

**REMOVAL OF ACENAPHTHENE FROM AQUEOUS SOLUTION
USING MAGNESIUM OXIDE NANOPARTICLES
BY NEEM EXTRACT SYNTHESIS**

Thesis submitted in partial fulfillment of the requirements for the
award of

Master of Science in Chemistry

by

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M.Sc., Chemistry (2021-2023)**



**SATHYABAMA INSTITUTE OF SCIENCE AND
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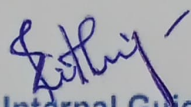
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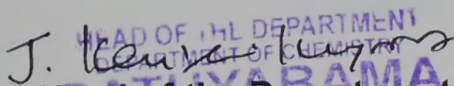
This is to certify that this Project Report is the bonafide work of Ms. F. AARTHI INFANT MARY (41910001) who carried out the project entitled "REMOVAL OF ACENAPHTHENE FROM AQUEOUS SOLUTION USING MAGNESIUM OXIDE NANOPARTICLES BY NEEM EXTRACT SYNTHESIS" under our supervision from JANUARY 2023 to MAY 2023.


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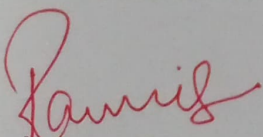

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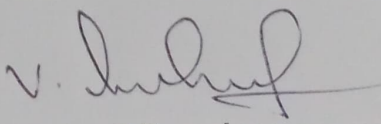
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DECLARATION

I **F. AARTHI INFANT MARY (41910001)**, hereby declare that, Project Report entitled “**REMOVAL OF ACENAPHTHENE FROM AQUEOUS SOLUTION USING MAGNESIUM OXIDE NANOPARTICLES BY NEEM EXTRACT SYNTHESIS**” done by me under the guidance of **Dr. T. Krithiga, M.Sc., M.Phil., 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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F. AARTHI INFANT MARY

ABSTRACT

Magnesium oxide nanoparticles have been synthesized by sol-gel method. The synthesized nanoparticle has been characterized by FTIR, XRD, BET surface area and SEM. XRD studies show that magnesium oxide nanoparticle as hexagonal structure. The SEM analysis confirms the formation of spherical particles which are agglomerated. FTIR confirms the presence of Mg – O stretching bond. BET(Brunauer-Emmett-Teller) theory is used to evaluate the adsorption data and generate a specific surface area result. The synthesized Magnesium oxide nanoparticle was used as adsorbent for the removal of the acenaphthene from the aqueous solution. This includes adsorption isotherm effect of temperature, pH initial concentration, catalyst weight, time on stream. Freundlich isotherm was best fitted. Optimized condition for adsorption studies is pH at 6, temperature 60°C, initial concentration at 0.15, time of contact 30 minutes. The concentration of Acenaphthene in aqueous solution after adsorption were studied by UV-Visible Spectroscopy.

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LIST OF ABBREVIATIONS

UV-VIS	- Ultra Violet Visible
XRD	- X-Ray Diffraction
FTIR	- Fourier Transform Infrared Spectroscopy
SEM	- Scanning electron Microscope
BET	- Brunauer-Emmett-Teller
CNT	- Carbon Nano Tubes
MGO	- Magnesium Oxide
MM	- Millimeter
PPM	- Parts Per Million
RPM	- Revolutions Per Minute
pH	- Potential of Hydrogen
MMNC	- Metal Matrix Nano Composites
CMNC	- Ceramic Matrix Nano Composites

CHAPTER 1

INTRODUCTION

1.1 NANO PARTICLES:-

From the turn of the century, researchers have known about nanotechnology. Materials of all kinds were created at the nanoscale through nanotechnology. Nanoparticles (NPs) are a diverse class of materials that comprise substances that are particulate and have at least one dimension that is less than 100 nanometer.

A nanoparticle is a tiny particle with a size that can range from 1 to 100 nanometers. It is invisible to human eyes. According to their bigger substance, they have fundamentally different physical and chemical characteristics. (Ibrahim Khan:2019)

1. 1 . 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. Semiconductor nanoparticles
- IV. Polymeric nanoparticles
- V. Lipid-based nanoparticles
- VI. Metallic oxide nanoparticles

i) Carbon-Based Nanoparticles

They are separated into two different categories of materials:

- ❖ Carbon nanotubes (CNTs)
- ❖ Fullerenes

All carbon nanotubes (CNTs) are a tube of Graphene sheets. They outperform steel by a factor of a thousand. Nanomaterials made of globular hollow cages, like allotropic forms of carbon, are found in Fullerenes.

ii) Ceramic Nanoparticles

Oxides, carbides, carbonates, and phosphates make up these ceramic nanoparticles. They have a high level of heat and chemical inertness resistance. For a number of diseases such as bacterial infections, cancer, etc., they are simply utilized as a medication delivery mechanism.

iii) 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 bandgap particles. Semiconductor materials have characteristics that fall between those of metals and nonmetals, hence they have a wide range of uses in the literature.

iv) Polymeric Nanoparticles

These polymeric nanoparticles have a structure similar to a nanocapsule or a nanosphere and are based on organic materials depending on fabrication techniques. Whereas nanocapsule particles have a core-shell structure, nanosphere particles have a matrix-like structure.

v) Lipid- Based Nanoparticles

Lipid acts as the solid core of lipid-based nanoparticles. It has a diameter ranging from 10 to 100 nanometer and is spherical in shape. Surfactants and Emulsifiers stabilize the nanoparticles' exterior surfaces. They can be utilized as drug carriers and deliverers in the biological industry.

vi) Metal-Oxides Nanoparticles

Chemical, Electrochemical, or Photochemical techniques can be used to create these nanoparticles. These nanoparticles are used in research, bio-molecule detection and exclusion, environmental analysis, and bio-analytical applications. Mostly through its use in the treatment of waste water, industrial and household waste, soil sediments, and atmospheric pollutants, metal oxide nanoparticles play a crucial position in environmental rehabilitation. (Khalid Saeed:2019)

1.1.2 Classifications Of Nanoparticles:-

i) Organic Nanoparticles

Organic nanoparticles are superior to other nanoparticle platforms in many ways, such as their ability to self-assemble adjuvants and antigens under physiologically benign conditions and their chemical diversity, which allows them to accommodate a wide range of modalities, compositions, sizes, shapes, and surface functionalist. These nanoparticles are biocompatible, biodegradable, and generally non-toxic, organic nanoparticles have been used as vaccination platforms in a wide range of various applications.

ii) Inorganic Nanoparticles

Inorganic nanoparticles have primarily been utilized in cancer photo-thermal therapy or as imaging contrast agents. Inorganic nanoparticle vaccine development in preclinical environments has recently attracted attention. The majority of inorganic materials have properties that make them perfect for antigen delivery in vaccines, including reduced particle size, increased stability, controlled tun-ability, enhanced permeability, large drug loading, and triggered release profiles. In this section, we include a brief overview of the recent advancements in the use of these inorganic nanoparticles as transporters and adjuvants in vaccines.

iii)Hybrid Nanoparticles

Hybrid nanomaterials are defined as unique chemical conjugates of organic and/or inorganic materials. Due to the difficulty of having more options for multifunctional materials, the inclusion of nano-sized materials has further expanded the extraordinary properties of hybrid materials. Due to the materials' chemical and physical features, which are leading to advances with high added value, multifunctional applications have attracted a lot of interests. These materials are classified as innovative advanced materials applicable to a huge diversity of applications including catalysis, sensors, ionics, energy conversion and storage. (Idrees Khan:2019)

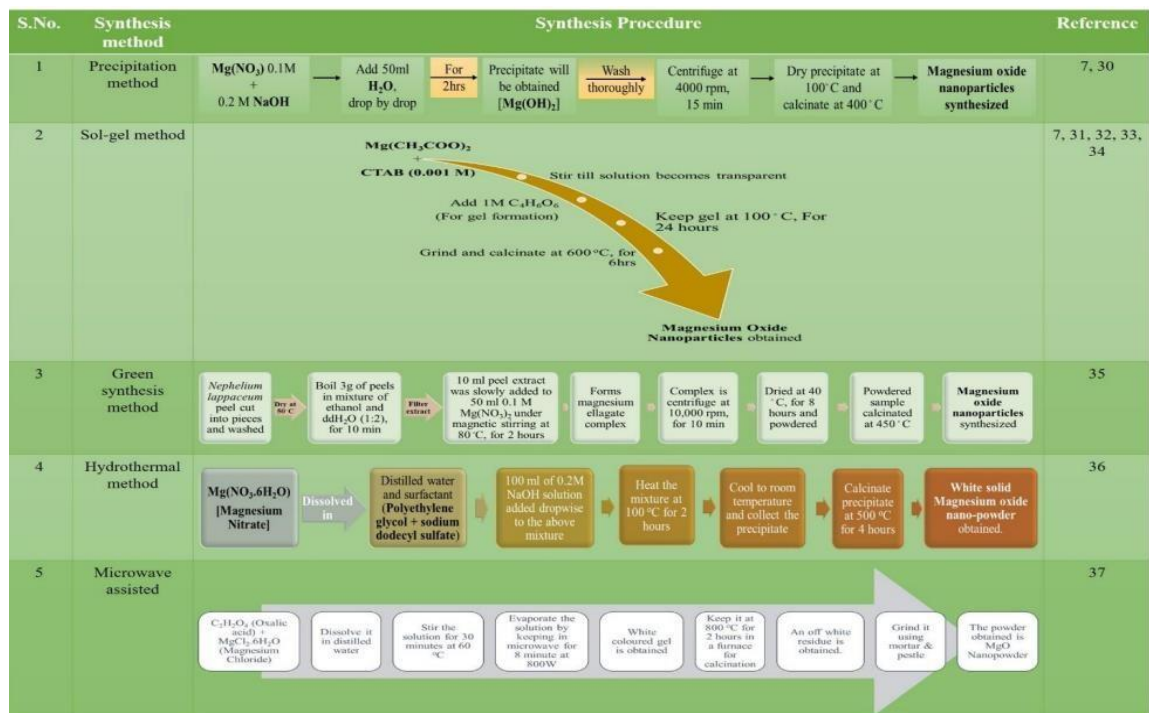


ORGANIC, INORGANIC AND HYBRID NANOPARTICLES

1.1.3 Magnesium Oxide Nanoparticle

Magnesium oxide nanoparticles are non-toxic and capable of adsorption. They have a high melting point, are very pure, and very hard. The size, morphology of oxide particles can be adjusted utilizing characteristics such as pH, ionic strength, and varying calcination temperature.

