SYNTHESIS OF CATALYST Al-MCM-41 BY HYDROTHERMAL PROCESS

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SYNTHESIS OF CATALYST Al-MCM-41
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By

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This is to certify that **MANJIT SINGH** (Roll No 20/006), **BISHAL GHOSHAL** (Roll No 20/158) , **SAURAV SWARGIARY** (Roll No 20/201) and AMBIKA MAIBONGSA (Roll No 20/428) of B.Tech. 7th Semester have jointly carried out the project entitled “**Synthesis of Catalyst Al-MCM-41 By Hydrothermal Process** ” under my supervision and submitted the report in partial fulfillment of the requirement for the Degree of Bachelor of Technology in Chemical Engineering of Assam Science and Technology University, which may be accepted.

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DECLARATION

WE,

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7th semester, Department of Chemical Engineering, Assam Engineering College, sincerely declare that the project report titled **"Synthesis of Catalyst Al-MCM-41 By Hydrothermal Process"** under the guidance of **Dr. Ujwala Hujuri** is submitted to the **Department of Chemical Engineering, Assam Engineering College** in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering from Assam Engineering College.

We declare that this project has not been published previously elsewhere and is a result of our own efforts and has been taken solely for the academic purpose and all the information provided in the project is true and correct to the best of our knowledge.

Date :
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ABSTRACT

In present times, accumulation of plastic waste has been one of the major issue to be addressed. Devising methodologies for efficient treatment of plastic waste has been a need of the hour. One of such methodologies is converting the plastic back to its monomeric form, which is called pyrolysis. The issue in this process is extreme temperature conditions and low energy efficiency. For this catalyst specifically of the zeolite's class can be employed. To address this issue, in this project Al-MCM-41 has been synthesized which can be used as a catalyst in catalytic pyrolysis of plastic. The hydrothermal process has been used for the synthesis of the catalyst and the synthesized samples has been characterized using XRD, BET, FTIR and TGA. There has been a considerable yield of the synthesized catalyst. The samples are crystalline nature an had shown a considerable pore volume. All the necessary parameters has been fulfilled as analyzed by the characterization processes. The synthesized catalyst can be used for catalytic pyrolysis of plastic.

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List of abbreviations

- **MCM-41**: Mobile Composition of Matter no. 4**1**
- **ZSM-5:** Zeolite Socony Mobil-5
- **LDPE:** Light Density Polypropylene
- **XRD:** X-Ray Diffraction
- **TGA:** Thermogravimetric Analysis
- **FTIR**: Fourier-Transform /infrared
- **TEOS:** Tetraethoxysilane
- **BET:** Brunauer-Emmet-Teller

Chapter 1

Introduction

1.1 Importance of the work

Plastic being one of the most durable and versatile materials available in market, it has been widely used and preferred for variety of uses. But increased use of plastic has lead to a huge accumulation of waste plastic. This increased amount of plastic waste has resulted in severe environmental repercussions as plastics are non-degradable waste and once it comes in the ecology, it remains undegraded for millions of years. Majority of these waste plastic are commodity plastics specifically LDPE (Low Density Poly Ethylene) which is used in almost every domain.

So, in such a scenario, recycling of these plastic waste and reduction in accumulated plastic in the ecology has become need of the hour. Plethora of techniques for recycling of plastic waste has emerged among which pyrolysis is one of the most efficient technique. Pyrolysis is a tertiary technique wherein the plastic waste is converted into liquid fuels and gases. But pyrolysis has a major drawback as it requires very high temperature for the degradation of the plastics. Moreover, selecting reactors which can withstand such high temperatures is another major challenge regarding the pyrolysis technique. Additionally, such high operating conditions are not economically feasible. So, to reduce the extremely high degradation temperature of the pyrolysis process, catalyst is added to the plastic waste during the process. One of such catalysts is zeolites. Zeolites are widely used for cracking the hydrocarbons present in the plastic. Al-MCM-41, a mesoporous zeolite, is one such catalyst which significantly reduces the degradation in catalytic pyrolysis of plastics. In this project, Al-MCM-41 catalyst has been synthesized by hydrothermal method and characterized for surface area, pore size, crystallinity and the functional groups.

