

**SYNTHESIS, CHARACTERIZATION OF MAGNESIUM OXIDE/CARBON
NANOCOMPOSITE AND IT'S ADSORPTIVE EFFICIENCY FOR THE
REMOVAL OF IBUPROFEN FROM AQUEOUS SOLUTION**

**Submitted in partial fulfilment of the requirements for the award
of Master of Science in CHEMISTRY**

By

PRAVEEN RAJ N

(REGISTER NO : 40910008)



DEPARTMENT OF CHEMISTRY

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with Grade "A" by NAAC

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119



SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)



Accredited with "A" grade by NAAC

Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai – 600 119

www.sathyabama.ac.in

DEPARTMENT OF CHEMISTRY

BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **Mr. PRAVEEN RAJ N (40910008)** who carried out the project entitled **“SYNTHESIS, CHARACTERIZATION OF MAGNESIUM OXIDE/CARBON NANOCOMPOSITE AND IT'S ADSORPTIVE EFFICIENCY FOR REMOVAL OF IBUPROFEN FROM THE AQUEOUS SOLUTION”** under our supervision from November 2021 to May 2022.

Internal Guide

Dr. T. KRITHIGA, M.Sc., Ph.D.,
Associate Professor
Department of Chemistry

Head of the Department

Dr. J. KARTHIKEYAN, M.Sc., Ph.D.,
Associate Professor
Department of Chemistry

Submitted for Viva voce Examination held on _____

Internal examiner

External Examiner

DECLARATION

I **N. PRAVEEN RAJ (40910008)**, hereby declare that the Project Report entitled **“SYNTHESIS, CHARACTERIZATION OF MAGNESIUM OXIDE/CARBON NANOCOMPOSITE AND IT’S ADSORPTIVE EFFICIENCY FOR REMOVAL OF IBUPROFEN FROM THE AQUEOUS SOLUTION”** 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**.

DATE:
PLACE:

N. PRAVEENRAJ
SIGNATURE OF THE CANDIDATE

ACKNOWLEDGEMENT

I am pleased to acknowledge my sincere thanks to Board of Management of **SATHYABAMA** for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I convey my thanks to **Dr. J KARTHIKEYAN, Head of the Department, Dept. of Chemistry** for providing me necessary support and details at the right time during the progressive reviews.

I would like to express my sincere and deep sense of gratitude to my Project Guide's **Dr. T. Krithiga** for their valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

I wish to express my thanks to all Teaching and Non-teaching staff members of the Department of Chemistry who were helpful in many ways for the completion of the project.

N. PRAVEENRAJ

ABSTRACT

IBP is a non-steroidal anti-inflammatory drug released into the environment through hospital and medical effluents, pharmaceutical waste water and veterinary use. The objective of this project was to study the adsorption experiments were conducted for the removal of IBP using the Nano-composite and It is experimented by preparing different ppm by dissolving 1000mg in 1L of solution of volumetric flask. From the prepared solution, and made up to different concentration. The concentration of IBP after treatment was quantified using a UV-vis spectroscopy. The experiment was showed at PH of 4. The functional groups present in the product and its structure was confirmed by the spectral studies.

TABLE OF CONTENTS

Chapter No	Title	Pages No
	ABSTRACT	i
	LIST OF ABBREVIATIONS	v
	LIST OF FIGURES	vi
1	INTRODUCTION	
1.1	CATALYST	1
1.1.1	TYPES OF CATALYST	1
1.1.2	HOMOGENEOUS CATALYST	1
1.1.3	HETEROGENEOUS CATALYST	1
1.1.4	HETEROGENIZED HOMOGENEOUS CATALYST	2
1.1.5	BIO CATALYST	2
1.2	NANOPARTICLES	3
1.2.1	TYPES OF NANOPARTICLES	3
1.2.2	CARBON BASED NANOPARTICLES	4
1.2.3	CERAMIC NANOPARTICLES	4
1.2.4	SEMICONDUCTOR NANOPARTICLES	4
1.2.5	POLYMERIC NANOPARTICLES	5
1.2.6	LIPID BASED NANOPARTICLES	5
1.2.7	METAL OXIDE NANOPARTICLES	6
1.2.8	MAGNESIUM OXIDE NANOPARTICLES	6
1.3	CARBON AS CATALYST SUPPORT	6
1.4	NANOCOMPOSITES	6
1.4.1	POLYMER NANOCOMPOSITE	7
1.4.2	NONPOLYMER NANOCOMPOSITE	8
1.4.3	METAL BASED NANOCOMPOSITE	8
1.4.4	MAGNESIUM OXIDE/CARBON NANOCOMPOSITE	8
1.5	PHARMACEUTICAL WASTE	9

1.5.1 PHARMACEUTICAL WASTE DISPOSAL	9
1.5.2 REMOVAL OF PHARMACEUTICAL COMPOUNDMUNICIPAL WASTE WATER TREATMENT PLANTS	9
1.6 MAGNESIUM OXIDE	10
2 LITERATURE SURVEY	11
3 AIM & SCOPE	
3.1 AIM	13
3.2 SCOPE	13
4 MATERIALS AND METHODS	
4.1 MATERIALS	14
4.2 CHARACTERIZATION TECHNIQUES	
4.2.1 FTIR	14
4.2.2 NMR	15
4.2.3 XRD	17
4.2.4 SEM	18
4.2.5 BET	20
4.3 METHADODOLOGY	
4.3.1 SYNTHESIS OF MgO NANOPARTICLE	21
4.3.2 SYNTHESIS OF MgO/C NANOCOMPOSITE	22
4.3.3 CHARACTERIZING OF THE NANO MgO/C COMPOSITE	22
4.3.4 PREPARATION SOLUTION IBUPROFEN DRUGS	23
4.4 EFFECT OF ADSORPTION PARAMETERS	
4.4.1 EFFECT OF IBUPROFEN DRUG CONCENTRATION	23
5 RESULTS & DISCUSSION	
5.1 CHARACTERIZATION OF MAGNESIUM OXIDE NANOPARTICLE	24
5.1.1 FTIR Spectrum Magnesium Oxide nanoparticles	24
5.1.2 SEM and EDAX image of Magnesium oxide nanoparticle	25

5.1.3 XRD pattern of magnesium oxide nanoparticle	27
5.2 ADSORPTION ISOTHERM	27
5.3 EFFECT OF ADSORPTION PARAMETERS	
5.3.1 Effect of Ibuprofen drug concentration	29
5.3.2 Effect of temperature on removal efficiency	29
5.3.3 Effect of weight of catalyst amount	30
5.3.4 Effect of time on removal efficiency	31
5.3.5 Effect of pH on adsorption of ibuprofen	31
6 CONCLUSIONS	33
REFERENCE	34

LIST OF ABBREVIATION

FTIR	-	Fourier Transform Infrared Spectroscopy
SEM	-	Scanning Electron Microscopy
BET	-	Brunauer-Emmett-Teller
UV	-	Ultraviolet Visible Spectroscopy
XRD	-	X-Ray Diffraction Spectroscopy
NMR	-	Nuclear Magnetic Resonance Spectroscopy

LIST OF FIGURES

Figure No	Title	Page No
1.6	Structure of Magnesium Oxide	10
4.3.1	Fourier Transform Infrared Spectrometer	15
4.3.2	Nuclear Magnetic Resonance Spectrometer	16
4.3.3	X-ray diffractometer	17
4.3.4	Scanning Electron Microscopy	19
4.3.5	Brunauer-Emmett-Teller	21
5.1.1	FTIR Spectrum of Magnesium Oxide nanoparticles	24
5.1.2	SEM image of Magnesium Oxide nanoparticles	25
5.1.3	EDAX image of Magnesium Oxide nanoparticles	26
5.1.4	XRD pattern of magnesium oxide nanoparticle	27
5.2	Adsorption isotherm	28
5.3.1	Effect of Ibuprofen on drug concentration	29
5.3.2	Effect of temperature on removal efficiency	30
5.3.3	Effect of weight of catalyst amount	30
5.3.4	Effect of time on removal efficiency	31
5.3.5	Effect of pH on adsorption of Ibuprofen	32

CHAPTER 1

INTRODUCTION

1.1 CATALYST

Any substance that increases the rate of a reaction without itself being consumed. Enzymes are naturally occurring catalysts responsible for many essential biochemical reactions. [Masel, et al, 2001]

1.1.1 TYPES OF CATALYST

Primarily categorized into four types. They are,

- Homogeneous,
- Heterogeneous (solid),
- Heterogenized homogeneous catalyst and
- Biocatalysts.

1.1.2 HOMOGENEOUS CATALYST

In homogeneous catalysis, reaction mixture and catalyst both are available in a same phase. Both catalyst and reactants show high homogeneity which brings about high interaction between them that prompts high reactivity and selectivity of the reaction under mild reaction conditions. A few instances of homogeneous catalysts are bronsted and lewis acids, transition metals, organometallic complexes, organocatalyst. A few notable chemical processes that happen through homogeneous catalysis are carbonylation, oxidation, hydrocyanation, metathesis, and hydrogenation. [Berzillius, et al, 2017]

1.1.3 HETEROGENEOUS CATALYST

It will be catalysis where the phase of catalysts varies from that of the reactants or products. The process appears differently in relation to homogeneous catalysis where the reactants, items and catalyst exist in a similar phase. Stage distinguishes between not only solid, liquid, and gas components, yet in addition immiscible mixtures (for example oil and water), or anywhere an interface is available.

