

SYNTHESIS, CHARACTERIZATION OF MAGNESIUM OXIDE/ CARBON NANOCOMPOSITE AND ITS ADSORPTIVE EFFICIENCY FOR THE REMOVAL OF CIPROFLOXACIN FROM THE AQUEOUS SOLUTION

Submitted in partial fulfilment of the requirements for the award of

Master of Science in Chemistry

by

T. NISHANTH RAJ

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**DEPARTMENT OF CHEMISTRY
SCHOOL OF SCIENCE AND HUMANITIES**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE

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MAY - 2022



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **T. NISHANTH RAJ (40910007)** who carried out the project entitled "**SYNTHESIS, CHARACTERIZATION OF MAGNESIUM OXIDE/ CARBON NANOCOMPOSITE AND ITS ADSORPTIVE EFFICENCY FOR REMOVAL OF CIPROFLOXACIN FROM THE AQUEOUS SOLUTION**" under our supervision from November 2021 to May 2022.

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DECLARATION

I **T. NISHANTH RAJ (40910007)**, hereby declare that the Project Report entitled “**SYNTHESIS, CHARACTERIZATION OF MAGNESIUM OXIDE/ CARBON NANOCOMPOSITE AND ITS ADSORPTIVE EFFECIENCY FOR THE REMOVAL OF CIPROFLOXACIN**” done by me under the guidance of **Dr. T. Krithiga, M.Sc., Ph.D.**, Associate professor, Department of Chemistry at **Sathyabama Institute of Science and Technology**, Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai – 600119 is submitted in partial fulfillment of the requirements for the award of Master of Science degree in Chemistry.

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T. NISHANTH RAJ

ABSTRACT

Magnesium oxide nanoparticles have been synthesized by sol-gel method. The synthesized nanoparticle has been characterized by FTIR, XRD and SEM with EDAX. XRD studies show that zinc oxide nanoparticle as hexagonal wurtzite structure. The SEM analysis confirms the formation of spherical particles which are agglomerated. FTIR confirms the presence of Mg – O stretching bond. EDAX confirms the presence of Magnesium and oxygen in the formed catalyst. The synthesized Magnesium oxide nanoparticle was used a catalyst for the removal of the ciprofloxacin from the aqueous solution. Th. The synthesized product was characterized by FTIR and NMR spectroscopy techniques. The functional groups present in the product and its structure was confirmed by the spectral studies.

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LIST OF ABBREVIATIONS

NMR	- Nuclear Magnetic resonance Spectroscopy
XRD	- X-Ray Diffraction
FTIR	- Fourier Transform Infrared Spectroscopy
SEM	- Scanning electron Microscope
BET	- Braeuer-Emmette-Teller

CHAPTER 1

INTRODUCTION

1.1 CATALYST

A Catalyst is a substance that speeds up the chemical reaction without involving during the reaction. When the catalyst is added to chemical reactions, reduce the activation energy and increase the reaction rate. The catalyst is not consumed in the reaction process, the amount of catalyst does not change during the reaction. To reach the transition state of the reaction the catalyst lowers the energy by allowing more molecular interactions to achieve that state. However, catalysts do not affect the reaction progresses. Even though catalysts affect reaction kinetics, the equilibrium state remains unaffected.

Catalysis is a process of adding a catalyst to ease the reaction using the catalyst leads to fastening the chemical reaction.

1.1.1 TYPES OF CATALYSTS: -

Mainly catalysts are four types,

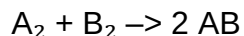
- ❖ Homogeneous catalyst
- ❖ Heterogeneous catalyst
- ❖ Heterogenized homogeneous catalyst
- ❖ Biocatalyst

1.1.2 HOMOGENEOUS CATALYST: -

In homogeneous catalysis reaction mixture and catalyst both are in same state. Homogeneous catalyst has high interaction because the catalyst and reactant have high homogeneity among them. Some of the Example for homogenous catalyst, Acid catalysis, organometallic catalysis, and enzymatic catalysis are examples of homogeneous catalysis. Here is the Some important chemical process that occurs through homogenous catalysis are carbonylation oxidation and hydrogenation.

An advantage of homogeneous catalysis is that the catalyst mixes into the reaction mixture and allows the interaction between catalyst and reactant molecules at high

degree. However, the heterogeneous catalysis is different, the homogeneous catalyst is often irrecoverable after the reaction has run to completion.



1.1.3 HETEROGENOUS CATALYST: -

Heterogenous catalysis is a catalysis that the reactant or product and catalyst differ from the states. Example of heterogenous catalyst is Haber- Bosch process for the synthesis of ammonia. In Heterogenous catalyst we can easily separate the product and recover the catalyst used so they are widely used in industrial process. Typically, solid phase heterogenous catalysts are used in order to work the chemical reaction between two gaseous reactants. In such reactions, the catalysis takes place over the following three steps:

- The gaseous reactants are adsorbed on the surface of the solid heterogenous catalyst.
- The absorbances of reactants in the chemical reaction, resulting in the formation of the product.
- The product compound is disinserted from the surface of the catalyst, resulting in the regeneration of the active catalytic surface.

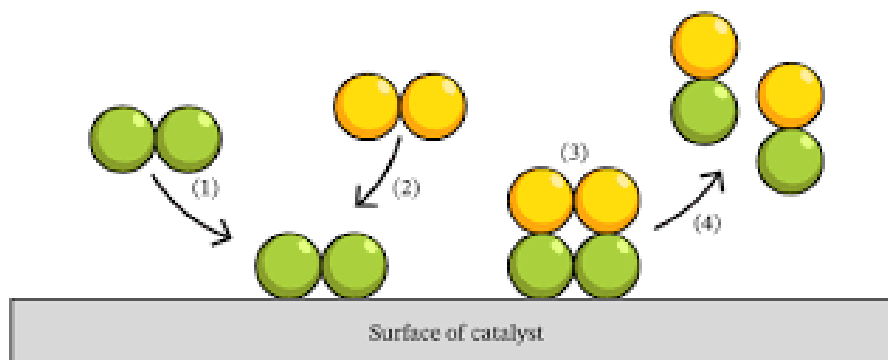


Fig 1.1 Heterogenous catalyst

Source: <https://www.nagwa.com/en/explainers/576190657252/>

Heterogenous catalysts are very useful because they can be used in production of several commercially important products on a relatively large scale. For example, Oxides of iron placed on alumina (a chemical compound with the formula Al_2O_3) are widely used as heterogenous catalysts in the Haber process for the industrial production of ammonia.

1.1.4 HETEROGENIZED HOMOGENEOUS CATALYSTS: -

Heterogeneous catalyst is different from the homogeneous counterpart are much more difficult to develop practically because of their complexity.

1.1.5 BIOCATALYST: -

Some specific chemical reactions are carried outside the living cells by catalysis using natural proteins or nucleic acid is called bio catalysis. From plant, microbes and animal tissues the enzymes are obtained. They are high selectivity, high efficiency and eco- friendly.

1.2 NANOPARTICLES: -

A nanoparticle is a small particle which have the range between 1 to 100 nanometres in size. Human cant sees it with naked eyes. They exhibit significantly different physical and chemical properties to their larger material.

1..2.1 TYPES OF NANOPARTICLES: -

They are classified into different types based on their size, morphology, physical and chemical properties,

- I. Carbon- based nanoparticles
- II. Ceramic nanoparticles
- III. Metallic-oxide nanoparticles
- IV. Semiconductor nanoparticles
- V. Polymeric nanoparticles
- VI. Lipid-based nanoparticles

1.2.2 CARBON-BASED NANOPARTICLES: -

They are further classified into two main materials.

- ❖ Carbon nanotubes (CNTs)
- ❖ Fullerenes.

Carbon nanotubes (CNTs) is nothing but graphene sheets rolled into a tube. They are 1000 times stronger than steel.

1.2.3 CERAMIC NANOPARTICLES: -

These ceramic nanoparticles are made up of oxides, carbides, carbonates and phosphates. They are highly resistance to heat and chemical inertness. They are crudely used as drug delivery system for a number of diseases like bacterial infections, cancer etc.....

1.2.4 SEMICONDUCTOR NANOPARTICLES: -

These semiconductor nanoparticles are found in the periodic table in groups 2-6, 2-4. they are used in photocatalysis photo-optics because they have wide bandages particles.

1.2.5 POLYMERIC NANOPARTICLES: -

These polymeric nanoparticles are based on organic depending of preparation methods, they have structure like nano capsular or nanosphere. Nanosphere particles has a structure like matrix whereas the nano capsular particles have core- shell structure.

1.2.6 LIPID- BASED NANOPARTICLES

Lipid-based nanoparticles are consisting of solid core made of lipid. It is spherical shaped with a diameter of 10 to 100nm. The external surface of these nanoparticle is stabilized by surfactants and emulsifier. They can be used in biomedical field as drug carrier and delivery.

1.2.7 METAL-OXIDES NANOPARTICLES:

These nanoparticles can be prepared by chemical, electrochemical or photochemical methods. These nanoparticles have applications in research area, detection and ignoring of biomolecules and in environmental and bioanalytical applications. Metal oxide nanoparticles play vital role in environmental remediation by treating waste water industrial and domestic waste, soil sediments as well as atmospheric pollution.

1.2.8 MAGNESIUM OXIDE NANOPARTICLE

Magnesium oxide nanoparticles is widely used among the other metal oxides nanoparticles that widely being used in various fields because they have promising structure material in biological implants due to their high strength to weight ratio, low density, good functionality, recycling activity, nontoxic and hygroscopic nature Magnesium oxide nanoparticles are adsorbing and non- toxic. They are like white powdered substance. They are highly hard, highly pure, and they have high melting point. Magnesium oxide nanoparticles can be used in as electronics, catalyst, ceramics, petrochemicals products, coating and numerous other fields. There are few methods to synthesis by methods like sol-gel, precipitation method, and green synthesis and it is widely used as toxic Waste remediation, antibacterial materials, removal of industrial pollutants and anti-cancer activities.

