INDUSTRIAL TRAINING REPORT

on

STUDIES ON REACTORS, CATALYSTS AND REACTION APPLICATIONS

completed at

CSIR-INSTITUTE OF PETROLEUM (IIP) CERTIFIED INSTITUTE

(ISO 9001: 2015)

DEHRADUN (INDIA) DURING THE PERIOD

3rd June, 2024 – 21st July, 2024

Under the guidance of

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Submitted in partial fulfillment of the requirements for Summer Training Internship

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Declaration

I extend my heartfelt appreciation to **Dr. Harender Singh Bisht, Director of the Indian Institute of Petroleum, Dehradun**, for granting me the opportunity to conduct research at this esteemed institute.
I am deeply grateful to **my mentor, Dr. N.Viswanadham**, whose unwavering guidance and encouragement introduced me to the fascinating field of chemical technology research. His mentorship has been instrumental in shaping my understanding and approach.
I would like to acknowledge and thank **Mr. Amit Sharma, Dr. Anup Prakash Tathod and Dr. A. Selvamani** for their wholehearted support and timely assistance throughout the course of my project. Additionally, I express my sincere appreciation to the research assistants who diligently contributed to the success of this project.

I hereby declare that this written submission reflects my own ideas expressed in **my own words**. Where I have included the ideas and words of others, I have appropriately cited and referenced the original sources. I affirm that I have adhered to all principles of academic honesty and integrity. **There has been no misrepresentation, fabrication, or falsification of any idea, data, fact, or source in my submission.**. I acknowledge that any violation of the above principles may result in disciplinary action by the Institute. Furthermore, failure to properly cite sources or obtain necessary permissions may lead to penal action from the concerned parties.

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Priyanshu Bist	**Supervisor**
Date: 19-07-2024	**Date: 19-07-2024**
Place: CSIR-IIP, Dehra Dun	**Place: CSIR-IIP, Dehra Dun**

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About CSIR-IIP DehraDun

The CSIR-Indian Institute of Petroleum (CSIR-IIP) in Dehradun is a premier research laboratory under the Council of Scientific and Industrial Research (CSIR). Since its establishment in 1960, CSIR-IIP has been at the forefront of research and development in the downstream sector of hydrocarbons and related industries. The institute boasts a team of highly skilled and experienced professionals and is equipped with advanced facilities, including state-of-the-art pilot plants.

CSIR-IIP is dedicated to delivering globally competitive technologies and services for the hydrocarbon and related industries.

CSIR-IIP has successfully developed and transferred over fifty technologies to industries in the areas of petroleum refining, natural gas, petrochemicals, chemicals, and the utilization of petroleum products. The institute also provides services to the petroleum industry, assists in the formulation of standards for petroleum products with BIS, and conducts techno-economic feasibility studies and market demand surveys.

In summary, CSIR-IIP Dehradun continues to play a crucial role in advancing research and development for the downstream sector of the hydrocarbon industry, providing essential technologies, training, and services to enhance the efficiency and sustainability of the industry.

Abstract

In this process, catalysts do not change at all, as they facilitate the occurrence of chemical reactions at a greater rate. Modern day industries prefer heterogeneous catalysts to homogeneous ones because they are easy to handle and recycle.

Characterization and optimization present difficulties in spite of advantages like easier separation and reusability, since they occur in different phase with respect to reactants and products. Zeolites like ZSM-5 is one such example of heterogeneous catalyst which is characterized by a porous crystalline three-dimensional framework consisting of silicon, aluminum, oxygen and alkali or alkaline earth metals. Its structure has been tailored for capturing polluting substances hence making it ideal for many chemical processes.

One important use of zeolites includes converting naphtha feedstock into reformates that contain high levels of benzene, toluene xylene (BTX) and other aromatic compounds which are needed for gasoline blending purposes as well as petrochemical operations. Hence zeolites can be used in transformations of naphtha feeds into reformates with high concentration of benzene, toluene xylene (BTX) and other aromatics required for gasoline mixing and petrochemical applications.

This has given rise to advanced technologies such as High-Pressure Micro Reactor use in these reactions. Also looked into will be the importance of reactors such as High-Pressure Micro Reactors used in catalytic processes.