Magnesium oxide nanoparticles are used more frequently than other metal oxide nanoparticles in a variety of industries because they offer promise as a structure material for biological implants due to its favorable properties, including their high strength to weight ratio, low density, excellent functionality, and nontoxic nature. These MgO nanoparticles can be employed in as electronics, catalyst, ceramics, petrochemicals products, coating and several other sectors. There are few synthesis techniques, including sol-gel, precipitation and green synthesis techniques which are frequently used in the removal of industrial pollutants, eradication of cancer, remediation of toxic waste.



Magnesium Oxide Synthesis Process

1.2 METAL OXIDE

Metal oxides are crystalline solids that comprise a metal cation and an oxide anion.

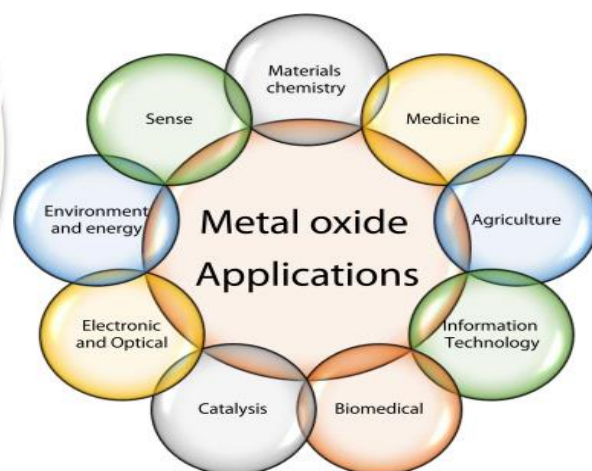
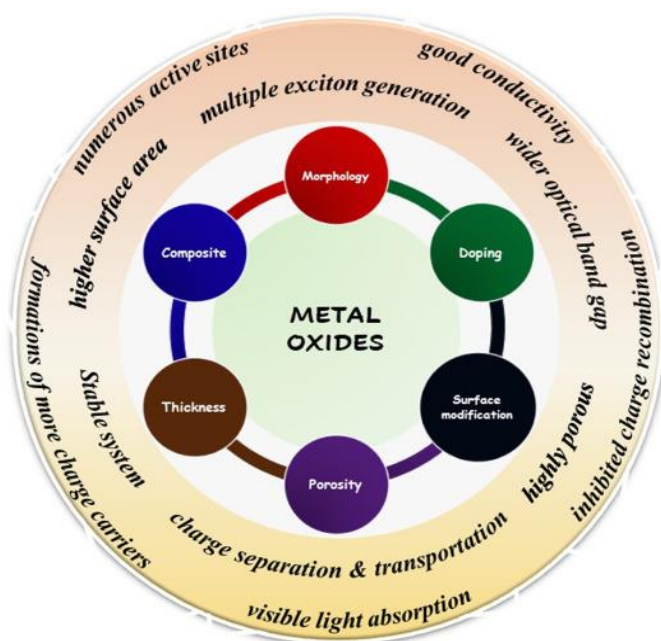
Normally, they interact with water to form bases or with acids to form salts. Metal oxides exhibit wide range of functional capabilities that are greatly influenced by their crystal structure, morphology, composition, intrinsic flaws, doping, etc.

One of the most significant and well-known solid catalysts are metal oxides. Because Metal Oxides have a variety of structural characteristics, they stand out among inorganic solid-state materials. Certain metal groups, especially transition metals, have drawn a lot of interest due to configuration of their valence electron. These factors also affect their optical, electrical, chemical, and catalytic properties.

Along with the development of new catalytic methods for non-thermal reactions, energy storage techniques, and environmental remediation through the removal or breakdown of hazardous chemicals released during manufacturing processes, metal oxides play a significant role in decarbonization strategies, have grown to be significant factor in industrial expansion.

One of the key factors to consider when thinking about metal oxide-based materials is the scalable from laboratory to industrial scale. Some gas sensors require higher operating temperatures, and metal oxides are a suitable sensing material in such situations as they exhibit high-temperature stability as well as inertness to varying humidity.

Metal Oxides are also relatively simple to summarize, the cost of making up gas sensors from metal oxides is relatively low, and they also show higher operating temperatures. The mechanics of ions of nutritional elements and pollutants are well understood and the interaction of metal oxides in soil systems involving the sorption of ions from mixtures has received less attention than research on synthetic or pure metal oxides, though. (Roman Viter:2019)

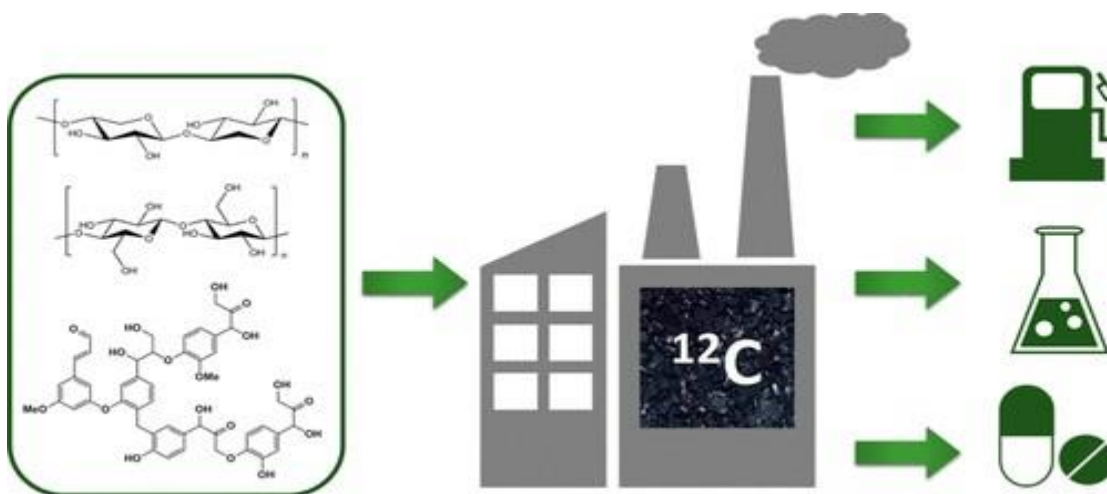


METAL OXIDE AND METAL OXIDE APPLICATIONS

1.3 CARBON AS CATALYST

Due to its abundant surface area, superior electron conductivity, and chemical inertness, carbon can be utilized as a catalyst as well as a catalyst support for chemical and enzymatic reactions. (Dushyant Shekhawat:2011)

The catalytic activity of carbon is enhanced when it is chemically combined with enzymes and nanoparticles.



Carbon Catalyst

1.4 NANOCOMPOSITES

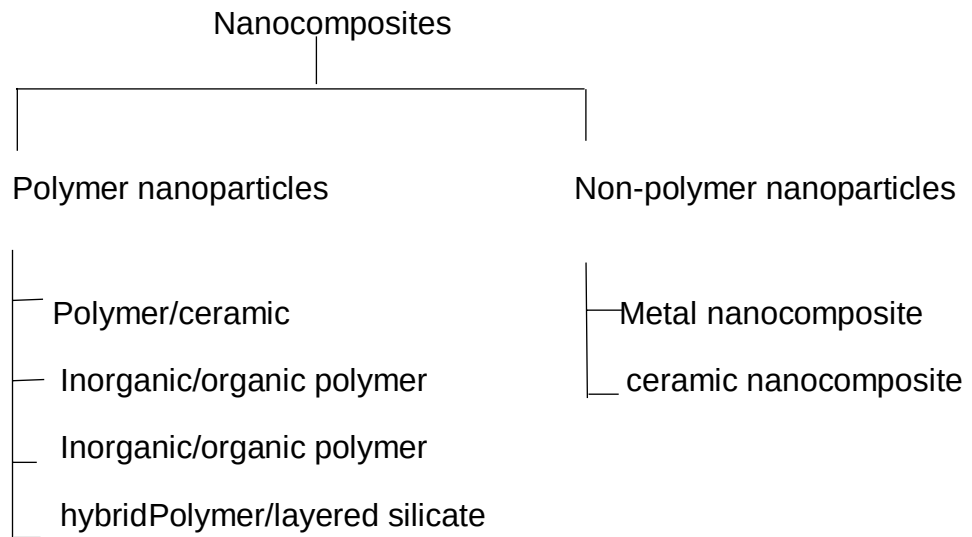
Generally, nanocomposites are based on the concept of creating a very large interface between the nanosized building blocks and the polymer matrix, the main problems of nanocomposite are usually homogeneous distribution of the particle.