Heterogeneous catalysis ordinarily includes solid phase catalysts and gas phase reactants. For this situation, there is a cycle of molecular adsorption, reaction, and desorption happening at the catalyst surface. Thermodynamics, mass exchange, and hotness move impact the rate (kinetics) of reaction. [Berzillius, et al, 2017]

1.1.4 HETEROGENIZED HOMOGENEOUS CATALYST

Heterogeneous catalysts in contrast to their homogeneous partners are substantially harder to for all intents and purposes create. One analysis is their intricacy, which blocks their analysis at a molecular level and advancement through structure-reactivity connections. Moreover, customary heterogeneous catalysts (metal oxides or upheld metals) show less selectivity and reactivity. To overcome these issues, the homogeneous catalyst is joined onto the solid supports to set up their heterogenic analogs. As of now, the strong upheld homogeneous catalysts are generally perceived and very much took advantage of in intellectual and industrial research. The point of this approach is to cover the positive features of both homogeneous (selectivity and reactivity) and heterogeneous catalyst (reproducibility) and this can be accomplished through the immobilization of catalyst like metal complexes, organometallic compounds on the strong surface either through physisorption or chemisorption. Covalent uniting of reactant dynamic species on strong surfaces is viewed as the most preferred approach for planning heterogenized homogeneous catalyst. [Ostwald, et al, 2017]

1.1.5 BIOCATALYST

It is used to catalyze explicit chemical reaction outside the living cells is called biocatalyst. Compounds are acquired from creature tissues, plants and microorganisms (yeast, microbes or organisms). High selectivity, high productivity, eco-agreeableness and gentle response conditions are the main impetuses for their huge scope use and making biocatalysts an option in contrast to ordinary industrial catalyst. Critical advancement in the field of protein designing and sub-atomic development has changed the universe of bio catalysis for the modern scale syntheses of fine chemicals, dynamic fixings (APIs) biofuels (for example lipase for the development of biodiesel from vegetable oil), dairy industry (for example protease, lipase for lactose expulsion), baking

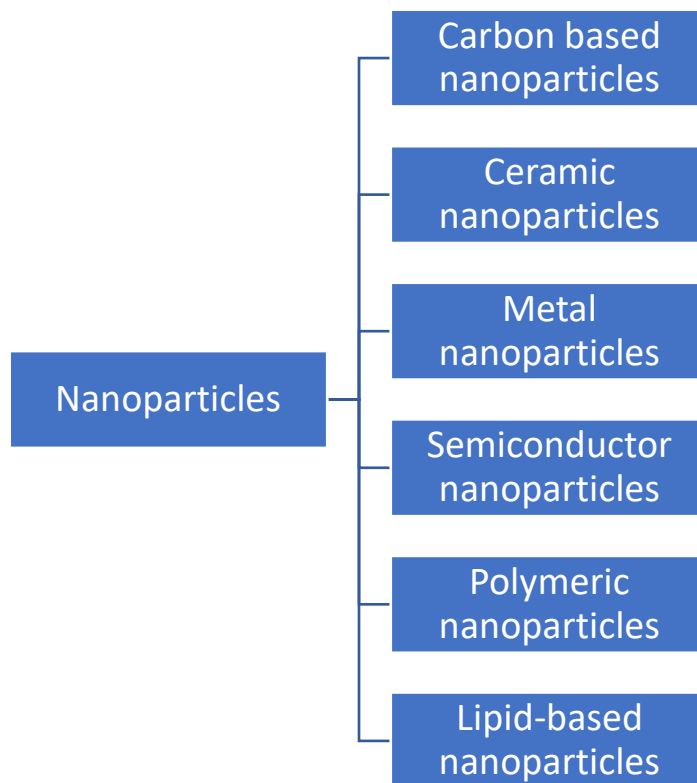
industry (for example amylase for bread delicate quality and volume, glucose oxidase for batter fortifying), cleanser producing (for example proteinase, lipase, amylase used to eliminate stains of proteins, fats, starch, individually). [Ostwald, et al, 2017]

1.2 NANOPARTICLES

A nanoparticle is a little molecule that reaches between 1 to 100nm in size. Imperceptible by the natural eye, nanoparticles can display altogether unique physical and chemical properties to their bigger material partners. [Schue, F.O, et al, 2012]

1.2.1 TYPES OF NANOPARTICLES

Nanoparticles can be classified into different types according to the size, morphology, physical and chemical properties. [Khan, et al, 2017]



1.2.2 CARBON BASED NANOPARTICLES

They are incorporate two primary materials,

- carbon nanotubes (CNTs) and
- fullerenes.

CNTs are only graphene sheets moved into a cylinder. These materials are essentially utilized for the primary support as they are multiple times more grounded than steel. [Khan, et al, 2017]

1.2.3 CERAMIC NANOPARTICLES

inorganic solids comprised of oxides, carbides, carbonates and phosphates. These nanoparticles have high hotness opposition and chemical inactivity. They have applications in photocatalysis, photodegradation of dyes, drug conveyance, and imaging.

By controlling a portion of the characteristics of ceramic nanoparticles like size, surface region, porosity, surface to volume ratio, and so forth, they proceed as a good drug delivery agent. These nanoparticles have been utilized actually as a medication conveyance framework for various illnesses like bacterial infections, glaucoma, cancer, and so on. [Khalid Saeed, et al, 2017]

1.2.4 SEMICONDUCTOR NANOPARTICLES

Semiconductor nanoparticles have properties like those of metals and non-metals. They are found in the periodic table in group II-VI, III-V or IV-VI. These particles have wide +bandgaps, which on tuning shows various properties. They are utilized in photocatalysis, gadgets, photograph optics and water dividing applications.

A few instances of semiconductor nanoparticles are GaN, GaP, InP, InAs from group III-V, ZnO, ZnS, CdS, CdSe, CdTe are II-VI semiconductors and silicon and germanium are from group IV. [Khalid Saeed, et al, 2017]

1.2.5 POLYMERIC NANOPARTICLES

Polymeric nanoparticles are organic based nanoparticles. Contingent on the strategy for preparation, these have structures formed like nano capsular or nanospheres. A nanosphere molecule has a matrix like structure though the nano capsular molecule has mid shell morphology. In the former, the active compounds and the polymer are consistently scattered though in the last the active compounds are confined and surrounded by a polymer shell.

A portion of the benefits of polymeric nanoparticles are controlled delivery, protection of drug molecules, capacity to join treatment and imaging, explicit focusing on and some more. They have applications in drug delivery and diagnostics. The drug deliveries with polymeric nanoparticles are profoundly biodegradable and biocompatible.

Polymeric nanoparticles are particles within the size range from 1 to 1000 nm and can be loaded with active compounds entrapped within or surface-adsorbed onto the polymeric core. Profoundly. The expression "nanoparticle" represents both nano capsules and nanospheres, which are recognized by the morphological design. [Idrees Khan, et al, 2017]

1.2.6 LIPID BASED NANOPARTICLES

Lipid nanoparticles are by and large round in shape with a diameter ranging from 10 to 100nm. It comprises of a solid core made of lipid and a matrix containing soluble lipophilic particles. The external core of these nanoparticles is stabilized by surfactants and emulsifiers. These nanoparticles have application in the biomedical field as a medication transporter and conveyance and RNA discharge in cancer therapy.. These particles are in the size scope of 50-1000 nm. [Idrees Khan, et al, 2017]

1.2.7 METAL OXIDE NANOPARTICLES

Metal oxide nanoparticles are made of absolutely metal precursors. These nanoparticles assume a critical part in numerous areas of physics, chemistry and material sciences. Thermal elements are equipped for framing a wide scope of oxide compounds. Metal oxide nanoparticles are mostly gotten by hydrolysis of metal salts at room temperature or temperatures not more prominent than 100°C. [Jolivet, et al, 2010]