1.2 Low Density Polyethylene:

LDPE is a thermoplastic made from the monomer ethylene. It is produced using a high pressure process via free radical polymerization. It is a commodity plastic and is widely used due to its durability and low cost. It is used almost in every domain and hence contribute the highest share towards total accumulation of waste plastic. When exposed to sunlight, the plastic produces a significant amount of two green-house gases namely methane and ethylene. Also, upon heating it releases a huge amount of carcinogenic gases. So proper treatment of these plastics is a huge challenge in the modern society.

1.3 Pyrolysis of waste plastic:

Pyrolysis is a thermochemical process wherein the waste plastic is heated at a very high temperature leading to its degradation and eventually breaking down of the bonds to form fuel and gas. Pyrolysis is a promising technique for conversion of Solid Waste Plastic to more usable material such as gas fuel or fuel oil or to high feedstock for the chemical industry. Degradation of the waste plastic materials by heating in an inert atmosphere- pyrolysis is usually conducted at a moderate temperature between $400-800^{o}$C [*k.Miteva et al.*]. Obtained products are volatile condensable hydrocarbon oil and a non-condensable high calorific value gas.

Pyrolysis can be thermal or catalytic. The advantages of catalytic pyrolysis are degradation at a lower temperature (lower energy consumption), increased selectivity, faster-cracking reactions, shorter residence time, inhibition of the formation of undesirable products, increased product yield and production of liquid products with a lower boiling point. [*Francis Anene et al.*]

Catalytic pyrolysis is a way to improve the product yield and selectivity and reduce the pyrolysis temperature. The lower reaction temperature, which may lower the energy requirement of the process, is due to the lowered activation energy of the pyrolysis reactions. A catalyst may favour the yield of lighter hydrocarbons, gasoline range products and gases. The distribution of the products obtained depends on the type of polymer, their sources and their structures. [*Yan et al.*]

Yan et al. studied the thermal cracking of virgin and waste plastics PP and low-density polyethylene (LDPE) in a semi-batch reactor, under atmospheric pressure at 460 ◦C. The thermal pyrolysis of LDPE and PP at 460°C gave a high yield of liquid fraction (84wt%) in both cases. The gas chromatography analysis of the oil yield obtained from the pyrolysis of PP contained 58% gasoline range hydrocarbons, while the oil obtained from pyrolysis of LDPE contained 21 wt % of gasoline range hydrocarbons.

1.4 Zeolites as a catalyst for plastic pyrolysis:

Zeolites are crystalline inorganic polymers consisting of SiO_4 and AlO_4 tetrahedra, having a structure filled with ions and water molecules. Zeolite are hydrated Alumino-Silicates of alkaline or alkaline-earth metals. Major use category of synthetic zeolites is catalyst, detergent or molecular sieve. Zeolites are microporous in nature with a typical diameter or 0.3-0.8 nm (as per journal of natural gas and science engineering, vol 22). Zeolites are mainly 3D network of Al, Si and O bonded mainly in Al-O-Al, Si-O-Si and Al-O-Si linkages. General formula of zeolites is $Mn^{+}(AlO2)$ (SiO2) YH2O, where Mn+ is either H+ or metal ions (Na+). Si/Al ratio in zeolites are variable. Si/Al ratio greater than 3 indicates high silica zeolites with hydrophobic nature. The huge network of co-valent bonds in zeolites accounts for its chemical and physical stability. Synthetic zeolites are ion exchangeable and can be used as solid acid catalysts. Shape and size selectivity is a vital consideration for many industrial catalytic processes using zeolites. Selectivity can be based on the size/ shape of the reactant, product or intermediate. In order to provide such size/ shape selective behaviour, the catalyst must have uniform pores with molecular dimensions. There are two class of zeolites namely natural zeolites and synthetic zeolites. [*Krol et al.*]

1.4.1Natural Zeolites:

Natural zeolites are hydrothermal and of mainly volcanic origin. They can occur both in crystallized forms and amorphous forms and is found in igneous and metamorphic rocks, as well as in grains of smaller diameters accumulated in sedimentary rocks. Ocean bottom sediments are relatively huge and rich in zeolites, but these deposits are so far inaccessible to humans. However, these minerals may also constitute important components of tuffs or clay. Such surface retention of zeolite sediments, and therefore relatively simple mining using the opencast method, creates perfect conditions for their wider use. It should be mentioned here that the zeolites naturally occurring in nature, possess operational significance. Examples are:

clinoptilolite; mordenite; chabazite. [*Krol et al.*]