S.No.	Synthesis method	Synthesis Procedure	Reference
1	Precipitation method	$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 0.1M + 0.2 M NaOH → Add 50ml H_2O , drop by drop → For 2hrs → Precipitate will be obtained $[\text{Mg}(\text{OH})_2]$ → Wash thoroughly → Centrifuge at 4000 rpm, 15 min → Dry precipitate at 100 °C and calcinate at 400 °C → Magnesium oxide nanoparticles synthesized	7, 30
2	Sol-gel method	$\text{Mg}(\text{CH}_3\text{COO})_2$ + CTAB (0.001 M) → Stir till solution becomes transparent → Add 1M $\text{C}_4\text{H}_9\text{O}_2$ (For gel formation) → Keep gel at 100 °C, For 24 hours → Grind and calcinate at 600 °C, for 6hrs → Magnesium Oxide Nanoparticles obtained	7, 31, 32, 33, 34
3	Green synthesis method	Nephelium lappaceum peel cut into pieces and washed → Dry at 60 °C → Boil 3g of peels in mixture of ethanol and ddH ₂ O (1:2), for 10 min → Filter extract → 10 ml peel extract was slowly added to 50 ml 0.1 M $\text{Mg}(\text{NO}_3)_2$ under magnetic stirring at 80 °C, for 2 hours → Forms magnesium ellagate complex → Complex is centrifuge at 10,000 rpm, for 10 min → Dried at 40 °C, for 8 hours and powdered → Powdered sample calcinated at 450 °C → Magnesium oxide nanoparticles synthesized	35
4	Hydrothermal method	$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ [Magnesium Nitrate] → Dissolved in Distilled water and surfactant (Polyethylene glycol + sodium dodecyl sulfate) → 100 ml of 0.2M NaOH solution added dropwise to the above mixture → Heat the mixture at 100 °C for 2 hours → Cool to room temperature and collect the precipitate → Calcinate precipitate at 500 °C for 4 hours → White solid Magnesium oxide nano-powder obtained	36
5	Microwave assisted	$\text{C}_2\text{H}_2\text{O}_4$ (Oxalic acid) + $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (Magnesium Chloride) → Dissolve it in distilled water → Stir the solution for 30 minutes at 60 °C → Evaporate the solution by keeping in microwave for 8 minute at 800W → White coloured gel is obtained → Keep it at 800 °C for 2 hours in a furnace for calcination → An off white residue is obtained → Grind it using mortar & pestle → The powder obtained is MgO Nanopowder	37

Fig 1.2 Magnesium Oxide Synthesis Process

Source: https://www.researchgate.net/publication/343017457_Recent_Applications_Of_Magnesium_Oxide_MgO_Nanoparticles_In_Various_Domains

1.3 CARBON AS CATALYST

Carbon can be used as both catalyst and catalyst support for the chemical and enzymatic reactions because they have large surface area, excellent electron conductivity and chemical inertness. When the carbon is chemically mixed with nanoparticles and enzymes the catalytic activity of carbon is increased.

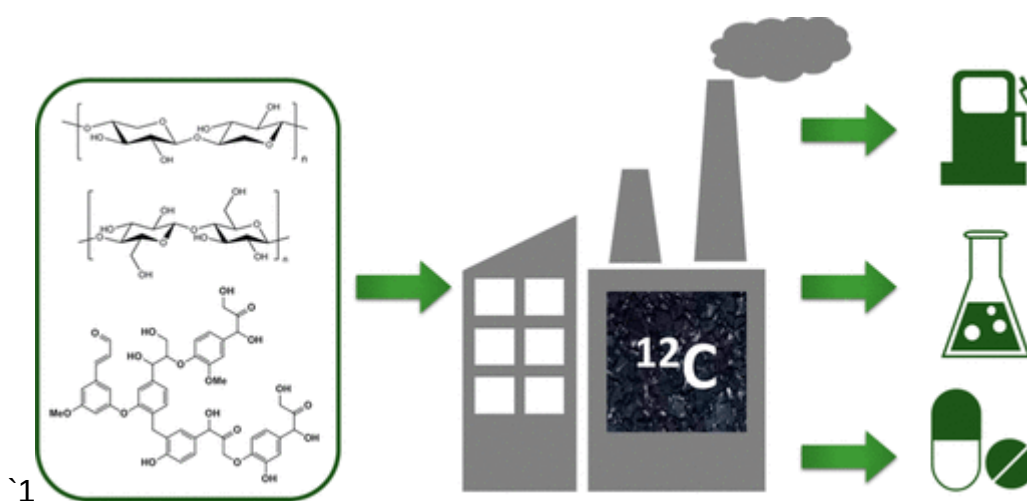


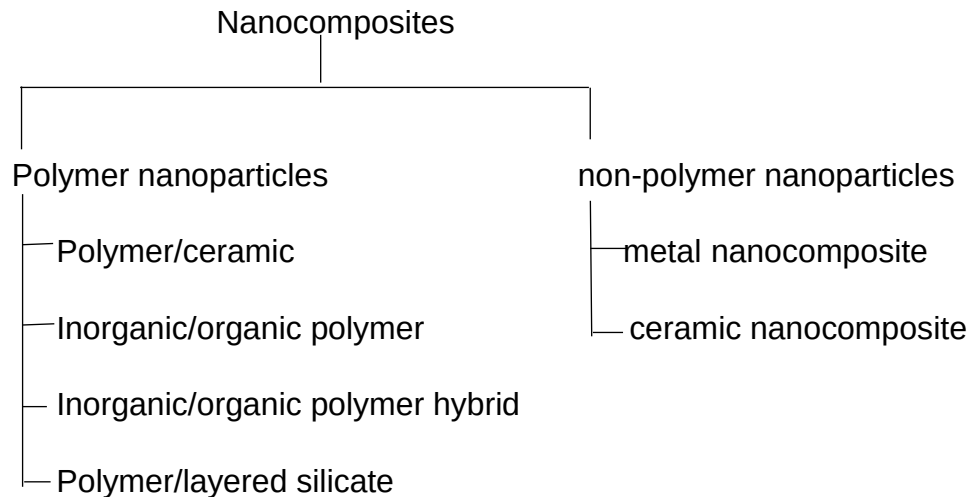
Fig 1.3 Carbon Catalyst

Source: <https://pubs.acs.org/doi/10.1021/cs5008393#:~:text=Carbon%20plays%20a%20dual%20role,conductivity%2C%20and%20relative%20chemical%20inertness>

1.4 NANOCOMPOSITES

General nanocomposites are based on the concept of creating a very large interface between the nanosized-building blocks and the polymer matrix, the main problems of nanocomposite are usually homogeneous distribution of the particle. Nanocomposites are made up of two or more constituent materials with notable different physical or chemical properties after the combination. Its procedure a material has different characteristics from the individual components. If at least one of the component constituents is nanometric then it is nanocomposite. They are heterogeneous materials thus their properties are determined by the same

factors as in traditional composites. Their structure is more complicated than the micro composites.



1.4.1 POLYMERIC NANOCOMPOSITE

Polymeric nanocomposite consists of polymer or co polymer with nanoparticles speared in the polymer phase. They are of different shapes like fibre, platelets and spheroids from those at least one of its one dimension should be in the range of 1-50nm. They consist of multiphase system. In plastic production polymer nanocomposites cover 95%. They are done by controlled manner of compounding.

1.4.2 NON-POLYMER NANOCOMPOSITES

the nanocomposite which is not consist of any polymer compounds in it is called non polymer nanocomposites.

1.4.3 METALLIC OXIDE NANOCOMPOSITE

Bimetallic nanocomposites are used in the form of alloy or core shell structure because they have vast catalytic properties, they are characterized by,

- ❖ Super plasticity
- ❖ Lower melting point
- ❖ Increased strength and hardness
- ❖ Improved magnetic properties

- ❖ Increased electric resistivity etc.....

1.4.4 MAGNESIUM OXIDE/CARBON NANOCOMPOSITES

Here the catalyst is prepared by taking glucose (500mg) as the carbon source and Mg (NO₃)₂ in different constitution like (0, 50, 100 and 200mg) respectively. They are mixed in the inert salt of KCL (1g) and LiCl (1g) they are used as the molten salts for porous carbon synthesis, then the mixture is calcined in a tube furnace under nitrogen atmosphere in room temperature to 800°C at 5°C min⁻¹ and maintained at the temperature for 2hr after calcination it is exposed to more magnesium oxide. After calcination is done the carbonaceous material is washed out to remove the salt and dry to weight by using different weight range of Mg loaded with Carbon as carbon catalyst, Mg (500mg)/ carbon catalyst, Mg (100mg)/ carbon catalyst, Mg (200mg)/ carbon catalyst. Respectively and the general name of these catalyst is Mg(x)/ carbon where x stands for the weight of Mg load.

1.5 PHARMACEUTICAL WASTE

Pharmaceutical waste is used or unused expired drug or medicine pharmaceuticals, home use person care products and medications used or disposed in garage. It can be classified as hazardous or non-hazardous depending on its risk to humans and the environment. There are different methods required for the disposal of hazardous versus non-hazardous pharmaceuticals.

1.5.1 HOW TO DISPOSE OF PHARMACEUTICAL WASTE

Pharmaceutical waste cannot be disposed in the normal way. We should take some special care and precautions must be taken while handling and disposing of pharmaceutical waste due to its dangerous nature. If pharmaceutical waste is not disposed properly, the effect is catastrophic, not only for the peoples for also to the animals and plants.

1.5.2 RISKS OF IMPROPER PHARMACEUTICAL WASTE DISPOSAL

If a drug is disposed incorrectly, dangerous chemicals can leach into the surroundings environments and contaminate ground water, drinking water, plants life and so on. This may cause health problems in the society. Improper disposal

methods may include washing drugs down sinks, flushing them down toilets, throwing them away in trash or dumping.

