

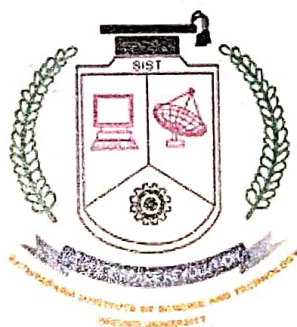
SILVER DECORATED IRON OXIDE FOR TEXTILE EFFLUENT TREATMENT

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

by

B.GOKULAKANNAN

(REGISTER NO : 40910008)



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

SATHYABAMA

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

ACCREDITED WITH GRADE "A" BY NAAC

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI-600119

MAY- 2023



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Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai – 600 119
www.sathyabama.ac.in

DEPARTMENT OF CHEMISTRY

BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of
Mr.B.GOKULAKANNAN (41910008) who carried out the project entitled "**SILVER
DECORATED IRON OXIDE FOR TEXTILE EFFLUENT TREATMENT**" under my /
our supervision **FROM NOVEMBER-2022 TO MAY 2023**

Internal Guide

(Dr. S. SUNITHA)

External Guide

(Dr. G. MURUGADOSS)


HEAD OF THE DEPARTMENT
DEPARTMENT OF CHEMISTRY
SATHYABAMA
INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)
Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai – 600 119
08-05-2023
Head of the Department (Name in Capital Letters with Seal & Signature)

Submitted for Viva voce Examination held on

8-5-2023

Internal examiner

External Examiner

DECLARATION

ACKNOWLEDGEMENT

I Mr.B.GOKULAKANNAN student of SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY hereby declare that the Project Report entitled "SILVER DECORATED IRON OXIDE FOR TEXTILE EFFLUENT TREATMENT " done by me under the guidance of Dr. S.SUNITHA, M.Sc., M.Phil., Ph.D. and Dr. G.MURUGADOSS, M.Sc., Ph.D. Scientist-E at International Research Centre, Sathyabama Institute of Science and Technology, Chennai is submitted in partial fulfillment of the requirements for the award of Master of Science degree in CHEMISTRY.

SUNITHA M.Sc., M.Phil., Ph.D. and Dr. G. Murugadoss M.Sc., Ph.D. Scientist-E at International Research Centre, Sathyabama Institute of Science and Technology (External guide) for their valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

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

DATE: 08/05/2023

PLACE: CHENNAI

B. Gokulanan

SIGNATURE OF THE CANDIDATE

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

GOKULAKANNAN B

SILVER DECORATED IRON OXIDE FOR TEXTILE EFFLUENT TREATMENT

VISION

**“TO EXPLORE NEW FRONTIERS IN CHEMICAL SCIENCE
THROUGH INNOVATION AND COLLABORATION”**

ABSTRACT

silver-magnetite oxide ($\text{Ag-Fe}_3\text{O}_4$) nanocomposites were synthesized by the chemical precipitation method using different ratios of silver nitrate and iron oxide. The structural, morphological, and optical properties of nanocomposites were analysed by X-ray diffraction (XRD), Fourier transform infrared spectra (FT-IR), Scanning electron microscope (SEM), UV-Vis spectra. ($\text{Ag-Fe}_3\text{O}_4$) has a strongly quenching in UV intensity indicating stability between electrons and holes (e^-/h^+). The photocatalytic activities of $\text{Ag-Fe}_3\text{O}_4$ nanocomposites were investigated by the degradation of fast green in aqueous solution under visible light in different condition of pH, initial concentration and different trapping agents for free radicals. The results shows that the photodegradation efficiency of silver compound with presence of magnetite improved. The optical results show enhancement of nanocomposite absorption under the visible region with a high life time between e^-/h^+ at the optimum ratio between ($\text{Ag-Fe}_3\text{O}_4$), which can applied to obtain high photocatalytic degradation of fast green under visible light. Finally, the catalytic activity has been stabled when such photocatalyst was utilized again for several cycles.

Key words : Silver, Iron oxide, photocatalyst, photoluminescence

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CHAPTER-1

INTRODUCTION

Water pollution due to discharge of textile effluents from textile dyeing mills are one of the major environmental problem, in the world today. Water-containing dye molecules affect human health and aquatic life.

Colour effluents are responsible for the receiving aquatic ecosystem poses aesthetic problem and serious ecological problems. Therefore a numbers of techniques aimed at preferential removal dyes from wastewater have been developed.

The present study includes brief discussion about mixed metal nanoparticles, properties of nanoparticles, nanocatalysis and different synthesis method of nanoparticles.

Metal oxides are formed as a consequence of co-ordination tendency of metal ions so that oxide ions form co-ordination sphere around metal ions and give rise to close packed structure. The transition metals have the special properties of formation of coloured compounds and show magnetic properties.

