

**REMOVAL OF NAPHTHALENE FROM AQUEOUS
SOLUTION USING ZnO NANOPARTICLES
BY NEEM EXTRACT SYNTHESIS**

Thesis submitted
in partial fulfilment of the
requirements for the award of
MASTER OF SCIENCE IN CHEMISTRY

By

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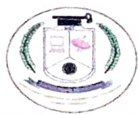
MSc., Chemistry (2021-23)



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This is to certify that this project report is the bonafide work of V.Suchitra (41910020) "REMOVAL OF NAPHTHALENE USING ZnO NANOPARTICLES BY NEEM EXTRACT SYNTHESIS" who carried out the project entitled under our supervision from November 2022 to May 2023.

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ABSTRACT

Zinc oxide nanoparticles have been synthesized by neem extract. FTIR, XRD, and SEM with EDAX were used to characterize the synthesized nanoparticle. And it shows that zinc oxide nanoparticles as a hexagonal wurtzite structure. The SEM analysis confirms the formation of spherical particles which are agglomerated. FTIR confirms the presence of Zn-O stretching bond. BET Isotherm theory is used to evaluate the adsorption data and generate a specific surface area result. 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 Naphthalene in aqueous solution after adsorption was studied by UV-Visible Spectroscopy.

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LIST OF ABBREVIATION

FTIR	- FOURIER TRANSFORM INFRARED SPECTROSCOPY
SEM	- SCANNING ELECTRON MICROSCOPE
BET	- BRAEUEER-EMMETTE-TELLER
XRD	- X-RAY DIFFRACTION
UV-VIS	-ULTRAVIOLET VISIBLE
NM	-NANOMETER
0D	-ZERO DENSITY
1D	-ONE DIMENSIONAL
2D	-TWO DIMENSIONAL
3D	-THREE DIMENSIONAL
NP	-NANOPARTICLE
CNT	-CARBON NANOTUBES
ZNO	-ZINC OXIDE
MM	-MILLIMETRE
PPM	-PARTS PER MILLION
RPM	-REVOLUTIONS PER MINUTE
PH	-POTENTIAL OF HYDROGEN
DNA	-DEOXYRIBONUCLEIC ACID
MMNC	-METAL MATRIX NANOCOMPOSITES
CMNC	-CERAMIC MATRIX NANOCOMPOSITES
PMNC	-POLYMER MATRIX NANOCOMPOSITES

CHAPTER 1

INTRODUCTION

1.1 METAL OXIDE

Metal oxides and mixed metal oxides have numerous uses in medicine as well as being catalysts, adsorbents, superconductors, semiconductors, ceramics, and antifungal agents.

In several fields of chemistry, physical science, and materials science, metal oxides are extremely significant.

Metal ions have a tendency to coordinate, which causes oxide ions to form a coordination sphere around the metal ions and result in a densely packed structure, which leads to the formation of metal oxides.

Metal/Mixed metal oxides have wide applications as catalyst because of their high surface area and reactive sites. Number of scientists and academicians are using metal/mixed metal oxides as catalyst in various organic reactions.

The transition metals and their compounds are used as catalysts in chemical industry and in battery industries.

Besides, these compounds can be used in formation of interstitial compounds and alloy formation. The transition metals have the special properties of formation of coloured compounds and show magnetic properties.

Metals of d-block elements are used for many industrial applications. They behave as catalysts, superconducting materials, sensors, ceramics, phosphors, crystalline lasers etc.

For example, zinc oxide sintered together with other metal oxide additives have been made into nonlinear resistors, which are called Varistors for surge suppressing function. The suppressing function has been applied for switching and for protection of random voltage protections.

1.2 NANOPARTICLES:

We now live in a time of rapid scientific innovation known as nanotechnology. The typical particle size that surrounds a sample is between 1 and 1000 nm (1 nm = 10^{-9} m). Nanoparticles can mean several different things. One of them claims that a material can be classified as “nanomaterial” if at least one measurement is more than 100 nm.

Another meaning of “nanomaterial” is a substance that, after being “miniaturized” to the nanoscale, exhibits altered physicochemical qualities in comparison to a mass-produced item. (naturally visible). In contrast to their particular particles at bigger scales, the nanoparticles exhibit unique physical, chemical, and biological features at the nanoscale.

This happens because of a larger surface area to the volume, increase in reactivity or stability in a chemical reaction, improved mechanical strength, and so on. These properties of nanoparticles have prompted its utilization in different applications.

The nanoparticles differ from shapes, sizes and structure. It can vary from 1 nm to 100nm in size.

The surface can be uniform or unpredictable with surface varieties. Some nanoparticles are translucent or shapeless with single or multi gem solids either free or agglomerated. Various synthesis techniques are either being created or improved to upgrade the properties and lessen the production costs.

A few methodologies have been developed to quantify explicit nanoparticles in order to improve their physical, chemical, optical, and mechanical properties. A significant increase in instrumentation has resulted in improved nanoparticle 2rganized2zation and application.

1.3 TYPES OF NANOPARTICLES:

Nanomaterials differ from bulk materials in several ways, including their vast surface area, capacity to customize their functions for specific applications, and high surface energy.

1.3.1 ZERO-DIMENSIONAL NANOPARTICLE:

Compare with bulk high-dimensional nanomaterials, 0D nanomaterials are mostly spherical or quasi-spherical nanoparticles with a diameter of less than 100 nm.

1.3.2 ONE-DIMENSIONAL NANOPARTICLE:

In the nanoscale range, atoms are 3rganized in one dimension. The nanoscale is only one dimension. Nanowires, nanorods, and nanotubes thin films and coatings are some of the examples.

1.3.3 TWO-DIMENSIONAL NANOPARTICLES:

Atoms are arranged in two-dimensional nano scale range. Examples are nanotubes, biopolymers, nanowires.

1.3.4 THREE-DIMENSIONAL NANOPARTICLES:

Atoms are arranged in three-dimensional nano scale range. Examples are fullerenes.

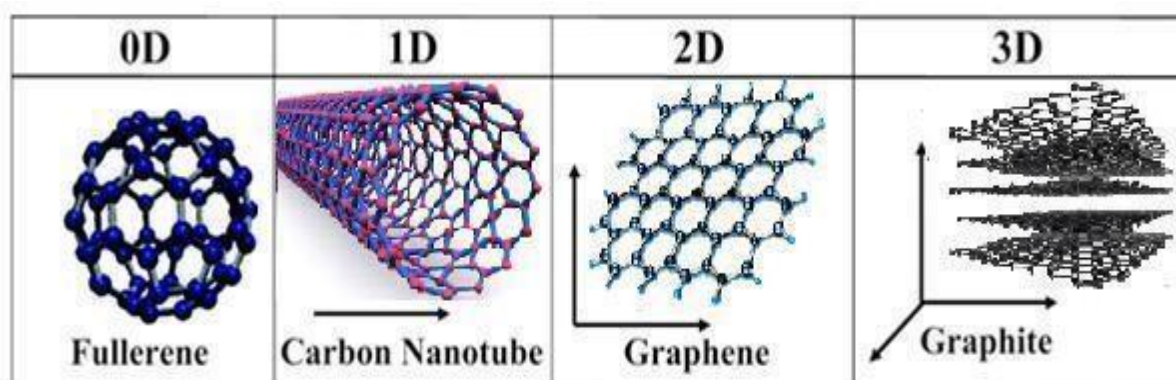


Fig 1.3 Types of nanoparticles

1.4 CLASSIFICATION OF NANOPARTICLES:

1.4.1 CARBAN-BASED NANOPARTICLES:

Two important kinds of carbon-based NPs are fullerenes and carbon nanotubes (CNTs). Nanomaterials comprised of globular hollow cages, like allotropic forms of carbon, are found in fullerenes. Due to their electrical conductivity, high strength, structure, electron affinity, and adaptability, they have generated a significant amount of economic interest. These materials have pentagonal and hexagonal carbon units that are organized and each of which is sp^2 hybridized. Some well-known fullerenes with the formulas C_{60} and C_{70} with diameters of 7.114 and 7.648 nm, respectively.

As a result, they are referred to as single-walled (SWNTs), double-walled (DWNTs), or multi-walled carbon nanotubes (MWNTs). Individually, they are produced by an electric arc on metal nanoparticles and carbon precursors. by using Chemical Vapour Deposition, they are created. (CVD).

These materials are used in a variety of applications, such as filler and as organic and inorganic catalyst, because of their distinctive physical, chemical, and mechanical qualities.

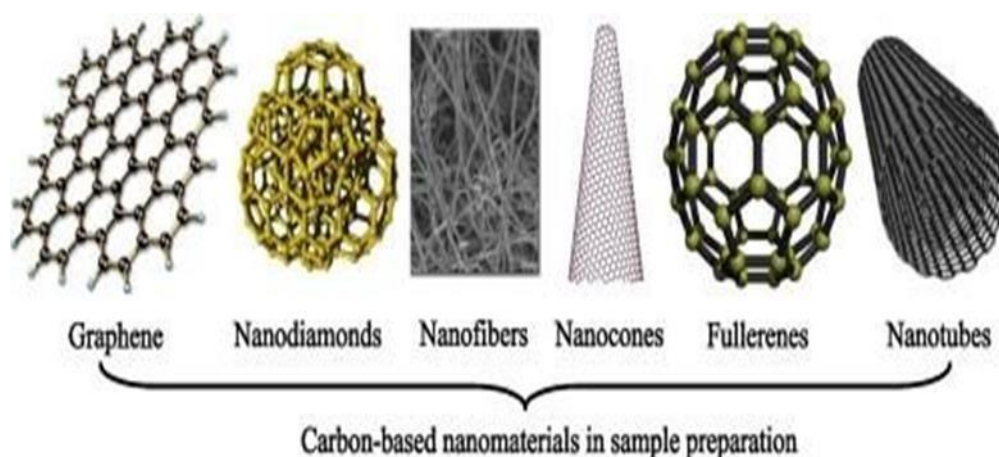


Fig 1.4 Carbon based nanomaterials

Nanomaterials have a variety of significant uses, including in the fields of aerospace and chemical engineering, optics, solar hydrogen, fuel cells, batteries, sensors, power generation, automotive engineering, consumer electronics, thermoelectric devices, and pharmaceuticals.