1.2.8 MAGNESIUM OXIDE NANOPARTICLES

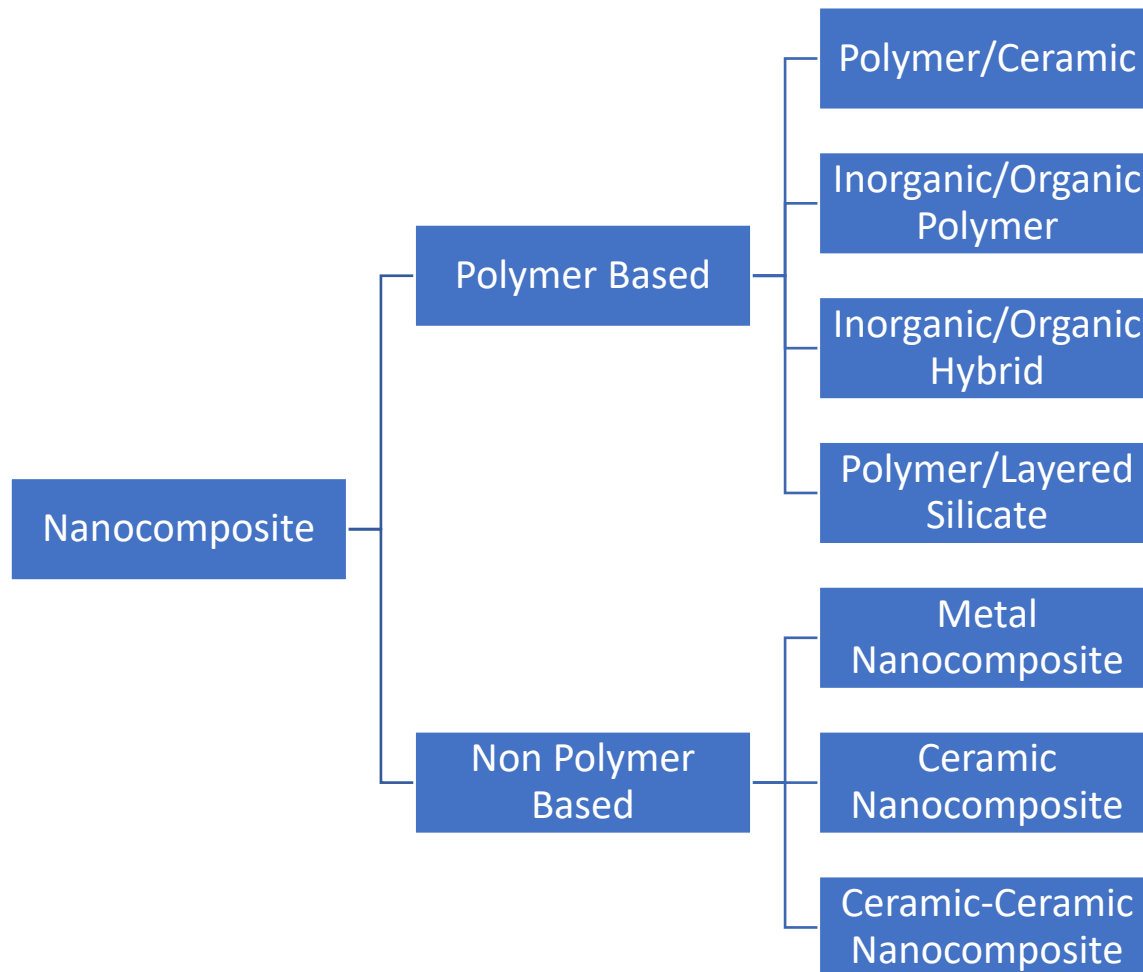
Magnesium oxide (MgO) nanoparticles have attracted interest for use in biomedical applications because of their antimicrobial properties. They are accounted for to have antimicrobial action against both Gram-positive and Gram-negative bacteria, spores, and infections, and can be prepared from economic precursors. [K.Krishnamoorthy, et al, 2012]

1.3 CARBON AS CATALYST SUPPORT

Carbon plays a dual role as a catalyst or a catalyst support for chemical and enzymatic biomass transformation reactions due to its large specific surface area, high porosity, excellent electron conductivity, and relative chemical inertness. Beneficially, carbon materials can be ready from residual biomass, an attractive property for decreasing the so-called "carbon-footprint" of a biomass change process. [Narayanamoorthy]

1.4 NANOCOMPOSITE

A nanocomposite is a multiphase material where, as opposed to micro composites, one of the stages has one, two, or three elements of under 100nm, or the composite stages have nanoscale distances between them. It is found in nature, for example in the structure of the abalone shell and bone. [O Kamigaito, et al, 1991]



1.4.1 POLYMER NANOCOMPOSITE

polymer nanocomposites consist of a polymer or copolymer having nanoparticles or nanofillers scattered in the polymer matrix. These might be of various shape (e.g., platelets, fibers, spheroids), however something like one dimension should be in the range of 1-50 nm. These PNC's have a place with the classification of multi-phase systems (MPS, viz. blends, composites, and foams) that consume almost 95% of plastics creation. These system require controlled mixing/compounding, adjustment of

the accomplished dispersion, orientation of the scattered phase, and the intensifying techniques for all MPS, including PNC, are comparable. Then again, polymer can be infiltrated into 1D, 2D, 3D preform making high content polymer nanocomposites. [Walsh, et al, 2019]

1.4.2 NONPOLYMER NANOCOMPOSITE

The nanocomposites in which the arrangements contain no polymers or polymer-determined materials are called non-polymer-based nanocomposites. Non-polymer-based nanocomposites are otherwise called inorganic nanocomposites.

1.4.3 METAL BASED NANOCOMPOSITE

Bimetallic nanoparticles being researched in detail in the form of combination or core shell structures because of their better catalytic properties and advancement in optical properties connected with individual and separate metals

They can be described by:

- Lower melting points,
- Increased strength and hardness,
- Improved magnetic properties. [Fam DWH, et al, 2011]

1.4.4 MAGNESIUM OXIDE/CARBON NANOCOMPOSITE

MgO adorned carbon nanofiber (MgO @CNFs) nanocomposite was prepared by electrospinning and carbonization technique. Then the nanocomposite was casted on carbon ionic fluid anode (CILE) with the accompanying immobilization of hemoglobin (Hb) by Nafion polymer film. [Hamza, et al, 2020]

1.5 PHARMACEUTICAL WASTE

Drug squander" (otherwise known as PPCPs), which incorporates utilized and unused lapsed solution drugs, home-utilize individual consideration items, and over-the-counter prescriptions, have arisen since the advancement of standard clinical waste guidelines just like another significant public and ecological wellbeing concern. Critical exploration has been performed overall and information have been gathered in regards to the complex issues related with this waste stream. In the United States, the EPA has performed different investigations and exploration connecting with the existence and impacts of PPCPs in the environment. [Anne Woolridge, et al, 2011]

1.5.1 PHARMACEUTICAL WASTE DISPOSAL

Disposal refers to the long-term placement of waste or treated waste. It is almost always off-site - away from where the waste is generated - and usually involves burial underground. Putting waste in storage containers at the site of generation is not a form of disposal. It is not a permanent solution. We classify disposal as the permanent resting place for the waste. Pharmaceutically active compounds are created to have specific physicochemical and biological properties. While such characteristics are necessary to deliver the desired therapeutic effect, many pharmaceutical products have an impact on the aquatic environment. [Voulvoulis N, et al, 2005]

1.5.2 REMOVAL OF PHARMACEUTICAL COMPOUND MUNICIPAL WASTE WATER TREATMENT PLANTS

- Sewage
- Aquaculture
- Waste disposal

- Animal husbandry & horticulture
- Environmental fate pharmaceuticals in marine environments. [Vidal N, et al, 2002]

1.6 MAGNESIUM OXIDE

It is an inorganic salt of magnesium formed with ions of magnesium and oxygen. One of many forms of magnesium available for purchase in supplement form. It's added to dietary supplements as well as over-the-counter medications used to treat constipation, indigestion, and headaches. [Jillian Kubala, et al, 2021]

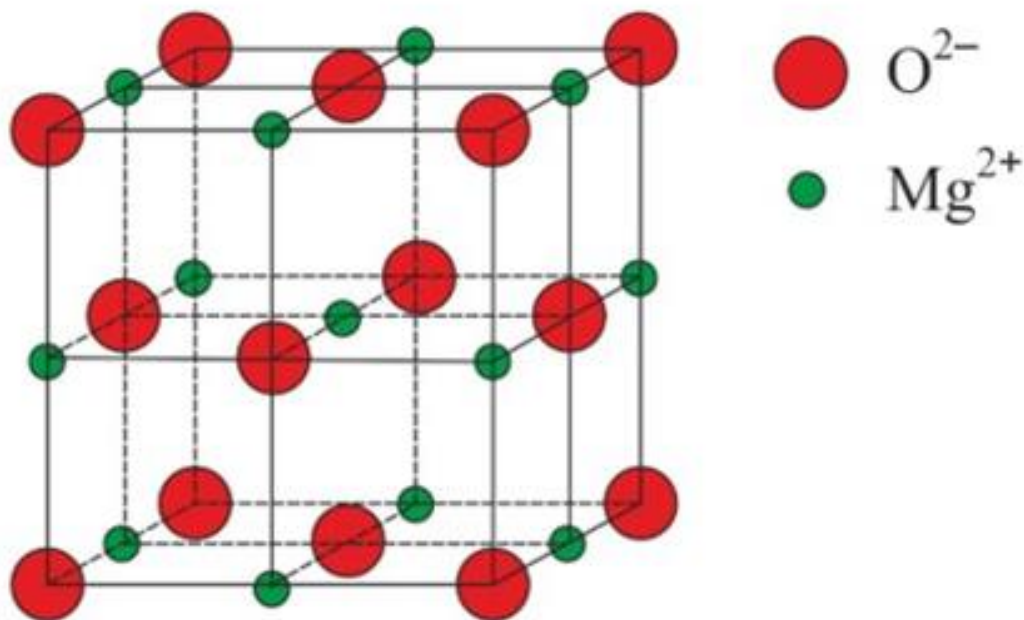


Fig 1.6 Structure of Magnesium Oxide

SOURCE: https://www.google.com/search?q=magnesium+oxide&sxsrf=ALiCzsaQ7YDpuLXLhMqde6z0OBYicI9CCw:1651232962106&source=lnms&tbm=isch&sa=X&ved=2ahUKEwjO-paRmrn3AhWHwzgGHfrADzYQ_AUoAXoECAIQAw&biw=1366&bih=657&dpr=1#imgrc=aCnAepAHham8_M&imgdii=WywPTAKr4ZW2XM

CHAPTER 2

LITERATURE SURVEY

R Prasanth, S Dinesh Kumar, A Jayalakshmi, G Singaravelu, K Govindaraju, V Ganesh Kumar has demonstrated new avenues to fight and prevent diseases using atomic-scale tailoring of materials. The present investigation describes the mangrove *Rhizophora lamarckii*'s property of synthesizing magnesium oxide nanoparticles. The newly synthesized magnesium oxide nanoparticle morphology is nanohexagonal and spherical. The particles range in dimensions between 20 and 50 nm and are crystalline in nature. The functional groups of the mangrove, amine, and alkane are found to act as reductants and stabilizers.