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, therefore same factors that affect typical composites' properties also apply to them. Compared to micro composites, they have a more complex structure. The dispersed matrix and dispersion phase materials in nanocomposites can be used to classify them. Through the use of creative synthetic techniques, it is now possible to produce a wide variety of fascinating new materials with new features due to this quickly developing sector.

They are multi-phase materials because they have many phases, and at least one of those phases should have diameters between 10 and 100 nanometer. Hence, the concept behind nanocomposite is to develop and produce new materials with remarkable flexibility and increase in their physical properties by using building blocks with dimensions in the nanoscale range. They are greatly valuable in a variety of industries, from small scale to huge to very large manufacturing units, thanks to the rapidly predicted need of these outstanding theoretically sophisticated materials. (Mahmood Reza Rahimi, Soleiman Mosleh:2022)

With a lot of benefits for humanity and the environment, these provide improved technologies in addition to better economic chances for a number of industrial sectors, including the automotive, building, electrical and electronics, food packaging, and technology transfer.



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 range of 1- 50 nanometer. They consist of multi phase system. In plastic production polymer nanocomposites cover 95%. They are done by controlled manner of compounding.

1.4.2 Non-Polymer Nanocomposite

The Non-polymer nanocomposites are those nanocomposites that may not contain any polymer ingredients. They are also known by the name inorganic nanocomposites as well.

1.4.3 Metallic Oxide Nanocomposite

Bimetallic nanocomposites are utilized in alloy or core shell structures due to their extensive catalytic capabilities, which include ,

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

- ❖ Increased electric resistivity etc..

1.4.4 Magnesium Oxide/Carbon Nanocomposites

Glucose (500 mg) and Mg (NO₃)₂ in various concentrations (0, 50, 100, and 200 milligram) are used to prepare the catalyst in this instance. They are combined with the inert salts KCL (1gram) and LICI (1gram), which are used to make the molten salts for the synthesis of porous carbon. The mixture is then calcined in a tube furnace at room temperature for two hours in a nitrogen atmosphere at 800°C at a rate of 5°C per minute. During calcination, the carbonaceous material is rinsed to remove salt and dried to weight using a range of Mg weights loaded with carbon as carbon catalysts, including Mg (500 milligram)/carbon catalyst, Mg (100 milligram)/carbon catalyst, and Mg (200 mg)/carbon catalyst. The word for these catalysts is Mg(x)/carbon, where x is the weight of the Mg load.

1.5 MAGNESIUM OXIDE

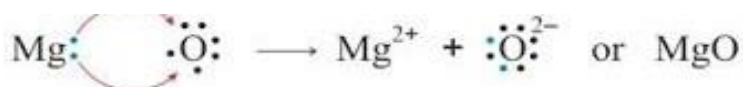
One of the most significant metal oxides in the field of catalysts is magnesium oxide (MgO), which is frequently used as a support. Magnesium Oxide is an oxide with a rock-salt structure, on each Mg²⁺ are surrounded by five O²⁻ ions, and typically the anhydrous surface of metal oxides possesses two active sites, the cation Mn⁺ and the anion is O²⁻. Due to its low reduction potential, Mg²⁺ is easily converted during catalytic testing to Mg¹⁺ and Mg⁰. Magnesium oxide is an inorganic salt that is created when magnesium ions combine with oxygen; it is often available as a supplement. In addition to dietary supplements, it is also included in over-the-counter drugs for headaches, indigestion, and insomnia.

Magnesium oxide and other inorganic salts of magnesium carbonate are high in elemental magnesium, which is the total amount of magnesium in a supplement. Because of their poor solubility rate, they are less bioavailable than other types of minerals. The amount of magnesium in magnesium oxide supplements is adequate, but our bodies do not readily absorb or use it. Magnesium is frequently used to treat medical issues like persistent constipation since it has potent laxative effects, various advantages, and may cause digestive complications like

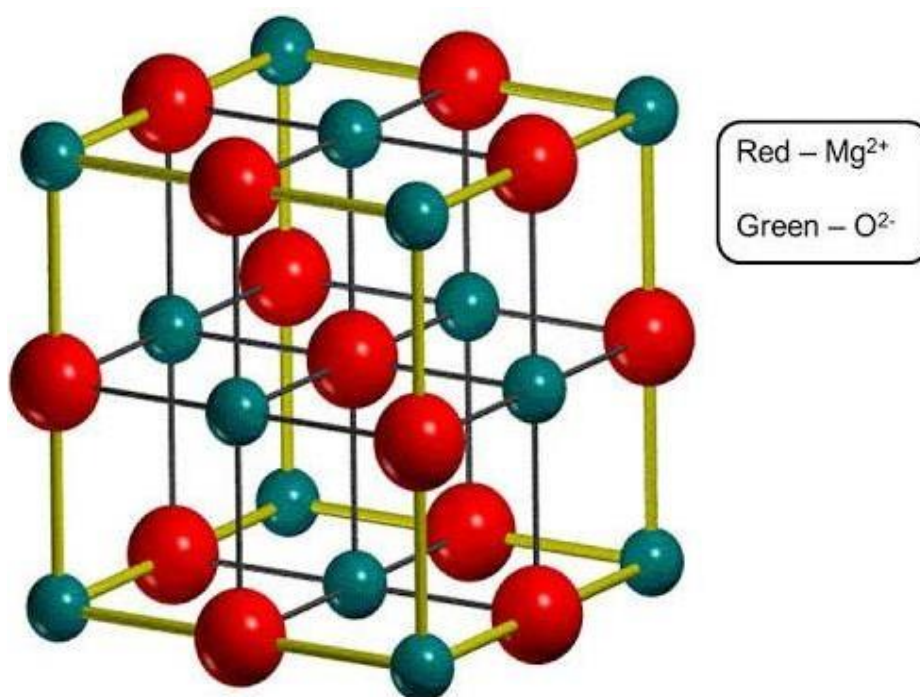
diarrhoea.

Magnesium oxide may benefit some people by lowering blood pressure, enhancing blood sugar control, treating migraines and constipation, and lowering stress and anxiety levels. Magnesium oxide, which is offered separately and as part of multivitamin packages, is one of the most extensively used supplement forms. .

Magnesium oxide dosing generally ranges from 250-1000 milligram per day, High doses of magnesium oxide may cause dangerously high blood magnesium levels, it may also cause side effects like bloating and reduce the effectiveness of certain medications.



Formation of MgO



Structure of MgO

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

CHAPTER 2

LITERATURE SURVEY

Aravind Kumar et al, (November 2019), illustrated how palm shell agricultural waste may be used to prepare activated carbon utilizing a chemical activation process and potassium hydroxide. On the palm shell, nano-magnesium oxide is applied. The effectiveness of the drop-wise process used to create the activated carbon composite to remove anthracene from the aqueous solution was evaluated. X-Ray diffraction demonstrated the amorphous nature of composites..

Neelam Thakur et al, (December 2022), illustrated a new synthetic strategies for Magnesium Oxide Nanoparticles. Conventional physical and chemical synthesis techniques was for highly technical equipment, extreme energy, pressure, and vacuum conditions, as well as a variety of dangerous substances. A safe and sustainable method of synthesizing nanoparticles from several biological sources, particularly plants and microorganisms.

D Tan et al, (March 2020), illustrated on the green synthesis of nanoparticles and their uses in a variety of disciplines are the foundation of the current review. By using non-toxic reagents and components, the green chemistry approach to the synthesis of nanoparticles has gained importance. Multiple spectroscopic methods, including TEM, SEM, FTIR, UV-Visible absorption spectroscopy, and others, can be used to characterize these synthesized nanoparticles.

Ayushi Jain et al,(August 2018), illustrated the colorimetric sensing of Fe^{3+} ions in water using magnesium oxide (MgO) nanoparticles where a novel and green method to synthesize MgO nanoparticles using *Syzygium aromaticum* extract (clove extract) as reducing agent. The synthesized MgO nanoparticles were examined using a number of methods, including Transmission Electron Microscopy (TEM), Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction (XRD), and Energy Dispersive X-ray Analysis (EDX). Using UV-Vis spectrophotometry, the MgO NPs were employed as a colorimetric probe for the sensitive.

Stuti Mittal, Arpita Roy, (May 2021), illustrated one of the potential applications of nanoparticles is their role in the removal of toxic pollutants such as industrial dyes, heavy metals, and degradation of toxic chemicals. Due to its dependability, environmental friendliness, affordability, and sustainability, green synthesis has proven to be advantageous. The removal of dyes by a variety of mechanisms, including photodegradation, adsorption, and ion exchange, is successfully accomplished by the use of a large number of nanoparticles as catalysts.