Contents

1 Introduction to Catalysts

A catalyst is a substance that facilitates a chemical reaction without being consumed in the process. It is introduced into the reaction in very small quantities compared to the reactants, typically in the range of 10^{-6} to 10^{-1} molar ratios. Despite participating in the reaction, the catalyst remains unchanged and does not appear in the final reaction products, which is why it is often noted on the reaction arrow in chemical equations (e.g., $A + B \rightarrow C + D$ [cat.]).

Catalysts do not affect the overall thermodynamics of the reactions they catalyze; they do not make an impossible reaction feasible. Instead, they provide an alternative reaction pathway with a lower activation energy, thereby increasing the rate of REXN. Key performance metrics for catalysts include TOF (the number of reactions facilitated per unit time by one catalyst site) and TON (a total number before deactivation).

2 Types of Catalysts:

Catalysts are generally classified into two main types: **homogeneous** and **heterogeneous**

2.1 Homogeneous Catalysts

In homogeneous catalysis, the catalyst is in the same phase as the reactants, commonly dissolve in a liquid. This allows for uniform reaction media and ease of study on mechanisms. However, it may be difficult to separate the catalysts from the reaction mixture. It includes common examples such as different transition metal complexes used in organic synthesis.

2.2 Heterogeneous catalysis

Involves catalysts that are in a different phase with reactant usually solid Catalysts and gas or liquid reactants. This kind of catalysis is frequently applied in industrial processes since separation of solid catalysts from reaction mixture can be done easily and these can be reused even under high temperature conditions. Some examples include ammonia (NH_3) production catalysts, selective oxidation of methane (CH_4) into methanol(CH_3))$andseveralprocessesfoundinpetrochemica$

3 Solid Acid Catalysts

Solid acids similar to liquid acids e.g., sulfuric and hydrochloric acids have catalytic properties on their surface. Their use supports sustainable chemical process development by enhancing productivity metrics and minimizing environmental impact. Diverse industrial sectors use solid acid catalysts for instance, catalytic cracking in petroleum refining to the synthesis of fine chemicals.

4 Introduction to Zeolites

Zeolites, named from the Greek words "Zeo" (boiling) and "Lithos" (stone), are crystalline solids that form naturally when molten lava interacts with seawater or freshwater containing dissolved salts and can also be synthesized in chemistry laboratories. They have a unique porous structure characterized by angstrom uniform-sized pores resembling a honeycomb. Most of the zeolite frames comprise silicon, oxygen and Aluminum atoms which are sometimes substituted by alkali or alkaline earth metals giving them negative electric charges that make them excellent adsorbents for molecular sieves.

4.1 Zeolite [ZSM-5]

ZSM-5 is an interesting composition among zeolites because of its structure. Moreover, it belongs to the pentasil family, characterized by the chemical formula NaAlSiO·16HO (0 ¡ x ¡ 27). The strong acidity results from its high SiO2/Al2O3 mole ratio that places it under the pentasil family of zeolites. This is why ZSM-5 is very receptive to shape-selective catalytic applications and this includees processes such as alkylation, cracking and isomerization within various sectors of chemistry. Its framework is made up of corner-sharing tetrahedral units consisting of aluminum and silica, thereby conferring upon it distinct absorptional and catalytic characteristics.

4.2 Structure and Properties of Zeolites

Zeolites have a crystal-like structure with even-sized pores. These pores measure 1 to 20 angstroms across. SiO4 and AlO4 tetrahedra share corners to create this structure. This setup lets zeolites pick up molecules based on their size and polarity. Zeolites work well as catalysts due to their large surface area and controlled acidity. For example when three-valent aluminum joins the framework, it brings extra negative charges. Cations can balance these charges. When protons (H+) act as cations, they give zeolites Bronsted acidity. Lewis acid sites form when aluminum atoms don't have hydroxyl groups.

4.3 Applications and Preference in High-Pressure Environments

Zeolites, like ZSM-5, play a key role in high-pressure industrial processes because of their strong crystal structure and steady catalytic performance in tough conditions. These materials are essential for jobs such as fluid catalytic cracking (FCC) in oil refining where they help turn heavy hydrocarbons into lighter more valuable products like gas and diesel. Their ability to keep their shape and stay active at high pressures and temperatures makes them the go-to catalysts in industrial reactors that need to be productive and reliable.