1.6 MAGNESIUM OXIDE

Magnesium oxide is one of the most important metal oxides in the field of catalyst, it is commonly used as support, MgO is an oxide with a rock-salt structure, on each Mg^{2+} is surrounded by five O^{2-} ions, usually the anhydrous surface of metal oxides possesses two active sites, the cation Mn^{+} and the anion is O^{2-} . Mg^{2+} has low reduction potential, which makes it easily reduced to Mg^{1+} to Mg^0 during catalytic testing, Magnesium oxide is an inorganic salt formed by Magnesium ions with oxygen. Magnesium is mostly available in supplement form. It is added to dietary supplements as well as over the counter medications used to treat constipation, indigestion, and headaches. Magnesium oxide and other inorganic salts of magnesium carbonate are high in elemental magnesium, which is the total amount of magnesium in a supplement. They have low solubility rate and making them as a less bioavailable than other forms of the minerals. Even though magnesium oxide supplement provides a good amount of magnesium, it is not the most available from our body to utilize. Magnesium has strong laxative effects it may lead to digestive effects like diarrhoea and it has several benefits and it is commonly used to treat medical conditions like chronic constipation. Magnesium oxide may help to treat migraine and constipation, reduce blood pressure, improve blood sugar management, and decrease levels of stress and anxiety in certain populations. Magnesium oxide dosing generally ranges from 250-1000 mg per day, High doses of magnesium oxide may cause dangerously high blood magnesium levels, it may also cause side effects like bloating and diarrhoea and reduce the effectiveness of certain medications.

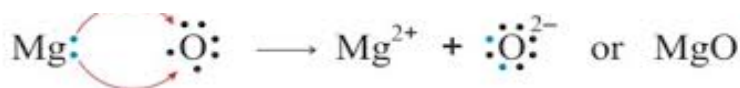


Fig: 1.4 Formation of MgO

Source: <https://www.embibe.com/exams/magnesium-oxide-formula/>

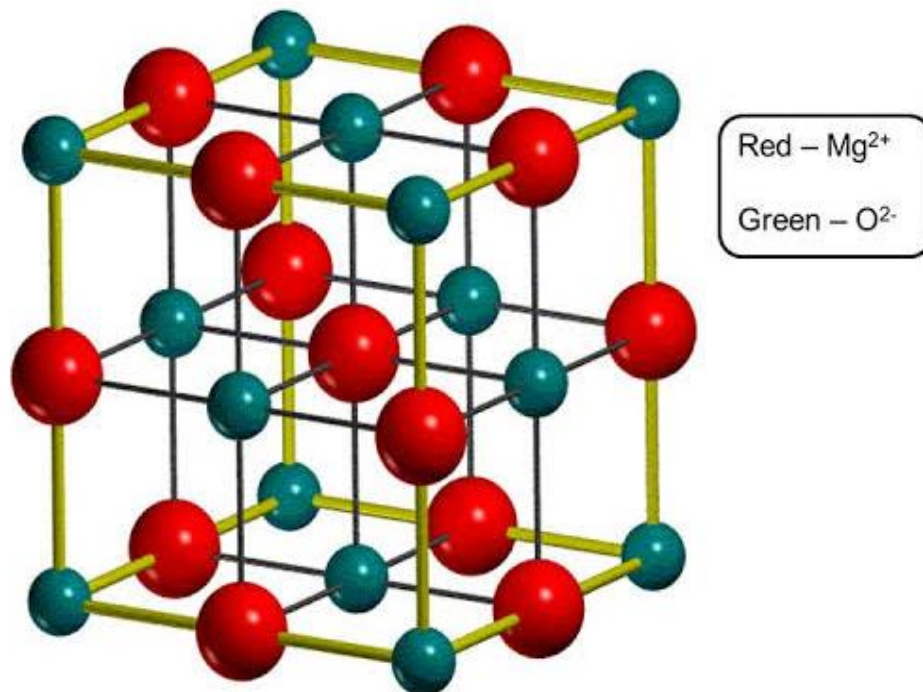


Fig 1.5 Structure of Magnesium oxide

Source: <https://www.chegg.com/homework-help/questions-and-answers/determine-lattice-constant-mgo-crystal-structure-nacl-given-r-mg-2-0078nmr-mg-2-0078nm-r-o-q23628839>

CHAPTER 2

LITERATURE SURVEY

Rizwan Wahab, S.G. Ansari, M.A. Dar, Y.S. Kim and H.S. Shin demonstrated the synthesis of Magnesium oxide nanoparticle by sol-gel method using Magnesium nitrate and sodium hydroxide at the room temperature. Hydrated Magnesium Oxide were heated in air at 300 and 500°C. crystallinity of the nanoparticle is obtained by X-ray diffraction. Morphology and size of the nanoparticle of the nano particle is shown at the range of 50-70nm powder composition was analysed by the FTIR spectroscopy

S. Abinaya, Helen P. Kavitha, M. Prakash, A. Muthukrishnaraj obtained the nanoparticle in less hazardous process. The Magnesium Oxide nanoparticle have been effectively produced by green synthesis which is environmentally friendly, non-toxic with greater stability.

Renata Dobrucka has demonstrated that the we can synthesis MgO nanoparticle using an alternative way to the traditional chemical method. Here the aim is to synthesis MgO nanoparticle using the Artemisia abrotanum herb water extract. It is characterized by UV-Visible spectroscopy, (FTIR) Fourier transform infrared spectroscopy, (XRD) X-ray diffraction, (SEM) scanning electron microscopy with EDS and (TEM) transmission electron microscopy. The crystallite structure of MgO is known XRD. The average size of the MgO nanoparticle was found in the range of 10nm using SEM. EDS confirms the signal characteristic of magnesium and oxygen. FTIR analysis confirmed the presence of active compounds responsible for the stabilization of MgO Nanoparticle

J Suresh, R Yuvakkumar, M Sundararajan, Sun Ig Hong demonstrated that Nephelium lappaceum L. peels was effectively used for the synthesis of Magnesium oxide nanoparticles as a natural ligation agent. The Crystallinity and spherical morphology were revealed by XRD and SEM. The successful formation of magnesium oxide nanoparticle was confirmed employing XRD, SEM-EDX and PSA analysis.

KR Nemade, SA Waghuley has synthesized MgO using solvent spray pyrolysis technique, since reaction can be run in liquid state without further heating. MgO extensively used as catalyst. The crystallinity structure of MgO pattern was confirmed by XRD.

Jagadeesan Aravind Kumar, Duvuru Joshua Amarnath. Thangavelu krithiga, Samson Sunny, Somasundaram Purna demonstrated that activated carbon can be prepared by palm shell agro waste by chemical activation method using potassium hydroxide. Nano-Magnesium oxide is placed onto palm shell. Activated carbon composite was produced by dropwise mechanism and its efficiency was tested for the removal of anthracene from the aqueous solution. Amorphous nature of composites was confirmed by X-Ray diffraction.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

The main aim of this project is to synthesis MgO nanoparticle. MgO nano powders were synthesized with well-defined morphology at the room temperature by using sol-gel method using magnesium nitrate and sodium hydroxide in a stoichiometric ratio via sol-gel method. MgO nano powder were characterized with the help of FTIR, XRD, SEM and to remove the Ciprofloxacin from the aqueous solution.

3.2 Scope

- Synthesizing of MgO/C by sol-gel method.
- Characterization of MgO/C by FTIR spectroscopy, XRD, SEM and EDAX.
- Removal of ciprofloxacin from aqueous solution.
- Conformation of product formed by characterization by FTIR.

CHAPTER 4

MATERIAL AND METHODOLOGY

4.1 MATERIALS:

Materials used in this reaction are magnesium nitrate, sodium hydroxide, deionised water, dextrose which were purchased from micro fine chemicals and it can be used without further purification.

4.2. CHARACTERIZATION TECHNIQUES:

4.2.1 Powder X-Ray Diffraction (XRD)

Since most materials have unique diffraction patterns, when an X-ray is incident on the crystal they scattered in accordance with Bragg's law, it diffracts and produces different patterns. Which assumes the diffraction to take place only when the distance travelled by the rays reflected from the successive planes of crystal, by scanning the sample through a range of 2θ angles, plotting the angular positions and intensities of the resultants diffracted peaks produced the pattern. Finally, matching the resultants diffraction peaks to characterize, finally the resultant patterns are also studied for the position of diffraction maxima, peak intensities, and the intensity distribution as a function of the diffraction angle, Here the diffracted pattern is obtained from a powder of the material. Compounds can be determined from its diffracted pattern. Powder diffraction is often easier and more convenient than single crystal diffraction, the purity of a sample can also be determined from its diffracted pattern, Powder X-ray diffraction is a characterization tool that offers a detailed view of the crystallographic structure and chemical composition of the sample. The particle size can be determined by using the Scherrer formula is

$$\text{Scherrer formula} = (D = K\lambda/\beta\cos\theta)$$

Where, K = e Shape factor (0.9)

λ = wavelength of X-rays,

β = corrected full width at half-maximum;

θ = angle of peak

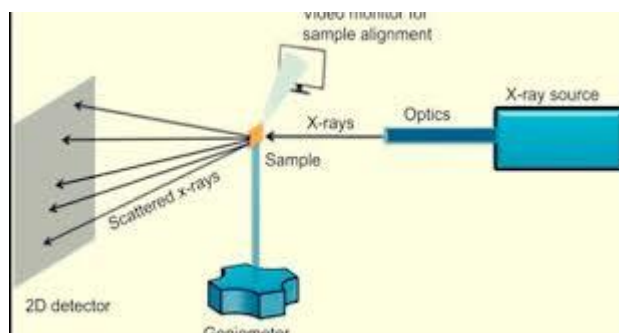


Fig 4.1 X-ray diffractometer

Source: <https://www.14impressions.in/2020/09/principle-instrumentation-types-and.html>

4.2.2 Fourier Transform Infrared Spectrometer

FTIR stands for Fourier transform infrared, it is a versatile tool for surface characterization of nanoparticle. It involves in various phenomena such as the adoption, reflection, and emission of light using the sample and it is used to identification of organic, inorganic, and polymeric materials using infrared light for scanning the samples. FTIR is useful in identifying and characterizing unknown materials, detection contaminants in a material, finding additives, and identifying decomposition and oxidation. FTIR spectrometer includes a source, sample cell, detector, amplifier, A/D convertor, and a computer. when IR radiation is passed through a sample, some radiation is absorbed by the sample and some passes through (is transmitted), radiation from the source reach the detector after it passes through the interferometer. The signal is amplified and converter to a digital signal by the A/D converter and Amplifier, then the signal is carried to the computer. The resultant signal obtained at the detector is a spectrum generally from the range of $4000\text{--}400\text{ cm}^{-1}$ which represents the fingerprint range of sample. For this purpose, the sample were mixed potassium bromide and made into pellets for analysis.



