintenance and cleaning procedures
1E			Reverse	Backflow due to incorrect	Disruption of heat transfer,	Correct valve positions, install check valves or non-

				valve positions	potential reactor overheating	return valves, and implement regular inspections and maintenance
1F			Sooner	Premature opening of hydrogen supply valve	Uncontrolled reaction, increased reaction rate	Implement interlocks or safety systems, improve control system response time, and enhance operator training and awareness
1G			Later	Delayed opening of CTA supply valve	Inadequate reaction time, reduced production	Improve response time of the CTA supply valve, monitor and optimize CTA feed scheduling, and implement backup systems

Table 39: HAZOP Analysis of Reactor Pressure

Item	Study node	Process parameters	Deviations (Guide words)	Possible Causes	Possible Consequences	Action Required
1A	**Reactor**	**Pressure**	No	Failure of pressure source or loss of steam supply	Incomplete or halted reaction, reduced production	Investigate and address the pressure source failure, ensure sufficient steam supply, and implement redundant pressure sources or backup systems
1B			More/Higher	Malfunction of pressure control valve or excessive steam flow	Over pressurization, potential reactor failure	Repair or replace the malfunctioning pressure control valve, install pressure relief valves or safety valves, and implement an alarm system to monitor and control pressure levels

1C			Less/Lower	Insufficient steam supply or pressure drop	Inadequate heat transfer, reduced reaction rate	Investigate and address the steam supply failure, check for blockages or leaks, and ensure sufficient steam pressure is maintained
1D			Part of	Uneven distribution of reactants or heat transfer	Inefficient reaction, inconsistent product quality	Optimize reactor design for better reactant and heat distribution, monitor and balance reactant flow rates, and ensure proper insulation and cooling mechanisms

1E			Reverse	Incorrect valve positions or backflow	Disruption of reaction, potential equipment damage	Correct valve positions to prevent reverse flow, install check valves or non-return valves, and implement regular inspections and maintenance of the valve systems
1F			Sooner	Premature increase in pressure due to rapid heating	Uncontrolled reaction, potential safety hazards	Implement interlocks or safety systems to prevent rapid heating, improve control system response time, and enhance operator training and awareness

1G			Later	Delayed increase in pressure due to slow heating	Inadequate reaction time, reduced production	Optimize heating processes, monitor and adjust heating rates, and ensure proper heat transfer mechanisms are in place

Table 40: HAZOP Analysis of Reactor Temperature

No.	Study node	Process parameters	Deviations (Guide words)	Possible Causes	Possible Consequences	Action Required
1A	**Reactor**	**Temperature**	No	Failure of heating system or control	Incomplete reaction, reduced production	Investigate and address the heating system failure, check for malfunctioning components, ensure proper power supply, and implement redundant heating systems or backup heaters
1B			Higher	Malfunction of temperature control system or excessive heating	Overheating, potential reactor failure	Repair or replace the malfunctioning temperature control system, install additional temperature sensors, implement automated systems for temperature control, and ensure proper insulation and cooling
1C			Less/Lower	Insufficient heating or cooling system performance	Inadequate heat transfer, reduced reaction rate	Investigate and address the heating or cooling system failure, check for blockages, leaks or malfunctioning components, and ensure proper calibration and

						maintenance of the temperature control equipment
1D			Part of	Uneven distribution of heat or reactants	Inefficient reaction, inconsistent product quality	Optimize reactor design for better heat and reactant distribution, monitor and balance temperature gradients, and ensure proper mixing and circulation mechanisms
1E			Reverse	Incorrect valve positions or backflow	Disruption of heat transfer, potential reactor overheating or cooling	Correct valve positions to prevent reverse flow, install check valves or non-return valves, and implement regular inspections and maintenance of the valve systems
1F			Sooner	Rapid increase in temperature due to process anomalies or control failure	Uncontrolled reaction, potential safety hazards	Implement interlocks or safety systems to prevent rapid temperature rise, improve control system response time, and enhance operator training and awareness
1H			Later	Delayed increase in temperat	Inadequate reaction time, reduced production	Optimize heating processes, monitor and adjust heating rates, and ensure proper heat