These materials have drawn interest in numerous fields, including biomedical applications, because of their exceptional mechanical, electrical, thermal, optical, and chemical capabilities and distinctive structural dimensions.

1.4.2 CERAMIC NANOPARTICLES:

These solid inorganic nanoparticles are made up of oxides, carbides, carbonates, and phosphates. Ceramic nanoparticles are used in biological imaging, drug administration, photocatalysis, and dye photodegradation because of their great heat resistance and chemical inertness.

By controlling their specific characteristics like size, surface area, porosity, surface-to-volume ratio, these nanomaterials perform as good drug delivery agents.

Ceramic nanoparticles have been used effectively as a drug delivery system for several diseases like bacterial infections, glaucoma, and cancer.

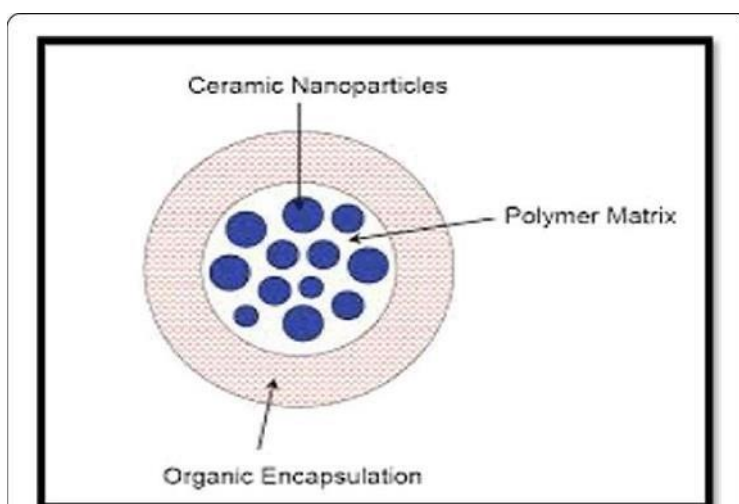


Fig 1.4.2 Ceramic nanoparticles

1.4.3 SEMICONDUCTORS:

Semiconductor nanoparticles are found in groups II-VI, III-V, or IV-VI of the periodic table and exhibit characteristics similar to those of both metals and non-metals. Wide bandgaps in these nanoparticles allow for the tweaking of many characteristics. GaN, GaP, InP, and InAs from groups III to V are some examples of semiconductor nanoparticles, as are ZnO, ZnS, CdS, CdSe, and CdTe from groups II to VI and silicon and germanium from group IV.

1.4.4 POLYMERIC NANOPARTICLES:

Polymeric nanoparticles are organic-based nanoparticles. Depending upon the preparation method, polymeric nanoparticles have structures shaped like nano-capsules or nanospheres.

A nanosphere nanoparticle has a matrix-like structure, whereas nano-capsules have core-shell morphology. In nanosphere polymeric nanoparticles, the active compounds and the polymer are uniformly dispersed while in nano-capsule nanoparticles, the active compounds are confined and surrounded by a polymer shell.

Some of the advantages of polymeric nanoparticles include controlled release, protection of drug molecules as they travel within the internal and external environment, the ability to combine therapy and imaging, and specific targeting.

Polymeric nanoparticles have applications in drug delivery and diagnostics. Drug delivery systems with polymeric nanoparticles are also highly biodegradable and biocompatible.

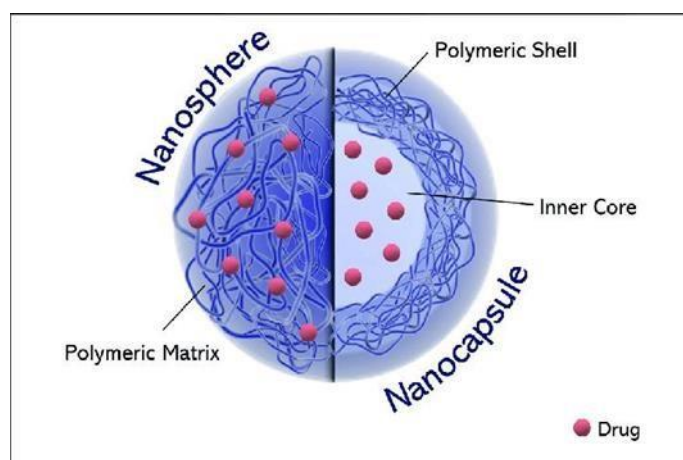


Fig1.4.4 Polymeric nanoparticles

1.4.5 LIPID-BASED NANOPARTICLES:

Lipid nanoparticles are generally spherical with a diameter ranging from 10 to 100nm. Their structure consists of a solid core made of lipid and a matrix containing soluble lipophilic molecules, and the external core is stabilized by surfactants and emulsifiers.

Lipid nanoparticles have applications in the biomedical field as drug carriers and RNA release in cancer therapy.



Fig 1.4.5 Lipid Based Nanoparticles

1.4.6 METAL-OXIDES NANOPARTICLES:

Metal precursors are used to create metal nanoparticles, which can then be produced chemically, electrochemically, or photochemically. By reducing the metal-ion precursors in solution using chemical reducing agents, metal nanoparticles are produced via chemical techniques. The resulting nanostructures have a high surface energy and can adsorb tiny molecules.

Metal nanoparticles are used in a variety of scientific disciplines, including biomolecular imaging and detection as well as environmental and analytical applications. For example, to improve SEM and provide excellent electron microscopy pictures, gold nanoparticles are employed to coat the sample prior to SEM examination.

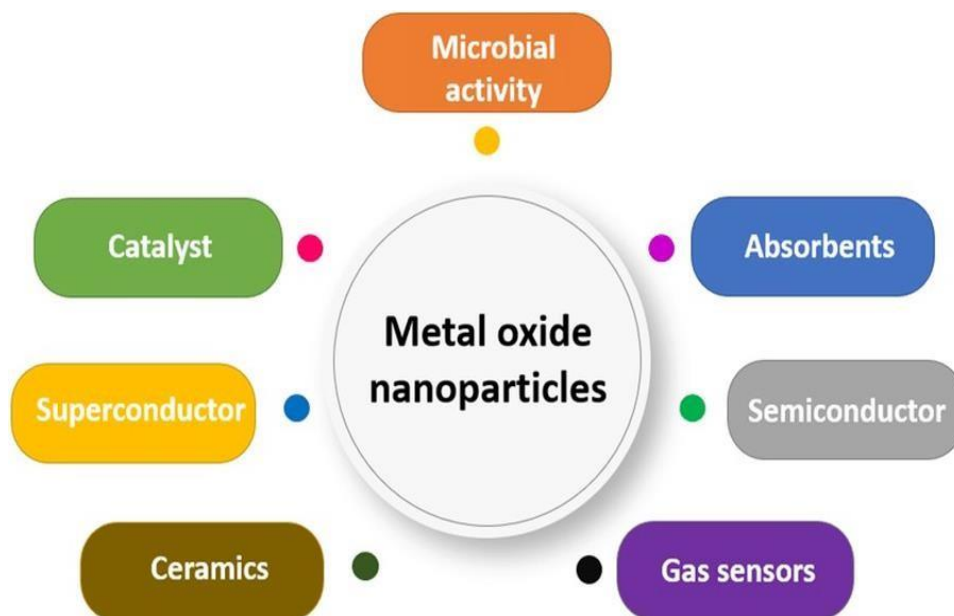


Fig 1.4.6 Metal Oxide Nanoparticles

1.4.7 ZINC OXIE NANOPARTICLES:

Zinc oxide nanoparticles are nanoparticles of zinc oxide (ZnO) that have diameters less than 100 nano meters. They have a large surface area relative to their size and high catalytic activity. The exact physical and chemical properties of zinc oxide nanoparticles depend on they are synthesized.

Laser ablation, hydrothermal processes, electrochemical depositions, the sol-gel method, chemical vapour deposition, thermal decomposition, two-step

mechanochemical-thermal synthesis, anodization, co-precipitation, electrophoretic deposition, and precipitation methods using solution concentration, pH, and washing medium are some of the potential production methods for ZnO nanoparticles. At ambient temperature, the wide-bandgap semiconductor ZnO has an energy gap of 3.37 eV. ZnO nanoparticles are one of the most produced nanomaterials, along with titanium dioxide nanoparticles and silicon dioxide nano particles. The most common use of ZnO nanoparticles is in sunscreen. They are used because they effectively absorb ultraviolet light, but possess a large enough bandgap to be completely transparent to visible light.

Additionally, its ability to eradicate dangerous germs from packaging and UV-protective materials like textiles is being studied. Since many businesses fail to

disclose the presence of nanoparticles in their products, it is challenging to estimate their production and widespread use in consumer goods.

Because of their small size, they are rapidly absorbed by tissues, making detection challenging. However, until damage occur, human skin is an efficient barrier to ZnO nanoparticles when used as a sunscreen, for example. ZnO nanoparticles may enter the system from inadvertent absorption of small amounts while applying sunscreen. When sunscreen is rinsed away, ZnO nanoparticles can enter runoff water and go up the food chain.

Since ZnO nanoparticles are a relatively novel substance, their potential for harm is under discussion. Due to their small size, nanoparticles may often move throughout the body and have been demonstrated in animal tests to pass through the blood-brain barrier, the placenta, individual cells, and their nuclei.