1.4.2Synthetic zeolites:

Zeolites have been recognized as minerals of natural origin, but currently more than one hundred different types of zeolite structures are known which can be obtained synthetically. Under natural conditions zeolites were formed as a result of the reaction of volcanic ash with the waters of the basic lakes. This process lasted several thousand years. In laboratory conditions, an attempt can be made to imitate hydrothermal processes using elevated temperature or pressure and using natural raw materials and/or synthetic silicates. The synthesis reaction requires appropriate equipment, clean substrates and energy. As a result, the price of the product may be much higher than the price of natural zeolite. Therefore, research often focuses on the search for cheaper and available substrates for the production of zeolites, while striving to reduce the cost of the reaction itself. The current trends in research on the synthesis of zeolites are shaped by environmental aspects, which implies the use of natural or waste raw materials for this purpose. [*Krol et al.*]

1.5 MCM-41

MCM-41 is a mesoporous material with a hierarchial structure from a family of aluminosilicate solids and can be used as catalyst or catalyst support. MCM-41 consists of a regular arrangement of cylindrical mesopores that form a one-dimensional pore system. It is characterized by an independently adjustable pore diameter, a sharp pore distribution, a large surface and a large pore volume. The pores are larger than with zeolites and the pore distribution can easily be adjusted [*Reichinger M. et al.*] The mesopores have a diameter of 2 nm to 6.5 nm. Contrary to zeolites, the framework of MCM-41 has no bronsted acid centers because there is no aluminium contained in the lattice. The acidity of alumina-doped MCM-41 therefore is comparable to that of the amorphous alumo-silicates. MCM-41 is not hydrothermally stable because of the slight wall thickness and the low degree of cross-linking of the silicate units [*Silaghi et al.*] MCM-41, as the zeolites, are widely used as catalytic cracking. MCM-41 type materials have been widely used as support of heterogeneous catalysts and also used for separations [*P chatterjee et al.*]

1.6 Al- MCM-41:

Al- MCM-41 is a mesoporous aluminosilicate and is synthesized using CTAB as a template. The Al added to MCM-41 creates bronsted acid sites and hence makes the catalyst even more efficient. Mesoporous silicates such as Al-MCM-41 have a large surface area and large pore size with an ordered array of cylindrical mesopores. This morphology increases the likelihood that a reactant molecule in solution can make contact with the surface of a catalyst and react. It also ensures that the reactant molecules are small enough to diffuse into large pores. As such, Al-MCM-41 is an excellent option for use as a catalyst or catalyst support, as an adsorbent, or as a component of guest host chemistry. [*Hongyun Chen et al.*]

Chapter 2

LITERATURE REVIEW:

A study by **Hongyun Chen, Siyao Fu, Liangjie Fu, Huming Yang and Deliang Chen [1]** suggested use of green silicon sources due to toxic nature of the synthetic silicon sources. They used nature perlite as silicon sources. The catalyst had been synthesized using hydrothermal process using CTAB as a template and subsequently calcining the sample.

A study by **M.C.Silaghi[2]** focussed on challenges on molecular aspects of dealumination and desilication of zeolites. microporous and crystalline aluminosilicates exhibit a strong Bronsted acidity making them attractive for processes such as hydrocracking and fluid catalytic cracking. However, micropores can induce diffusion limitations and confinement effects resulting in the formation of undesired side products. Posterior introduced mesopores, leading to the so called “hierarchical zeolites” can overcome these phenomena. They contain bi- or multimodal pore structures which can be post-synthetically introduced by partial dealumination and/or desilication of the framework

A study by **K.Miteva[3]** aims to convert waste polyolefin mixture and production of liquid fuel using mixture of Al_2O_3 and SiO_2 as catalyst. Waste plastics are pyrolyzed at a temperature of 400-500^oC and products were liquid fuel, gas and minor solid residue.

A study by **Azubuike Francis Anene [4]** aims to study the differences between thermal and catalytic pyrolysis. Virgin LDPE, HDPE, PP and mixtures of LDPE/PP were carried out in a 200ml labarotary scale batch reactor at a temperature of 460^oC in a Nitrogen environment. The amount of PP in the LDPE/PP mixture was varied and the reaction parameters were observed accordingly. The yield in catalytic pyrolysis w.r.t thermal cracking was significantly high with lower degradation temperature in the former.