Davoud Ahmadvand et al,(June 2023), illustrated the current study is the first to demonstrate a green chemistry approach for synthesizing magnesium oxide nanoparticles by using chitosan polymer as a stabilizer and reducing agent. The results of the TGA/DTA, FTIR, UV-Vis, XRD, and FESEM/EDX/PSA techniques were used to describe the synthesized MgO nanoparticles. The necessary functional groups were found throughout the synthesized MgO nanoparticles, based on FTIR spectra.

Usha Subbiah et al, (September 2019), illustrated the development of herbal medicine-incorporated nanoparticle formulations in dentistry, as the plant-mediated biogenic metal nanoparticles can overcome the drawbacks of herbal drugs. Many oral/dental diseases have been successfully treated using the green synthesis of metal nanoparticles like silver, copper, and gold nanoparticles, which is aided by the various medicinal plant extracts. In fact, it is more successful than using conventional materials. These are also present in common dental care items like mouthwash and toothpaste. However, there is a need for more research because we currently know insufficiently about the safety of nanomaterials. Drug resistance would be addressed when a variety of plant extracts were mixed with nanoparticles, among other problems.

Krithiga.T, J.Aravindkumar, G. Narendrakumar, P.Prakash, K.Balasankar, Purna Pushkala.D, Najat Marraiki, A.G. Ramu, Dongjin Choi, (September 2021), illustrated a water-based form of naphthalene, calcium oxide nanoparticles were found to be adsorptive in the presence of Ca^{2+} ions. With the help of calcium chloride in a solution concentration of 0.002-0.1 M, calcium oxide regeneration was shown to be more effective (90%) than before. The significance of naphthalene adsorption onto calcium oxide employing calcium chloride for regeneration is highlighted by the less hazardous naphthalene desorption along with SEM, FTIR, and XRD characterization data. The benefits of adding Ca^{2+} ions to the naphthalene solution for its quick adsorption were revealed, perhaps due to an increase in naphthalene molecule sorption capacity at lower concentrations.

S. Suchart Siengchin.L, (December 2017) - illustrated the metal oxide nanoparticles impact on the mechanical, thermal, bacterial-resistant, and barrier properties of biopolymers. The load transferability between the polymer and filler determines the characteristics of biocomposites containing metal oxides.

Understanding the fundamental interactions between the filler, polymer, and filler-filler in polymer composites is important for extending the variety of uses for these materials. Additionally, there are biopolymers made of metal oxides.

T. Shrinivas et, (August 2022), illustrated various attempts for green synthesis of metal oxide nanoparticles; revealing the significance of plant extracts in reducing metal sources to nanoparticles and applications in various scientific domains.

Neem, or *Azadirachta indica*, is an evergreen tree in the Meliaceae family.

Numerous phytochemicals, including flavonoids, glycosides, terpenes, and triterpenes, are said to be present in its leaves. The usage of neem leaves' aqueous extract revealed that its polar phytochemicals were involved in decreasing the metal oxides and stabilising their nanoparticles. It should be emphasized that metal oxide nanoparticles outperform aqueous leaf extract in terms of antibacterial activity and photocatalytic potential.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

The main aim of this project is to remove the acenaphthene from aqueous solution using neem extract with synthesized Magnesium Oxide nanoparticles. Magnesium Oxide nanoparticles were synthesized with well-defined morphology at the room temperature by using green synthesis method, using magnesium nitrate and sodium hydroxide in a stoichiometric ratio via this method. MgO nano powder were characterized with the help of FTIR, XRD, BET surface area and SEM to remove Acenaphthene from the aqueous solution.

3.2 Scope

- Synthesizing of MgO by green synthesis using neem extract.
- Characterization of MgO by FTIR spectroscopy, XRD, SEM, BET and UV visible spectroscopy..
- Removal of acenaphthene from the aqueous solution using MgO nanoparticles.
- To study the adsorption isotherms.
- Optimization of reaction parameters such as the Effect of Initial concentration of acenaphthene, Effect of Temperature, Effect of pH and Time on stream.

CHAPTER 4

MATERIAL AND METHODOLOGY

4.1 MATERIALS:

Materials used in this reaction are dried neem leaves, Magnesium Nitrate, Sodium Hydroxide, Distilled Water, which were purchased from scientific chemicals and it can be used as such.

4.2 CHARACTERIZATION TECHNIQUES:

4.2.1 Powder X-Ray Diffraction (XRD)

Based on the fact that most materials have different diffraction patterns, when an X-ray is incident on the crystal they scattered according to Bragg's law, it diffracts and forms diverse patterns. By scanning the sample through a variety of 2 angles and graphing the angular positions and intensities of the resulting diffracted peaks, which posits that diffraction only occurs when the distance travelled by the rays reflected of the successive planes of crystal, the pattern was formed. The patterns that are produced are analyzed for the positions of the diffraction maxima, peak intensities, and the intensity distribution as a function of the diffraction angle. This is done by matching the resulting diffraction peaks to characterize the patterns. In this case, the material's powder is used to create the diffracted pattern. Its diffracted pattern can be used to identify compounds. The purity of a sample can also be examined from its diffracted pattern, and powder diffraction is often simpler and more practical than single crystal diffraction. A characterization technology known as powder X-ray diffraction provides a thorough view of the sample's crystalline structure and chemical composition. The particle size can be determined by using the Scherrer formula is

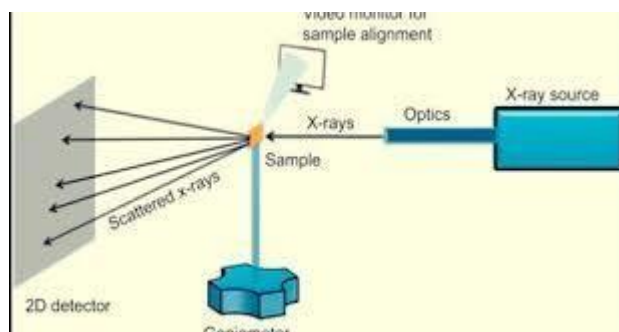
$$\text{Scherrer formula} = (k\lambda/\beta \cos \theta)$$

where, K = e Shape factor (0.9)

λ = wavelength of X-rays,

β = corrected full width at half-maximum;

θ = angle of peak



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 flexible method for characterizing the surface of nanoparticles. It uses infrared light to scan samples and utilizes a number of phenomena, including the absorption, reflection, and emission of light utilizing the sample to identify organic, inorganic, and polymeric components. FTIR can be used to identify and characterize unknown components, find additives, detect impurities in a material, and determine decomposition and oxidation. A source, sample cell, detector, amplifier, A/D converter, and computer are all components of an FTIR spectrometer. When IR radiation through a sample, some of the radiation is absorbed by the sample and some of the radiation passes through (is transmitted). After passing through the interferometer, the source's radiation reaches the detector. The A/D converter and amplifier enhance the signal, convert it to a digital signal, and then transmit it to the computer. The final signal produced by the detector usually has a spectrum from 4000-400 cm^{-1} , which corresponds to the fingerprint range of the sample. The sample was combined with potassium bromide and formed into pellets for this purpose.



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 concentrated beam of high-energy electrons to produce a range of surface-generated signals that reveal information about the sample, including its external morphology, chemical composition, crystalline structure, and orientation of the materials that make up the sample. SEM with EDX can identify the particle's primary atomic composition from a high-resolution image of the particle's size and surface texture, but it cannot identify the colors of the particle. One of the primary instruments used for in-depth micro area analysis is the SEM. It features a powerful stereoscopic view and great magnification. SEM photographs are more magnified and have better definitions than those obtained through optical microscopy. Only elements with atomic coefficients less than 5 can be measured using a SEM. We can obtain photos of the sample's chosen point locations using SEM. This method is particularly helpful when determining chemical composition in a qualitative manner. In a filtering mode using SEM techniques and with an amplification range of 50 nm to 100 nm, zones with a width of about 1 cm to 5 microns can be captured in a SEM examination system.



Scanning Electron Microscopy

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

4.2.4 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 includes multiple layers of gas adsorption on the adsorbent surface. Because nitrogen is so pure and interacts so strongly with most materials, it is frequently utilized in BET surface area analysis. The surface is chilled using liquid N₂ to obtain the adsorption since the contact between solid and gaseous phases is typically weak. Nitrogen gas in a known quantity is delivered into the sample cell. The sample is taken out of the nitrogen atmosphere once the adsorption layers have developed, and it is then heated to cause the adsorbed nitrogen to be liberated from the material and measured. BET isotherm, which shows the amount of gas adsorbed as a function of relative pressure, is used to display the collected data.