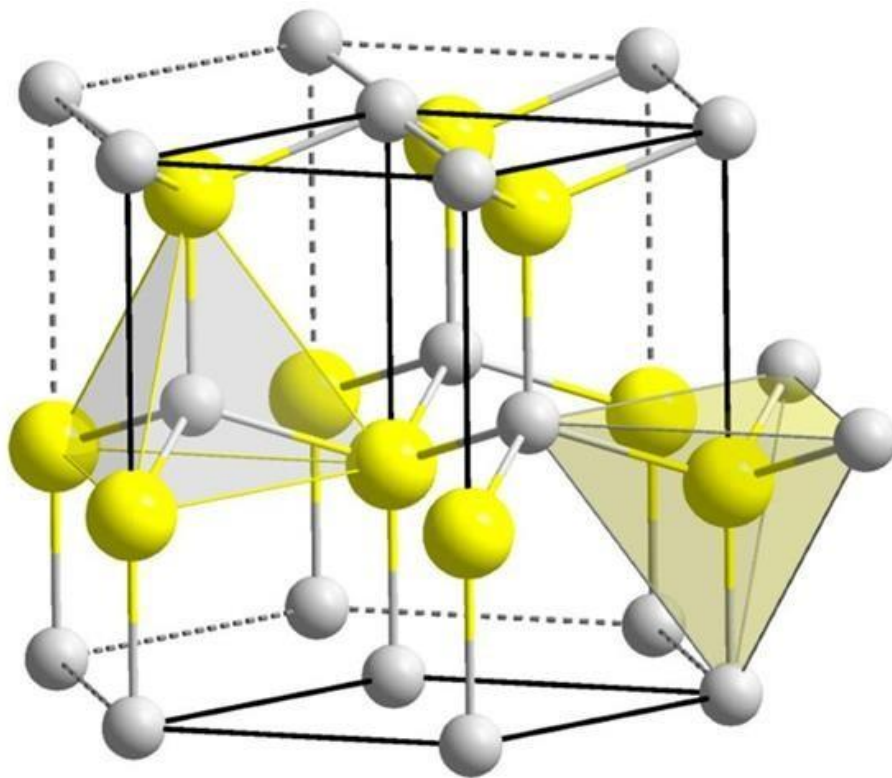


Fig 1.4.7 Zinc Oxide Nanoparticles

1.5 NANOCOMPOSITES:

A nanocomposite combines two or more materials – of which at least one is a nanomaterial – with different physical and chemical properties. Nanocomposite materials are designed to exhibit properties that exceed, sometimes drastically, the capabilities of the sum of their constituent parts.

As in the case of micro composites, nanocomposite materials can be classified, according to their matrix materials, in three different categories.

- Ceramic Matrix Nanocomposites (CMNC);
- Metal Matrix Nanocomposites (MMNC)
- Polymer Matrix Nanocomposites (PMNC).

1.5.1 CERMARIC MATRIX NANOCOMPOSTION:

The most common methodologies, as used for micro composites, are Conventional powder method; Polymer precursor route; Spray pyrolysis; Vapour techniques (CVD and PVD) and Chemical methods, which include the sol-gel process, colloidal and precipitation approaches and the template synthesis.

1.5.2 METAL MATRIX NANOCOMPOSTION:

Metal matrix composites (MMCs) or nanocomposites (MMNCs) are a class of advanced materials and structures wherein a light-weight metal such as aluminum or magnesium alloy is reinforced by high-strength reinforcing particles in order to produce light-weight high-strength composite materials.

1.5.3 POLYMER MATRIX NANOPARTICLES:

Polymer matrix composites are used as components in a wide range of medical devices; such as MRI scanners, C scanners, X-ray couches, mammography plates, tables, surgical target tools, wheelchairs, and prosthetics.

1.6 ZINC OXIDE:

Zinc Oxide is an inorganic compound which is also known as Calamine or Zinc White. It is naturally found as a mineral zincite. It is mostly produced synthetically. It is a chemical compound with mild astringent and antiseptic action which works as a topical protectant. It has wide applications in bandages, ointments, pastes, and dental cement.

Zinc white crystallizes mainly in two forms viz cubic zinc blende and hexagonal wurtzite. The most common and stable structure under ambient conditions is wurtzite. Zincblende can be stabilized by growing zinc oxide on substrates which have a cubic lattice structure. The oxide and zinc centres are tetrahedral.

Calamine is a white solid which is insoluble in water and has no smell. Crude zinc oxide appears yellow-grey in colour and exists in a granular solid form with no odor. In its natural form, it is obtained as a mineral zincite which consists of manganese (Mn) and some other impurities which make it appear yellow-red color. In its crystalline form, it is thermochromic which on heating in the presence of air changes its color from white to yellow and on cooling it turns white in color. It is an amphoteric oxide which is insoluble in water. It dissolves in acids and alkalis.

People allergic to ZnO should not use products made of zinc oxide. It may lead to skin irritation, redness of the skin or worsening rashes.

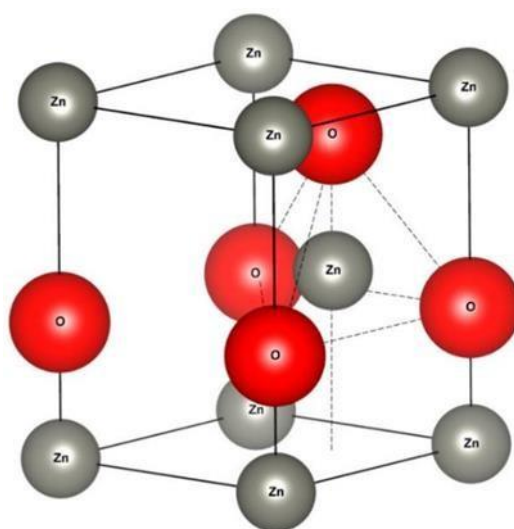
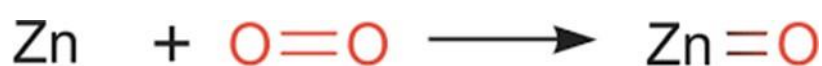


Fig 1.6 Zinc Oxide

1.7 APPLICATION OF NANOPARTICLES:

- Nanoparticles are now being used in the manufacture of scratchproof eyeglasses, crack-resistant paints, anti-graffiti coatings for walls, transparent sunscreens, stain-repellent fabrics, self-cleaning windows and ceramic coatings for solar cells.
- Nanoparticles are widely used in drug delivery system. Certain drugs are not metabolized in the first pass. To resolve this, nanoparticles can be modified, and they also allow for regulated, long-term drug release from the matrix.
- When they're adhered to short DNA segments, they can also be used to detect genetic sequences. Nano-technology allows damaged tissue to be repaired or replicated. The use of nanotechnology can revolutionise organ transplantation.
- The need for new antimicrobial agents has risen. Because of their inbuilt antimicrobial properties, nanoparticles (NPs) are now being considered as an effective nanotechnology for regulating harmful micro-organisms.

CHAPTER 2

LITERATURE SURVEY

Amrutha S. (11 Jan 2020) Green Synthesis of Zinc Oxide Nanoparticles using Neem (*Azadirachta indica*) and Tulasi (*Ocimum tenuiflorum*) Leaf Extract and their Characterization. The bio-active molecules present in the plant extracts are utilized for the synthesis of zinc oxide nanoparticles from its bulk molecules. A study was carried out to synthesis zinc oxide nanoparticles from neem (*Azadirachta indica*) and tulasi (*Ocimum tenuiflorum*) leaf extracts.

Ashwini Jayachandran (November 2021) Green synthesis and characterization of zinc oxide nanoparticles using *Cayratia pedata* leaf extract. The nanoparticles were successfully synthesized using the Nitrate derivative of Zinc and plant extract of the indigenous medicinal plant *Cayratia pedata*. 0.1 mM of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was made to react with the plant extract at different concentrations, and the reaction temperature was maintained at 55 °C, 65 °C, and 75°C. The yellow coloured paste obtained was wholly dried, collected, and packed for further analysis. In the UV visible spectrometer (UV–Vis) absorption peak was observed at 320 nm, which is specific for Zinc oxide nanoparticles.

Agarwal, S. (March 2017) Green synthesis of zinc oxide nanoparticles – An eco-friendly approach. Zinc oxide nanoparticles (ZnO NPs) had been in recent studies due to its large bandwidth and high exciton binding energy and it has potential applications like antibacterial, antifungal, anti-diabetic, anti-inflammatory, wound healing, antioxidant and optic properties. Due to the large rate of toxic chemicals and extreme environment employed in the physical and chemical production of these NPs, green methods employing the use of plants, fungus, bacteria, and algae have been adopted.

Dandan Liu, Zhansheng Wu (August 2016) La and N co-doped TiO_2 nanoparticles supported on activated carbon (TiO_2/AC) were synthesized through a microwave-assisted sol–gel method for the synergistic removal of naphthalene solution by photocatalytic degradation. The results provided valuable information on the mechanism of naphthalene decomposition.

Elvis Fosso Kankeu, Ajay Kumar Mishra(10 October 2017) There has been significant development of nanotechnology and expansion of the application in various sectors of activity including water treatment. The application of nanotechnology currently allows the removal of recalcitrant pollutants through adsorption or photocatalytic degradation. Metal oxide nanomaterials, as well as carbon nanoparticles exhibit suitable properties for photocatalytic degradation and adsorption processes.

Harshiny Muthukumar, Ashish Gire(April 2019) Biogenic synthesis of nano-biomaterial for toxic naphthalene photocatalytic degradation optimization and kinetics studies. ZnO nanoparticles were synthesized using *Amaranthus dubius* aqueous leaf extract as a reducing agent. The physicochemical properties of the nanoparticles such as size, shape, surface area and band energy were determined by analytical techniques. The characterization confirmed that Fe single bond ZnO were spherical shape with wurtzite phase structure, smaller size, and large surface with less aggregation. The photocatalytic efficiency of ZnO and ZnO were evaluated by degradation of naphthalene. The effects of various operating parameters including solution pH, photocatalyst and naphthalene concentration on the degradation efficiency and the rate of degradation were investigated.

Huanhuan Wang, Peijiang Zhou (March 2018) Synthesis of Rectorite/Fe₃O₄/ZnO Composites and Their Application for the Removal of Methylene Blue Dye. The adsorption isotherm of the dye on the composite fitted well to the Langmuir model, with a maximum adsorption of 35.1 mg/g.