Fig 4.2 Fourier Transformer Infrared Spectrometer

Source: <https://blogs.getty.edu/iris/conservation-tools-fourier-transform-infrared-spectroscopy-ftir/>

4.2.3 Scanning Electron Microscopy

Scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface, the signal comes out from the electron- sample interaction reveal the information about the sample including external morphology, chemical composition, and crystalline structure and orientation of materials making up the sample. it a high-resolution image of the size and surface texture of the particle, SEM equipped with EDX can determine the main atomic composition of pf the particle, particle colours cannot be determined by SEM. SEM/ESEM-EDX. SEM is one of the main testing instruments for in situ analysis of micro areas. It has high magnification, and strong stereoscopic view. Photographs taken by SEM have higher magnification and clearer definitions than those by optical microscopy. SEM can be able to measure only elements with atomic coefficients less than 5. Using SEM we can get the selected point location images of the sample. This approach is especially useful in qualitative determining chemical composition. In SEM examination system the zones territory from around 1cm to 5 microns in width can be imaged in a filtering mode utilizing SEM methods and having amplification run from spatial goals of 50nm to 100nm.

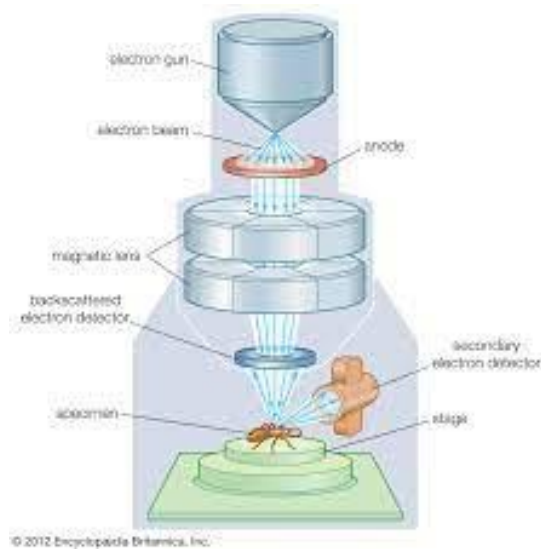


Fig 4.3 Scanning Electron Microscopy

Source: <https://www.britannica.com/technology/scanning-electron-microscope>

4.2.4 Nuclear Magnetic Resonance (NMR)

When a nucleus that possesses a magnetic moment (Such as a hydrogen nucleus ^1H , or carbon nucleus ^{13}C) is placed in a strong magnetic field, it begins to spin like a spinning top.

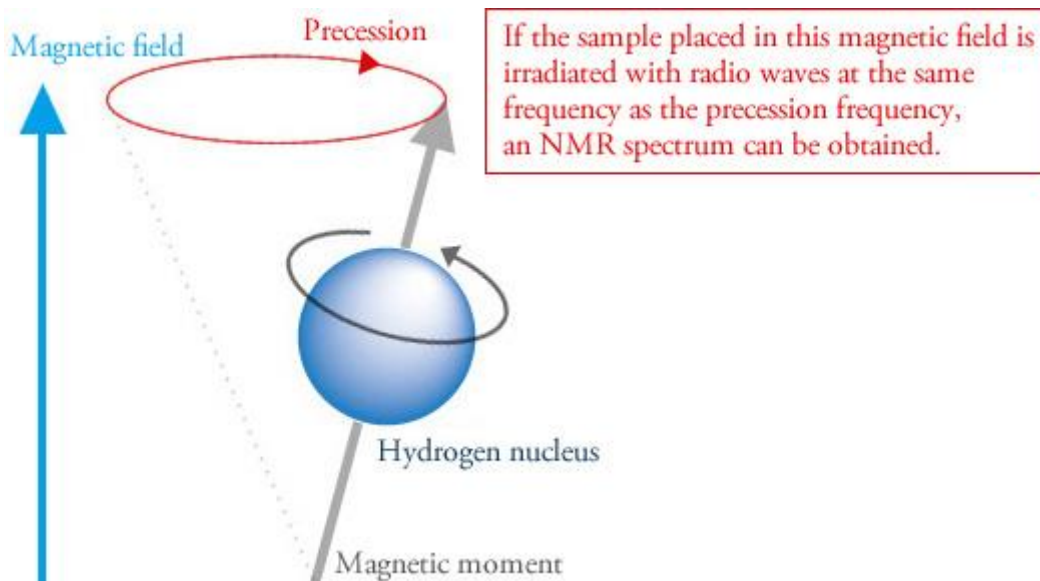


Fig 4.4 spinning momentum

Source: <https://www.jeol.co.jp/en/products/nmr/basics.html>

Nuclear magnetic resonance is technique that exploits the magnetic properties of certain atomic nuclei and can be used to determine the physical and chemical properties of atoms or the molecules. They can provide detailed information about the structure, dynamics, reaction state, and chemical environment of molecules. The electronic structure of a molecule and its individual functional group can be found by the intermolecular magnetic field around an atom in a molecule changes the resonance frequency. They are usually performed on the diamagnetic compounds. The nuclear magnetic resonance spectra of inorganic compounds are more complicated the organic because other nuclei also have nuclear magnetic moments. Solid-state nuclear magnetic resonance spectroscopy and even single-crystal nuclear magnetic resonance spectroscopy can be used on material that simply do not dissolve in any solvent. The theory and application of the NMR response in porous rocks are described, and the sample preparation methods, test equipment, measurement parameters, test procedure, data interpretation techniques and data reporting requirements for the principal NMR tests on core are details.

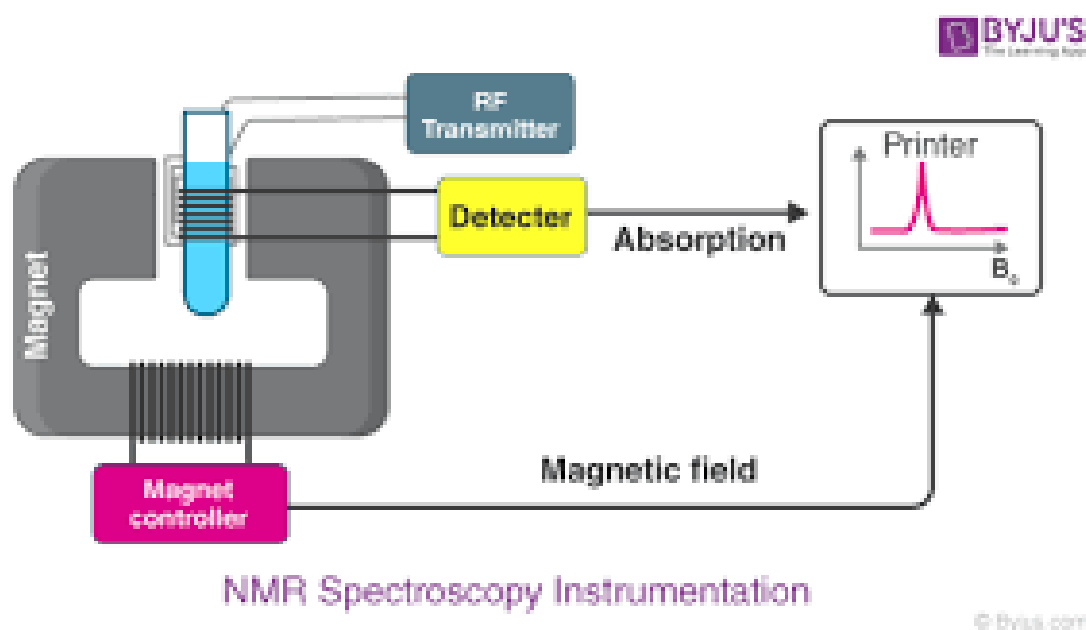


Fig 4.5 Nuclear Magnetic Resonance

Source: <https://www.jeol.co.jp/en/products/nmr/basics.html>

4.2.5 Braeuer-Emmette-Teller (BET)

The Braeuer-Emmette-Teller method is commonly applied to calculate the specific surface area on the basis of nitrogen adsorption isotherm measurements at 77K. the BET model carries multilayer adsorption of gas on the adsorbent surface. In BET surface area analysis, nitrogen is usually used because nitrogen is highly pure and its strong interaction with most solids. Because interaction between solid and gaseous phases is usually weak, the surface is cooled using liquid N₂ to get the adsorption. Known amount of nitrogen gas is injected to the sample cell. After the adsorption layers formed, the sample is removed from the nitrogen atmosphere and heated to cause the adsorbed nitrogen to be released from the material and quantified. The data collected is displayed in the form of BET isotherm, which plots the amount of gas adsorbed as the function of the relative pressure.



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Fig 4.6 Braeuer-Emmette-Teller

Source: <https://www.jinaipu.com/eng/newsInfo.aspx?parentid=940&catid=944&newsid=11948>

There are five types of isotherms is possible,

Type I Isotherm

Type 1 is a pseudo-Langmuir isotherm because it depicts monolayer adsorption, A type one is obtained when P/P_0 is the partial pressure value and C is BET constant. This type of isotherm is given with those pore diameters less than 2nm.