Saeid Taghavi Fardood, Ali Ramazani, Sang Woo Joo has demonstrated that they can synthesis MgO nanoparticles using Arabic gum as a biotemplate and magnesium nitrate as the magnesium source by the sol-gel method. This method has many advantages such as nontoxic, economic viability, ease to scale up, less time consuming and environmental friendly approach for the synthesis of MgO nanoparticles without using any organic chemicals. Nanoparticles were characterized by FTIR, XRD. The average crystallite size of MgO nanoparticles was calculated using Scherrer formula and Williamson Hall equation. The XRD analysis revealed the formation of cubic phase MgO with average particle size of 14 (Scherrer formula) and 5.3 (Williamson Hall equation) nm.

Mohammad Moslem Imani, Mohsen Safaei demonstrated the synthesis of Magnesium oxide nanoparticles by coprecipitation method, and their antibacterial activity was evaluated using colony-forming unit (CFU) and disk diffusion. Morphology, crystalline structure, and size of synthesized nanoparticles were investigated using FTIR, XRD and

SEM. The optimum conditions (0.2 M magnesium nitrate, 2 M NaOH, and 90 min stirring time) for the synthesis of magnesium oxide nanoparticles with the greatest antibacterial activity were determined using the Taguchi method.

Gaurav Sharma, Rajgovind Soni, Nakuleshwar Dut Jasuja has demonstrated that phytoassisted synthesis of magnesium oxide nanoparticles with an aqueous extract of *Swertia chirayaita*. The stable magnesium oxide nanoparticles (MgONPs) formed by this method were spherical particles that were <20 nm in size. The method is cost-effective and eco-friendly. MgONPs from phytoassisted synthesis were characterized by UV-visible (UV-vis) spectroscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD). The UV-vis absorbance spectrum of the synthesized MgONPs displayed a surface plasmon resonance at 510 nm.

CHAPTER 3

AIM & 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

- Brought an Ibuprofen drug from apollo pharmacy.
- Preparation of Ibuprofen solution.
- Synthesizing MgO/C nanoparticle using sol gel method.
- Confirmation of product formed by characterizing by FTIR, NMR spectroscopy.
- Removal of ibuprofen from aqueous solution.
- Characterization of synthesized MgO/c nanoparticle by FTIR spectroscopy, XRD, SEM and EDX.

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS

Materials used in this reaction are Magnesium nitrate, Sodium hydroxide, dextrose and Ibuprofen which were purchased from our college lab and apollo pharmacy and used as such without further purification.

4.2 CHARACTERIZATION TECHNIQUES

4.2.1 FTIR (FOURIER TRANSFORM INFRARED SPECTROSCOPY)

FTIR is a strategy used to get an infrared spectrum of absorption or emission of a solid, fluid or gas. An FTIR spectrometer all the while gathers high-resolution spectral information over a wide spectral range. This presents a critical benefit over a dispersive spectrometer, which estimates intensity over a restricted range of wavelengths all at once.^[21] The term Fourier-change infrared spectroscopy starts from the way that a Fourier change is expected to change over the raw information into the genuine spectrum. FTIR spectroscopy is utilized to rapidly and definitively identify compounds such as compounded plastics, blends, fillers, paints, rubbers, coatings, resins, and adhesives. It very well may be applied across all phase of the item lifecycle including design, assembling, and failure analysis. Spectroscopy is the investigation of interaction of electromagnetic waves with matter. The surface functional groups of the samples were qualitatively analyzed by FTIR spectroscopy and kept in the range of 4000-600 cm^{-1} . For this reason, the samples were mixed with potassium bromide and made into pellets for analysis. [P. Mohamed Shameer, et al, 2019]



Fig 4.3.1 Fourier Transform Infrared Spectrometer

SOURCE: https://www.google.com/search?q=FTIR&tbm=isch&tbs=rimg:CYQ1C3qjJii-YcOwxUYQKhWWsqIMCqIIABAAOgQIARAA&hl=en&sa=X&ved=0CBsQullBahcKEwil_9v-nLb3AhUAAAAAHQAAAAAQBg&biw=1519&bih=688#imgrc=EpBKBu8apASyiM

4.2.2 NMR (NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY)

Nuclear magnetic resonance spectroscopy, most commonly known as NMR spectroscopy or magnetic resonance spectroscopy (MRS), is a spectroscopic technique to observe local magnetic fields around atomic nuclei. The sample is placed in a magnetic field and the NMR signal is produced by excitation of the nuclei sample with radio waves into nuclear magnetic resonance, which is detected with sensitive radio receivers. The intramolecular magnetic field around an atom in a molecule changes the resonance frequency, thus giving access to details of the electronic structure of a molecule and its individual functional groups. As the fields are unique or highly characteristic to individual compounds, in modern organic chemistry practice, NMR spectroscopy is the definitive method to identify monomolecular organic compounds.

The principle of NMR usually involves three sequential steps:

1. The alignment (polarization) of the magnetic nuclear spins in an applied, constant magnetic field B_0 .

2. The perturbation of this alignment of the nuclear spins by a weak oscillating magnetic field, usually referred to as a radio-frequency (RF) pulse.
3. Detection and analysis of the electromagnetic waves emitted by the nuclei of the sample as a result of this perturbation. [Hoult D, et al, 1997]



Fig 4.3.2 Nuclear Magnetic Resonance Spectrometer

SOURCE: https://www.google.com/search?q=NMR&tbm=isch&ved=2ahUKEwiHu7rGnbb3AhVlidgFHfteAZoQ2-cCegQIABAA&og=NMR&gs_lcp=CgNpbWcQAziHCCMQ7wMQJzIECAAQQzIFCAAQgAQyBQgAEIAEMgUIABCABDIFCAAQgAQyBQgAEIAEMgUIABCABDIFCAAQgAQyBQgAEIAEOgoIABCxAXCDARBDoggIABCABBCxAzoICAAQsQMgQgWE6BAgAEAM6CwgAEIAEELEDEIMBUKsMWLYRYNYkaABwAHgAgAHbBogB5BOSAQkzLTEuMC4yLjGYAQcQAQGgAQtnD3Mtd2l6LWltZ8ABAQ&sclient=img&ei=r0FqYsenOMiS4t4P-72F0Ak&bih=688&biw=1519&hl=en#imgsrc=1b4E4341IfNM&imgdii=k6sNHMp3NM9hDM

4.2.3 XRD (X-RAY DIFFRACTION)

X-ray diffraction, or XRD, is a technique for analysing the molecular or atomic structure of materials. It is non-horrendous, and works most actually with materials that are entirely, or part, crystalline. The technique is much of the time known as x-ray powder diffraction in light of the fact that the material being analysed typically is a finely ground down to a uniform state. Diffraction is when light bends slightly as it passes around the edge of an object or encounters an obstacle or aperture. How much it happens relies upon the relative size of a wavelength compared to the dimensions of the obstacle or gap it experiences. [T.Y.Inan, et al, 2017]