MuthuKathija.M (June 2022) Green synthesis of zinc oxide nanoparticles using *Pisonia Alba* leaf extract and its antibacterial activity. The reducing and capping agent of *Pisonia Alba* leaf extract were used in a green production of zinc oxide (ZnO) nanoparticles. UV–Vis, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy with EDS (SEM) were all used to examine the produced ZnO nanoparticles. UV–Vis spectroscopy verified the synthesis of ZnO nanoparticles and their resulting optical characteristics. The UV–Vis spectra showed that the ZnO nanoparticles formed at an absorption peak maximum of 378 nm.

Nthambeleni Mukwevho (January 2021) Removal of naphthalene from simulated wastewater through adsorption- photodegradation by ZnO/Ag/GO nanocomposite. ZnO/Ag/GO nanocomposite was synthesised and used as photocatalyst for effective photodegradation of naphthalene from simulated wastewater under visible light. Adsorption study of naphthalene prior to photocatalysis using synthesised material was thoroughly done by studying the kinetics and adsorption isotherm models.

Pranjali.P(March 2017) Synthesis and characterization of zinc oxide nanoparticles by using polyol chemistry for their antimicrobial and antibiofilm activity. The present investigation deals with facile polyol mediated synthesis and characterization of ZnO nanoparticles and their antimicrobial activities against pathogenic microorganisms.

Tamanna Bhuyan(May 2015) Biosynthesis of zinc oxide nanoparticles from *Azadirachta indica* for antibacterial and photocatalytic application. Lowcost, green synthesis of zinc oxide (ZnO) nanoparticles using 25% (w/v) of *Azadirachta indica* (Neem) leaf extract. The antibacterial activity of characterized samples was determined using different concentrations of biosynthesized ZnO nanoparticles (20 mg/mL, 40 mg/mL, 60 mg/mL, 80 mg/mL and 100 mg/mL).

Thana Shuga Aldeen(March 2022) ZnO nanoparticles prepared via a green synthesis approach: Physical properties, photocatalytic and antibacterial activity. The physical properties of the nanoparticles were examined using X-ray powder diffraction (XRD), and Fourier transform infrared (FTIR) spectroscopy. XRD investigations revealed that the ZnO NPs have a single phase wurtzite structure with P6₃mc space group.

Yasir Iqbal(December 2021) Green synthesis of ZnO and Ag-doped ZnO nanoparticles using *Azadirachta indica* leaves: Characterization and their potential antibacterial, antidiabetic, and wound-healing activities. ZnO nanoparticles (NPs) using the aqueous extract of *Azadirachta indica* (Neem) leaves as the reducing agent. Surface morphology was evaluated with scanning electron microscopy while crystalline size confirmed by X-ray diffractometer.

CHAPTER 3

AIM AND SCOPE

3.1 AIM:

The main aim is to remove the Naphthalene from aqueous solution using synthesized zinc oxide nanoparticle. ZnO nanoparticles were synthesized by neem extract. ZnO was prepared by using zinc acetate, sodium hydroxide and neem extract.

And to study Freundlich the adsorption isotherm and Optimization of reaction parameters of the Effect of Initial concentration of Naphthalene, Effect of Temperature, Effect of Ph, and Time of stream. ZnO nanoparticles were characterized with the help of FTIR, XRD, SEM .

3.2 SCOPE:

- Synthesize of ZnO by green synthesis using neem extract.
- Characterization of ZnO by FTIR spectroscopy, XRD, SEM and UV visible spectroscopy.
- Removal of naphthalene from aqueous solution by using ZnO Nanoparticles.
- To study Adsorption Isotherm.
- Optimization of reaction parameters such as the Effect of Initial concentration of Naphthalene, Effect of Temperature, Effect of pH and Time on stream.

CHAPTER 4

MATERIAL AND METHODOLOGY

4.1 MATERIALS:

In this process of synthesizing zinc oxide nanoparticle, zinc acetate, sodium hydroxide, deionized water and neem leaf are used.

4.1.1 PREPARATION OF NEEM EXTRACT:

Fresh neem leaf is washed and rinsed and dried for 3 Days. 5 gram of neem leaf in 100 ml distilled water is boiled at 80°C and stirred for 30 minutes. The boiled solution is cooled and filtered.

4.1.2 PREPARATION OF ZnO NANOPARTICLES:

30ml of leaf extract is mixed with 21.4 gms zinc acetate solution. And it is boiled at 80°C for 30 minutes and NaOH solution is added to adjust the pH and heated at 70°C for 30 minutes. A white crystalline precipitate of ZnO is obtained which is washed, filtered and dried in oven at 90°C.

4.1.3 REMOVAL OF NAPHTHALENE USING ZINC OXIDE NANOPARTICLES:

Using ZnO nanoparticle as a catalyst the catalytic activity of the synthesized catalyst is studied by adsorption Isotherm. Effect of initial concentration, Effect of pH, Effect of temperature, Effect of amount of catalyst was studied using UV Visible Spectroscopy.

About 0.15 mm from the concentration is added to (10, 20, 30, 40, 50 and 60 ppm) from drug is added to 25ml standard flask and kept in the shaking incubator for 1hr to study the effect of initial concentration. By studying the effect of Temperature solution by carrying various temperature solutions adsorbent amount 0.15 mm of catalyst and naphthalene concentration 30ppm, 100ml utilizing water bath at 60°C. The effect of adsorbent amount was studied through stirring of several weight with the drug concentration of 30ppm in 100ml utilizing water bath. For Time of Stream the naphthalene 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 naphthalene 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. 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.

4.2 CHARACTERIZATION TECHNIQUES:

Crystal structure, morphology, compositional information, and photocatalytic application of the synthesized nanoparticles were characterized using the following instruments. These include X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), Fourier Transform Infra-red spectroscopy (FTIR), UV-Vis spectroscopy.

4.2.1 Powder X-Ray Diffraction (XRD):

XRD is one the characterization technique that reveals the structural properties of nanoparticles. It provides information on the phase, chemical composition, symmetry of unit cells, crystallinity, lattice constant and properties of the nanoparticles. This technique is employed for the identification of both single and multiphase nanoparticles.

XRD is considered as one of the oldest non-destructive methods for the analysis of nanoparticles. The XRD pattern is influenced by the size and shape of the unit cell of the crystal. Consequently, properties of a material are dependent on their structure. Of about 90% solid materials are crystalline in nature and each crystalline has a significant X-ray diffraction pattern which can be used as fingerprint to detect the material.

Diffraction pattern is obtained only when the X-rays are interacted with the crystal. Information like symmetry of the unit cell, stress, strain, size of crystallite and growth orientation etc. are obtained from the diffraction patterns.

4.2.2 SCANNING ELECTRON MICROSCOPE (SEM):

High resolution scanning electron microscope (SEM) is a very useful for the characterization of size and shape of the nanoparticle. The sample preparation and acquisition of images are relatively easy and convenient. Since the SEM image is a two-dimensional (2D) representation of three-dimensional (3D) structures from a certain viewing angle, it does provide a certain amount of 3D detail that can be used to recreate the shape of a basic structure with sub-nm precision using model-based measurement.

Secondary electrons (SEs) and/or backscatter electrons (BSE) produced when the sample is bombarded by the primary electron (PE) beam provide the signal that causes a SEM image to be created.

A SEM's overall accelerating voltage is usually 30 kV. The SE has a limited escape depth in the region of a few nm owing to their low energy below 50 eV, while the BSE has a slightly higher escape depth in the range of a few nm.

The demand for SEM in laboratories to characterize nanoparticulate materials is increasing because of the application of nanoparticles in the commercial products.

Since the SEM image is a two-dimensional (2D) representation of three-dimensional (3D) structures from a certain viewing angle, it does provide a certain amount of 3D detail that can be used to recreate the shape of a basic structure with sub-nm precision using model-based measurement.