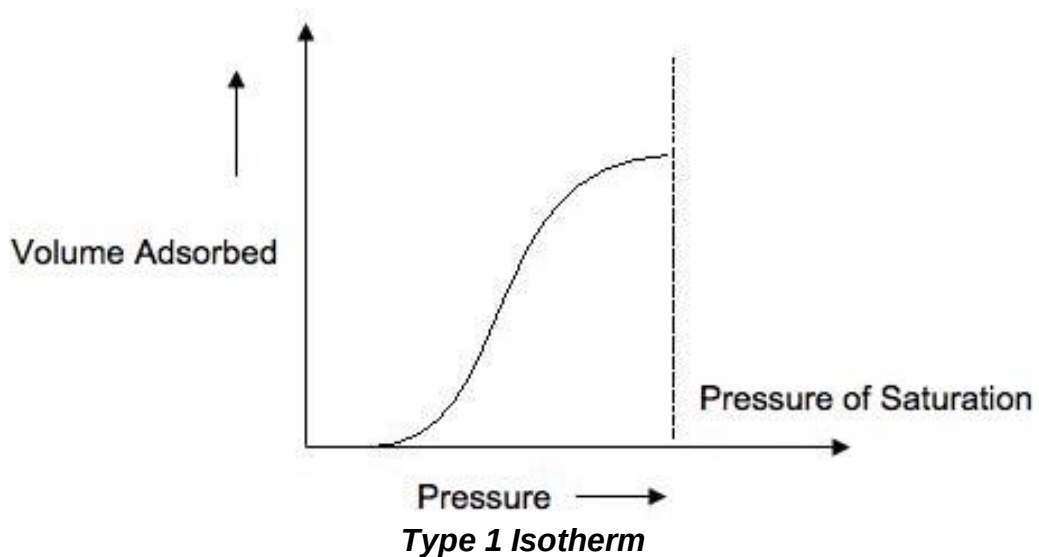


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There are five types of isotherms possible,

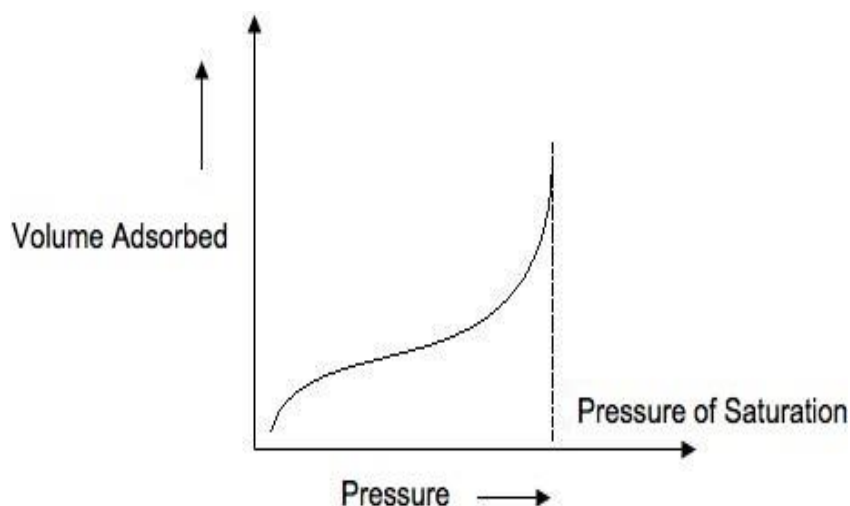
Type I Isotherm

A Type 1 is a pseudo-Langmuir isotherm, because it exhibits monolayer adsorption. Type 1 arises when P/P_0 equals the partial pressure value and C equals the BET constant. This type of isotherm is found when the pore diameter is less than 2 nm.



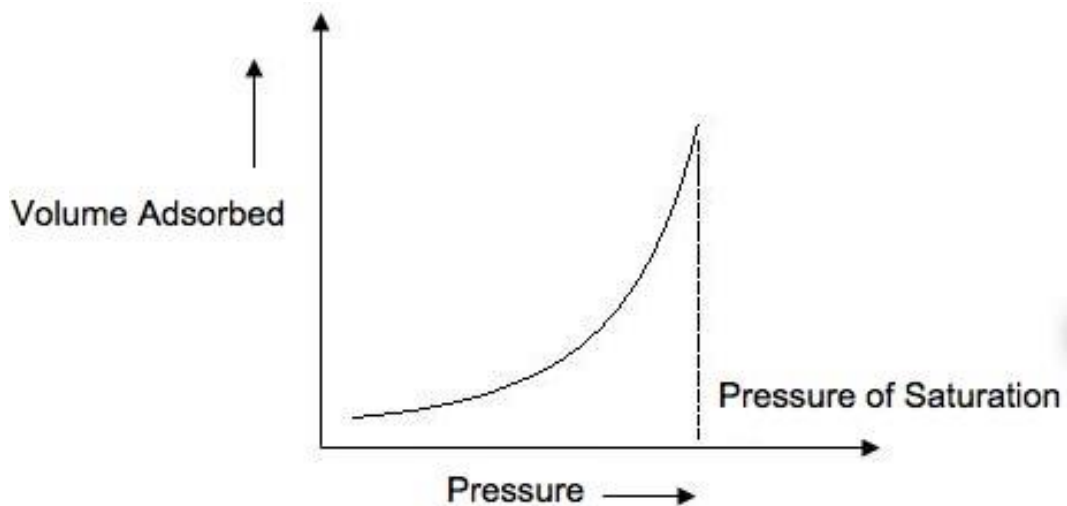
Type II Isotherm

A type II isotherm differs from a type II isotherm. Creation of monolayer is indicated by flat area in centre. Type II is formed when $c > 1$ in BET equation.



Type III Isotherm

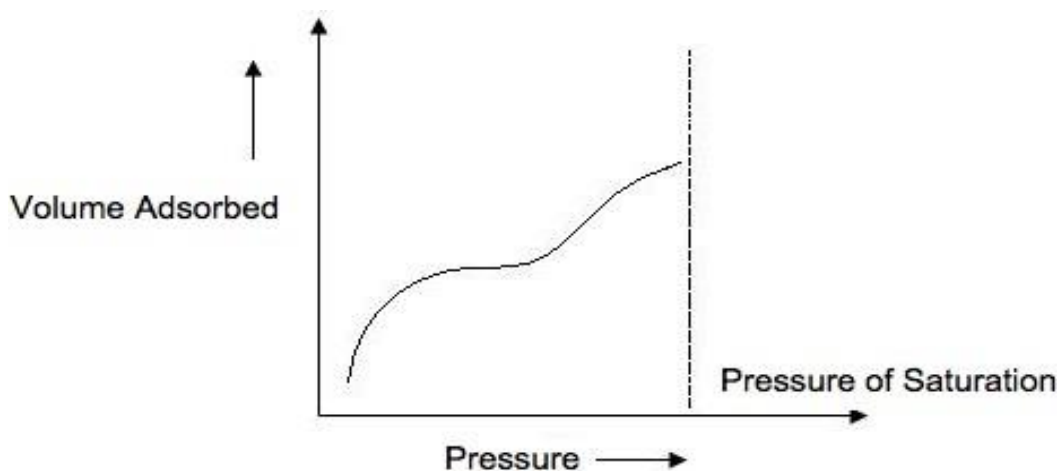
In type III isotherm, there is no asymptote in the curve, the isotherm is obtained when the $c < 1$ and exhibit the creation of a multilayer.



Type 3 isotherm

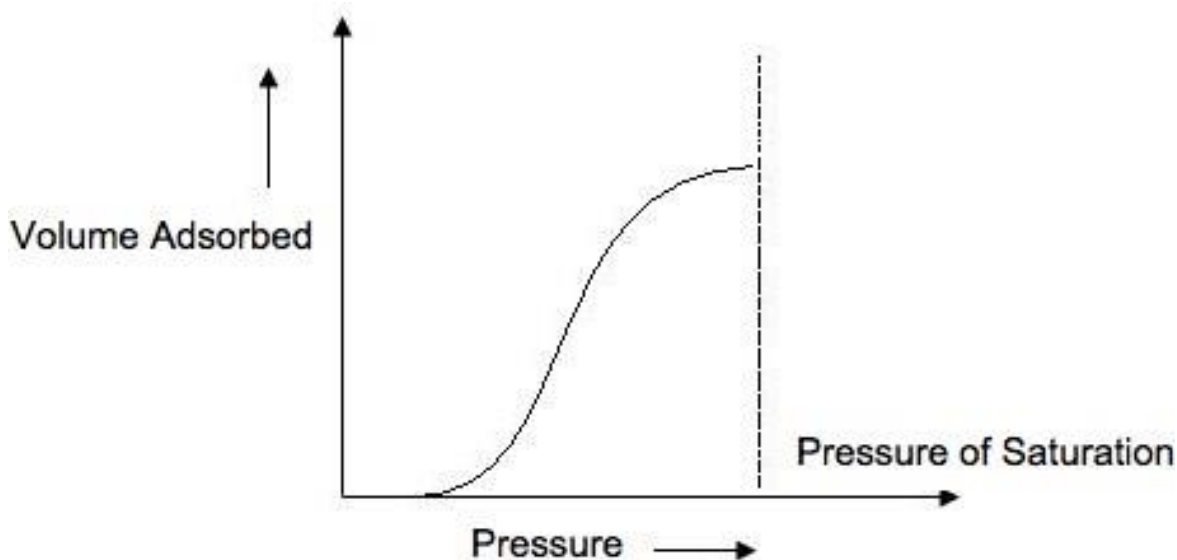
TYPE IV Isotherm

Type IV isotherm occur when capillary condensation occurs. At a pressure below the gas's saturation pressure, gases condense in solid's microscopic capillary pores. It demonstrates the production of a monolayer at lower pressure, followed by the formation of a multilayer. mesoporous materials, which have pores with sizes of 2 to 50 nm, are characterized by their surface areas using BET.