5 Preparation of Metal Zn-ZSM-5 Catalyst Extrudate

ZSM-5 short for Zeolite Socony Mobil-5, plays a key role as a catalyst in many chemical processes. Its popularity stems from its strong acidity and special structural features. To make metal-incorporated ZSM-5 catalysts, researchers use one of two approaches: the physical method or the ion exchange method.

5.1 Extrudate Preparation Components:

- **HZSM-5:** This zeolite component serves as the catalyst base. To get the best results, we mix HZSM-5 with Boehmite/binder powder of Alumina in a 3:2 ratio. Here's why:

  Mechanical Strength: Alumina makes the extrudate stronger.

  Dilution of Acidity: Alumina also tones down the zeolite's acidity making it fit better for certain reactions.

  Binder Powder of Alumina: This inactive part glues things together and gives the extrudate extra strength.

- **Glacial Acetic Acid (GAA):** This substance helps things stick together. We add a small amount (3 percent) of GAA to clean water to create a tight well-mixed slurry.

5.2 Physical Method (Incipient Metal Incorporation)

The physical method is a simple way to add metal to the catalyst after creating the initial extrudate. Here's how it works:

- **Mixing ZSM-5 and Binder:**
  Begin by combining ZSM-5 with a binder, like alumina, in a clean dry mortar. Pour a small amount of glacial acetic acid solution into the mix. This creates a thick sticky paste called a slurry.

- **Extrudate Formation:**
  Push the slurry through an extruder to shape it into the wanted form. The extruder ensures uniform shapes, which are key for effective catalytic reactions.

- **Drying and Calcination:**
  Let the shaped extrudate dry at room temperature. This step takes a few hours and gets rid of any leftover moisture.
  After it's dry, put the extrudate in a furnace and heat it up (a process called calcination)

for a few hours. This heating makes the extrudate stronger and ready to incorporate metal.

- **Metal Incorporation:**
  Put the dry extrudate in a mix with the metal salt, like zinc nitrate or gallium salt. Shake the mix a bit so the metal salt spreads out and soaks into the extrudate.

- **Final Drying and Calcination:**
  Dry the extrudate with metal in it one more time.
  Heat the extrudate hot in a furnace to finish up. This last step makes sure the metal sticks well in the catalyst structure.

- **Shaping:**
  Shape the catalyst to fit what you want to use it for. You can now use the extrudate in different chemical reactions.

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5.3 Ion Exchange Method

The ion exchange method puts the metal right into the ZSM-5 framework by swapping ions inside the structure. Here's a step-by-step guide to do this:

- **Getting the Metal Salt Solution Ready**

  Make a solution using a metal salt, like zinc nitrate or gallium salt. You'll use this solution to bring the metal into the ZSM-5 structure.

- **Blending with ZSM-5:**

  Put the ZSM-5 and the metal salt solution together in a round-bottom flask. Heat the mixture with a heating mantle. This process helps ions swap letting metal ions take the place of some ions in the ZSM-5 structure.

- **Drying:**

  Let the mixture dry at room temperature for a few hours. This first drying step gets rid of extra moisture.

  After this, put the mixture in an oven to dry it more. This step makes sure the mixture is dry before you move on.

- **Extrudate Formation:**

  Use an extruder to shape the dried stuff into the catalyst form you want. The extruder helps make even shapes, which you need for the catalyst to work well.

- **Final Calcination:**

  Put the shaped catalyst in an electric oven and heat it up hot. This last heating step makes sure the metal is mixed into the ZSM-5 structure and the catalyst is ready to use in chemical reactions.

6 How to Load the Catalyst

The steps below show how to put a Zn-ZSM-5 catalyst into a high-pressure reactor pilot plant. This helps to place the catalyst right for the best reaction conditions.

