Synthesis and Determination of Active Acidic/Basic Sites on Zn Loaded Zeolite (ZSM-5) Catalyst & Its Role in Catalytic Reforming of Naphtha Using HPMR Reactor

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Abstract: The crystalline aluminosilicate ZSM-5 (Zeolite Socony Mobil) has a high Si to Al ratio. It is utilized as a catalytic support material. Zeolite has huge industrial applications. They are commonly employed as heterogeneous catalyst for hydrocarbon isomerization reactions in the petroleum sector. In general, zeolite is synthesized as a powder with low mechanical strength. Powdered ZSM-5 and binder (alumina) were combined, and glacial acetic acid (peptide agent) was added. Mix them thoroughly before loading them into the extruder to form the shape. After drying for one day to let the acetic acid drain, they were heated for eight hours and calcined to remove the liquid from the pores, which aids in the formation of pores/active sites. By mixing the $ZnNO_3$ solution with the sample, the wet impregnation process is utilized to load Zn metal on extruded ZSM-5. Zn is chosen for loading because it favours cyclization and forming aromatic compounds. After many hours of such wet contact, the suspension evaporates, and the chemical is deposited randomly inside and outside the zeolite pores. Further heating and calcination are carried out. TPD (temperature-programmed desorption) of H/ZSM-5 is performed under catalyst characteristics. TPD of H/ZSM-5 reveals that it comprises primarily highly acidic sites with temperatures above 500°C. The prepared catalyst is employed in the naphtha reforming process in the HPMR (High-Pressure Micro Reactor) to produce BTX as the product.

Keywords: Alumino-Silicate, Heterogeneous, Peptide Agent, Isomerization, Naphtha Reforming

1. Introduction

A catalyst is a substance that increases the rate of the reaction at which a chemical system approaches equilibrium, without being substantially consumed in the process. Hence a catalyst can be recovered chemically unchanged at the end of the reaction it has been used to speed up or catalyse.

For chemicals to react, their bonds must be rearranged, because the bonds in the products are different from those in the reactants [1]. The slowest step in the bond rearrangement produces what is termed a transition state.

A chemical species that is neither a reactant nor a product, but is an intermediate between the two:

$$\text{Reactant} \rightleftarrows \text{Transition State} \rightleftarrows \text{Product}$$

Figure 1. Flow diagram of conversion of reactant into product via transition state.

Catalytic processes have great significance and about 90% of all chemical industry involves catalytic processes. Of all the industrial catalytic processes, approximately 80% involve the use of solid catalysts in this 17% homogeneous catalysts are used, and rest 3% are biocatalysts. Thus, heterogeneous catalysts, particularly solid catalysts, dominate the industrial catalytic [2] processes. The contributions of homogeneous catalytic processes in the

chemical industry are significantly smaller than that of heterogeneous catalytic processes.

Catalytic reforming is a major conversion process in petroleum refinery and petrochemical industries. It is a well-known type of petroleum refinery operation that is employed to improve the octane rating of straight-run gasoline [3]. The reforming process is a catalytic process that converts low-octane naphtha into higher-octane reformate products for gasoline blending and aromatic-rich reformate for aromatic production. Basically, the process re-arranges or restructures the hydrocarbon molecules [4] in the naphtha feedstocks as well as breaking some of the molecules into smaller molecules. Naphtha feeds to catalytic reforming include heavy straight run naphtha. It transforms low-octane naphtha into high-octane motor gasoline blending stock and aromatics rich in benzene, toluene, and xylene [5] with hydrogen and liquefied petroleum gas as a byproduct. Aromatics especially benzene toluene, and xylenes (para and ortho-xylene) are important petrochemical feedstocks for the manufacture of synthetic fibres, pesticides, explosives, surfactants, and synthetic rubber. Aromatics are either processed in Refinery in Catalytic reforming are processed separately [6] in petrochemical complex for manufacture of para-xylene required for DMT/PTA plant.

Quality of naphtha and impurities present in naphtha are very crucial in quality of aromatics as well as long life of catalyst. With the fast-growing demand in aromatics and demand of high - octane numbers, catalytic reforming [7] is likely to remain one of the most important unit processes in the petroleum and petrochemical industry. Feed consists of Heavy straight run gasoline (HSR), Naphtha, Heavy hydro cracker naphtha and Naphtha containing (C_6-C_{11}) chain paraffins, olefins, naphthene & aromatics [8]. Zeolite catalysts have been found to be highly useful in reforming and other naphtha upgrading processes. Zeolite Beta, for example, has been found to be suitable for use as a catalyst for hydrocracking naphtha under mild conditions. zeolite catalyst containing at least one noble metal and one alkali metal useful for catalytic reforming [9]. Zeolites useful include high silica/alumina zeolite beta, ZSM-23, and other similar ZSM-5 type zeolites. The noble metal loading should be in range of 0.1-1% weight, and the alkali metal loading should exceed the cationic exchange capacity of the zeolite component if the desirous improved process results are to be attained.