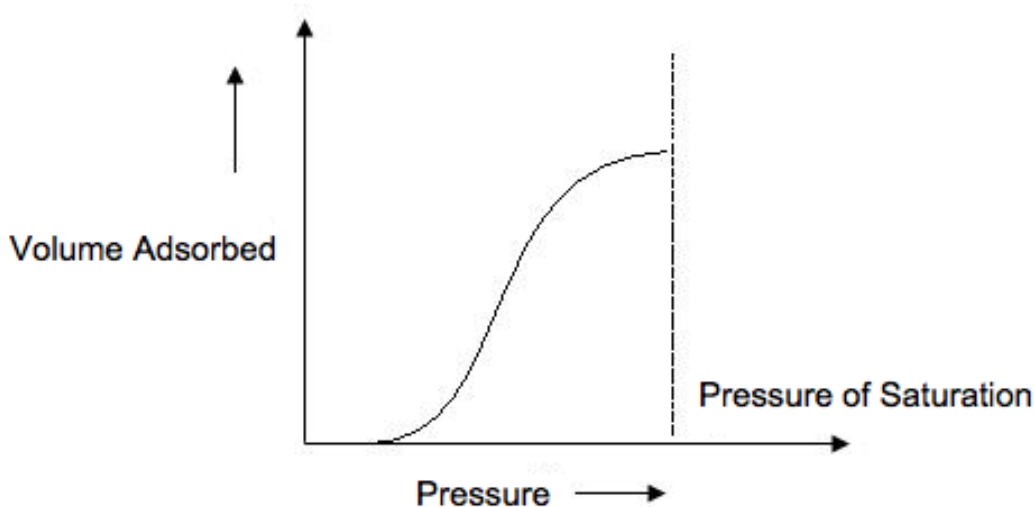


Fig 4.7 Type 1 Isotherm

Source: [https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_\(Barron\)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#](https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_(Barron)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#)

Type II Isotherm

A type II isotherm is different than the Langmuir model. The flat region in the middle indicates the formation of a monolayer. A type II isotherm is obtained when $c > 1$ in the BET equation.

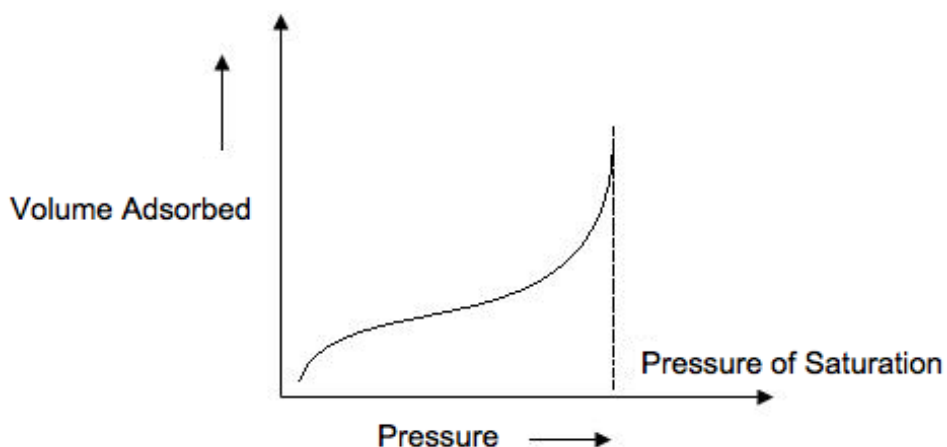


Fig 4.8 Type II isotherm

Source: [https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_\(Barron\)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#](https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_(Barron)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#)

Type III Isotherm

A type III isotherm is obtained when the $c < 1$ and shows the formation of a multilayer. Because there is no asymptote in the curve.

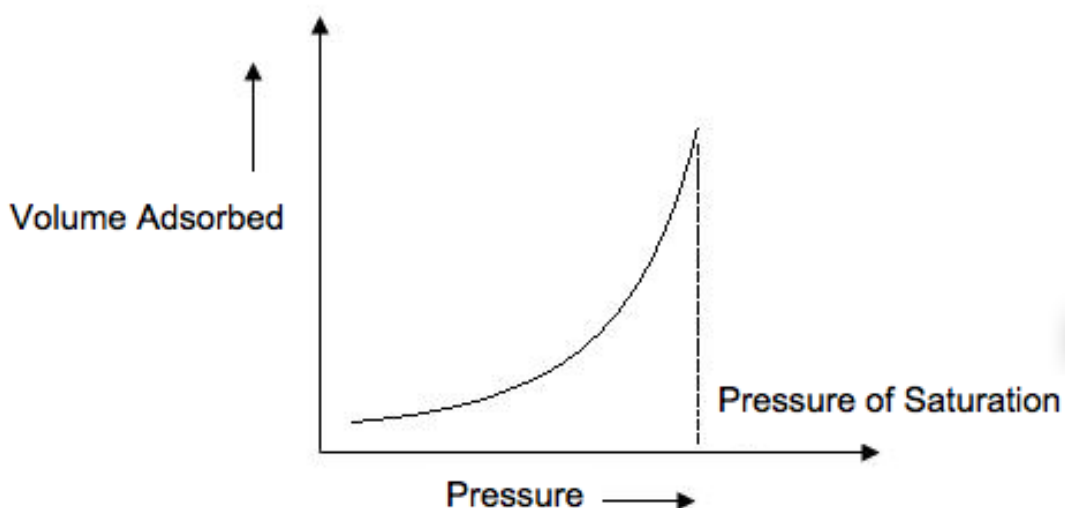


Fig 4.9 Type III isotherm

Source: [https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_\(Barron\)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#](https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_(Barron)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#)

TYPE IV Isotherm

Type IV isotherm occurs when capillary condensation occurs. Gases condense in the tiny capillary pores of the solid at the pressure below the saturation pressure of the gas. At lower pressure it shows the formation of a monolayer followed by formation of multilayer. BET surface area characterization of mesoporous materials, which are materials with pore diameters between 2-50 nm, gives this type of isotherm.

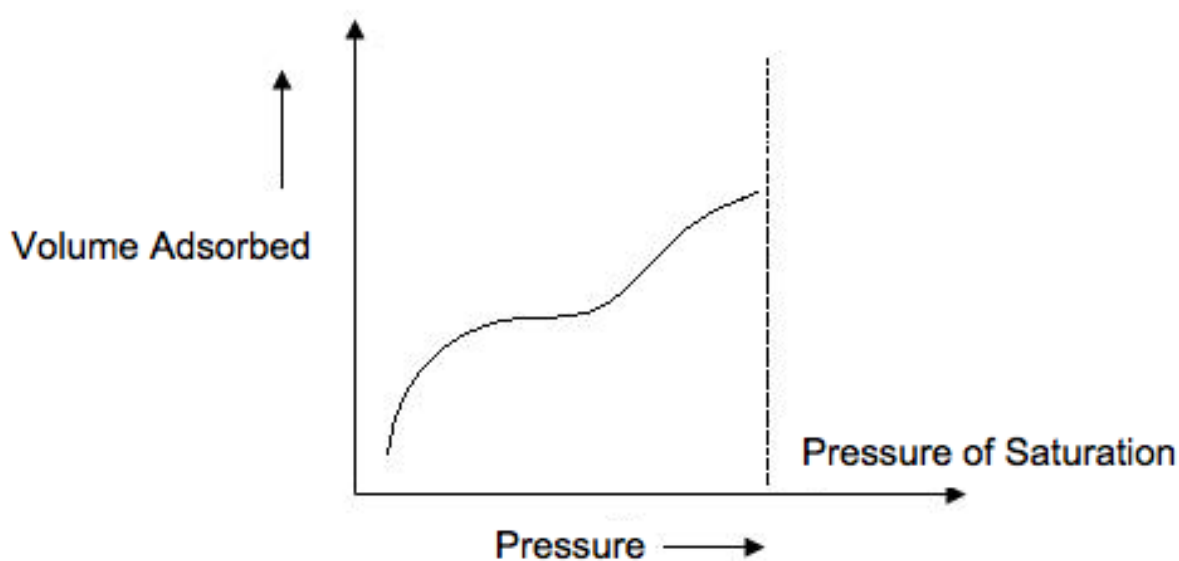


Fig 4.10 Type IV isotherm

Source: [https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_\(Barron\)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#](https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_(Barron)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#)

Type V Isotherm

Type V isotherm are very similar to type IV isotherm and are not applicable to BET.

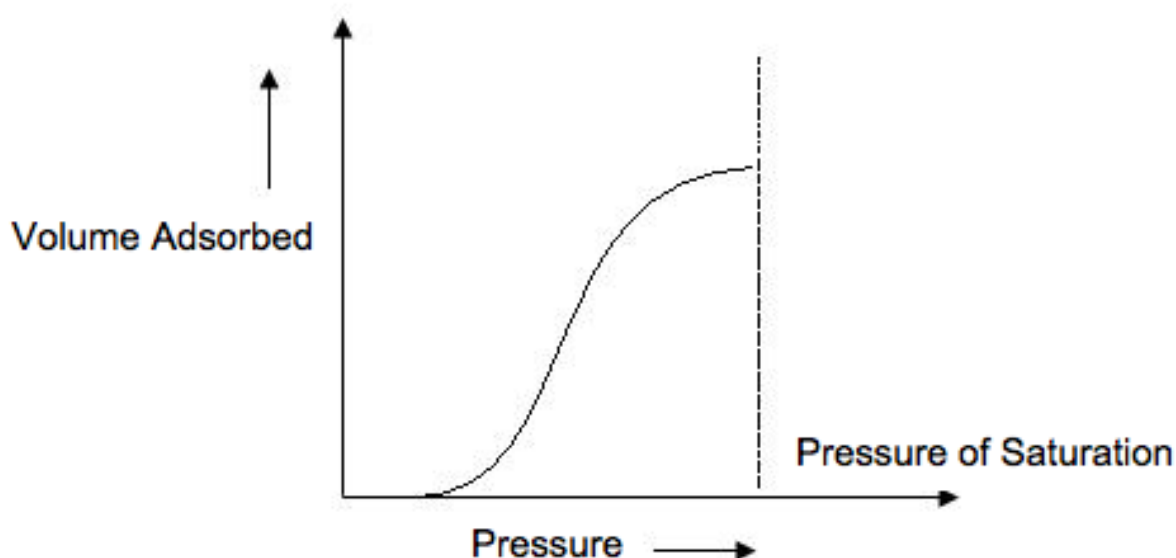


Fig 4.11 Type V Isotherm

Source: [https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical Methods in Chemistry and Nano Science \(Barron\)/02%3A Physical and Thermal Analysis/2.03%3A BET Surface Area Analysis of Nanoparticles#](https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Physical_Methods_in_Chemistry_and_Nano_Science_(Barron)/02%3A_Physical_and_Thermal_Analysis/2.03%3A_BET_Surface_Area_Analysis_of_Nanoparticles#)

4.3 METHODOLOGY:

4.3.1 Synthesis of MgO nanoparticles

0.1M of NaOH is prepared by dissolving 0.300g of NaOH in 100ml distilled water. Take 40ml of prepared NaOH solution in a beaker and place it in hot plated magnetic stirrer when the solution gets boiled then MgNO_3 is added drop by drop (mag stirrer 30°C). A gel like consistency is formed turn off the stirrer and transfers it to a crucible. Place the crucible in oven for (80°C) the moisture to evaporate (approximately 3hrs). Then filter it, wash it, and dry it. And then weight it.