6.1 Parts

- Zn-ZSM-5 zeolite catalyst

- Alpha-alumina
- High-pressure reactor with a reactor rod

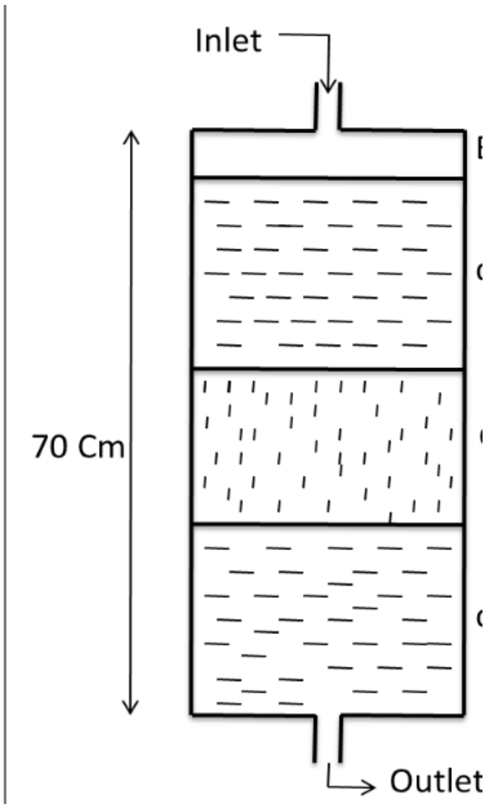
6.2 Machine Details

- Reactor rod size: 70 cm long, 13 cm wide
- Catalyst amount: 7 g
- The total weight of alpha-alumina is 50 g
  - Upper zone has 25 g of alpha-alumina
  - Lower zone has 25 g of alpha-alumina

6.3 Procedure

6.3.1 To Get the Catalyst and Reactor Ready

1. Weigh 7 g of the Zn-ZSM-5 catalyst.
2. Weigh 50 g of alpha-alumina splitting it into two equal parts of 25 g each.



6.3.2 Loading the Reactor Rod

1. The reactor rod has three separate parts: top, middle, and bottom.

   Upper Zone: Start by putting 25 g of alpha-alumina in the top part of the reactor rod. This creates a base to support the catalyst and helps keep everything in place.

   Middle Zone: Put 7 g of Zn-ZSM-5 catalyst in the middle zone. This zone is super important because it's where most of the catalytic stuff happens.

   Lower Zone: Fill the bottom zone of the reactor rod with the leftover 25 g of alpha-alumina. This helps hold up the catalyst and lets the reactants and products flow better.

2. Remember to keep some room in the reactor rod called the "empty space." This space is key to let gases move and make sure reactants mix well with the catalyst. It helps stop pressure from building up too much and allows for better control of the reaction

setting.

6.3.3 Pressurization with Nitrogen Gas

1. Once you've put in the catalyst and alpha-alumina, close up the reactor rod tight.
2. Start to pressurize the system using nitrogen gas. First, put in nitrogen gas to clean out any air and create a neutral environment inside the reactor.
3. pump nitrogen gas to raise the pressure to 15 bar. This helps the system get to the right working pressure without any sudden jumps.
4. Keep a close eye on the system to spot any pressure drops, which might mean there are leaks. Do all the needed checks and tweaks to make sure the system doesn't leak.

6.3.4 Final Checks

1. After you hit 15 bar, keep it there and watch to see if it stays steady. Make sure there are no leaks and the pressure doesn't change.
2. When the system is stable and doesn't leak, it's all set for the catalytic reactions to happen under controlled settings.

7 Start up Procedure:

7.1 Initial Pressurization and Leak Testing

1. The system goes through pressurization and depressurization with ultra high-purity nitrogen gas up to 25 kg/cm^2 after loading the catalyst. This step has the purpose to check for leaks and to make sure the system is airtight.
2. The system undergoes depressurization to 10 bars once the leak testing is done.

7.2 Heating and Nitrogen Flow

1. They heat up the reactor bit by bit at 60 °C every hour till it gets to 500 °C. This slow heating is key to stop sudden temperature changes and make sure the heat spreads .
2. As it's heating, they keep pumping in nitrogen gas at 30 liters per hour. This steady flow of nitrogen helps to spread the heat in the whole reactor.

7.3 Feed Vessel Preparation

1. They pump up the feed vessel with nitrogen gas to 2 bars. This pressure keeps the feed from turning into vapor and gives enough force to push the feed to the pump easily.
2. The feed has a weight hourly space velocity (WHSV) of 3 when it starts.