Table 1. *Naphtha Composition Percentage.*

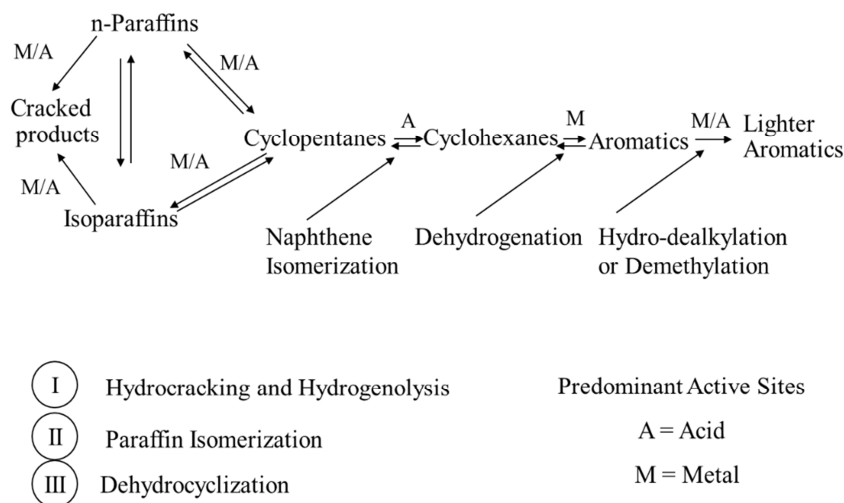| Hydrocarbon Type	Feed (%)	Product (%)
Paraffins	30-70	30-50
Olefins	0-2	0-2
Naphthene	20-60	0-3
Aromatics	7-20	45-60

Figure 2. *General Reforming Reaction Scheme.*

1.1. Bi-functional Catalyst

Bi-functional catalyst are such type of catalyst which possess two type of sites (two different functionality), hence they are able to catalyse two different type of reactions.

They basically contain lewis acid & lewis base.

1.1.1. Metallic Site
The metal (ex. Pt) promotes the dehydrogenation and hydrogenation reactions.

1.1.2. Acidic Site
The acidic site promotes the cracking & isomerization.

A unique advantage of this type of catalyst is that they can also convert the highly reactive intermediates that usually leads to carbonaceous deposits and loss of yield, thus selectivity of desired products can be increased.

Generally hydro-cracking process requires a bi-functional catalyst such as Pt/Al_2O_3.

1.2. Zeolites

Zeolites are hydrated aluminosilicate minerals made from interlinked tetrahedra of alumina (AlO_4) and silica (SiO_4). The atomic structures of zeolites are based on three-dimensional frameworks of silica and alumina

tetrahedra [10], that is silicon or aluminium ions surrounded by four oxygen ions in a tetrahedral configuration Each oxygen is bonded to two adjacent silicon or aluminium ions, linking them together. They are microporous and are commonly used as commercial adsorbents and catalysts. They occur naturally but are also produced industrially on a large scale. The naturally occurring zeolites are an important group of minerals for industrial [11] and other purposes. Since silicon typically exits in a 4+ oxidation state, the silicon-oxygen tetrahedra are electrically neutral. However, in zeolites, aluminium typically exists in the 3+ oxidation state so that aluminium-oxygen tetrahedra form centres that are electrically deficient one electron. Thus, zeolite frameworks are typically anionic [12], and charge compensating cations populate the pores to maintain electrical neutrality. These cations can participate in ion-exchange processes, and this yields some important properties for zeolites. They are widely used as ion-exchange beds in domestic and commercial water purification softening, and other applications. Many synthetic zeolites (around 150) have been designed for specific purposes, the best known of which are zeolite A (commonly used as a laundry detergent), zeolites X and Y (two different types of faujasites used for catalytic cracking), and the petroleum catalyst ZSM-5 (a branded name for pentasil-zeolite) [13].
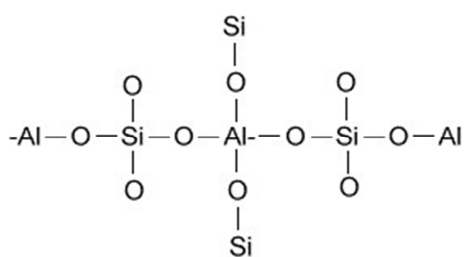

Figure 3. Zeolite Structure.