Yan [5] studied the thermal cracking of virgin and waste plastics PP and low-density polyethylene (LDPE) in a semi-batch reactor, under atmospheric pressure at 460 ◦C. The thermal pyrolysis of LDPE and PP at 460^oC gave a high yield of liquid fraction (84wt%) in both cases. The gas chromatography analysis of the oil yield obtained from the pyrolysis of PP contained 58% gasoline range hydrocarbons, while the oil obtained from pyrolysis of LDPE contained 21 wt % of gasoline range hydrocarbons.

A study by **P.Chatterjee[6]** suggested that the catalytic aerobic α-oxidation of amines in water is an atom economic and green alternative to current methods of amide synthesis. Basically, the inference that we got out of it is the bronsted sites in Al-MCM-41 increases the catalytic activity making it an economically feasible option as far as industrial production is concerned.

A research by **Magdalena Krol [7]** aims to study the geopolymer-zeolite composites. Such kind of hybrid materials connect the advantageous properties of both constituents: geopolymer serves as a strong and durable support for zeolites when zeolites provides high surface area, porosity and adsorption capacity.

A study by **Benedikt Lindlar, Andreas Kogelbauer, Roel Prins [8]** investigated on the effects of pH adjustment at a value of 11 during Al-MCM-41 synthesis with regard to changes in structure, chemical composition and catalytic activity are described. Long range order, surface area, and pore size distribution improved remarkably when the pH of the synthesis gel was adjusted immediately.

A research by **Qiang Wei, Pengfei Zhang, Xiaodong Lu, Xiaung Fan, Yitong Yan, Lin Wang[9]** aims to synthesize Ni-modified ZSM-5 and also it aims to study the catalyst's activity in n-Octane hydroconversion. The modification is basically done by in-situ synthesis method. The activity of the modified catalyst was found to be enhanced and the pore volumes were increased.

A study by **Cang-Yen Chen, Sandra L Burkett, Hong-Xin Li, Mark E Davis [**10**]** aimed at studying different characterization methods to study mesoporous materials. The solids obtained during the synthesis of MCM-41 are investigated by X-Ray powder diffraction, thermogravimetric analysis and NMR Spectroscopy.

Chapter 3

3.1 METHODOLOGY FOR SYNTHESIS:

There are two methods of synthesis of Al-mcm-41:

i) Sol-gel method

ii) Hydrothermal method

3.1.1 Sol-gel method:

The sol gel method is a wet chemical technique used for the fabrication of both glassy and ceramic material. In this process the sol(solution) evolves gradually towards the formation of a gel like network containing both a liquid phase and a solid phase.

In material science, sol gel process is a method for the production of porous solid materials from small molecules. For e.g. metal alkoxides, nitrides or sulfides by colloidal route, this method cannot be defined as a single technique because of broad variety of procedures exist. The sol-gel process is a versatile technique employed in material synthesis, involving sequential hydrolysis and condensation reactions of precursor molecules. Initially, metal alkoxides, often chosen as precursors, undergo hydrolysis, breaking metal-oxygen bonds and producing metal hydroxides. Subsequent condensation reactions lead to the formation of a three-dimensional network within a liquid sol, ultimately transforming it into a gel. The gel, consisting of interconnected particles, is then subjected to a drying process, eliminating the liquid phase and resulting in a porous material. Heat treatment, or calcination, is often applied to remove residual organic components and enhance structural integrity. This method provides precise control over composition, morphology, and porosity, making it invaluable in producing materials with tailored properties, notably in fields like ceramics, optics, and electronics.

3.1.2 Hydrothermal method

The hydrothermal process involves a chemical reaction or material synthesis that occurs in a closed system under high-pressure and high-temperature conditions using water as the solvent. It's commonly used in various fields, including material synthesis, mineral formation, and the production of nanoparticles. In material synthesis, the hydrothermal method typically involves mixing precursor chemicals or compounds in a solvent, sealing them in a reactor vessel, and subjecting them to elevated temperatures and pressures for a specified duration. This controlled environment promotes the formation of desired compounds or materials by facilitating reactions that might not occur under normal conditions. The conditions within the hydrothermal system, such as temperature, pressure, and time, play crucial roles in determining the properties and characteristics of the synthesized materials. Adjusting these parameters can influence the phase, structure, size, and morphology of the final product.