Besides these they are excellent photoactive materials and work as photosensitizer. Mixed metal oxide (MMO) electrodes are devices with useful properties for chemical electrolysis.

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.

Metal oxides/ Mixed metal oxides have wide applications as catalyst, Adsorbents, Superconductors, semiconductors, ceramics, antifungal agents and also have spacious applications in medicines

The review also gives a concise discussion of azo dyes, degradation method of dyes and applications of metal nanoparticles in different environmental remediation process. The experimental section gives depiction of several instruments, characterization, analytical techniques and also describing the details of the reagents, chemicals, their solutions with other specifications in the kinetic study of the degradation of dyes.

1.1 NANOPARTICLES

These days, we live in an escalated advancement of science known as nanotechnology. The normal size of particles framing a sample in the range of 1–1000 nm ($1\text{ nm} = 10^{-9}\text{ m}$). There are so meanings for nanoparticles. One of them says that, whose at any rate one measurement is more than 100 nm, then such material can be called a "nanomaterial".

Another definition of "nanomaterial" is a material which after "miniaturization" to nanoscale uncovers changed physicochemical properties in contrast with the mass item (naturally visible). The nanoparticles display novel physical, chemical, and biological properties at the nanoscale contrasted with their specific particles at higher scales.

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

The nanoparticles differ in shape, size, 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 techniques are adjusted to accomplish measure explicit nanoparticles to increment their physical, chemical, optical, and mechanical properties. A huge advancement in instrumentation has prompted an improvement in nanoparticle characterization and application. Based on the application like imaging, diagnosis, or treatment various kinds of nanoparticles have been proposed, some of them are utilized for multiple purposes.

Nanoparticles come under two main categories such as organic and inorganic nanoparticles. The primary group includes micelles, dendrimers, liposomes, hybrid, and minimized polymeric nanoparticles. The second group includes silica, fullerenes, quantum dots, and gold nanoparticles.

1.2 CLASSIFICATION OF NANOPARTICLES

Nanoparticles can be classified into different types according to their size, morphology, physical and chemical properties. Some of them are carbon-based nanoparticles, ceramic nanoparticles, metal nanoparticles, semiconductor nanoparticles, polymeric nanoparticles, and lipid-based nanoparticles[1]. nano species derived from carbon (carbon nanotubes, fullerenes, graphene) metallic nanoparticles (silver, gold, copper) oxide nanoparticles (silica, titanium dioxide, iron oxides, copper oxide, zinc oxide, aluminum oxide) polymeric nanoparticles (ion exchangers, reactive and complexed polymers) proteins, and enzymes. Notably, the iron oxide nanoparticles with magnetic properties (maghemite or magnetite) generated particular interest in studies on photocatalytic degradation on nanomaterials.

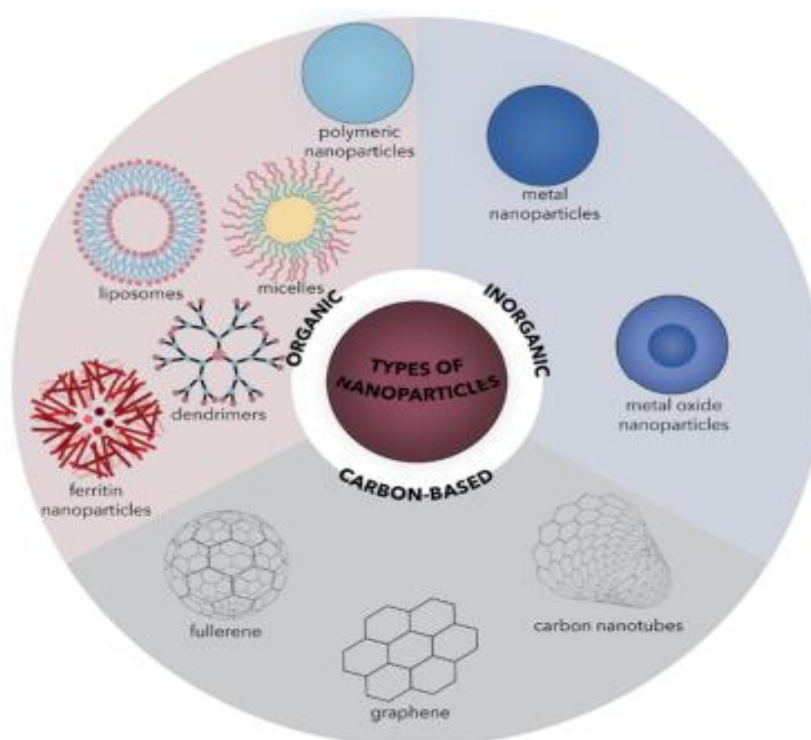


Figure 1.2: Classification of nanoparticles(NPs)

1.2.1 CARBON-BASED NANOPARTICLES

Carbon-based nanoparticles are of two main materials: carbon nanotubes (CNTs) and fullerenes. CNTs are nothing but graphene sheets rolled into a tube. These materials are mainly used for structural reinforcement as they are 100 times stronger than steel [2].