Type V Isotherm

Type V isotherms are not applicable to BET but are very similar to type IV isotherms.



Type 5 Isotherm

Source: [2.3: BET Surface Area Analysis of Nanoparticles - Chemistry LibreTexts](#)

4.3 METHODOLOGY:

4.3.1 Preparation of extract

The neem leaves are washed and rinsed well with water and dried in sunlight for 3-4 days. 5 gram of dried leaves is taken and 100 ml of distilled water is added in round-bottomed flask. Then it is heated and stirred using magnetic stirrer at 70° C and the boiled solution is cooled and filtered to obtain the extract.

4.3.2 Preparation of Magnesium Oxide nanoparticles

30ml of leaf extract is mixed with 100 ml of magnesium nitrate solution and heated at 80° C. The pH of the solution is adjusted to 11 using 1M NaOH is added while stirring and heated at 70° C for 30 minutes. The precipitate of magnesium oxide is obtained which is washed using water, filtered and dried in hot air oven at 90° C and the MgO nanoparticles are obtained which is used as the catalyst.

4.3.3 Removal of acenaphthene from aqueous solution

Adsorption experiment was conducted for the removal of acenaphthene using the polycyclic aromatic hydrocarbon. It is experimented in 25ml standard flasks which contained a specific quantity of the catalyst and 25 ml of hexane.

4.3.4 Adsorptive efficiency of adsorption parameters

Adsorptive Efficiency of Acenaphthene Concentration [10-60ppm]

About (0.025g, 0.05g, 0.075g, 0.1g, 0.125g, 0.15g) from acenaphthene is added in 25 ml beakers. These containers were tightly sealed and continuously agitated at rotational speed of 120 rpm in a shaker incubator.

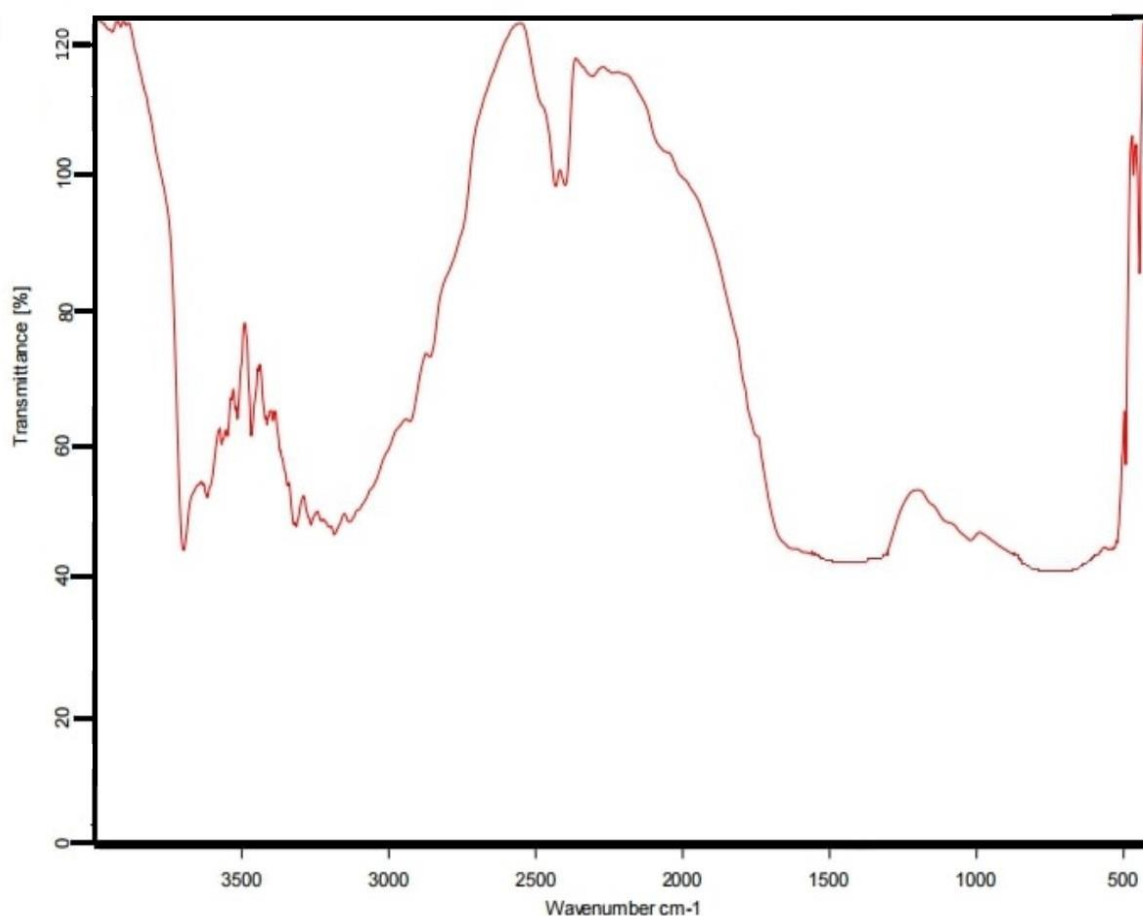
CHAPTER 5

RESULT AND DISCUSSION

5.1 CHARACTERIZATION OF MgO NANO PARTICLE

5.1.1 FTIR Spectrum MgO Nanoparticle

The FTIR spectrum of Magnesium oxide nanoparticles are shown below. The IR vibrational frequency obtained at 3849 cm^{-1} , 2300 cm^{-1} , 1417 cm^{-1} and 480 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 480 cm^{-1} is the characteristic peak for MgO nanoparticle.(ie: Metal oxide peak).

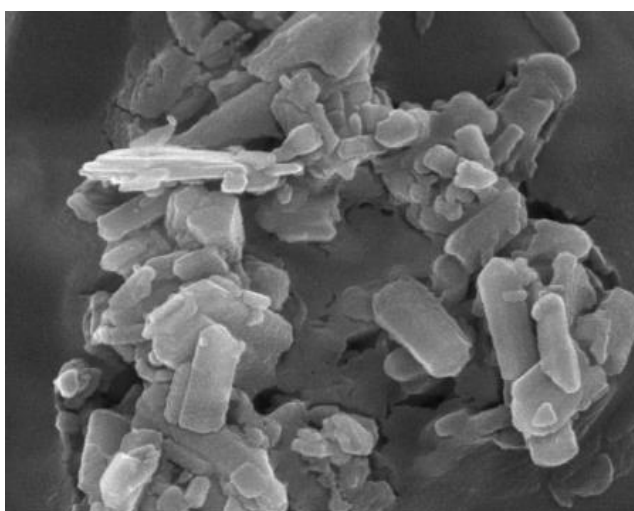


FTIR spectrum of MgO Nanoparticle

Vibrational frequency	Functional group identification
3850 cm^{-1}	O-H stretching of absorbable water molecule on magnesium oxide
2300 cm^{-1}	C=O stretching
1417 cm^{-1}	C-O
480 cm^{-1}	Mg-O stretching

5.1.2 SEM image of MgO Nanoparticle

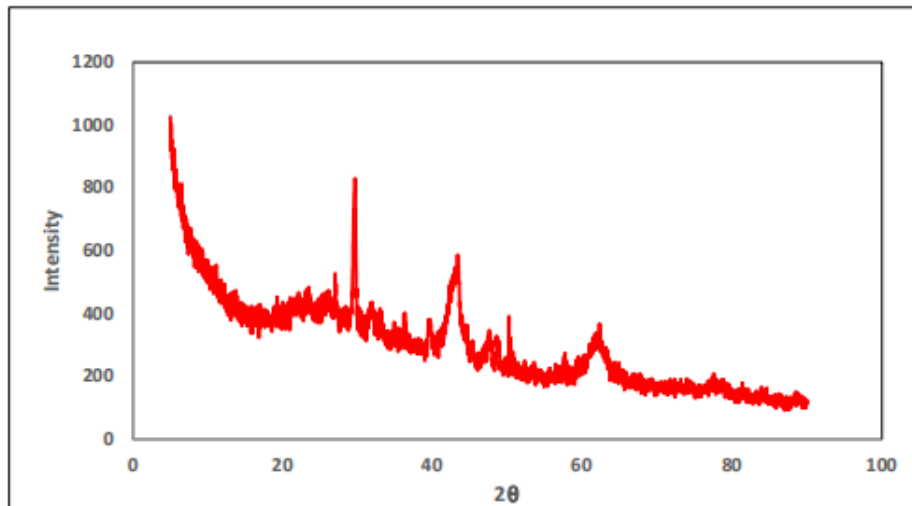
The SEM image of Magnesium oxide nanoparticle is shown in the picture. The image explains that magnesium oxide nanoparticle obtained have spherical shape. The SEM image also shows the presence of agglomerates of MgO nanoparticles. The result revealed size of 23 nm and has a spherical hexagonal shape.