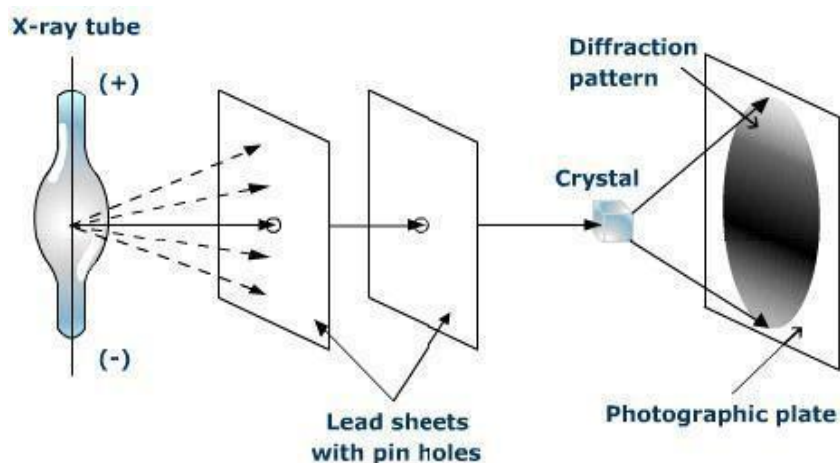


Fig 4.3.3 X-ray diffractometer

SOURCE: https://www.google.com/search?q=xrd+diffractometer&tbm=isch&ved=2ahUKEwibkla0n7b3AhWvG7cAHbkkCWwQ2-cCegQIABAA&oq=xrd+dif&gs_lcp=CgNpbWcQARgAMgQIABBDMgUIABCABDIFCAAQgAQyBQgAEIAEMgUIABCABDIFCAAQgAQyBQgAEIAEMgUIABCABDIGCAAQBRAeMgYIABAIEB46BwgjEO8DECdQ2AtYzB5qxEdoAHAAeACAACMDiAHABplBBzMuMS40LTGYAQCgAQGgAQtn3Mtd2l6LWltZ8ABAQ&sclient=img&ei=okNqYpu3C6-33LUPucmk4AY&bih=688&biw=1519&hl=en#imgsrc=32G6KV7y_7zFBM&imgdii=Qgo0hgYSEm5j8M

4.2.4 SEM (SCANNING ELECTRON MICROSCOPY)

A sort of electron microscope that instrument produces pictures of a sample by filtering the surface with a focused beam of electrons. The electrons cooperate with particles in the sample, producing different signals that contain data about the surface topography and composition of the example.

SEM utilizes a focused beam of high energy electrons to create an assortment of signals at the surface of solid specimens. The signals that get from electron-test interactions uncover data about the sample including outer morphology (surface), chemical composition, and crystalline structure and orientation of materials making up the sample. In many applications, information is gathered over a chose region of the outer surface of the sample, and a 2-dimensional image is produced that shows spatial variations in these properties. Areas ranging from around 1 cm to 5 microns in width can be imaged in a scanning mode utilizing regular SEM procedures (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is additionally fit for performing analyses of chosen point areas on the sample; this approach is particularly valuable in qualitatively or semi-quantitatively deciding chemical compositions (utilizing EDS), crystalline structure and crystal orientations (utilizing EBSD). The design and function of the SEM is basically the same as the EPMA and impressive cross-over in abilities exists between the two instruments. [Goldstein, et al, 2003]

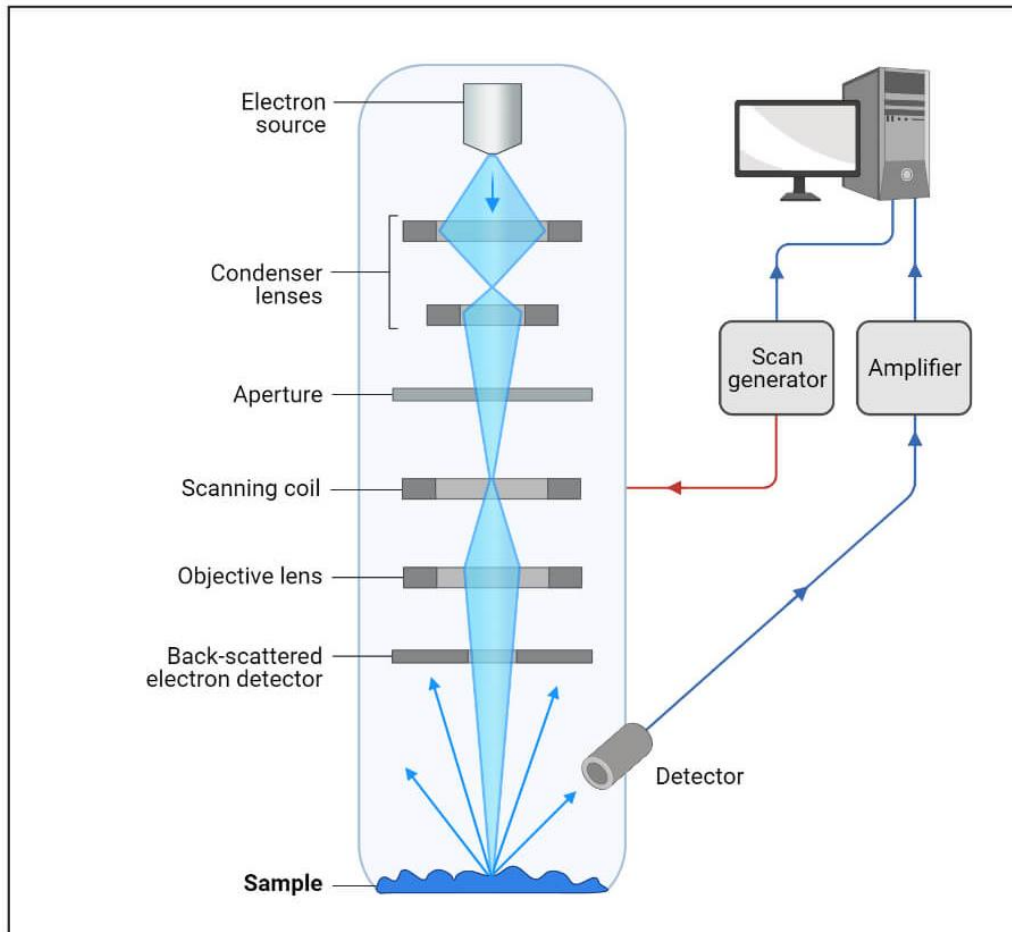


Fig 4.3.4 Scanning Electron Microscopy

SOURCE: https://www.google.com/search?q=sem+spectroscopy&tbm=isch&ved=2ahUKEwiooqyfoLb3AhVRk9gFHUSODLwQ2-cCegQIABAA&oq=SEM&gs_lcp=CgNpbWcQARgBMgcIlxDvAxAnMgcIlxDvAxAnMgQIABBDmQIABBDmGgIABCABBCxAzIECAAQQzIECAAQQzIECAAQQzIECAAQQzIECAAQQsQNOAFgAYPqOaABwAHgAqAFTiAFTkgEBMZgBAKoBC2d3cy13aXotaW1nwAEB&sclient=img&ei=g0RqYujUDNGm4t4PxJyy4As&bih=688&biw=1519&hl=en#imgrc=eXNEbyUcTD8UM

4.2.5 BET (Brunauer-Emmett-Teller)

This hypothesis intends to make sense of the physical adsorption of gas particles on a solid surface and fills in as the reason for a significant analysis strategy for the estimation of the particular surface area of materials. The perceptions are frequently alluded to as physical adsorption or physisorption. BET hypothesis applies to systems of multilayer adsorption that generally uses a testing gas (called the adsorbate) that don't respond chemically with the adsorptive (the material whereupon the gas appends to and the gas stage is known as the adsorptive) to evaluate explicit surface region. Nitrogen is the most generally utilized vaporous adsorbate for testing surface. Thus, standard BET analysis is most frequently led at the boiling temperature of N₂ (77 K). Other examining adsorbates are likewise used, though once in a while, permitting the estimation of surface region at various temperatures and estimation scales. These incorporate argon, carbon dioxide, and water. Explicit surface region is a scale-subordinate property, with no single genuine worth of specific surface region perceptible, and hence amounts of explicit still up in the air through BET hypothesis might rely upon the adsorbate particle used and its adsorption cross section. [Mahmoud Nasrollahzadeh, et al, 2019]



Fig 4.3.5 Brunauer-Emmett-Teller

SOURCE: https://www.google.com/search?q=bet+spectroscopy&tbm=isch&ved=2ahUKEwjR8OO5obb3AhW1jNgFHcZ2C_4Q2-cCegQIABAA&og=bet+spectroscopy&gs_lcp=CgNpbWcQAziHCCMQ7wMQJzoECAAQGD0GCAAQBxAeOgclABCxAxBDOgUIABCABDoECAAQQ1D7B1jBGmCQIWgAcAB4AIABkgaAdYckgENMS4wLjluMS4yLjluMZgBAKABAaoBC2d3cy13aXotaW1nwAEB&sclient=img&ei=x0VgYpHxBLWZ4t4Pxu2t8A8&bih=688&biw=1519&hl=en#imgsrc=f40xsuYPKtCD4M&imgdii=gmaYGb_ELPKqSM

4.3 METHODOLOGY

4.3.1 SYNTHESIS OF MgO NANOPARTICLE

- Molar of $Mgno_3$ is Prepared by dissolving 1.432g of $Mgno_3$ salt in 100ml of distilled water
- 0.1 Molar of NaOH is prepared by dissolving 0.300g of NaOH in 100ml of distilled water
- Take 40ml of the prepared NaOH solution in a beaker & place it in a hot plate magnetic stirrer. When the solution begins to boil, add 40ml of $Mgno_3$ solution drop by drop to the dextrose solution. (Magnetic Stirrer Temp: $30^{\circ}C$)
- A Gel like consistence is obtained, turn off the stirrer & transfer the contents to a crucible.
- Place the crucible in oven ($80^{\circ}C$) for the moister to evaporate (Approximately 3Hrs)

4.3.2 SYNTHESIS OF MgO/C NANOCOMPOSITES

- 18g of dextrose (glucose) is Dissolved in 100ml of distilled water in a standard flask.
- Molar of Mgno3 is prepared by dissolving 1.432g of Mgno3 salt in 100ml of distilled water
- Take 40ml of the prepared dextrose solution in a beaker & place it in a hot plate magnetic stirrer. When the solution begins to boil, add 40ml of Mgno3 solution drop by drop to the dextrose solution. (Magnetic Stirrer Temp: 30°C)
- A Gel like consistence is obtained, turn off the stirrer & 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 7mins, an amorphous powder is obtained.
- Then place the crucible at 600°C for calcination to occur.