1.3. ZSM-5 (Zeolite Socony Mobil–5)

There are many naturally occurring synthetic zeolites, each with its own structure. Commercially available pore sizes range from about 3 to about 8. Some commercial materials are: A, Beta, Mordenite, Y, ZSM-5. But we are working on this material and will discuss further with different loading metals and different silica and aluminum ratios of ZSM-5. ZSM-5 is an aluminum-silicate zeolite [14] of the pentasil family of zeolites. A pentasil unit consists of eight five-member rings. In these rings, the vertices are Al or Si, and it is assumed that O is bound between the vertices. Its chemical formula is $Na_nAl_nSi_{96-n}O_{192} \cdot 16H_2O$ ($0 < n < 27$). The ZSM-5 has a high silicon to aluminum ratio. When an Al^{3+} cation replaces a Si^{4+} cation, an additional positive charge is required to keep the material charge neutral. When proton (H+) is used as a cation, the material becomes very acidic. Thus, the acidity is proportional to the Al content. The reaction and catalytic chemistry of ZSM-5 is due to its acidity [15]. It is widely used in the petroleum industry as a heterogeneous catalyst for hydrocarbon isomerization reactions. ZSM-5 is used as a catalytic support material.

2. Preparation of Extruded ZSM-5

a. Taken a 15g of ZSM-5 & 10g of binder (Al-Si) in crucible using weighing machine.
b. Mixed them in mortar pestle.
c. Prepare homogeneous of glacial acetic acid 3%v/v i.e. 7.5ml in 250ml deionised water.
d. Added drop by drop of glacial acetic (peptide agent) in the mixture.
e. Prepare a slurry of the mixture.
f. Loaded the mixture into the extruder from the bottom.
g. Applying pressure on the piston, we get the thread like structure.

*Figure 4. Synthesis of Extruded ZSM-5.*

Table 2. *Temperature range for Calcination.*

Steps	Temp. range (°C)	Time (min.)	Operation
1.	30-100	40	Heating
2.	100-250	60	Heating
3.	250-500	60	Heating
4.	500-500	240	Calcination
5.	500-30	-	cooling

Wet impregnation of Zn metal on ZSM-5 Catalyst:

a. Take 10 g of extruded ZSM-5.

b. Take 0.5g Zinc nitrate & mix it with 7ml D. I. water to obtain homogeneous solution.

On solvation, the $ZnNO_3$ dissociate into respective ions.

$$ZnNO_3 + H_2O \rightarrow Zn^{2+} + NO_3^{2-}$$
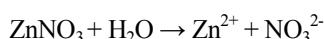
c. Added homogenous solution $ZnNO_3$ into breaker containing ZSM-5.

d. Add in such a way that it complete water retention volume.

e. Stirrer the mixture in such way that their extruded ZSM-5 adsorb all the solution.

f. Left the solution to dry & heat it at 110°C for 6hrs.

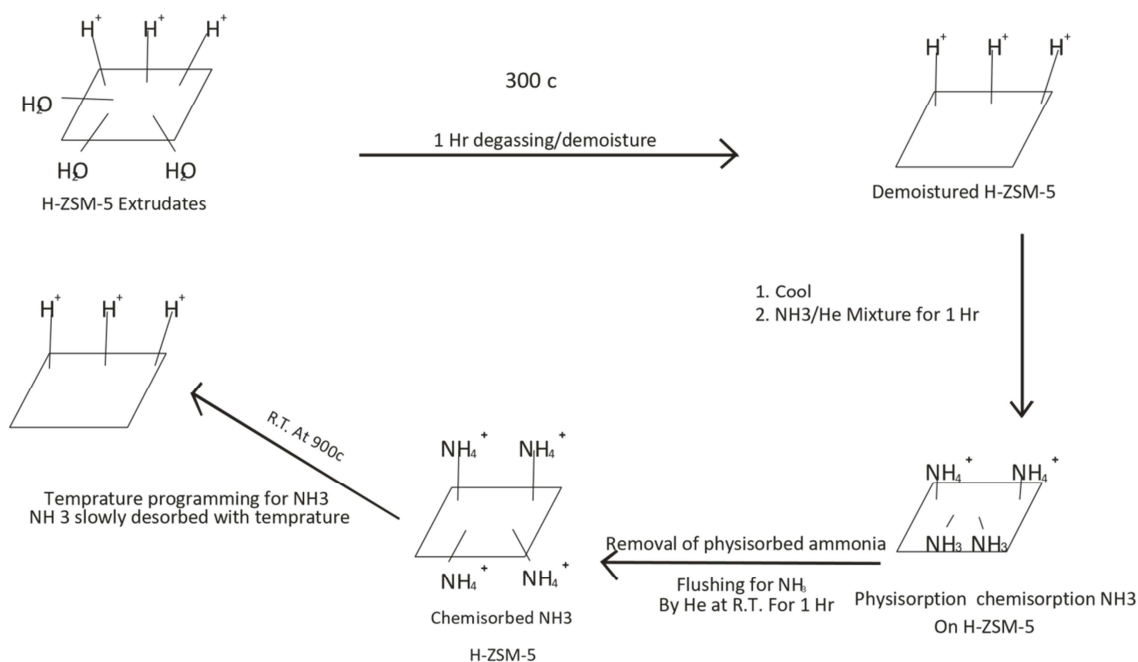# 3. TPD (Temperature Programmed Desorption) Analysis