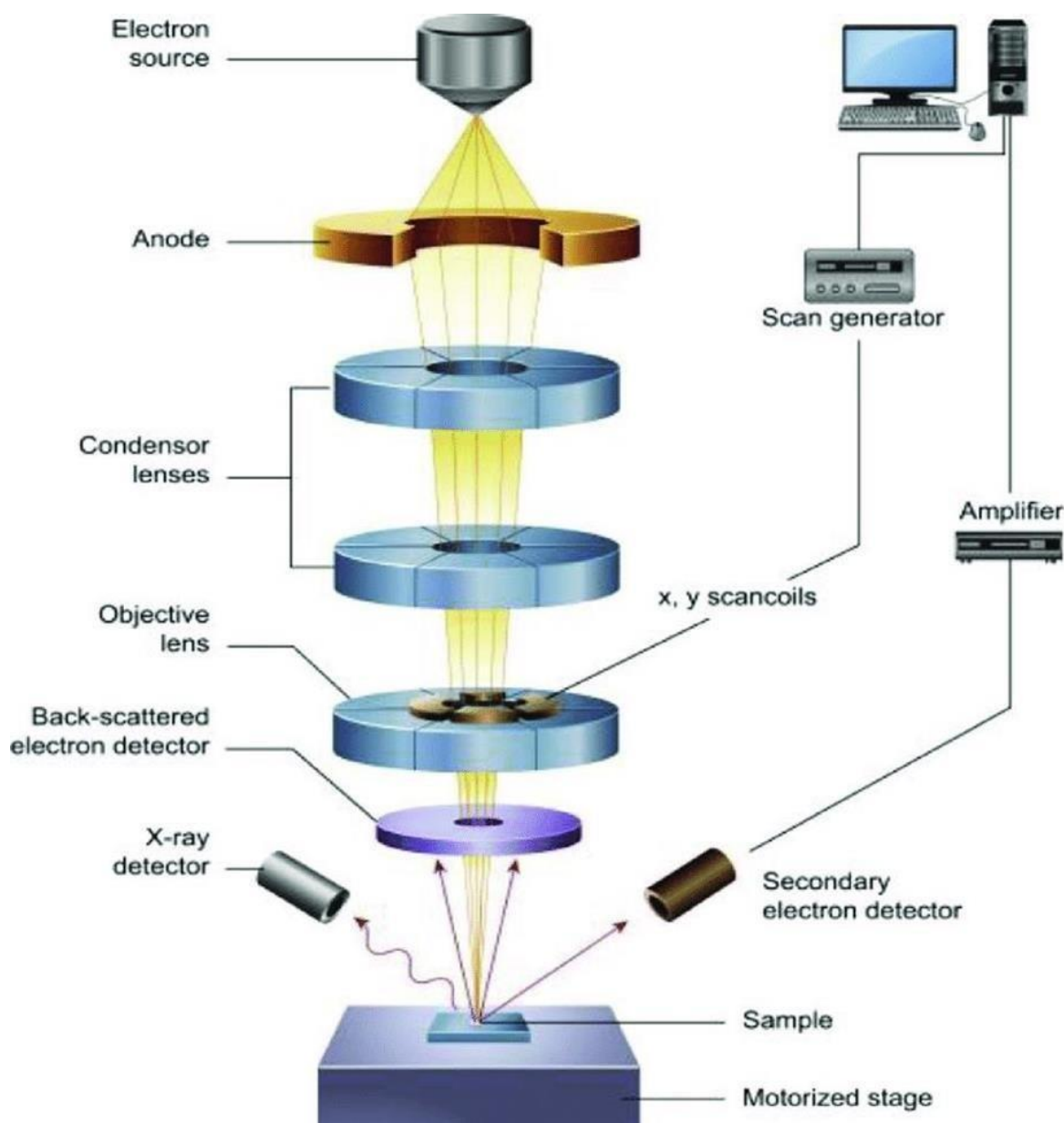


Fig 4.2.1 Scanning Electron Microscope

4.2.3 Fourier Transform Infrared Spectrometer:

Fourier transform infrared (FTIR) spectroscopy is a very efficient non- destructive characterization technique for surface characterization of nanoparticles' surfaces, providing a specific setup is connected to the spectrometer.

It is used to determine the functional groups. The chemical structure of the nanoparticles' surface and the surface reactive sites responsible for surface reactivity can be determined under particular conditions.

The structure and bands of the synthesized nanoparticles, FTIR may be used to test

a variety of components, including bulk or thin films, liquids, solids, pastes, powders, fibers, and other materials. FTIR analysis may be used for quantitative (amount) analysis as well as qualitative (identification) analysis of materials where appropriate criteria are used. FTIR can test samples up to 11 mm in diameter and weigh in bulk or the top 1 mm of the sample.

The difficulty in FTIR characterization is mainly because of high degree of overlapping of the IR absorption bands which makes difficult for the attribution of certain functional groups, despite the fact that up to date computer- searchable databases are available.

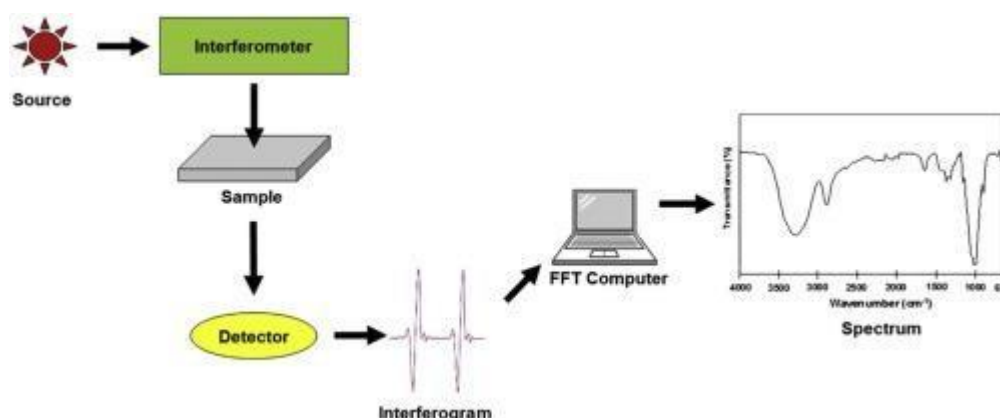


Fig 4.2.2 Fourier Transformed Infrared Spectroscopy

Source: <https://www.sciencedirect.com/topics/engineering/fourier-transform-infrared-spectrometer>

4.2.4 BET SURFACE AREA ANALYSIS

The Braeuer-Emmette-Teller method is commonly applied to calculate the specific surface area on the basis of nitrogen adsorption isotherm measurements at 77K. the BET model carries multilayer adsorption of gas on the adsorbent surface. In BET surface area analysis, nitrogen is usually used because nitrogen is highly pure and its strong interaction with most solids.

Because interaction between solid and gaseous phases is usually weak, the surface is cooled using liquid N₂ to get the adsorption. Known amount of nitrogen gas is injected to the sample cell. After the adsorption layers formed, the sample is removed from the nitrogen atmosphere and heated t cause the adsorbed nitrogen to be released from the material and quantified.

The data collected is displayed in the form of BET isotherm, which plots the amount of gas adsorbed as the function of the relative pressure.