3.2 Advantage of Hydrothermal process over Sol-gel process:

The hydrothermal process offers several advantages over the sol-gel process for the synthesis of Al-MCM-41:

- Better control over framework aluminum content:

Hydrothermal synthesis allows for more precise control of the aluminum content in the framework compared to the sol-gel method. This is crucial as the aluminum content significantly affects the material's properties such as acidity, thermal stability, and catalytic activity.

In the hydrothermal process, aluminum can be directly incorporated into the framework during synthesis by using aluminum sources like aluminum iso-propoxide or sodium aluminate. This leads to a more uniform distribution of aluminum compared to post-synthesis modification in the sol-gel process.

- Higher crystallinity and thermal stability:

Hydrothermally synthesized Al-MCM-41 generally exhibits higher crystallinity and thermal stability compared to the sol-gel counterpart. This is because the hydrothermal process promotes the formation of ordered and defect-free structures, leading to improved material stability at elevated temperatures.

Higher crystallinity translates to better pore size distribution and accessibility, which are important factors for catalytic applications.

- Reduced risk of dealumination:

One of the major drawbacks of the sol-gel method for Al-MCM-41 synthesis is the risk of dealumination during the removal of the surfactant. This occurs due to the hydrolysis of aluminum species, leading to a loss of aluminum from the framework and decreased acidity.

Hydrothermal synthesis can mitigate dealumination by employing milder conditions for surfactant removal, like low-temperature calcination or solvent extraction. This helps preserve the aluminum content and improve the acidity of the final material.

- Simpler and less time-consuming:

Generally, the hydrothermal synthesis of Al-MCM-41 is simpler and less time-consuming compared to the sol-gel process. It typically involves fewer steps and uses less complex reagents, making the process easier to scale up for industrial applications.

3.3 FACTORS AFFECTING THE OPERATING CONDITIONS:

- **Moisture**

If the synthetic Al mcm-41 is subjected to air, water, molecules may get absorbed at the Al-site of the zeolite and their associated acid sites in the zeolite’s interior, and hence can decrease its activity [*]. Controlling the moisture content during the synthesis of Al-MCM-41 is crucial for achieving the desired properties, such as high surface area, large pore volume, and ordered structure.

- **Temperature**

Temperature plays a significant role in the synthesis of Al mcm-41. The temperature when the solution is in autoclave should strictly be less than 300ºC as it affects the autoclave(teflon) [*]. Temperature affecting various aspects like Crystallization and ordering and Si/Al ratio impact.

- **Speed of mixing**

Slow mixing results in excess foam formation and raw crystal formation (Observed during the experiment time). Rapid mixing gives the desire results (less foam formation). [*]