7.4 Stabilization and Material Balance Periods

1. The system runs for 2 hours under the wanted test conditions. This first part is called the stabilization period. During this time, the system settles into a steady state.
2. After stabilizing, the system moves into the material balance period. This also takes 2 hours. In this time, workers collect gas samples to analyze.

7.5 Startup of Air Compressor

1. The air compressor gets turned on and emptied before starting the unit to make sure it doesn't have any water or other stuff in it.

8 Reaction Process

8.1 Reduction of the Catalyst

To get rid of oxygen molecules in Zn-ZSM-5 and create active Zn^{2+} that's not attached to anything, we need to reduce it. This step is super important because this Zn^{2+} is what makes reactions happen in the High Pressure Micro Reactor (HPMR).

$$ZnO + H_2 \rightarrow Zn^{2+} + H_2O \tag{1}$$

The catalyst gets cleaned with nitrogen before it's reduced. This gets rid of any water that forms during regeneration and reduction. To reduce the catalyst, they use a 20 L/H hydrogen atmosphere. The hydrogen pressure is 7 bar, and the temperature is 500°C.

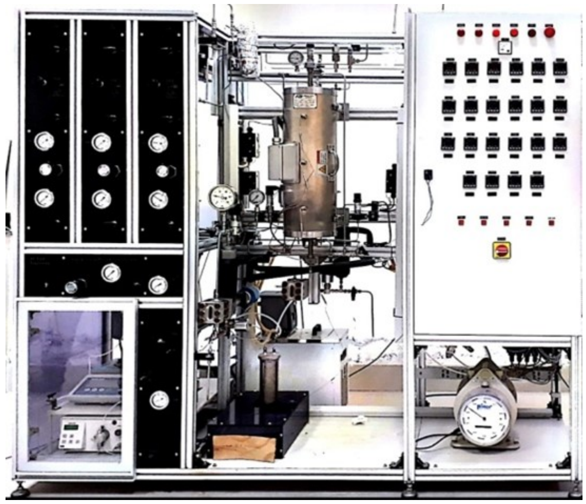
8.2 Catalytic Reforming

Catalytic reforming changes low-octane naphthas into high-octane gasoline parts called reformates. This is a big deal in making gasoline. A bunch of reactions happen at the same time during this process. These include cracking, polymerization, dehydrogenation, and isomerization. The naphtha feedstock's properties and the catalysts used have an impact on reformate production. The feedstock's makeup - its paraffin, olefin, naphthene, and aromatic content - determines these properties. Reformates are useful to blend gasoline and process petrochemicals. Hydrogen, a key by-product of reforming, gets separated from the reformate. People recycle it to use in other processes. Catalytic reforming will stay one of the most important unit processes in the petroleum and petrochemical industry. This is because people want more aromatics and higher octane numbers.

9 High Pressure Micro Reactor (HPMR) Plant Overview

The High Pressure Micro Reactor (HPMR) is a complex high-pressure reactor built to handle small-batch reaction chemistry. It's super helpful for research, to develop processes, and to test stuff when you're working with reagents, catalysts, or other important materials. This cool plant lets you run things or , which makes it easy to use and flexible. The HPMR can hold more than 22 grams of catalyst, so you can do experiments like they do in big industries. This ability gives you useful science info, including cool stuff about reactions and how materials behave.

10 What the Plant Looks Like



The HPMR can handle lots of different feedstock samples and work at different temperatures, pressures, and flow rates for both feedstock and gases. It has a max working pressure of about 70 bars and can get as hot as 700°C. A computer interface helps control and watch the system's temperature, pressure, and flow rate , while also keeping equipment data. You can run the HPMR through system software, but there's also a choice to control it by hand using a PID (Proportional Integral Derivative) control panel. The HPMR plant has three main areas:

1. **Feed Zone**
2. **Reaction Zone**
3. **Product Zone**

10.1 Feed Zone

The Feed Zone has two parts: Liquid Feed Section and Gas Feed Section. **Liquid Feed Section:** The Liquid Feed Section has a feed tank that can hold 2 liters. It sits on a weighing balance. A plunger-type pump that goes back and forth moves liquid feed into the reaction zone. The liquid feed goes through different operations and control equipment. These are needed to feed .