4.3.3 CHARACTERIZING OF THE NANO MgO CARBON COMPOSITE

Nano MgO carbon composite was characterized by SEM, XRD, FTIR, BET and UV spectrometric techniques. Morphology and size distribution nature of nano MgO carbon composite, the phase distribution synthesized nano – MgO carbon composite was determined using D8 advanced powder X-ray diffraction instrument at potential of 30KV with a supply of 15mA current passing Cu-K α radiation at a wavelength of 1.54Å. The analyzed material is finally grounded. X-ray powder diffraction is analytical technique method primarily used for phase identification of a crystalline material and that can provide information about the cell dimension. The surface interdict functional groups of nano-MgO carbon composition were determined using FTIR by Bruker instrument in the range of 400-4000cm⁻¹ with KBr pellets.

4.3.4 PREPARATION SOLUTION IBUPROFEN DRUGS

Adsorption experiments were conducted for the removal of Ibuprofen using the nano-composite. It is experimented by 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 make up to 100ml, 2ml in 100ml, 3ml in 100ml, 4ml in 100ml, 5ml in 100ml standard flask).

4.4 EFFECT OF ADSORPTION PARAMETERS

4.4.1 EFFECT OF IBUPROFEN 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 4.

CHAPTER 5

RESULT AND DISCUSSION

5.1 CHARACTERIZATION OF MAGNESIUM OXIDE NANOPARTICLE

5.1.1 FTIR SPECTRUM MAGNESIUM OXIDE NANOPARTICLES

The FTIR spectrum of Magnesium oxide nanoparticles are shown in Fig.5.1.1 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.

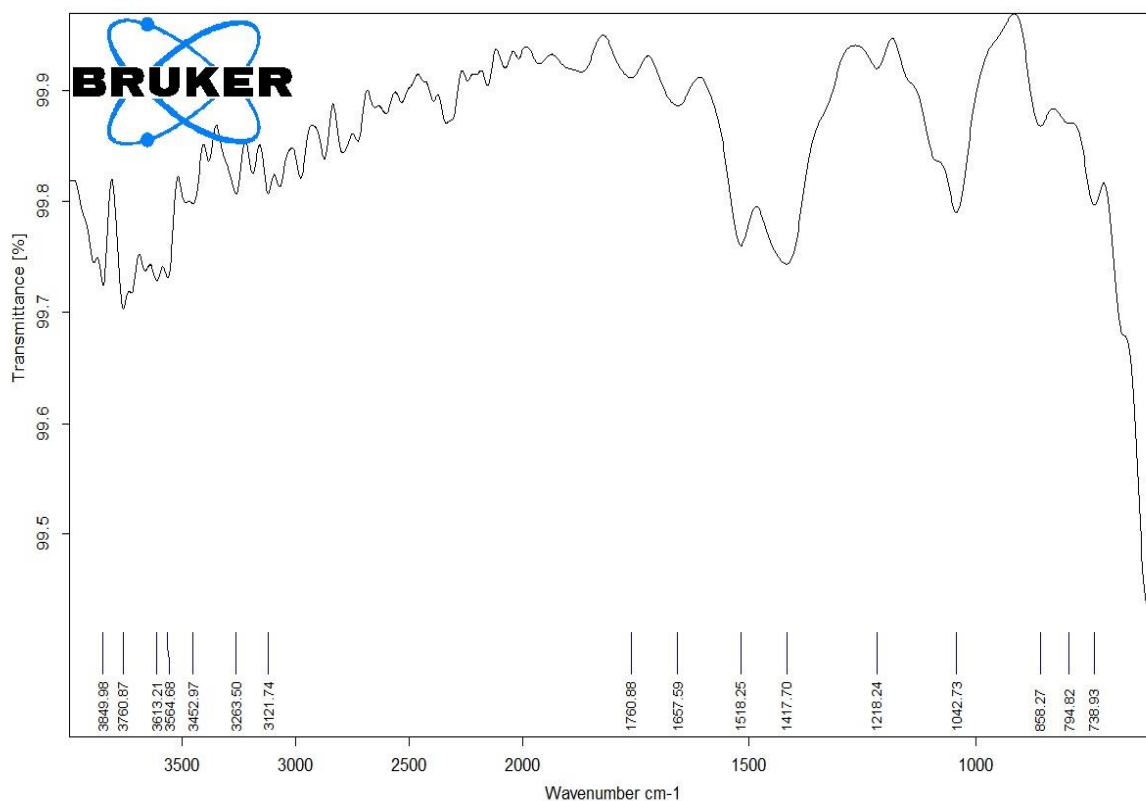


Fig 5.1.1 FTIR spectrum of Magnesium oxide nanoparticle

Vibrational frequency	Functional group identification
3849 cm^{-1}	O-H stretching of adsorbable 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 MAGNESIUM OXIDE NANOPARTICLE

The SEM image of Magnesium oxide nanoparticle is shown in the Fig. 5.1.2 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.

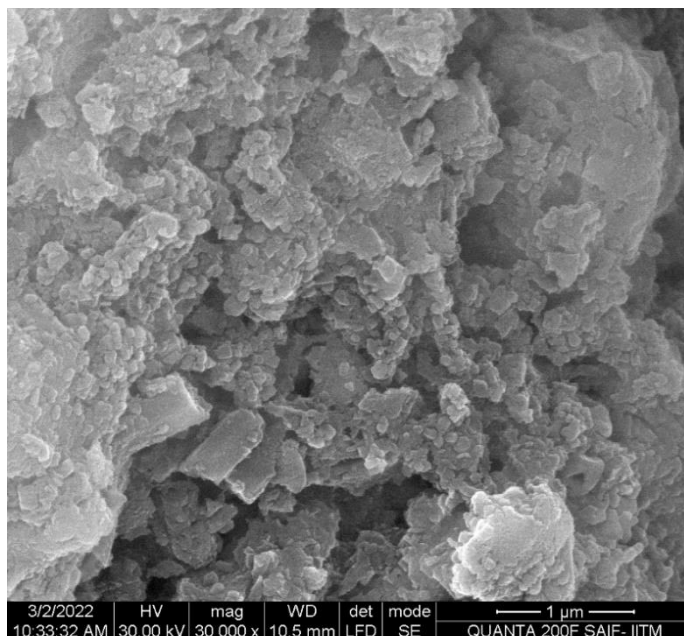
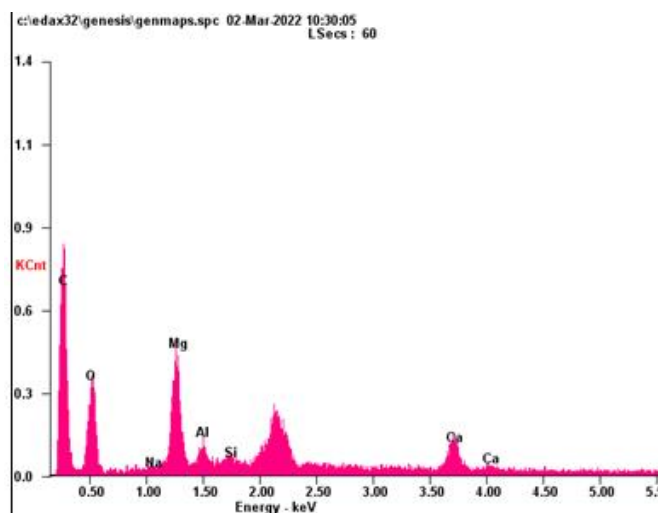


Fig 5.1.2 SEM image of Magnesium oxide nanoparticles



<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

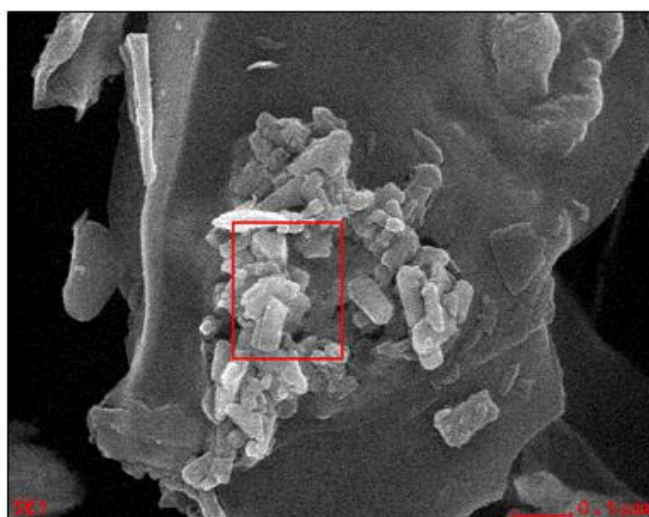


Fig 5.1.3 EDAX image of Magnesium oxide nanoparticle

5.1.4 XRD PATTERN OF MAGNESIUM OXIDE NANOPARTICLE

The XRD pattern of Magnesium oxide nanoparticle is shown in the Fig 5.1.3 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.