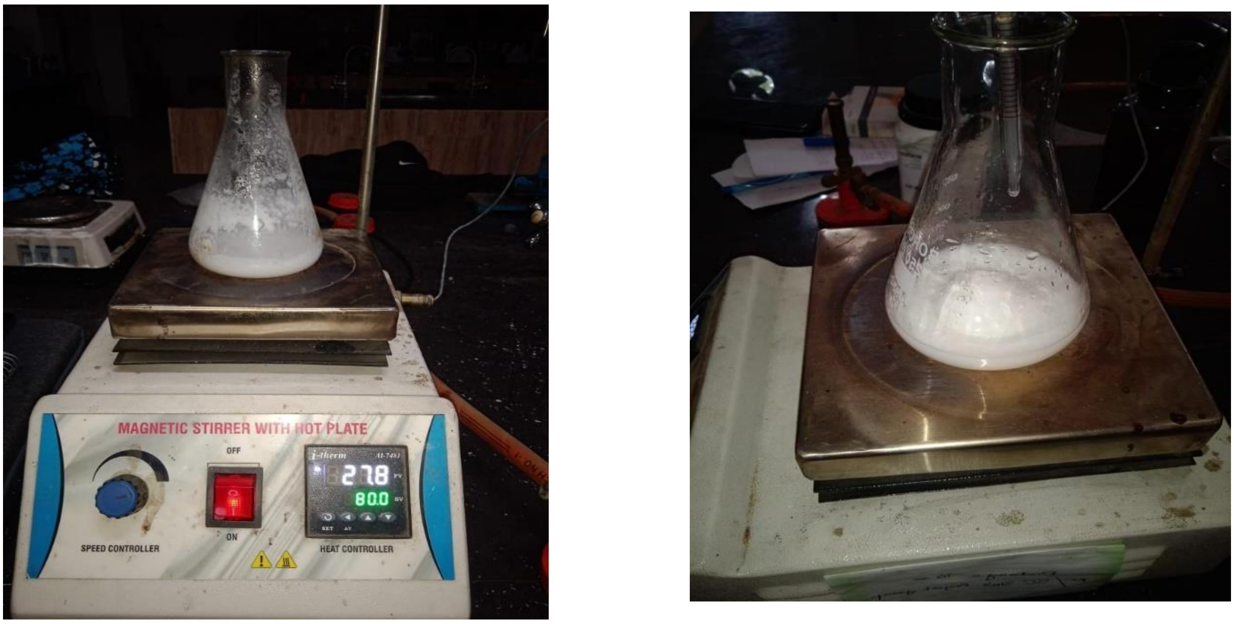


Fig : Mixing of sample

- **Foam formation**

Foam formation is a huge issue resulting in lower activity of the synthesized catalyst. Useof Anti-foaming agent before adding surfactant and a previous aging of the mixture ofSi and Al sources prior to the synthesis improves the Al-MCM-41 material.

Fig : Foam formation sample

- **Time:**

it has been observed while doing the experiments that when we keep the solution in the autoclave for higher amount of time and comparatively lesser temperature, crystalformation was prominent. But when the same solution is kept for lesser amount of time at a comparatively higher temperature, the solid formed was less crystalline.

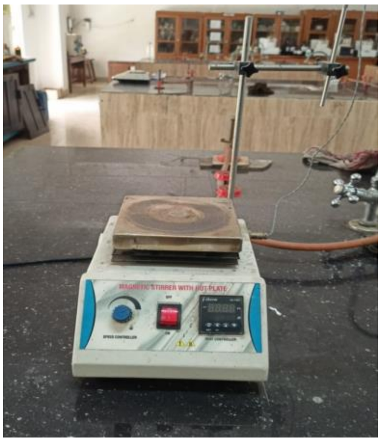
Chapter 4

Experimentation

4.1 Materials

4.1.1 For synthesis of Al-mcm-41-

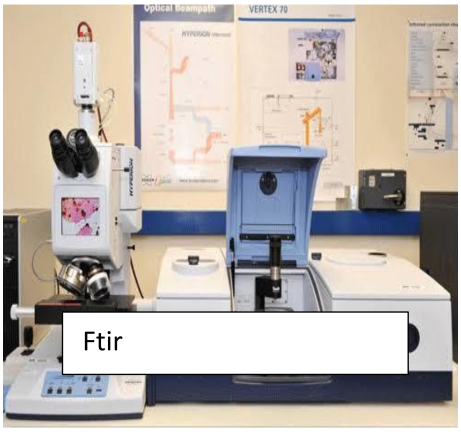
- N-cetyl-N,N,N-trimethylammonium bromide($C_{19}H_{42}BrN$)
- Tetraethoxysilane
- Aluminium Sulphate
- Distilled water
- Heated magnetic stirrer
- Teflon lined autoclave
- Filter paper
- Beakers
- Funnel
- Cylinders
- Programmable muffle furnace
- Hot air oven


MAGNETIC STIRRER WITH HOT PLATE


HOT AIR OVEN

4.1.2 For analysis-

- Thermogravimetric Analyser (TGA)
- Bet surface area Analyser (BET)
- Fourier transform infra-red spectroscopy (FTIR)
- X-ray diffractometer (XRD)



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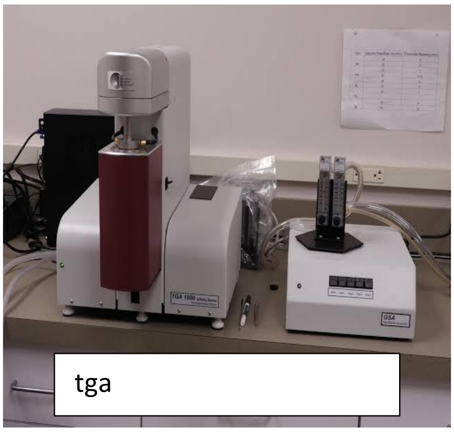
bet



tga

4.2 Method

Synthesis of Al-mcm-41(hydrothermal method)

Three batches of hydrothermal Al-mcm-41 catalyst with varying parameters (Temperature) are synthesized according to the procedure described by (reference/citation).