It is technique developed specially in the field of catalysis, this technique allows one to study the interactions of reaction of gases with solid surfaces, thus being a powerful tool for both the evaluation of active sites on catalysis surfaces [16] and the understanding of the mechanism of catalytic reactions including adsorption. It determines the number, type and strength of active site available on the surface of catalyst [17] from measurement of the amount of gas desorbed at various temperature.

At certain temperature, the heat overcome the activation energy; therefore, the bond between the adsorbate and adsorbent will break and the adsorbed species desorb. If different active metals are present, they usually desorb the adsorbed species at different temperature [18]. These desorbed molecules enter the stream of inert carrier gas and are swept to the detector, which measure the gas concentrations. The volume of desorbed species, combined with the stoichiometry factor and the temperature at which pre-adsorbed species desorb, yields the number and strength of active sites.

3.1. Procedure for TPD

a. Turn on the chemisorb system, eurotherm system and the software in the PC system.

b. Start the flow of helium gas in both preparation as well as in carrier gas in chemisorb unit.

c. Detach the quartz-U-tube from the chemisorb unit.

d. Unloading of U-tube followed by cleaning (through acetone).

e. Place little quantity of quartz wool into the tube just to prevent catalyst to flow away.

f. Pour the weighed amount of catalyst and placed into the glass tube with help of biceptor.

g. Place the Quartz tube in furnace in the chemisorb unit and lock down it and insert the thermocouple into the tube.

h. Degassing of catalyst started through eurotherm upto 300°C at the rate of 10°/min (for 30 min) followed by dwelling (60 min) and cooling upto room temperature.

i. Introduction of ammonia (Mode -preparation).

j. Desorption of physios- absorbed ammonia by helium flushing for 1 hour at room temperature.

k. Stop helium gas in preparation and set a programme in eurotherm Temperature= 900°C, Rate= 10°C/min.

On the basis of heat energy consumed for desorption they are characterised into

Different categories

1. **Less interaction** ⟶ : **less acidic sites** ⟶ **0-110ºC**
2. **Average interaction** ⟶ **medium acidic sites** ⟶ **110-450ºC**
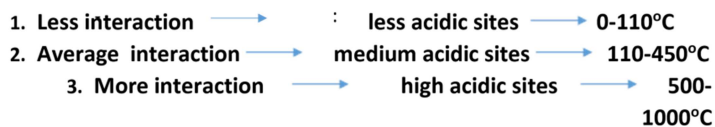3. **More interaction** ⟶ **high acidic sites** ⟶ **500-1000ºC**

Figure 5. *Flow Diagram of TPD.*

3.2. Experimental Conditions

Analysis Type: Temperature Programmed Desorption
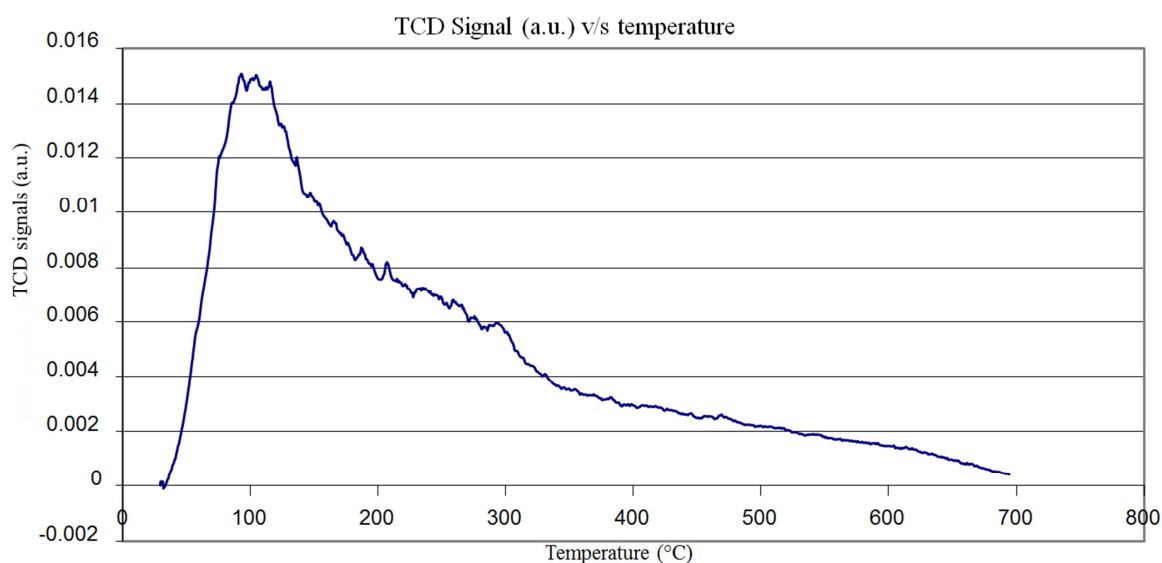Measured Flow rate: 20.00ml STP/min