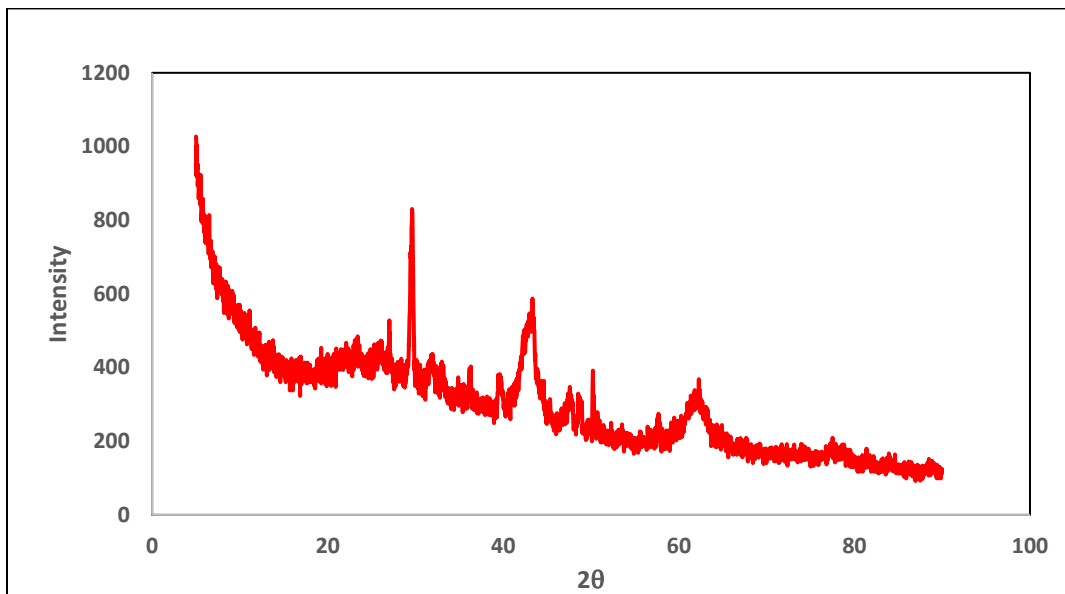


Fig 5.1.3 XRD pattern for MgO/C nanocomposite

5.2 ADSORPTION ISOTHERM

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

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

$$\frac{q_e = (C_0 - C_e)}{M} = K_f C_e^{1/n}$$

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

C_0 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 ibuprofen 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 ibuprofen 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 ibuprofen 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

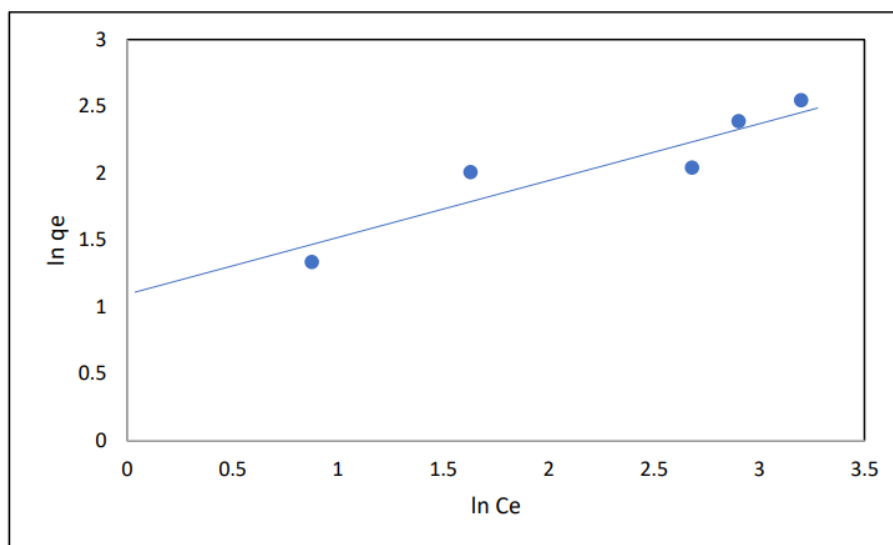


Fig 5.2 Adsorption isotherm

5.3 EFFECT OF ADSORPTION PARAMETERS

5.3.1 EFFECT OF IBUPROFEN 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.

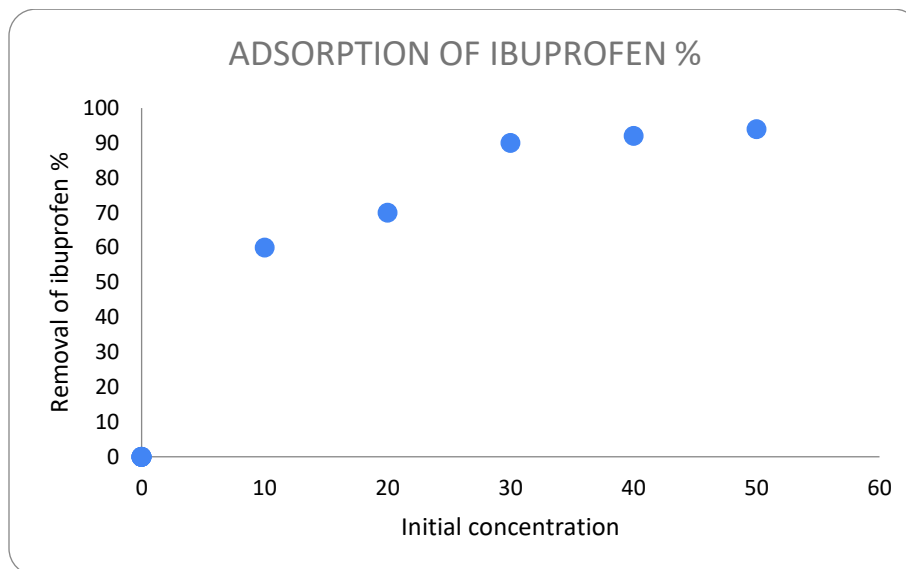


Fig 5.3.1 Effect of Ibuprofen on drug concentration

5.3.2 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 Ibuprofen drug concentration 30ppm, 100ml utilizing water bath at 30°C. the experiment was carried at the pH of 4 with the time of 2h at a constant speed of 120 rpm.

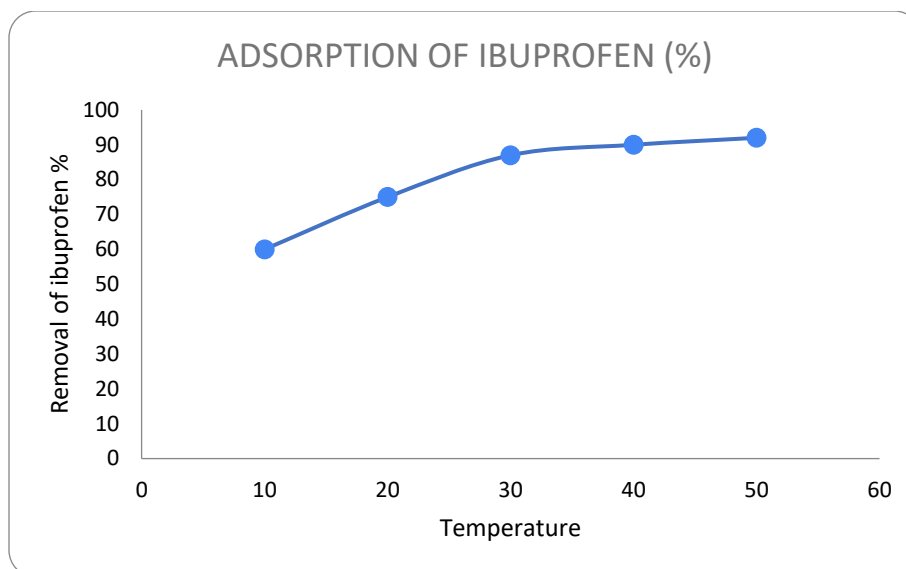


Fig 5.3.2 Effect of temperature on removal efficiency

5.3.3 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 4. contact time at 2hr a constant speed at 120 rpm.