4.3.2 Synthesis of MgO/C Nano composites: -

18g of dextrose(glucose) is dissolved in 100 ml of distilled water in a std flask. 0.1M of MgONO3 solution is prepared by dissolving 1.432g of MgNO3 salt in 100 of distilled water. Take 40ml of the prepared dextrose solution in beaker and place it in hot plate magnetic stirrer. When the solution begins to boil add 40ml of MgNO3 solution drop by drop. A gel like consistency is obtained, then turn off the stirrer and transfer the contents to a crucible. Place the crucible in oven (80°C) for the moisture to evaporate (Approximately 3hrs). Then, place the crucible in muffle furnace at 400°C where the reaction takes place in 7 mins, an amorphous powder is obtained. Then, place the crucible at 500°C for calcination to occur.

4.3.3 Removal efficiency of ciprofloxacin drugs.

Adsorption experiments were conducted for the removal of ciprofloxacin using the Nano-composite. It is experimented by preparing 1ppm by dissolving 1000mg in 1L of solution of volumetric flask. From the prepared solution, and made up to different concentration like (1ml of solution is made up to 100ml, 2ml in 100ml, 3ml in 100ml, 4ml in 100ml, 5ml in 100ml std flask).

4.3.4 Adsorptive efficiency of adsorption parameters

Adsorptive Efficiency of Ciprofloxacin Drug Concentration [10-50ppm]

About (0.01g, 0.03g, 0.05g, 0.075g, 0.1g) from drug is added to 30ppm in 100ml beaker. These containers are sealed tightly and continuously distributed at the speed of 120rpm in a shaker at the temperature of 30°C. The experiment was showed at pH of 6.5.

CHAPTER 5

RESULT AND DISCUSSION

5.1 CHARACTERIZATION OF MgO/C NANO COMPOSITE

5.1.1 FTIR Spectrum MgO/C Nano composites

The FTIR spectrum of Magnesium oxide nanoparticles are shown in Fig.4.1 the IR vibrational frequency obtained at 3849 cm^{-1} , 1760 cm^{-1} , 1417 cm^{-1} , 1518 cm^{-1} , and 738 cm^{-1} correspond to O-H stretching of adsorbable water molecules on magnesium oxide, C=O stretching, water and CO₂ molecule adsorption on MgO surface, O-H in plane bending of carboxyl group adsorbed on MgO and metal – oxygen bond Mg-O stretching respectively. The peak at 738 cm^{-1} is the characteristic peak for MgO nanoparticles.

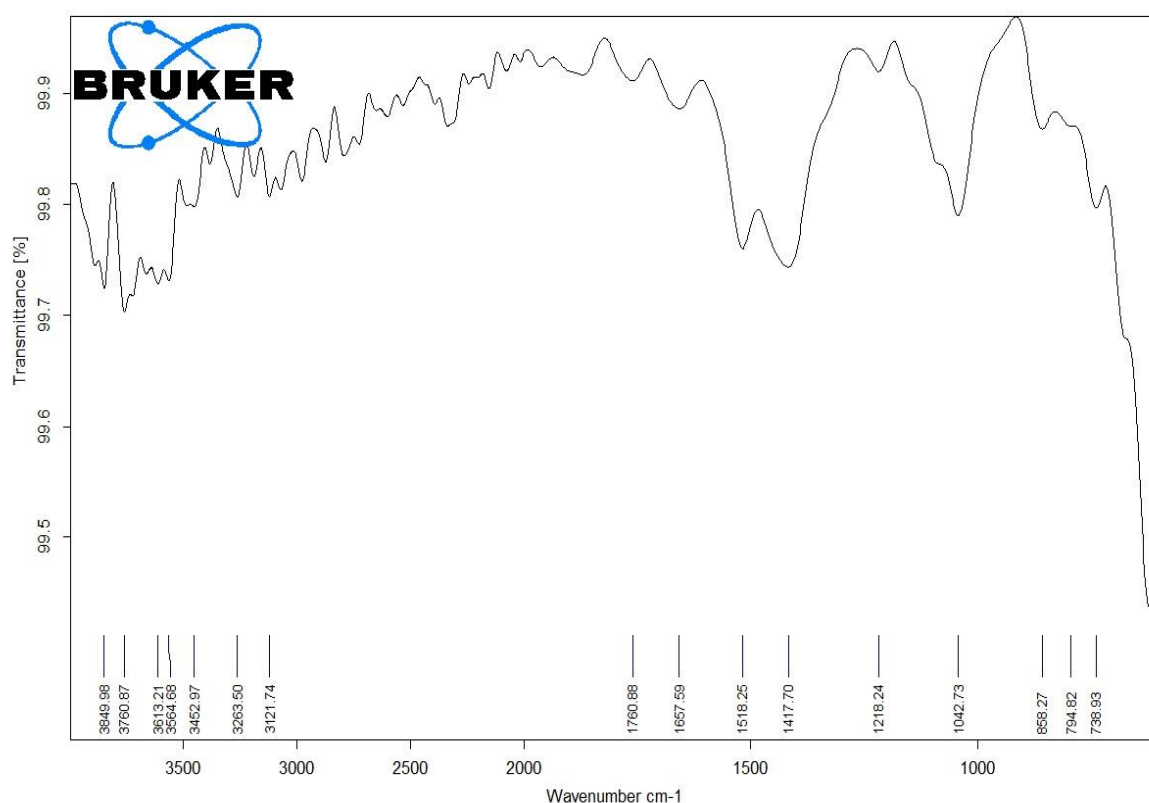


Fig 5.1 FTIR spectrum of MgO/C Nano composite

Vibrational frequency	Functional group identification
3849 cm^{-1}	O-H stretching of absorbable water molecule on magnesium oxide
1760 cm^{-1}	C=O stretching
1417 cm^{-1}	C-O
738 cm^{-1}	Mg-O stretching

5.1.2 SEM and EDAX image of MgO/C Nano composite

The SEM image of Magnesium oxide nanoparticle is shown in the Fig. 4.2. the image explains that magnesium oxide nanoparticle obtained have spherical shape. The SEM image also shows the presence of agglomerates of MgO nanoparticles. The EDAX spectra shows the presence of Mg and O in the MgO catalyst.

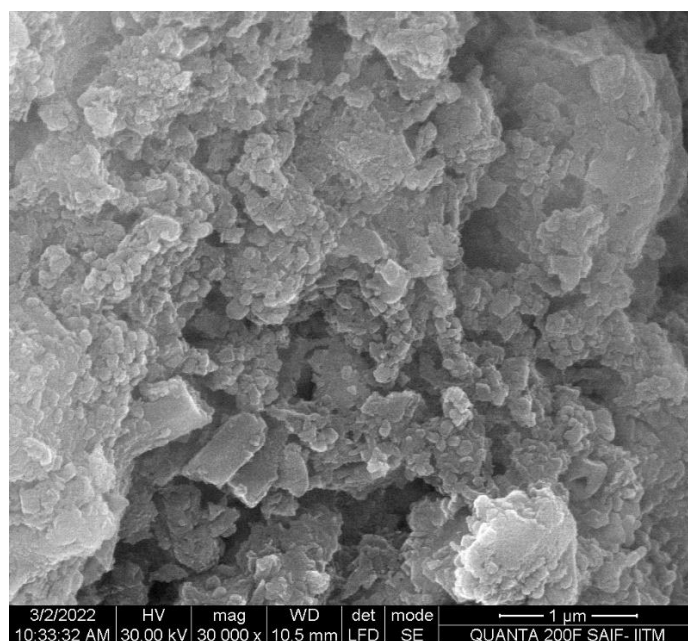
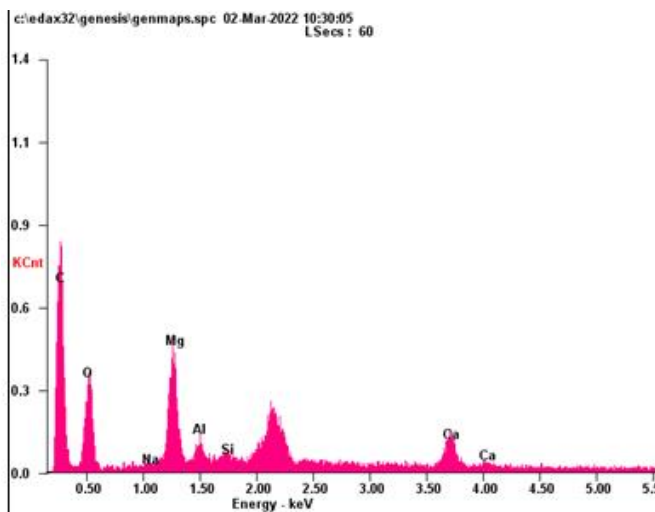