Feed Pump:

Plunger pumps move liquid through a cylinder using a plunger. These pumps work great for high-pressure needs thick fluids, and to control flow rates . This makes them a good fit for the reforming process. People also call these positive displacement pumps metering pumps. They're a type of pump that moves back and forth to do its job. The feed vessel gets a blanket of inert gas (nitrogen) to keep the liquid from breaking down and to make sure the fluid moves right from the tanks. This also stops the tank from getting dented and helps suck the liquid into the pump . To move feed from the pump to where the reaction happens, you need a pressure gauge, a pressure safety valve (PSV), and a three-way valve (TWV).

Gas Feed Section:

The Gas Feed Section has cylinders, regulators forward pressure regulators, mass flow controllers, filters, and different valves. These parts work together to control and move the needed gas into the plant. Pressure is super important for moving gases around. **Cylinders:** Gas cylinders come with pressure regulators that have two pressure gauges. One gauge shows how much pressure is inside the cylinder, and the other shows the pressure in the line. Each cylinder can hold 140 kg of gas. **Back Pressure Regulator (BPR):** The BPR has a function to relieve pressure. It opens when the system pressure goes above a set point. This bleeds off the working fluid at the needed rate to keep system pressure steady. If you turn the knob clockwise, it lowers the inlet pressure. Turning it counterclockwise does the opposite - it raises the inlet pressure. **Forward Pressure Regulator (FPR):** The FPR keeps the downstream pressure in check. To increase the pressure, you turn the knob clockwise. To decrease it, you turn it counterclockwise.

Mass Flow Controller (MFC): The MFC has an influence on the flow rate of fluids and gases. It has an inlet port, an outlet port, a mass flow sensor, and a control valve that's proportional. The MFC uses a closed-loop control system to adjust the valve. This system aims to get the right flow based on what the mass flow sensor tells it.

Manual Control Valves: The HPMR pilot plant uses different types of manual control valves such as:

1. **Plug Valve (PV and BV)**

2. **Needle Valve (NV)**

3. **2-Way and 3-Way Valves (2-WV and 3-WV)**

They put a special valve called a Check Valve at the cylinder's opening to make sure fluid only goes one way. **Safety Valve:** Safety valves play a key role in gas flow pipelines or gas storage tanks. When the pressure in the line gets too high, they open up on their own and let gas out. This stops damage to stuff and pipelines. To work right, the safety valve's set value should be lower than what the pipeline and tank were built to handle.

10.1.1 Reaction Zone

The Reaction Zone is where stuff happens. It's got a preheater and a reactor. Things heat up to between 300°C and 500°C, and the pressure changes from 10 bar to 60 bar. To make it work better, they heat the feed to 150°C before it goes into the reactor.
The reactor is a fixed-bed tube that's 70 inches long. This is where the reactions happen at the same temperature. They use electricity to keep it hot enough because the reforming reactions need heat to work.

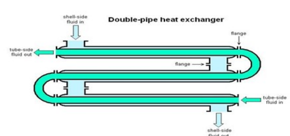
- **Preheater:** The preheater gets the feed hot enough - about 150°C - before it goes into the reactor. This makes sure the reaction happens as a gas.

- **Reactor:** The reactor makes heterogeneous reactions happen, with the catalyst as a solid and the feed as a vapor at 500°C. The reactor's temperature and pressure are key to the reaction, and three heaters between stages keep the heat even. The reactor has three parts: preheat, reaction, and post-heat. Thermocouples in all three areas measure both skin and core temperatures. Temperature safety switches (TSS) shut off the furnaces if it gets hotter than 700°C. The reactor pressure is the same as the plant pressure, with a top design pressure of 70 bars.

10.1.2 Product Zone

The Product Zone cools and separates the reaction products. A double-pipe spiral heat exchanger with a cold water bath cools the products. A two-phase separator then splits the chilled products into liquid and vapors. The vapors go through a pressure control valve (PCV) that controls the whole plant's pressure. They then enter a low-pressure separator to split any leftover liquid. The separator keeps a steady minimum liquid level to stop gaseous products from getting into the liquid product line.