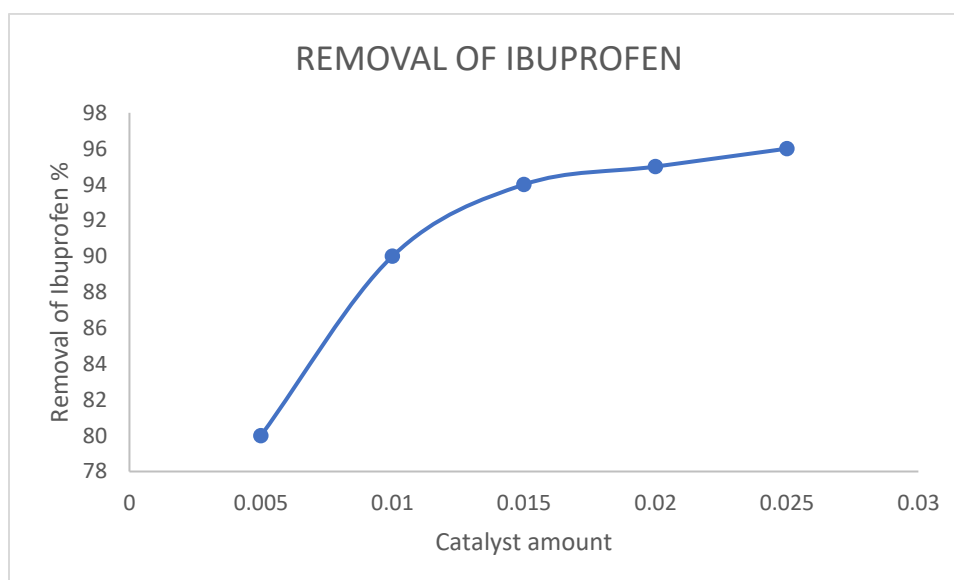


Fig 5.3.3 Effect of weight of catalyst amount

5.3.4 EFFECT OF TIME ON REMOVAL EFFICIENCY

We showed that the Ibuprofen 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 Ibuprofen 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 ibuprofen in the aqueous solution increases sharply for the first 30 mins and then decreases.

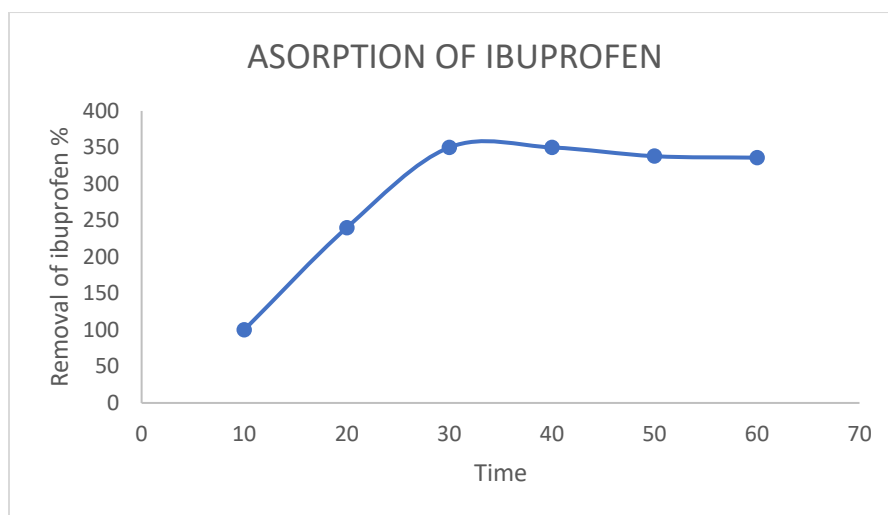


Fig 5.3.4 effect of time on removal efficiency

5.3.5 EFFECT OF PH ON ADSORPTION OF IBUPROFEN

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 Ibuprofen also increases. However, by increasing the pH from 7 to 12, the ibuprofen 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.

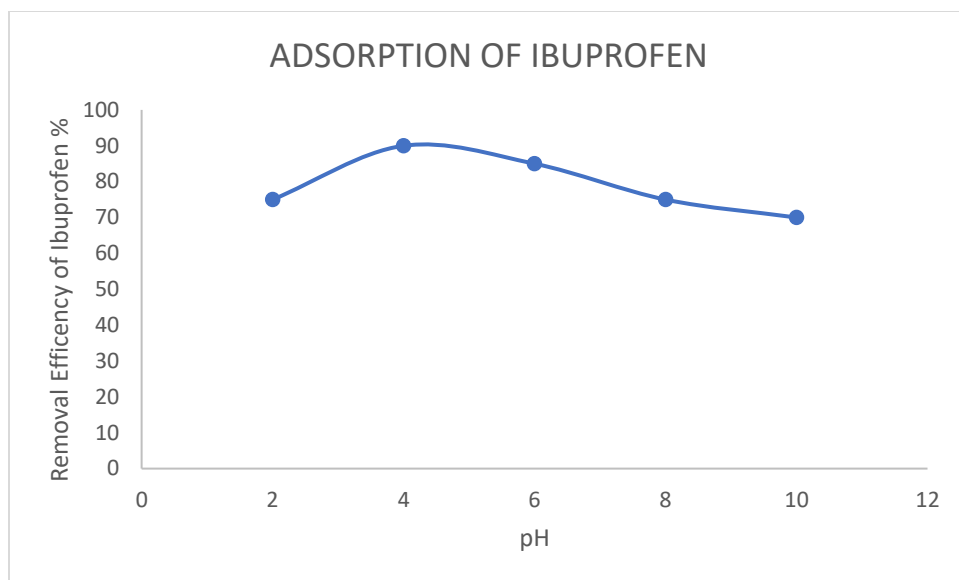


Fig 5.3.5 Effect of pH on adsorption of Ibuprofen

CHAPTER 6

CONCLUSION

The MgO/C Nano composite was using sol-gel method. The synthesized adsorbent materials is magnesium nitrate, dextrose and sodium hydroxide. The method relatively low temperature with carbon as support. XRD analysis shows that MgO/C has high crystalline nature. The characterization results show the Metal Oxygen stretching was confirmed by the functional group analysis.

REFERENCE

1. Andrew L. Shannon, Anne Woolridge. "Medical Waste", 2011. Pages 329-339.
2. Bound JP, Voulvoulis N. 'Household disposal as a pathway for aquatic contamination in the United Kingdom'. 2005.
3. Fam DWH, Palaniappan A, Tok AIY, Liedberg B, Mochhala SM. 'A review on technological aspects influencing commercialization of carbon nanotube sensors'. 2011.
4. Gaurav Sharma, Rajgovind Soni, Nakuleshwar Dut Jasuja. 'Phytoassisted synthesis of magnesium oxide nanoparticles with Swertia chirayaita'. April 2018.
5. Goldstein, J. 'Scanning electron microscopy and x-ray microanalysis'. Kluwer Academic/Plenum Publishers, 2003. Pages 689.
6. Harito C, Bavykin DV, Yuliarto B, Dipojono HK, Walsh FC. "Polymer Nanocomposites Having a High Filler Content: Synthesis, Structures, Properties, and Applications". 2009.
7. Hamza Abdalla Yones, Lin Zhu, Bo Shao, Siyue Zhang, Hui Xie, Xiaoqing Li, Wei Sun*, Published: 31 December 2020.
8. Hoult, D. I.; Bhakar, B. "NMR signal reception: Virtual photons and coherent spontaneous emission". Concepts in Magnetic Resonance. 1997. Pages 277–297.
9. Khan, Ibrahim, Khalid Saeed, and Idrees Khan. "Nanoparticles: Properties, applications and toxicities". 2017.
10. K. Krishnamoorthy, G. Manivannan, S.J. Kim, K. Jeyasubramanian, M. Premanathan, 'Antibacterial activity of MgO nanoparticles based on lipid peroxidation by oxygen vacancy'. 2012.
11. Kamigaito, O (1991). "What can be improved by nanometer composites?". J. Jpn. Soc. Powder Powder Metall. A, Concise encyclopedia of composites materials, Elsevier Science Ltd, 1994.
12. Masel, Richard I. 'Chemical Kinetics and Catalysis'. New York: Wiley-Interscience. 2001.

13. Mohammad Moslem Imani, Mohsen Safaei 'Optimized Synthesis of Magnesium Oxide Nanoparticles as Bactericidal Agents'. April 2019.
14. Mahmoud Nasrollahzadeh, ... Zahra Issaabadi, in 'Interface Science and Technology'. 2019.
15. P. Mohamed Shameer, P. Mohamed Nishath, in 'Advanced Biofuels'. 2019.
16. R Prasanth, S Dinesh Kumar, A Jayalakshmi, G Singaravelu, K Govindaraju, V Ganesh Kumar 'Green synthesis of magnesium oxide nanoparticles and their antibacterial activity'. September 2017.
17. Sally Gaw, Kevin V. 'Sources, impacts and trends of pharmaceutical in the marine and costal environment'. 19 November 2014.
18. Stokes, Debbie J. 'Principles and Practice of Variable Pressure Environmental Scanning Electron Microscopy' (VP-ESEM). 2008.
19. Schue, F.O, 'Terminology for bio related polymers and applications', 2012.
20. T.Y. Inan, in 'Recent Developments in Polymer Macro, Micro and Nano Blends'. 2017.
21. Vidal N, Poch M, Marté E, Rodríguez-Roda I. 'Evaluation on the environmental implications to include structural changes in a wastewater treatment plant'. J Chem Technol Biotechnol 77, 2002. Pages 1206–1211.