Fig 5.2 SEM image of MgO/C Nano composite



<i>Element</i>	<i>Wt%</i>	<i>At%</i>
<i>CK</i>	64.45	72.76
<i>OK</i>	26.68	22.61
<i>NaK</i>	00.41	00.24
<i>MgK</i>	06.02	03.36
<i>AlK</i>	00.97	00.49
<i>SiK</i>	00.32	00.15
<i>CaK</i>	01.16	00.39
<i>Matrix</i>	Correction	ZAF

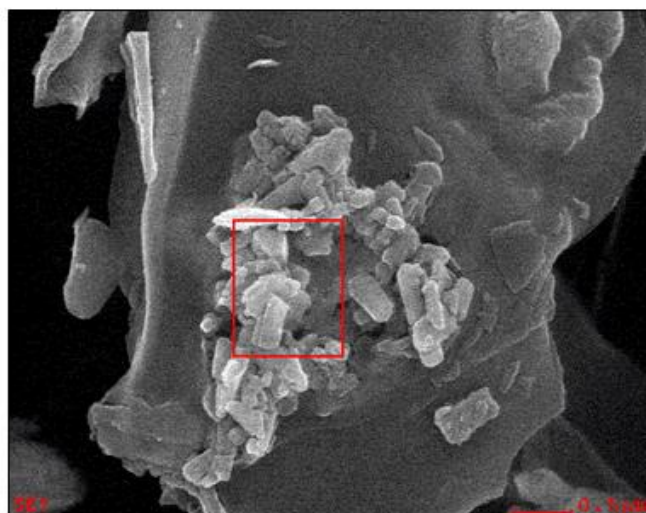


Fig 5.3 EDAX image of MgO/C Nano composite

5.1.3 XRD pattern of MgO/C Nano composite

The XRD pattern of Magnesium oxide nanoparticle is shown in the Fig 4.4 X-ray diffraction. The peak at the 41 and 61 ascribing to MgO were found in MgO/carbon. And the peak at 29 describes Carbon.

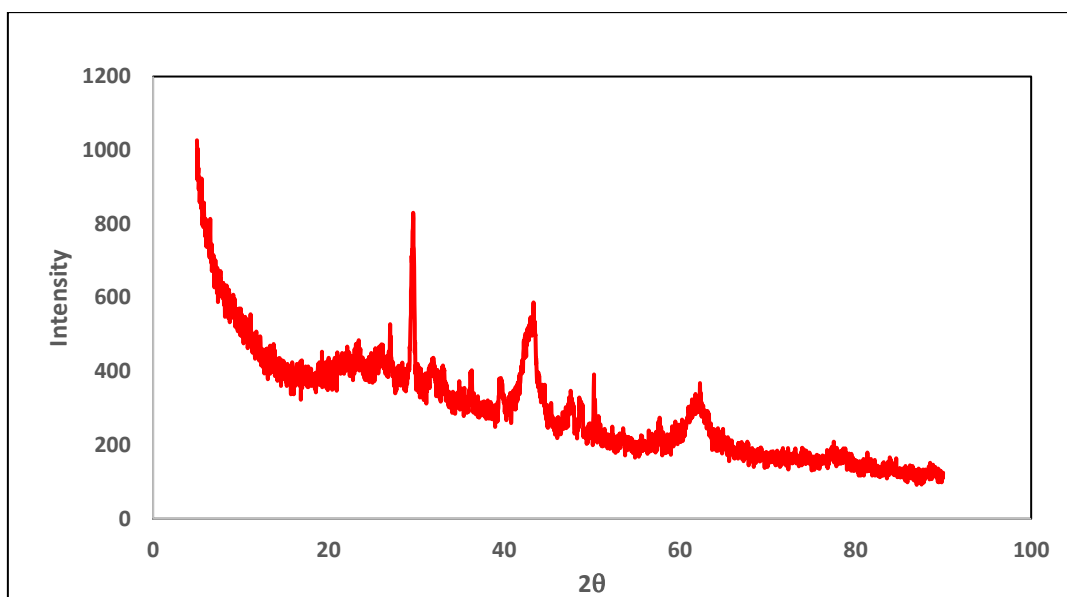


Fig 5.4 XRD pattern for MgO/C nanocomposite

5.2 ADSORPTION ISOTHERM:

Freundlich isotherm is used to examine the absorption of ciprofloxacin on the magnesium oxide doped activated carbon. The Freundlich isotherm is generally represented by,

$$q_e = K_f C_e^{1/n}$$

$$q_e = \frac{(C_o - C_e)}{M} = K_f C_e^{1/n}$$

Where q_e is the adsorptive capacity (mg g^{-1}),

C_o and C_e are the initial and equilibrium constant (mg L^{-1}) respectively,

k_f and n are Freundlich constant and it's related to adsorption capacity respectively

and M is the mass of adsorbent (g), the Freundlich isotherm is represented as

$$\ln q_e = \ln k_f + 1/n \ln C_e$$

The graph plotted between the $\ln q_e$ versus $\ln C_e$ for the adsorption of ciprofloxacin on the Magnesium oxide doped in active carbon is shown in figure 5.4. k_f and n

are determined from the slope and intercept of the liner plot. The Freundlich adsorption isotherm parameter for adsorption of ciprofloxacin from the aqueous solution using the adsorbent is shown in the table 5.2. Here the Freundlich isotherm proved to be the better fit from the regression coefficient. The heterogeneity of Magnesium oxide doped activated carbon adsorbent surface.

Figure 5.5 shows the effect of time on stream for the removal of ciprofloxacin by using Magnesium oxide doped activated carbon sample.

S.No	C _i (mg/L)	C _e (mg/L)	1/C _e	Log C _e	Ln C _e	q _e (mg/g)	1/q _e	Ln q _e
1	10	2.4	0.4167	0.38021124	0.87546874	3.8	0.26315	1.335
2	20	5.1	0.1961	0.70757018	1.62924054	7.45	0.13422	2.0082
3	30	14.6	0.0685	1.16435286	2.68102153	7.7	0.12987	2.0412
4	40	18.2	0.0549	1.26007139	2.90142159	10.9	0.09174	2.3888
5	50	24.5	0.0408	1.38916608	3.19867312	12.75	0.07843	2.5455

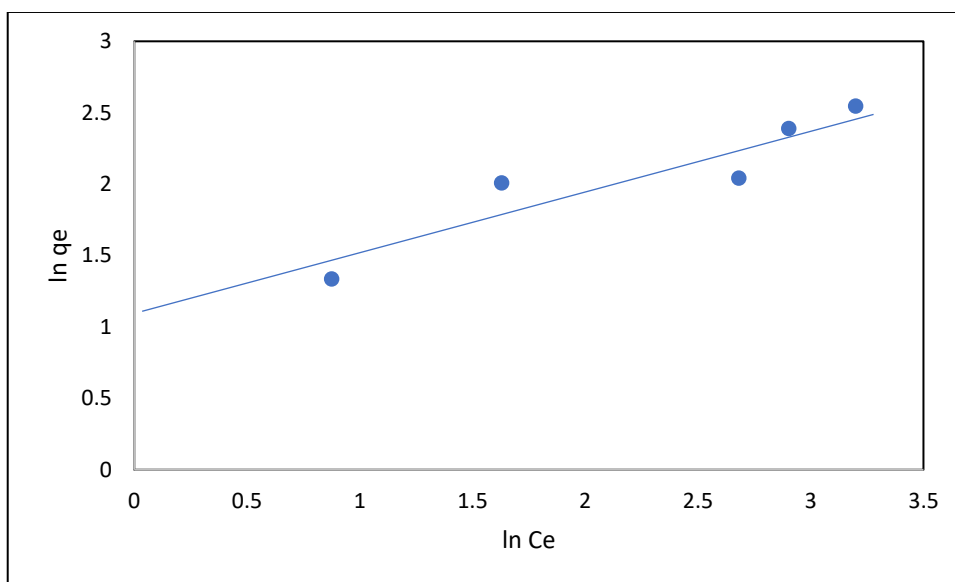


Fig 5.5 Adsorptive isotherm

5.3 EFFECT OF ADSORPTION PARAMETERS

Effect of Ciprofloxacin on drug concentration [10-50ppm]

About 0.05 gm from the concentration is added to (10, 20, 30, 40 and 50 ppm) from drug is added to 100ml conical flask and kept in the utilizing water at 30°C with constant stirring for 2hr.

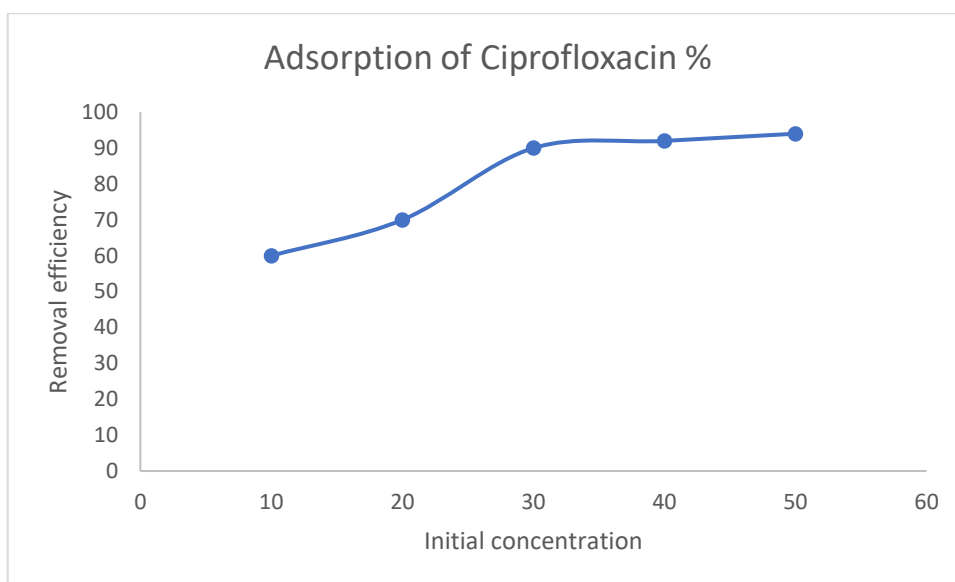


Fig 5.6 effect of ciprofloxacin on drug concentration

Effect of Temperature on removal efficiency [10-50°C]

The adsorption of ciprofloxacin is increased with an increase in temperature. As we know that while increasing the temperature, the molecular movement also increases and thus the collision frequency between the adsorbent and adsorbate. Thus the adsorption process can be enhanced and moreover when temperature is increased the contribution of activation energy of reaction also increased and decreases the chemical adsorption capacity while the physical adsorption capacity increases

Here we are studying the effect of Temperature solution by carrying various temperature solutions (10, 20, 30, 40 and 50) °C adsorbent amount 0.05 gm of catalyst and Ciprofloxacin drug concentration 30ppm, 100ml utilizing water bath at 30°C. the experiment was carried at the pH of 6.5 with the time of 2h at a constant speed of 120 rpm.