SEM image of MgO NanoParticle

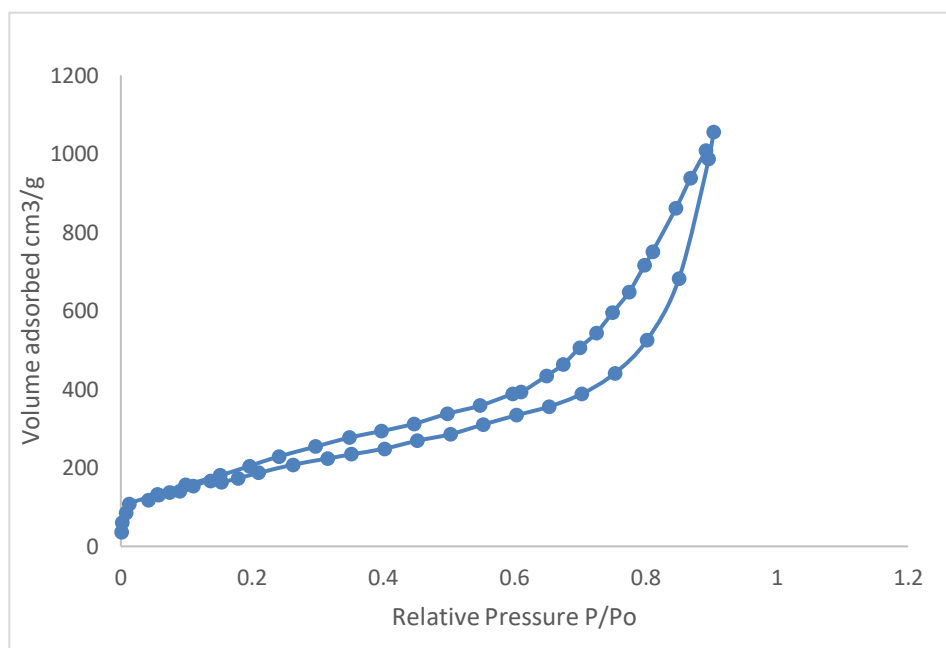
5.1.3 XRD pattern of MgO Nanoparticle

The XRD pattern of Magnesium oxide nanoparticle is shown in the diagram. X-ray diffraction. The peak at the 41 and 61 ascribing to MgO were found in MgO.



XRD pattern for MgO nanoparticles

5.1.4 BET Surface area of MgO Nanoparticle



The pore distribution and surface area of MgO nanoparticles were determined using an adsorption equipment maintained at 77 K (Quantachrome Autosorb Instrument - IQ Analyzer). The experimental samples of nanoparticle were degassed at a temperature of 200°C for 2 hour, prior to every analysis and the sample was allowed to run in triplicates. The surface distribution and porosity are essential parameters to estimate the performance efficiency. The resulting MgO had a relatively large surface area, it should be indicated. Typically, MgO nanostructures feature a surface area ranging from 3.4 m² /g to 17.6 m² /g, The surface area (69.8 m² /g).

5.2 FREUNDLICH ADSORPTION ISOTHERM:

Applying the Freundlich isotherm, the acenaphthene absorption on the magnesium oxide nanoparticles is analyzed. In general, the Freundlich isotherm is 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}),

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

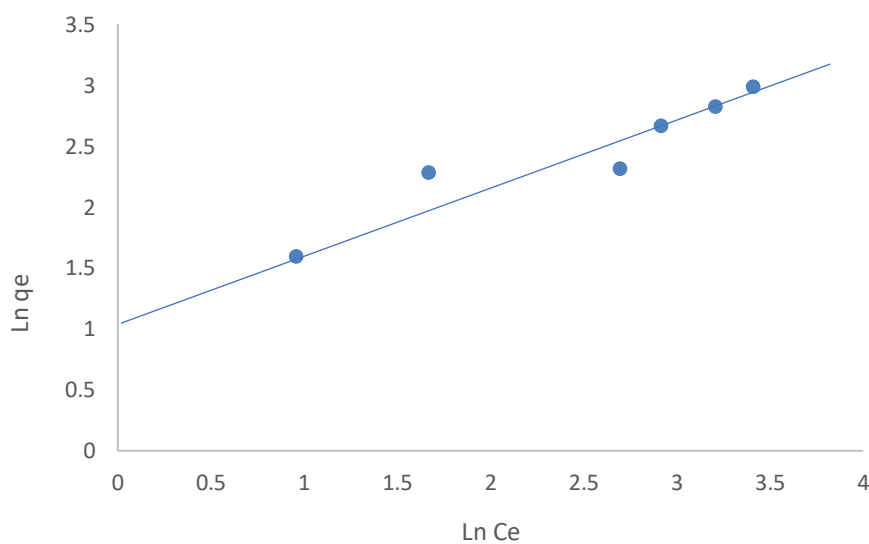
$$\ln q_e = \ln k_f + 1/n \ln C_e$$

The graph for the adsorption of acenaphthene on the magnesium oxide nanoparticles showed the correlation between $\ln q_e$ and $\ln C_e$. The slope and intercept of the liner plot are used to calculate k_f and n . The table displays the Freundlich adsorption isotherm parameter for acenaphthene removal from the aqueous solution using the adsorbent. In this case, the regression coefficient showed that the Freundlich isotherm gave a better fit. Magnesium oxide nanoparticle adsorbent surface heterogeneity.

This table shows the effect of time on stream for the removal of acenaphthene by using Magnesium oxide nanoparticle sample.

ANALYSIS OF ADSORPTION ISOTHERM OF ACENAPHTHENE

S.N o	C _o (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	2.6	0.3846	0.41497334	0.95551144	4.933333333	0.202702703	1.596014892
2	20	5.3	0.1886	0.72427586	1.66770682	9.8	0.102040816	2.282382386
3	30	14.8	0.0675	1.17026171	2.69462718	10.13333333	0.098684211	2.31583032
4	40	18.4	0.0543	1.26481782	2.91235066	14.4	0.069444444	2.667228207
5	50	24.7	0.0404	1.39269695	3.20680324	16.86666667	0.059288538	2.825339288
6	60	30.2	0.0331	1.48000694	3.40784192	19.86666667	0.05033557	2.989043285

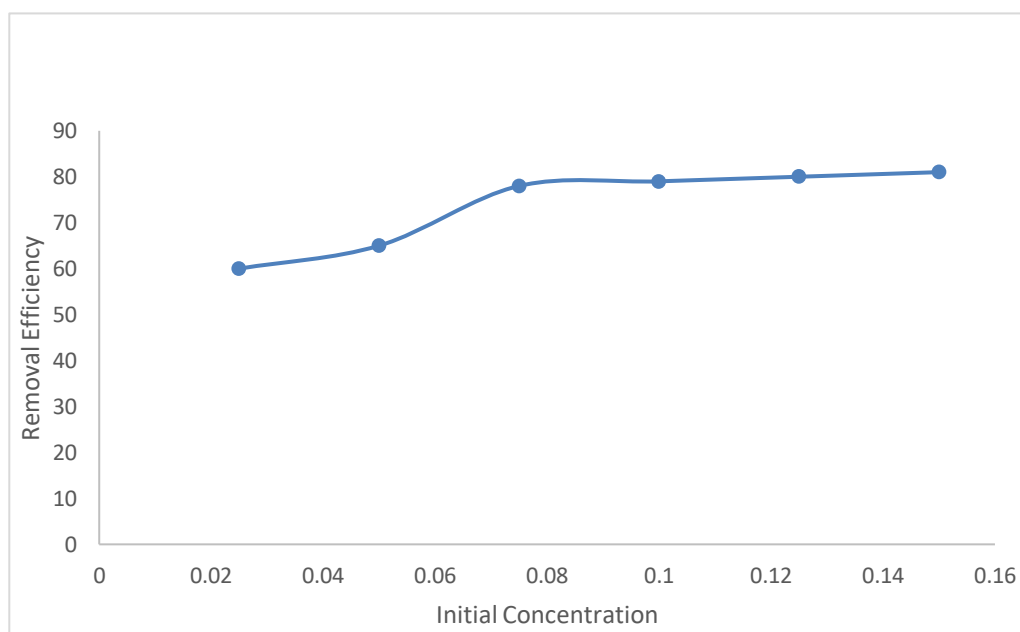


Freundlich Adsorption isotherm of Acenaphthene

5.3 EFFECT OF ADSORPTION PARAMETERS

Effect of Acenaphthene on initial concentration

About 2.5 mg, 5mg, 7.5mg, 10 mg, 12.5 mg and 15 mg from the concentration is added to 25ml standard flask and kept in the shaking incubator for 1hour.