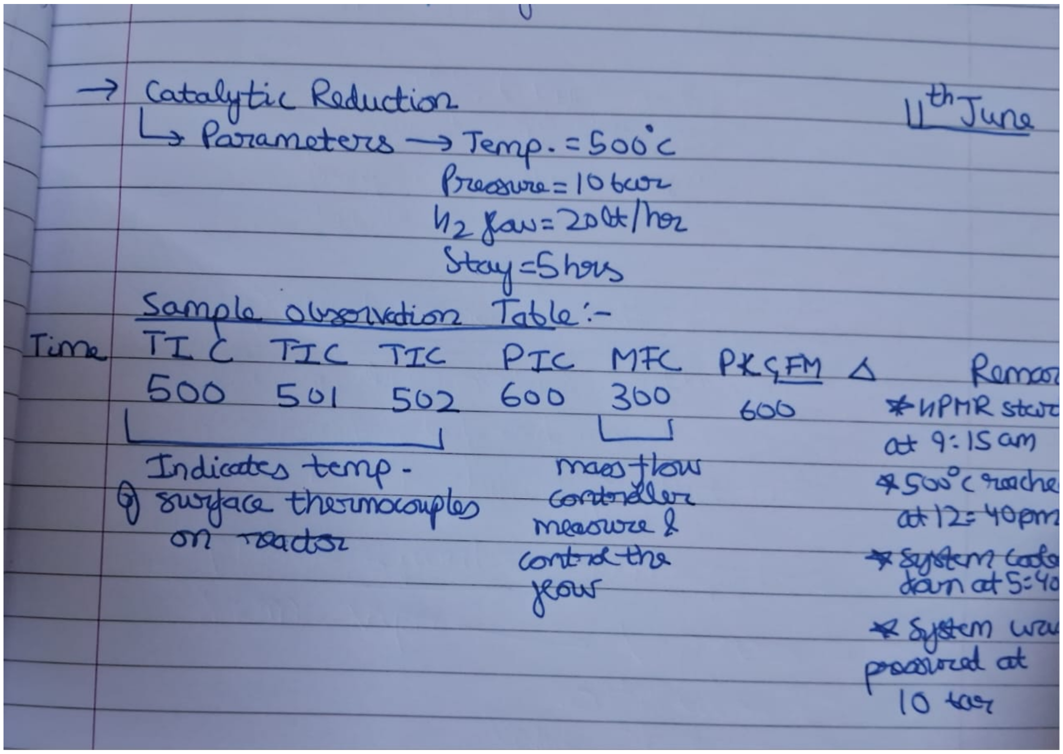
- **Double Pipe Heat Exchanger:** This exchanger has the job of cooling down the hot vapors coming from the reactor. To do this, it uses distilled water that's been chilled to 6°C.



- **High and Low Pressure Separators (HPS and LPS):** The high-pressure separator splits the product into lighter (vapor) and heavier (liquid) parts. After that, the leftover vapor from the HPS goes to the LPS through a PCV. The LPS then separates more vapor at low pressure, and they collect the liquid product in a tank. To keep things cool, the separators have a layer of glass wool and copper coils that carry water. These coils help to cool everything down. • Pressure Control Valve (PCV): The PCV has the job to control plant pressure. It's a diaphragm control valve that shuts with air and keeps vapor speed in check. An air compressor gives the needed pressure to make the PCV work right.

• Gas Flow Meter (GFM): The GFM keeps track of how much gas goes to the storage tank. It also makes sure things move by letting out extra pressure from the control valve.

11 Things we saw while training:



→ Catalytic Reduction

11th June

↳ Parameters → Temp. = 500°c

Pressure = 10 bar

H_2 flow = 20 lt/hr

Stay = 5 hrs

Sample observation Table:-

Time	TIC	TIC	TIC	PIC	MFC	PKGFM	Δ	Remar
	500	501	502	600	300	600		* HPMR star at 9:15 am * 500°c reache at 12:40pm * System coole down at 5:40 * System wa pressurized at 10 bar

Indicates temp. @ surface thermocouples on reactor

mass flow controller measure & control the flow

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