Figure 6. *TPD Plot.*

3.3. TPD REPORT

Table 3. *TPD temperature value, area & peak.*

Peak No.	Temperature at maximum	Area	Peak height
1.	92.8	0.06133	0.01504
2.	104.1	0.24788	0.01498
3.	188.5	0.00060	0.00064
4.	206.9	0.00065	0.00076
5.	234.9	0.00051	0.00040
6.	265.7	0.00041	0.00033
7.	296.6	0.00096	0.00061
8.	469.2	0.00037	0.00025
9.	539.0	0.00095	0.00158
10.	613.9	0.00042	0.00103

4. Result of TPD

The TPD result shows that Zn-ZSM-5 contains mainly less acidic sites covering a greater area at 104°C.

5. Introduction to High-Pressure Micro Reactor

The HPR-Micro Reactor is a high-pressure reactor specifically designed for small-batch reaction chemistry and is well suited for research, process development, and screening applications when reagents, catalysts, or other essential materials are expensive or available in very limited supply. Plant Overview: The High-Pressure Micro Reactor System is an advanced and updated plant with fully automatic and manual working provisions [19]. This plant provides a catalyst holding capacity of more than 20 grams. It is the micro version of the industrial plants so that we can perform experiments on an industrial scale. It also provides valuable data for the design of the full-scale plant. Scientific data about

reactions, and material properties, may also be available, but it is difficult to predict the behaviour of a process of any

complexity. Designers use data from the pilot plants to refine their design of large-scale setups and production quality.

Figure 7. *HPMR Reactor.*

5.1. Plant Description

HPMR is capable of handling a wide variety of feedstocks, and can operate at different temperatures, pressure, and flow rate of feedstocks and gases. It can be operated through system software; also it has the provision of manual operation from (PID) Control Panel.

5.2. Pilot Plant Specification

a. Feed Tank Capacity - 1500cm^3
b. Product Tank Capacity - 1000cm^3
c. Feed Pump Range – 1-50ml/hr
d. Maximum operating temperature - 700°C
e. Maximum operating pressure - 70 bar
f. Reactor Volume - 70cm^3
g. Gas Meter (Min & Max Range) – 2 To 120L/hr

5.3. HPMR Pilot Plant Is Mainly Divided into Three Major Zones

5.3.1. Feed Zone

Feed zone mainly comprises of Liquid feed section and a Gas feed section.

5.3.2. Liquid Feed Section

It consists of a feed storage tank of 1.5 Liters, mounted over a weighing balance. Liquid feed is pumped through a plunger-type reciprocating pump to the reaction zone [20]. The pumping of feed is driven through different unit operations and controlling equipment which plays an important role in proper feeding. (Plunger pumps are reciprocating pumps that use a plunger to move the liquid through a cylindrical chamber). The positive displacement pressurizes the liquid by reciprocating the motion of its member. The positive displacement pumps are used high-pressure sure requirements, very viscous fluids and for precise control of flow rate. Positive Displacement [21] pumps are used in the reforming process as we require precise

control of flow rate. Blanketing on Feed vessel - Feed Vessel is blanketed i e., blanketing is done through an inert gas (nitrogen) in the vessel containing any liquid feed to protect it from decomposition and also for transportation of fluid from the vessel. It further eliminates the chances of buckling in the tanks. Also helps in proper suction of liquid in pump. For the proper and safe transportation of feed from pump to the reaction zone a pressure gauge, pressure safety valve (PSV) and a three-way valve (TW) are required.

Figure 8. *Liquid feed section.*

5.4. Gas Feed Section

Pressure plays an important role in the transportation of gases. Gas Feed Section consists of Cylinders (Capacity 140 kg), Forward Pressure Regulators, Mass Flow Controllers and different types of valves which are used to control and transport required gas into the plant. Gas cylinders consist of pressure [22] regulator equipped with a primary pressure gauge and a secondary pressure gauge. Primary pressure gauge indicates pressure inside the cylinder whereas the

secondary pressure gauge indicates line pressure. Forward Pressure Regulators (FPR), It is used to maintain the downstream pressure. Rotating the knob clockwise and anticlockwise increases and decreases the pressure respectively.