- **Batch 1**

Following are the steps followed for the synthesis of batch 1 catalyst:

- 0.40g of NaOH and 0.60g of N-cetyl-N,N,N-trimethylammonium bromide($C_{19}H_{42}BrN$) dissolved in 32ml distilled water.
- 3.85g of Tetraethoxysilane was added to step 1 mixture.
- 0.37g of Aluminium Sulphate dissolved in 10ml of distilled water in a separate beaker and then added to the reaction mixture.
- Stirring continued for about 105 minutes at ambient temperature.
- Resulting mixture was stirred and heated at 80°C for 20 mins.
- The mixture was further stirred overnight and then transferred to Teflon-lined autoclave at **150°C** for 8 hours.
- Synthesized products were recovered by filtration and washed with about 2 litres of distilled water.
- Drying at 242K was done in a hot air oven for 4 hours.
- After drying, calcination of the sample is done at 823K for 12 hours at a rate of 2°C/min.

Sample obtained:

Batch 2

Following are the steps followed for the synthesis of batch 2 catalyst:

- 0.4g of NaOH and 0.60g of N-cetyl-N,N,N-trimethylammonium bromide($C_{19}H_{42}BrN$) dissolved in 32ml distilled water.
- 3.85g of tetraethoxysilane was added to step 1 mixture.
- 0.37g of alluminium Sulphate dissolved in 10 ml of distilled water in a separate beaker and then added to the reaction mixture.
- Stirring continued for about 105 minutes at ambient temperature.
- Resulting mixture was stirred and heated at 80ºC for 20minutes.
- The mixture was further stirred overnight and then transferred to a Teflon lined autoclave at **140ºC** for 12 hours.
- Synthesized products were recovered by filtration and washed with about 2 litres of distilled water.
- Drying at 343K was done in a hot air oven for 5 hours.
- After drying, calcination of the sample is done at 823k for 12hrs at a rate of 2ºC/min.

Sample obtained:

Batch 3

- 0.80g of NaOH and 1.2g of N-cetyl-N,N,N-trimetylammoniumbromide($C_{19}H_{42}BrN$) dissolved in 64ml distilled water.
- 7.7g of Tetraethoxysilane was added to step 1 mixture.
- 0.74g of Aluminium Sulphate dissolved in 20ml of distilled water in a separate beaker and then added to the reaction mixture.
- Stirring continued for about 105 minutes at ambient temperature.
- Resulting mixture was stirred and heated at 80ºC for 20 minutes.
- The mixture was further stirred overnight and then transferred to a Teflon lined autoclave at **130ºC** for 12 hours.
- Synthesized products were recovered by filtration and washed with about 2 litres of distilled water.
- Drying at 343K was done in a hot air oven for 5 hours.
- After drying, calcination of the sample is done at 823K for 12hours at a rate of 2ºC/min.

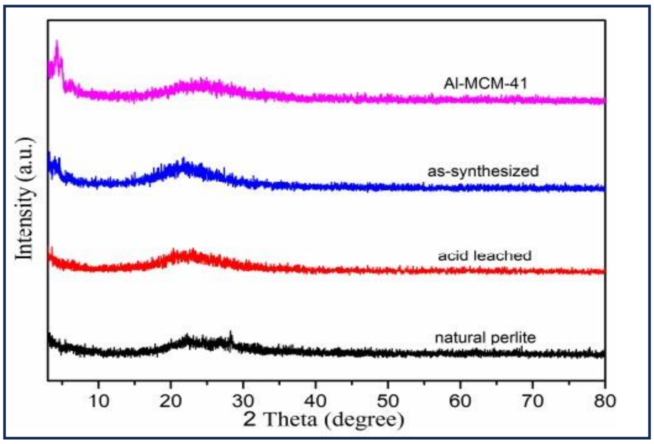
Sample obtained:

Chapter 5

Characterization techniques used

XRD

X-Ray diffraction analysis is a non-destructive technique that provides detailed information about the crystallographic structure, chemical composition and physical properties of material. XRD works based on the principle of Bragg's law, in the process, a monochromatic x-ray beam is directed at a sample. When X-rays encounter the electrons within the crystal lattice, they scatter in various directions. The scattered X-rays then interfere constructively if the path length difference between waves scattered from adjacent crystal planes is a multiple of the X-ray wavelength. This constructive interference results in a distinct diffraction pattern, typically captured on a detector. By analyzing the angles and intensities of these diffraction peaks, one can determine crucial information about the crystal structure, such as lattice spacing, phase composition, and orientation of the crystal lattice.



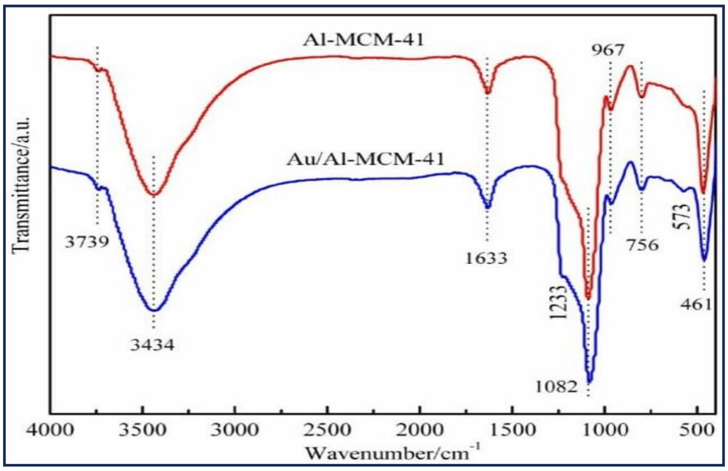
BET

Brunauer-Emmett-Teller (BET) analysis is a method employed to determine the specific surface area of a material, often porous substances like catalysts or adsorbents. The technique involves exposing the material to a gas, typically nitrogen, at various pressures and measuring the amount of gas adsorbed onto the material's surface.

The data obtained is then used to create a plot of the amount of adsorbed gas against the relative pressure. The resulting graph typically exhibits a characteristic shape with distinct regions. The BET equation is applied to fit the data, providing information about the monolayer capacity, multilayer adsorption, and ultimately, the specific surface area of the material.

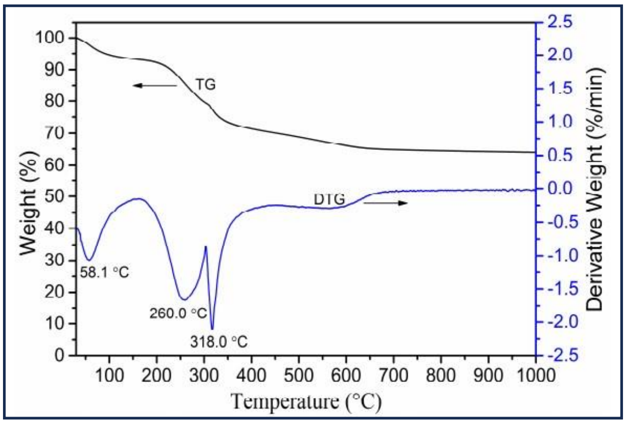
FTIR

Fourier transform infrared spectroscopy is a technique that used infrared radiation to identify and analyze the chemical composition of materials. FTIR works by shinning infrared light through a sample and measuring how much of the light is absorbed. The amount of absorption is related to the functional groups present in the sample, which can be used to identify the sample or to determine its chemical composition.



TGA

A thermogravimetric analyser continuously measures mass while the temperature of a sample is changed over time. In thermogravimetric analysis (TGA), a sample is continually weighed while heating, as an inert gas atmosphere is passed over it. The sample in this experiment was heated at a temperature range of (30-600) °C and at a heating rate of 10 degree per minute in an inert atmosphere of nitrogen. The loss of weight was measured as a function of temperature. For this, 3-5 mg of sample was used for each sample.[Hongyun Chen et al.]



Chapter 7

Results and Discussion

Chapter 8

Conclusion

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