Fig 4.2.3 BET SURFACE AREA ANALYZER

4.2.5 ADSORPTION ISOTHERM:

There are five types of isotherms is possible,

4.2.5.1 Type I Isotherm:

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

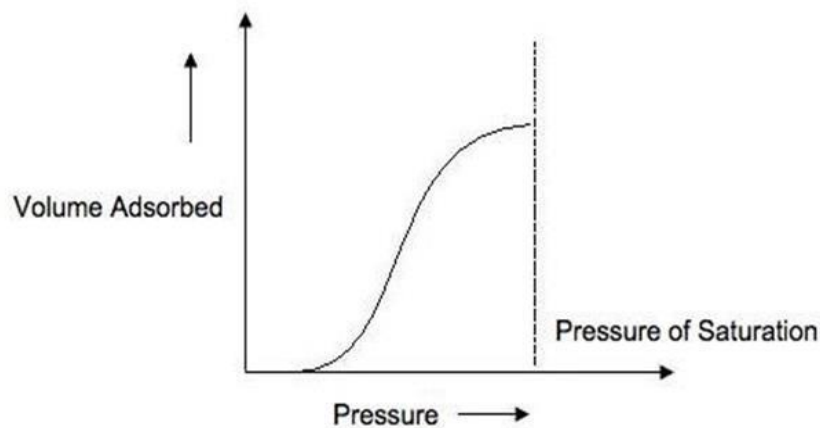


Fig 4.2.5.1 type I Isotherm

4.2.5.2 Type II Isotherm:

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

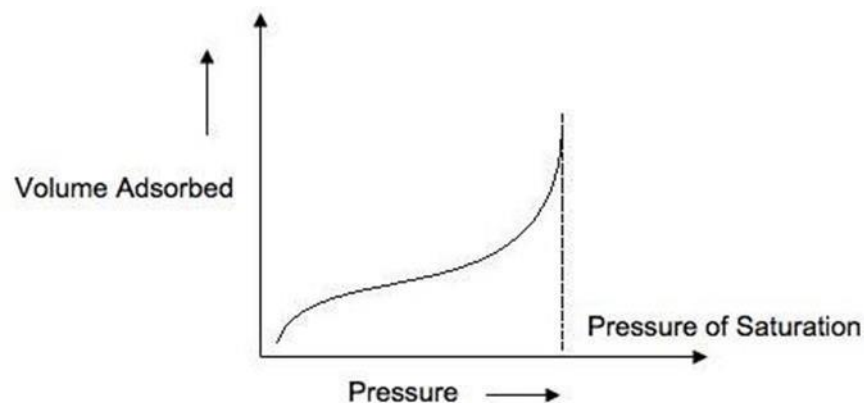


Fig 4.5.2.2 type II Isotherm

4.2.5.3 Type III Isotherm:

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

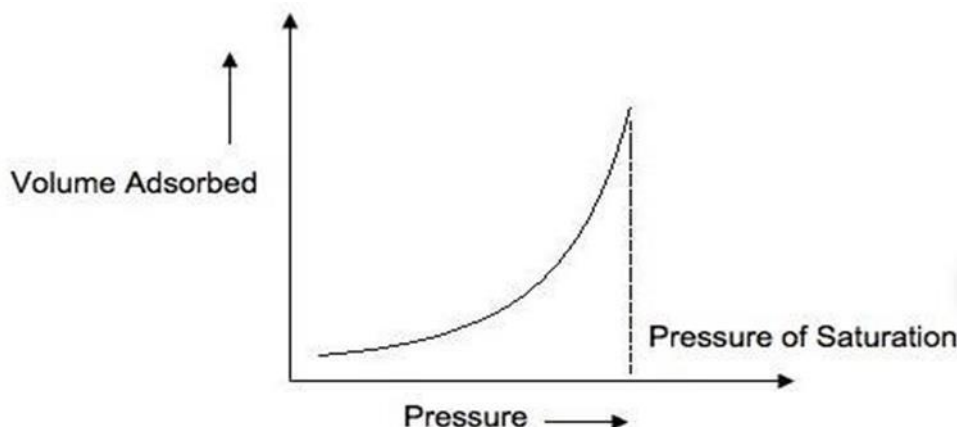


Fig 4.5.2.3 type III Isotherm

4.2.5.4 TYPE IV Isotherm

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

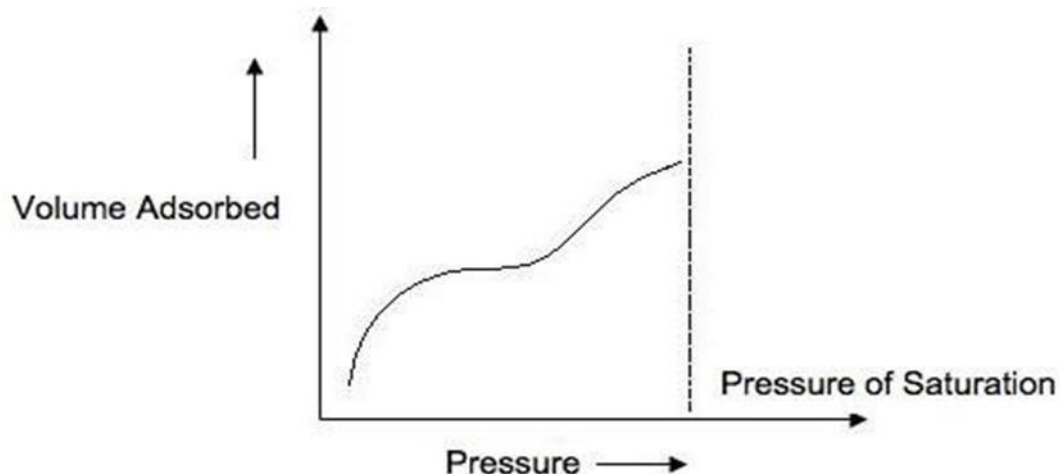


Fig 4.2.5.4 type IV Isotherm

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

4.2.5.5 Type V Isotherm

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

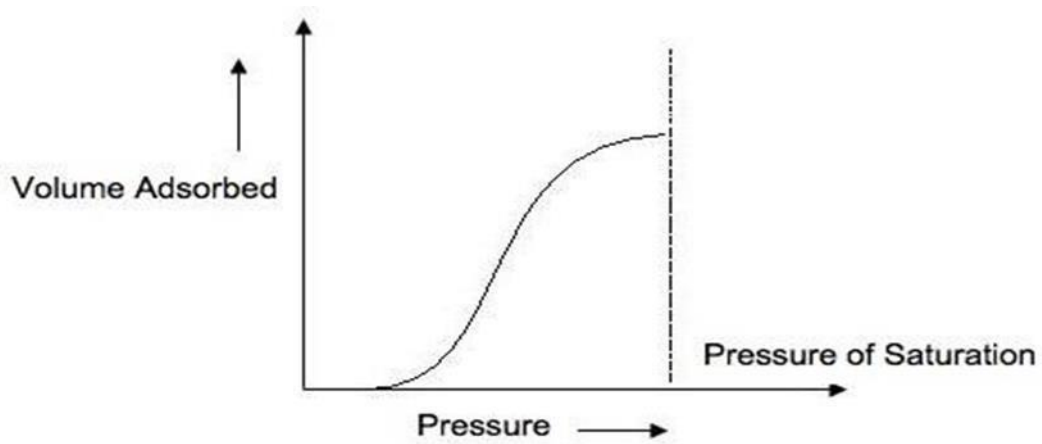


Fig 4.2.5.5 type V Isotherm

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

CHAPTER-5

RESULT AND DISCUSSION

Crystal structure, morphology, structural composition and functional group analysis of the given nanocomposite have been confirmed by characteristic techniques like XRD, SEM, UV-Vis spectroscopy, FTIR.

5.1 X-RAY DIFFRACTION (XRD):

X-ray diffraction (XRD) was used for detecting the crystalline phases represented in below figure.

ZnO is well indexed to hexagonal wurtzite structure with the planes (100), (110), and (221) corresponding to 31.7, 34.4 and 36.2.

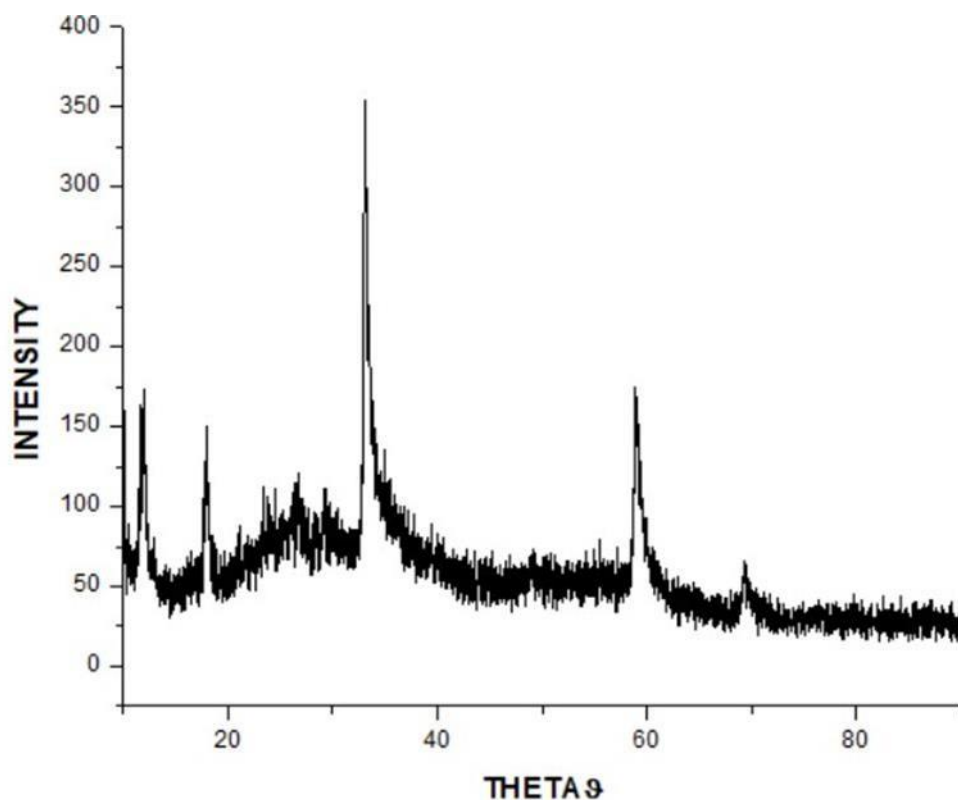


Fig 5.1 XRD of ZnO Nanoparticles

5.2 SCANNING ELECTRON MICROSCOPE (SEM):

The synthesized sample nanoparticles were detected and analyzed.

ZnO nanoparticles thus synthesized temperature was analyzed and the images are displayed in the below figure.

ZnO which is formed was crystalline in nature.

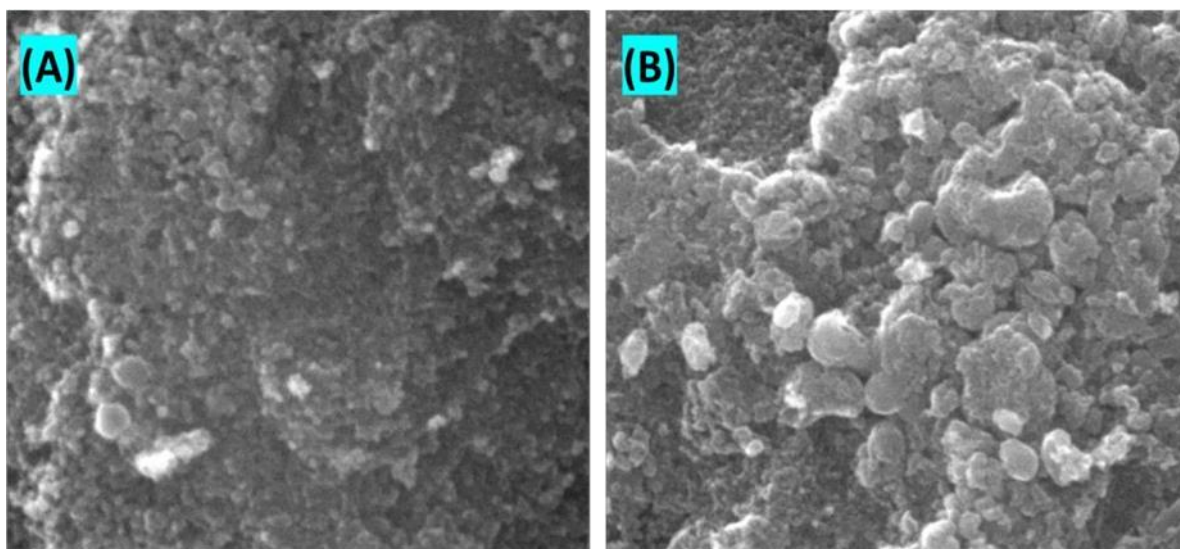


Fig 5.2 SEM Image of ZnO Nanoparticles

The result revealed size of 36 nm and has a spherical hexagonal shape. SEM analysis of ZnO nanoparticles revealed the size and shape of sample. The current study showed that ZnO nanoparticles have spherical and radial shape. The maximum and minimum particle size of ZnO nanoparticles was 28.63 nm and 61.42 nm, respectively.

5.3 FOURIER-TRANSFORM INFRARED SPECTROSCOPY:

This technique is used to determine the functional groups present in the given nanocomposite.

The FTIR spectrum of Zinc oxide nanoparticles are shown in Fig.

The IR vibrational frequency obtained at 3849 cm^{-1} , 1760 cm^{-1} , 1417 cm^{-1} and 738 cm^{-1} correspond to O-H stretching of adsorbable water molecules on Zinc oxide, C=O stretching, water and CO₂ molecule adsorption on ZnO surface, O-H in plane bending of carboxyl group adsorbed on ZnO and metal – oxygen bond Zn-O stretching respectively. The peak at 738 cm^{-1} is the characteristic peak for ZnO nanoparticles.