5.5. Mass Flow Controllers (MFC)

MFC is a device used to measure and control the flow rate of fluids and gases. The MFC can be given a set point from 0 to 100% of its full-scale range but is typically operated in the 10 to 90% of full scale where the best accuracy is achieved. All mass flow controllers have an inlet port, an outlet port [23], a mass flow sensor and a proportional control valve. The MFC is fitted with a closed loop control system which is given an input signal by the operator that it compares [24] to the value from the mass flow sensor and adjusts the proportional valve accordingly to achieve the required flow.

5.6. Reaction Zone

It is termed as the heart of the process as the reaction takes place in this zone. It mainly consists of a preheater and a reactor. The reaction takes [25] place in the temperature range of 300°C-600°C and pressure variation ranges up to 10 bar to 60 bar.

5.7. Preheater

To increase the efficiency, a preheater is used in the unit to preheat the feed (up to 150°C) before entering into the reactor in order to pre-vaporize the feed so that the reaction occurring in the reactor should be in the gas phase.

Figure 9. *Reaction Zone.*

5.8. Reactor

After heating, the feed from the pre-heater enters into the reactor, which is a fixed bed type tubular reactor. The reactions carried out in the reactor are heterogeneous reactions because the catalyst is in solid extrude form and feed is in vapor phase.

Figure 10. *Reactor.*

The reactions are performed in an isothermal environment and are electrically heated as the reforming [26] reactions are generally endothermic in nature. The reactor temperature and pressure play an important role for the reaction to occur. Thus, for heating, three inter-stage heaters are provided (The maximum reactor temperature is 600°C for all inter-stage heaters). The top zone of the reactor is the preheat zone, the middle zone [27] is the reaction zone and the last is the post-heat zone. Equal temperature is maintained in the entire zone throughout the reactor line and thermocouples are inserted in all three zones both to measure the outer (skin) as well as inner side temperature. Temperature safety switches (TSS) are also installed with all the zones [28] and they come in action when the furnace reaches its upper limit temperature (600°C). Pressure maintained inside the reactor is always equivalent of the plant pressure.

5.9. Product Zone

The product obtained after the reaction, get cooled in a double-pipe spiral heat exchanger by exchanging heat with cold water. The coolant used [29] in a double pipe heat exchanger is distilled water coming from a chilling water bath which is at a temperature 6°C. The chilled product enters into the High-Pressure Separator (HPS) where the lighter component (vapor phase) of the product separates from the heavier component (liquid phase) [30].

Figure 11. *Product Zone.*

The top product from the High-Pressure Separator (HPS) passes to Low-Pressure Separator (LPS) through a pressure

control valve (PCV). Again in the Low Pressure [31] Separator, the lighter component (vapor phase) of the product separates from the heavier component (liquid phase). The liquid product from both separators is collected into the product tank. The separator is covered with glass wool for insulation. The liquid indicator is provided [32] at the bottom of the first separator as it is necessary to maintain a certain amount of liquid level inside the separator otherwise the gas will come with the liquid product. There is a water-carrying copper coil wrapped around the separators for cooling.

5.10. Double Pipe Heat Exchanger

It is used in the HPMR plant to collect the product coming after the reaction. It is also known as a concentric pipe, hairpin, jacked pipe and jacketed U-tube heat exchanger. It consists of a single tube mounted inside another. One fluid flow in the inner pipe, while a second fluid flows in the [33] outer pipe annuals. They are suited for applications where either low flow rates, large temperature cross or small duties with high flow rates are involved. A double tube heat exchanger is able to achieve pure counter current flow thus allowing for a [34] temperature cross to be achieved whereby the cold fluid can be heated above the exit temperature of the hot fluid. Separators, High Pressure, and Low-Pressure Separator is used to separate out the phases (liquid and vapor) of the reaction product.
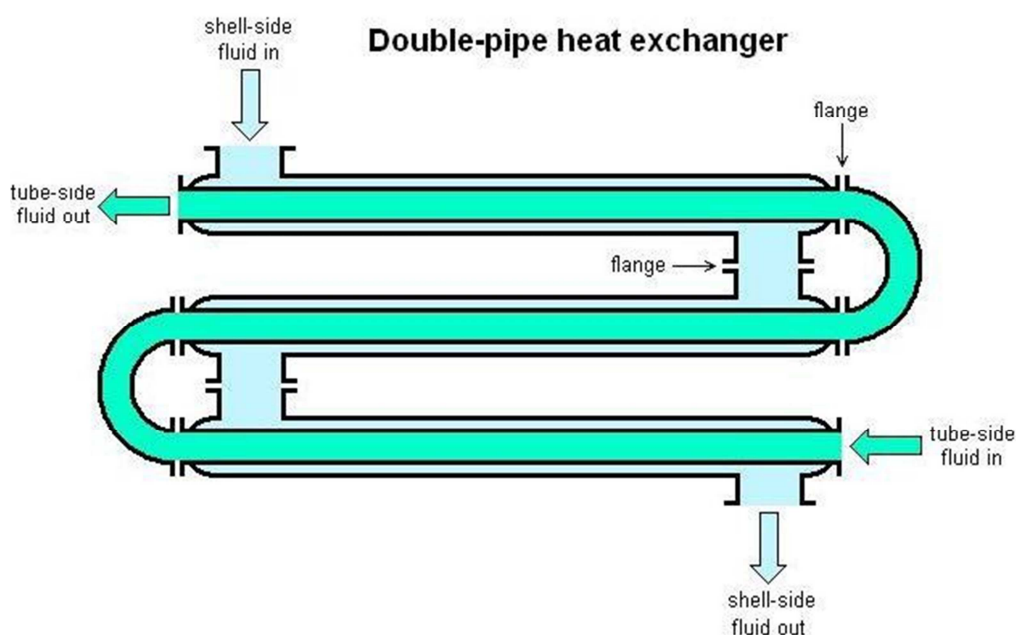