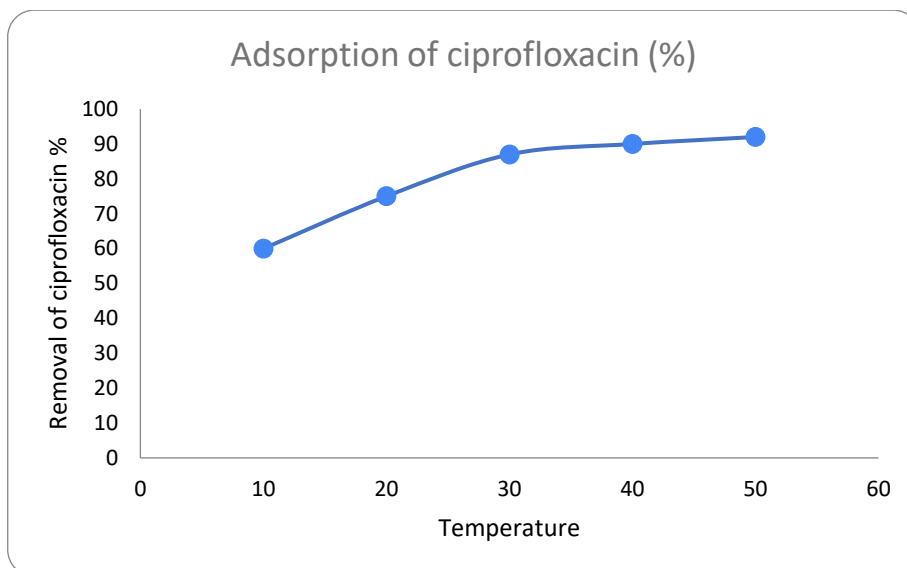


Fig 5.7 Effect of temperature on removal efficiency

Effect of weight of Catalyst amount [0.001-0.1]

The effect of adsorbent amount was studied through stirring of several weight (0.01, 0.03, 0.05, 0.075, 0.1) gm, with the drug concentration of 30ppm in 100ml utilizing water bath. The experiment was showed at pH 6.5. contact time at 2hr a constant speed at 120 rpm.

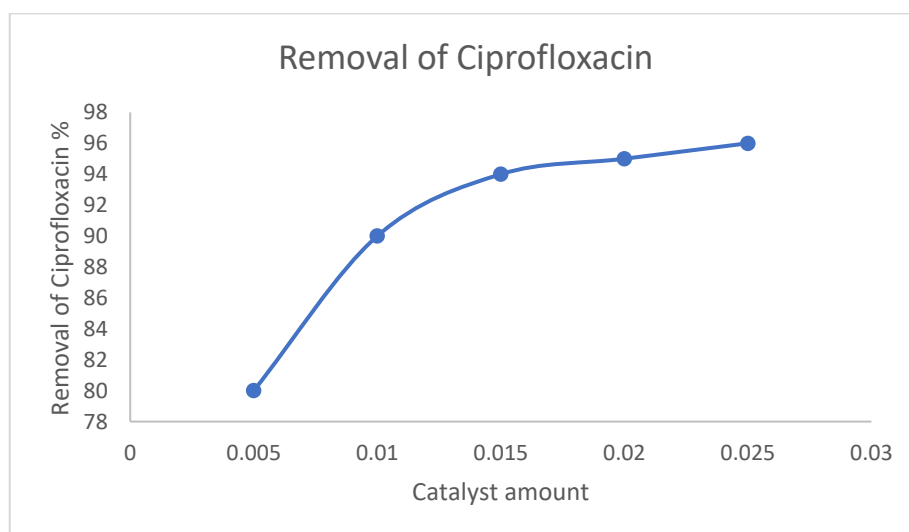


Fig 5.8 Effect of weight of catalyst amount

Effect of Time on removal efficiency

We showed that the ciprofloxacin adsorption is increased continuously with the increase certain period of time until it reaches the equilibrium and it decreases, we

observed that in earlier stages of the adsorption, the removal of ciprofloxacin is rapid. Which is may be due to the high number of active sites and due to the highest adsorption capacity if the nanotubes at the beginning of the adsorption. We observed that the ciprofloxacin in the aqueous solution increases sharply for the first 30 mins and then decreases.

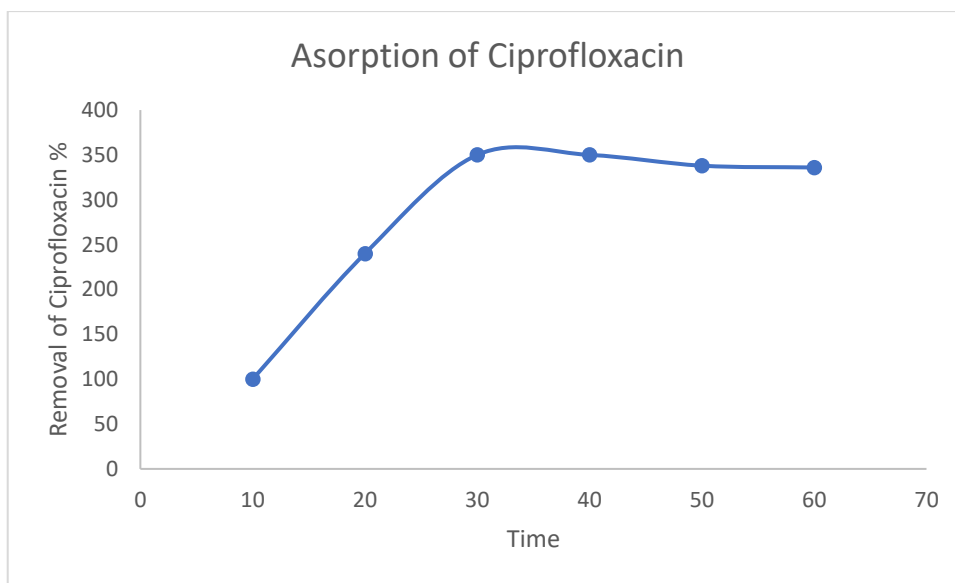


Fig 5.9 effect of time on removal efficiency

Effect of pH on adsorption of ciprofloxacin

One of the parameters that can affect the adsorption process is the pH of a solution because of the pH of the solution of the surface chemistry of the adsorbent, as well as the charges of adsorbent lowers, so that it can influence the chemical interaction between the adsorbent and adsorbate. The result shows that when the pH value increases from 2-6 and reaches the maximum pH of 6 the removal efficiency of ciprofloxacin also increases. However, by increasing the pH from 7 to 12, the ciprofloxacin adsorption decreases, these may due to the oxygen containing groups were deionized. So that it leads to the adsorption of H₂O molecules to the surface of adsorbent and results in decrease of removal efficiency.

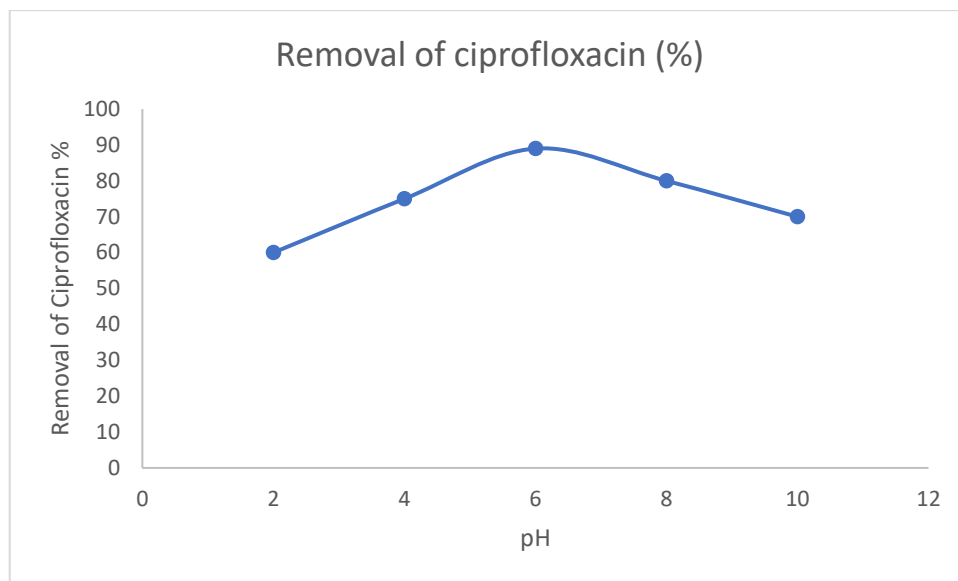


Fig 5.10 Effect of pH on adsorption of ciprofloxacin

CHAPTER 6

RESULT AND CONCLUSION

In summary, MgO/C Nano composite was synthesized using magnesium nitrate, dextrose and sodium hydroxide through Sol-Gel method at relatively low temperature (500°C) with carbon as support. XRD pattern revealed that MgO/C has high crystalline nature. Metal-Oxygen stretching was confirmed by the functional group analysis.

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