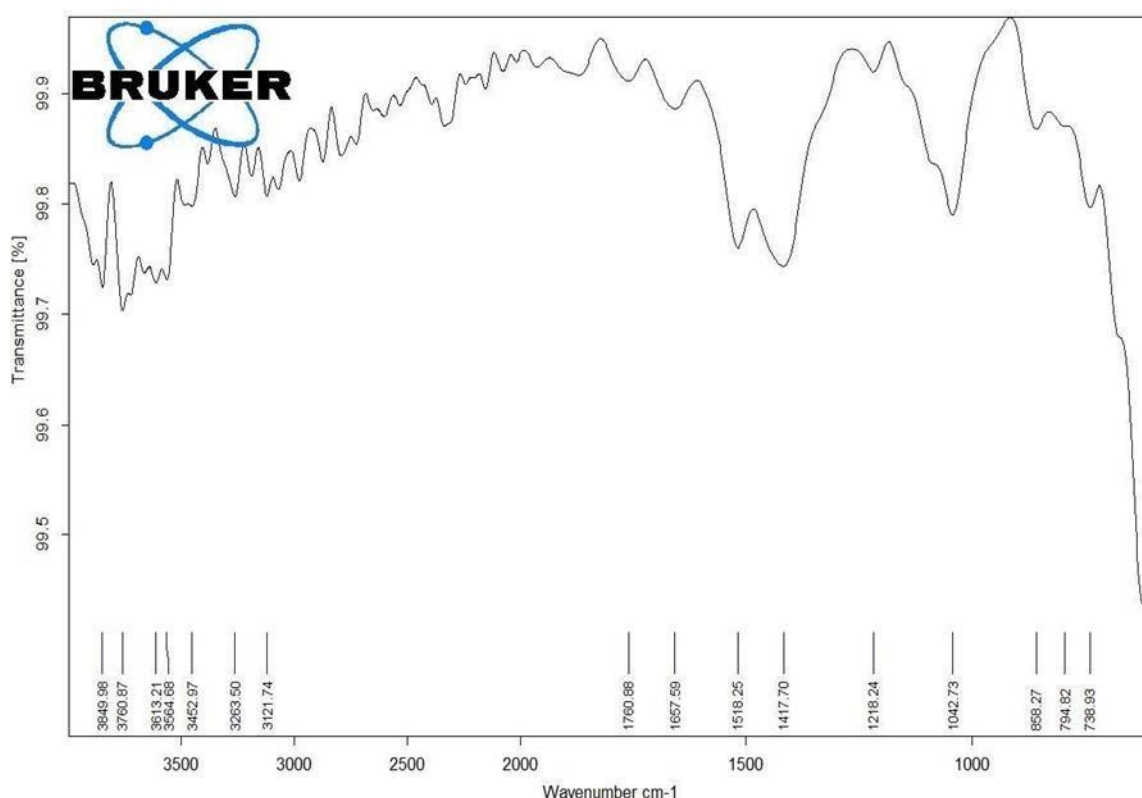


Fig 5.3 FTIR Spectrum of ZnO Nanoparticles

Table 5.1 FTIR ANALYSIS OF ZnO NANOPARTICLES

S.NO	VIBRATION FREQUENCY	FUNCTIONAL GROUP IDENTIFICATION
1	3849 cm^{-1}	O-H stretching of absorbable water molecule on zinc oxide
2	1760 cm^{-1}	C=O stretching
3	1417 cm^{-1}	C-O
4	738 cm^{-1}	Zn-O stretching

The above table shows that the various functional groups present at different wavenumbers.

5.4 BET ISOTHERM:

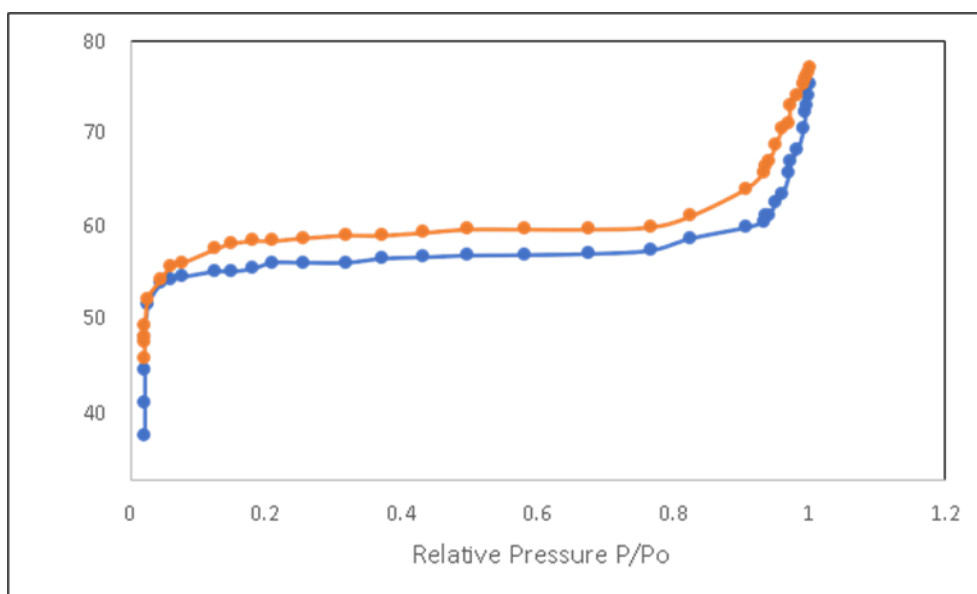


Fig 5.6 BET Isotherm

The BET interpretation and pore size estimation of ZnO nanoparticles claimed at 850 °C were determined using adsorption. The hexagonal wurtzite structure of nano ZnO-SPA is evaluated based on XRD pattern which also exhibit an average crystallite size

as 20.18 nm and high specific surface area as 52.99 m²/g. The SEM-supported morphological assessment confirms the formation of agglomerates of ultrafine ZnO rods and spherical particles. It should be noted that the resulting ZnO featured a very high surface area. Typically, ZnO nanostructures feature a surface area ranging from 3 m²/g to 50 m²/g. The surface area (78.1 m²/g).

5.5 ADSORPTION ISOTHERM:

Adsorption isotherms have been of immense importance to research dealing with environmental protection and adsorption techniques. The two primary methods used for predicting the adsorption capacity of a given material are known as the Freundlich and Langmuir isotherms.

An adsorption isotherm is a graph that represents the variation in the amount of adsorbate(x) adsorbed on the surface of the adsorbent with the change in pressure at a constant temperature.

5.5.1 FREUNDLICH ADSORPTION ISOTHERM:

Freundlich adsorption gives the variation in the quantity of gas adsorbed by a unit mass of solid adsorbent with the change in pressure of the system for a given temperature.²³

The expression for the Freundlich isotherm can be represented by the following equation,

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

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

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 naphthalene on the Zinc oxide. K_f and n determined from the slope and intercept of the liner plot.

The Freundlich adsorption isotherm parameter for adsorption of naphthalene from the aqueous solution using the adsorbent.

Here the Freundlich isotherm proved to be the better fit from the regression coefficient. The heterogeneity of zinc oxide activated adsorbent surface.

Figure shows the effect of time on stream for the removal of naphthalene by using Zinc oxide.

Table 5.2 Analysis of adsorption isotherm of naphthalene

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	3.6	0.2777	0.5563	1.2809	1.6	0.625	0.4700
2	20	6.3	0.1587	0.7781	1.8405	3.425	0.291970	1.2311
3	30	15.8	0.0632	1.1986	2.7600	3.55	0.281690	1.2669
4	40	19.4	0.0515	1.2878	2.9652	5.15	0.194174	1.6389
5	50	25.7	0.0389	1.4099	3.2464	6.075	0.164609	1.8041
6	60	31.2	0.0320	1.4941	3.4404	7.2	0.138888	1.9740

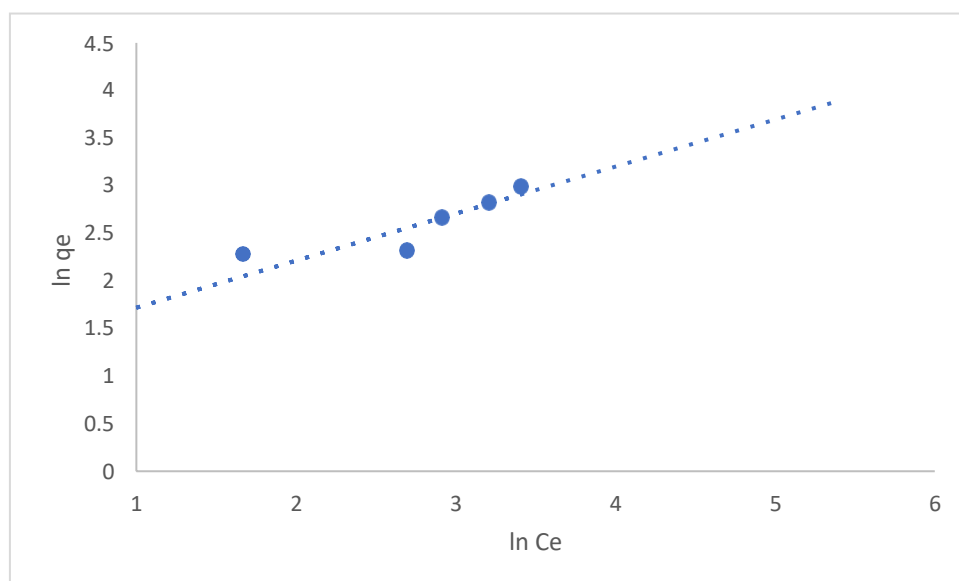


Fig 5.5 Freundlich Adsorption Isotherm of Naphthalene

5.6 EFFECT OF ADSORPTION PARAMETERS:

5.6.1 EFFECT OF INITIAL CONCENTRATION OF NAPHTHALENE:

About 0.15 mg from the concentration is added to (10, 20, 30, 40, 50 and 60 ppm) from drug is added to 25ml standard flask and kept in the shaking incubator for 1hr.

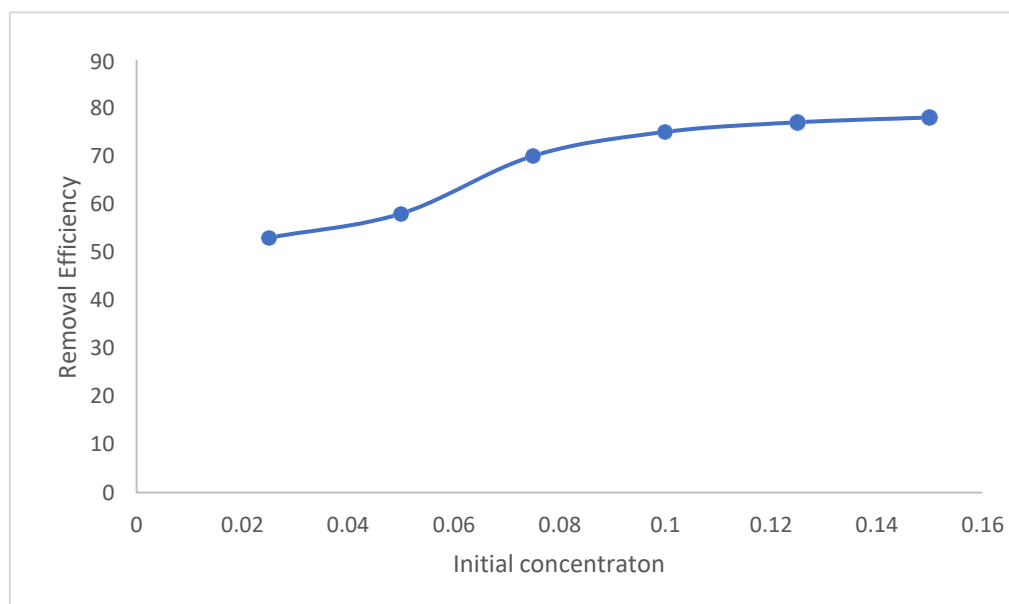


Fig 5.6.1 Effect Of Naphthalene Concentration

5.6.2 EFFECT OF TEMPERATURE ON REMOVAL EFFICIENCY:

The adsorption of naphthalene 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 0.15 mm of catalyst and naphthalene concentration 30ppm, 100ml utilizing water bath at 60°C. the experiment was carried at the pH of 6 with the time of 1h at a constant speed of 120 rpm.