Figure 12. *Heat Exchanger.*

5.11. Gas Flow Meter (GFM)

GFM is an instrument used to measure the volume of gas transported to a storage tank vessel. It maintains and receives the movement directly with the release of excess pressure from the control valve.
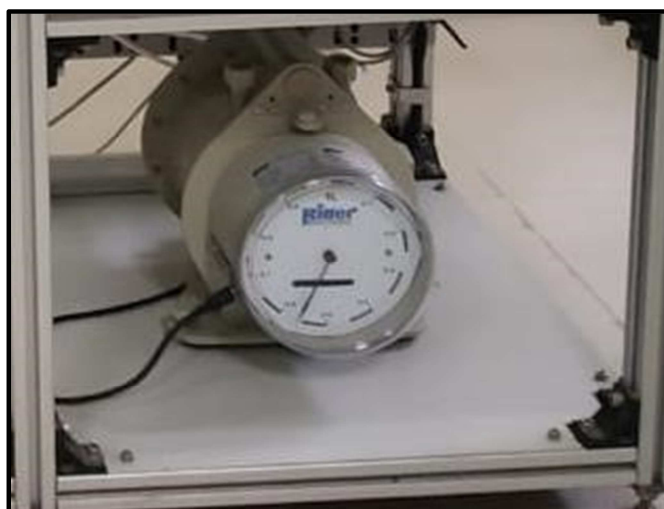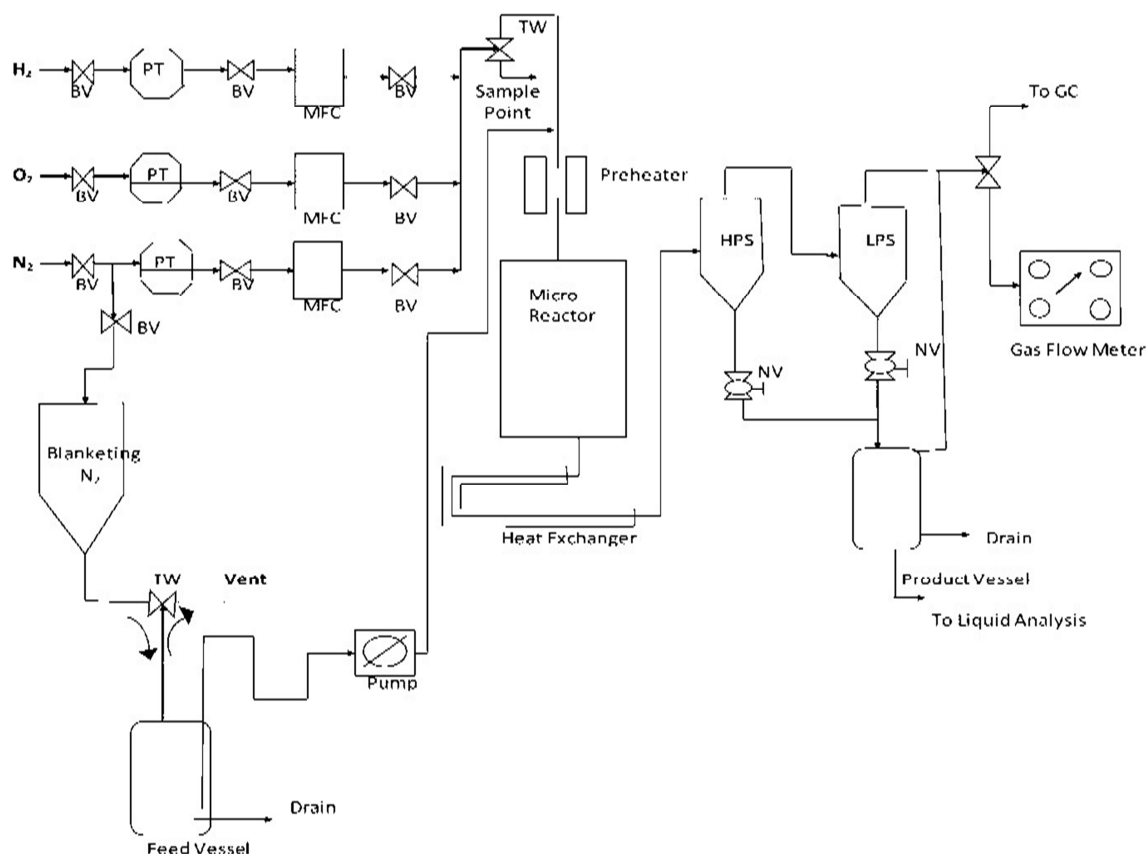