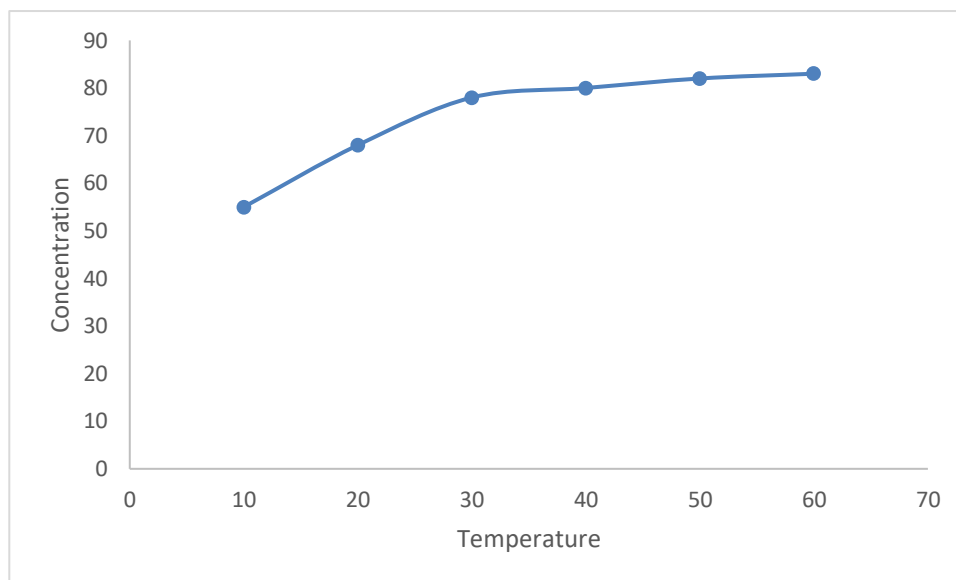
Effect of acenaphthene initial concentration

Effect of Temperature on removal efficiency

The adsorption of acenaphthene 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 (40, 50, 60, 70, 80 and 90) °C adsorbent amount 0.05 gm of catalyst and acenaphthene drug concentration, 25ml utilizing water bath at 60°C.

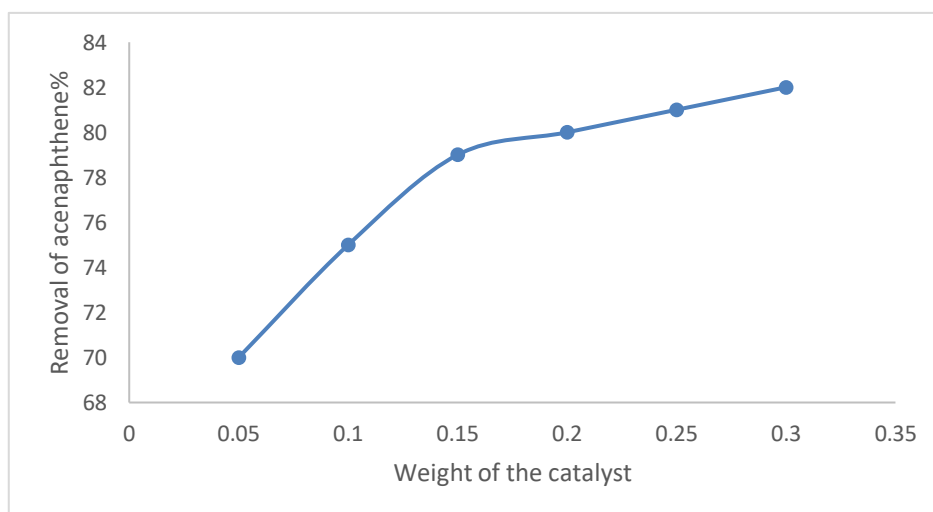
the experiment was carried at the pH of 6 with the time of 1hour at a constant speed of 120 rpm.



Effect of temperature on removal efficiency

Effect of amount of catalyst

The effect of adsorbent amount was studied through stirring of several weight (0.05, 0.1, 0.15, 0.2, 0.25, 0.3) gm, with drug concentration of 30ppm in 100ml utilizing water bath. The experiment was showed at pH 6 contact time at 1hour a constant speed at 120 rpm.

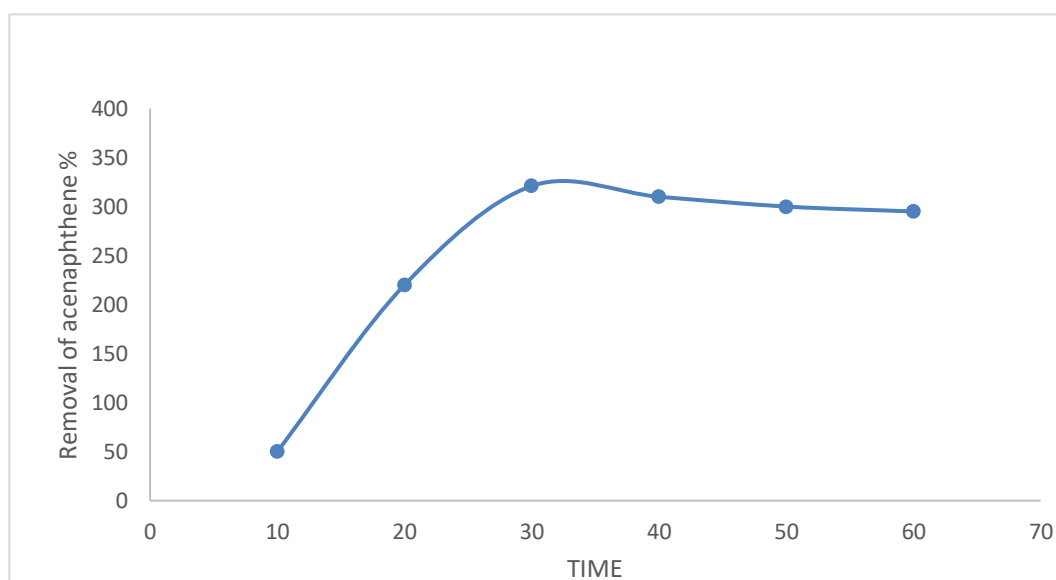


Effect of weight of catalyst amount

Effect of Time on stream

We showed that the acenaphthene 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 acenaphthene is rapid.

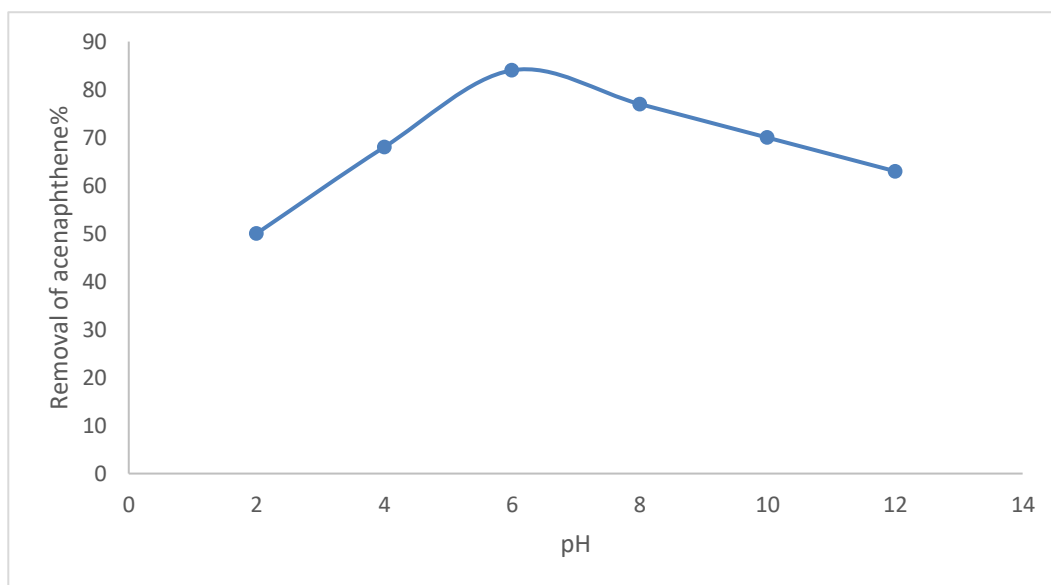
This is may be due to the high number of active sites and due to the highest adsorption capacity if the nanotubes at the beginning of the adsorption. We observed that the acenaphthene in the aqueous solution increases sharply for the first 30 minutes and then decrease.



Effect of time on stream

Effect of pH on adsorption of acenaphthene

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 acenaphthene also increases. However, by increasing the pH from 7 to 12, the acenaphthene adsorption decreases, these may due to the oxygen containing groups were deionized.



Effect of pH on adsorption of acenaphthene

CHAPTER 6

SUMMARY AND CONCLUSION

In summary, MgO nanoparticles was synthesized using magnesium nitrate, neem extract and sodium hydroxide through green synthesis method. To reduce the acidity Sodium hydroxide is used and its catalytic activity were studied by adsorption isotherm.

Crystalline structure and size was confirmed by XRD. The size of the MgO nanoparticles was calculated using Debye Scherrer formula. XRD pattern revealed that MgO has high crystalline nature. MgO stretching was confirmed by the functional group analysis.

The functional groups present in the product and its structure was confirmed by the spectral studies. This included Adsorption isotherms effect of temperature, pH, initial concentration, weight, time on stream. Freundlich isotherm was fitted best.

The SEM demonstrated the synthesized nanoparticles were in size of 23 nm.

Organic functional groups present on the surface of nanoparticles were identified using FT-IR spectroscopy.

The BET isotherm estimated the surface area and pore size of Magnesium Oxide nanoparticles. MgO nanostructures feature a surface area ranging from $3.4 \text{ m}^2\text{g}^{-1}$ to $17.6 \text{ m}^2\text{g}^{-1}$. The surface area ($69.8 \text{ m}^2\text{g}^{-1}$).

Fitted best results from Freundlich adsorption isotherm; Optimised pH at 6, temperature at 60°C , initial concentration at 0.15, on 30 minutes increases sharply and then decreases.

Therefore, the synthesized Magnesium Oxide nanoparticles are very efficient in all fields.

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