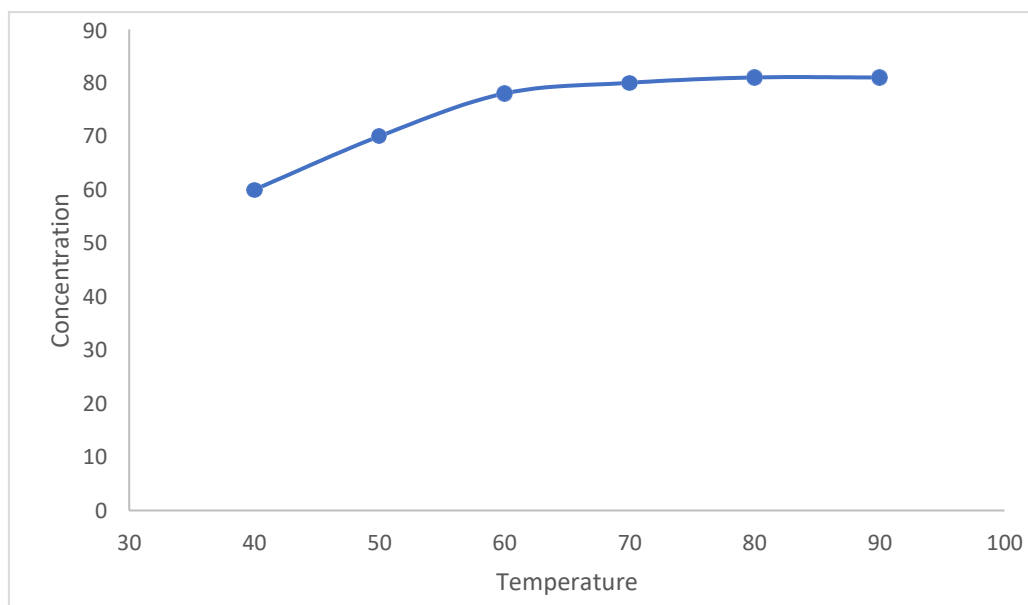


Fig 5.6.2 Effect of Temperature on removal efficiency

5.6.3 Effect of the amount catalyst:

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

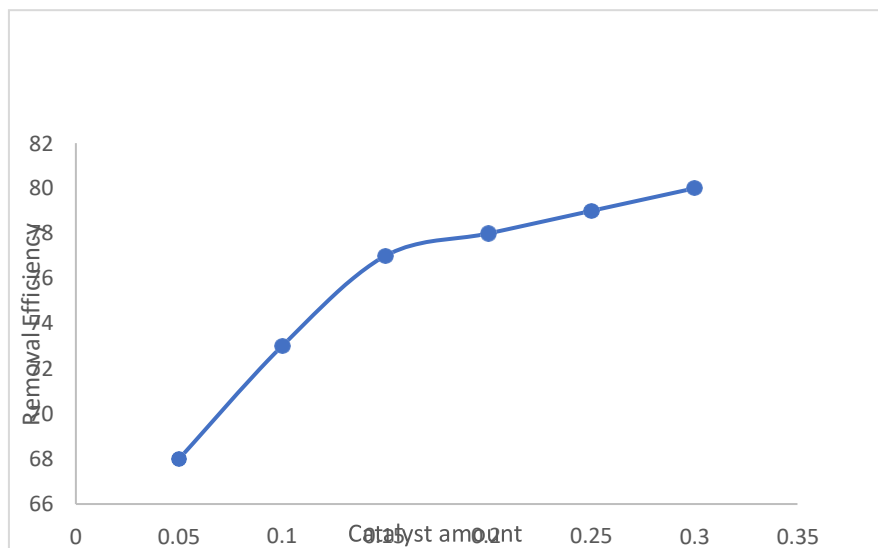


Fig 5.6.3 Effect of weight amount of catalyst

5.6.4 EFFECT OF pH ON ADSORPTION OF NAPHTHALENE:

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

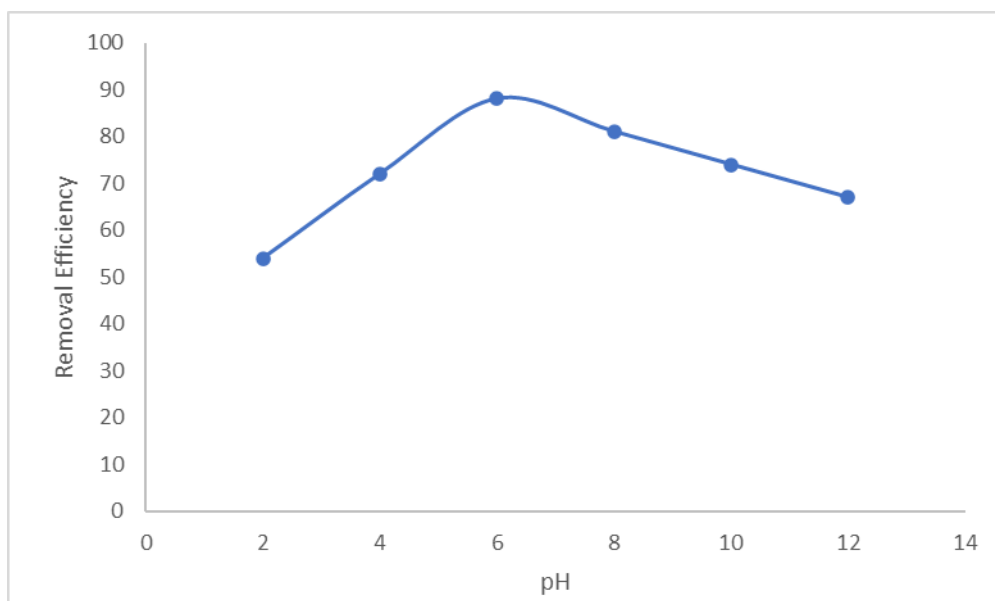


Fig 5.6.4 Effect of pH on adsorption of naphthalene

5.6.5 EFFECT ON TIME OF STREAM:

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

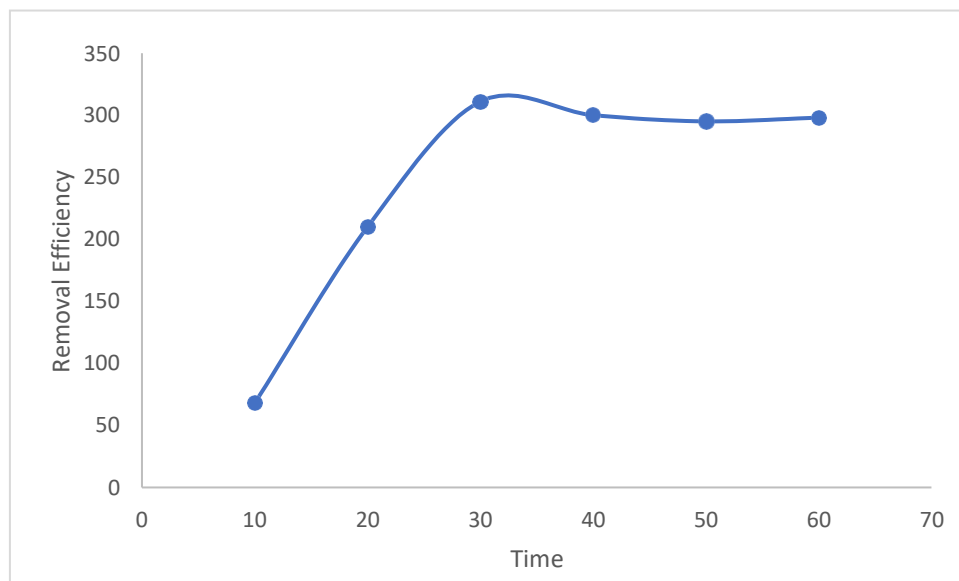


Fig 5.6.5 Effect on Time of Stream

CHAPTER 6

SUMMARY AND CONCLUSION

In summary, ZnO Nanoparticles was synthesized using Zinc acetate, neem extract and sodium hydroxide through green synthesis. 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 ZnO Nanoparticles was calculated using Debye Scherrer formula. XRD pattern revealed ZnO has to hexagonal wurtzite structure. Metal-Oxygen stretching was confirmed by the functional group analysis.

The SEM demonstrated the synthesized nanoparticles were in size of 36 nm and has a spherical hexagonal shape. The maximum and minimum particle size of ZnO nanoparticles was 28.63 nm and 61.42 nm.

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

The BET Isotherm estimated the surface area and pore size of Zinc Oxide nanoparticles. ZnO nanostructures feature a surface area ranging from 3 m² /g to 50 m² /g. The surface area (78.1 m² /g).

The concentration of Naphthalene in aqueous solution after adsorption were studied by UV-Visible Spectroscopy.

This includes adsorption isotherm effect of temperature, pH, initial concentration, weight, time on stream. Freundlich isotherm was best fitted; Optimized pH at 6, temperature 60°C, initial concentration at 0.15, first 30 minutes increases sharply and then decreases.

Therefore, the synthesized ZnO Nanoparticles was low cost and environmentally friendly absorbent for removal of PAC (Poly Aromatic compounds)

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