				ure due to slow heating or control issues		transfer mechanisms are in place

CONCLUSION

We were able to accomplish this by increasing the size of the purification facility so that it could accommodate the additional 40 tons/day of CTA, which resulted in an increase in the amount of PTA that was produced. This has resulted in additional revenue generation of $13 million with a relatively minimal cost of initial investment. Furthermore, the expansion helps to reduce the amount of wasted CTA that is produced when there is an excess.

capacity expansion: as a result of the expansion of the purification plant, the business was able to process a greater quantity of CTA, which in turn led to an increase in the quantity of PTA that was manufactured.

The new purification plant was built to be more efficient than the old one, which contributed not just to the increase in PTA production but also to the overall increase in production.

Better quality control: the newly constructed purification plant was outfitted with cutting-edge machinery that contributed to an improvement in the product's overall quality, which in this case was the PTA. Because of this, there was a subsequent rise in demand for the PTA goods offered by the company.

The increased production of PTA has brought about a variety of positive outcomes for the corporation, including the following:

As a direct result of the increased PTA production, the company has seen an increase in revenue of $13 million as a direct result of the higher output.

Profitability has increased as a result of the increased income and the decreased costs that are linked with the new purification facility. This has led to an improvement in the profitability of the business.

Enhanced market position: the firm's market position has been enhanced as a result of the increased production of PTA as well as the improvement in the quality of the PTA products that the company manufactures.

The corporation made a smart investment by expanding its facility that handles the purification process. As a direct consequence, we have seen a rise in sales, an improvement in profitability, and an improvement in our position in the market. Because of this investment, the company now has a solid foundation upon which to build its future expansion.

In addition to the benefits that were discussed previously, the enlargement of the plant that performs the purifying process has also assisted in cutting down on the amount of unused CTA that is created whenever there is a surplus. This is due to the fact that the newly constructed purification facility is more effective than its predecessor in terms of both efficiency and quality control. As a consequence of this, the business is in a position to generate a greater quantity of PTA from each ton of CTA that it employs. Both the company's financial situation and its impact on the surrounding environment have improved as a result of these changes.

RECOMMENDATIONS FOR FUTURE RESEARCH

1. Process Optimization:
   In the interest of enhancing PTA output, additional study may be directed on the optimization of the crystallization process. To achieve this goal, it may be necessary to investigate various process parameters, such as temperature, pressure, residence time, and cooling rates, in order to improve crystallization productivity and yield.
2. Development of a Comprehensive Kinetic Model for the Crystallization Process:
   The development of a comprehensive kinetic model for the crystallization process can provide useful insights into the underlying mechanisms and assist in the process of improving the process. In subsequent investigations, the formulation and verification of mathematical models may play a role in elucidating the nucleation, growth, and agglomeration behaviors that occur throughout the crystallization process.
3. Elimination of Impurities: The synthesis of PTA from CTA necessitates the elimination of impurities in order to reach the appropriate levels of purity. In the future, research may investigate more advanced methods of purification, such as adsorption, filtering, or membrane processes, with the goals of improving the effectiveness of impurity removal and reducing the overall expenses of the process.
4. Process Monitoring and Control: The development of innovative monitoring and control systems has the potential to improve the crystallization process's operational efficiency as well as its dependability. In the future, research can investigate the possibility of integrating advanced control algorithms with real-time process monitoring technologies, such as spectroscopy or imaging methods, in order to enable better process control and optimization.
5. Energy Efficiency: Investigating energy-efficient methods for the crystallization process can be beneficial to both the environment and the bottom line.
6. New Oxidation Process: A new process could also be developed perhaps using ionic liquids that could eliminate the need for hydro purification by oxidizing paraxylene such that 4-CBA is minimized.

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