Figure 13. *Gas Flow Meter.*

Figure 14. *Detailed Diagram of HPMR Reactor.*

6. Result of Naptha Reforming Reaction

Catalytic reforming result of Naptha (feed) in HPMR reactor shows that the product contains mainly aromatic hydrocarbons such as Benzene, Toluene & Xylene (BTX) detailed result of experiment given in Figure 15 and Figure 16.

Most of the naphthene (saturated hydrocarbons) converted in aromatic hydrocarbons.

Totals by Group Type & Carbon Number (in Mass Percent)

	Paraffins	I-Paraffins	Olefins	Naphthenes	Aromatics	Unknowns	Total
C1	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C2	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C4	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C5	0.3197	0.0431	0.0265	0.3602	0.0000	0.0000	0.7495
C6	2.5421	2.0633	0.0000	6.6607	1.6594	0.0000	12.9256
C7	7.2106	5.3607	0.0462	18.8130	7.5680	0.0000	38.9986
C8	6.9589	9.9144	0.0958	8.1053	7.3311	0.7554	33.1608
C9	2.5875	4.3072	0.3365	4.3523	1.3387	0.1577	13.0800
C10	0.1945	0.6377	0.0000	0.1437	0.0566	0.0292	1.0617
C11	0.0237	0.0000	0.0000	0.0000	0.0000	0.0000	0.0237
C12	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C13	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C14	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C15	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C16	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C17	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C18	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C19	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Total:	19.8371	22.3265	0.5051	38.4352	17.9538	0.9424	99.0576

Oxygenates 0.0000   Total C20+: 0.0000

Total Unknowns: 0.9424   Grand Total: 100.0000

Figure 15. *Composition of feed before reforming reaction.*

Figure 16. *Composition of the product after reforming reaction.*

Table 4. *Comparison of Reactant (feed) and Product Composition.*

	Aromatic C6 (Mass %)	Aromatic C7 (Mass %)	Aromatic C8 (Mass %)	Total Aromatic Hydrocarbon (Mass %)
Before	1.6594	7.5680	7.3311	17.9538
After	5.1867	25.0743	22.6471	62.2081

7. Conclusion

Final Result of Catalytic reforming of Naptha using Zn loaded ZSM-5, shows a drastic change from saturated to aromatic unsaturated hydrocarbons mass % mentioned is Table 4. I have been founded that mostly naphthalene hydrocarbons are converted in aromatic hydrocarbons.

Abbreviation

CV	Check valve
BV	Ball valve
PT	Pressure transmitter
HE-600	Heat exchanger
NV	Needle Valve
PG	Pressure gauge
TIC 500,501,502	Temperature controller of furnace
TSS 500,501,502	Safety temperature of furnace
MFC 100	Mass flow control for H_2
MFC 200	Mass flow control for O_2
MFC 300	Mass flow control for N_2
PT 100	Secondary pressure control H_2
PT 200	Secondary pressure control N_2
PT 300	Secondary pressure control for N_2
TI 500	Temperature indicator for reactor centre
TI 600	Feed inlet temperature before preheater
TIC 400	Temperature for preheater
PT 400	Pump head pressure indicator
PSV	Pressure safety valve
TW	Three-way valve
PT 500	Reactor inlet pressure indicator
PT 501	Reactor outlet pressure indicator
TI 401	Line preheater & reactor
TI 402	Line heater
PIC 600	Reactor main pressure controller
LIC 600	Liquid level control HPS
LIC 601	Liquid level control LPS
TIC	Temperature indicator control
BHU	Bulk head unit
WS	Weighing scale
PCV	Pressure control valve
LCV	Liquid control valve

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