Fullerenes contain nanomaterials that are made of globular empty enclosures like allotropic structures of carbon because of their electrical conductivity, high strength, structure, electron affinity, and flexibility. These materials possess pentagonal and hexagonal carbon units, while every carbon is sp^2 hybridized. CNTs are tubular, elongated, and 1–2nm in measurement. These can be anticipated as metallic or semiconducting dependent on their measurement telocity. These can be anticipated as metallic or semiconducting dependent on their measurement telocity [3]. The sheets can be single, double, or numerous walls and in this way, they are named single-walled (SWNTs), double-walled (DWNTs), or multi-walled carbon nanotubes (MWNTs)individually, they are synthesized by carbon precursors, by electric arc on metal nanoparticles. they are synthesized by Chemical Vapour Deposition (CVD).

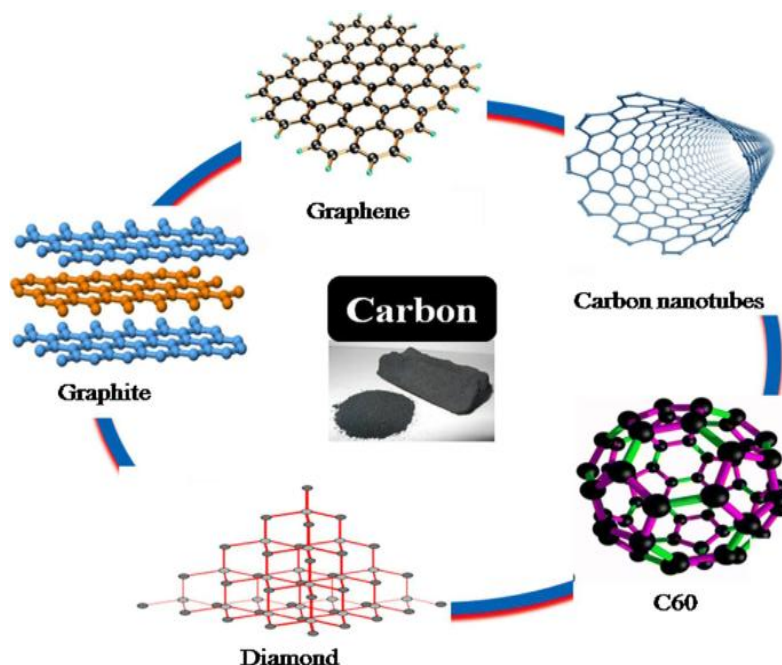


Figure 1.2.1 : Carbon based nanoparticles(NPs)

1.2.2 ORGANIC BASED NANOPARTICLES

These incorporate nanoparticles made for the most part from organic matter, barring carbon-based or inorganic-based nanoparticles.

The use of non-covalent weak interactions for the self-assembly and designs of atoms assists with changing the organic nanomaterials into specific structures, for example, dendrimers, micelles, liposomes, and polymer nanoparticles.

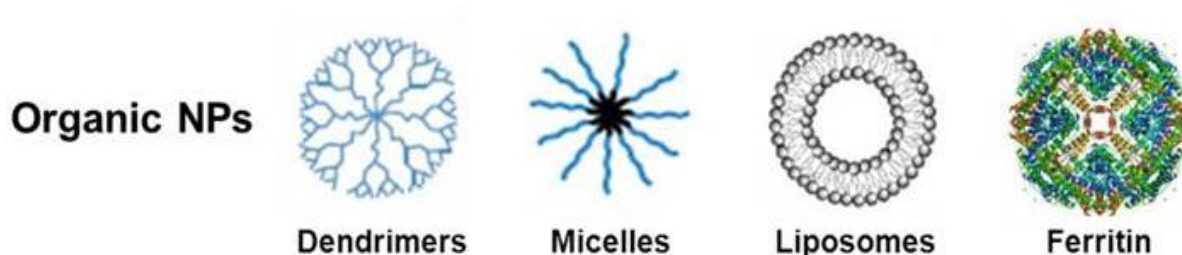


Figure 1.2.2 : organic based nanoparticles(NPs)

1.2.3 COMPOSITE BASED NANOPARTICLES

Composite nanomaterials are multiphase nanoparticles and nanostructured materials with one stage on the nanoscale measurement that can either consolidate nanoparticles with different nanoparticles or nanoparticles joined with bigger or with bulk materials or more complicated structures, for example, metal organic systems.

Nanocomposites are a class of nanomaterials wherein one or more phases at nano-sized dimensions (zero dimension, one dimension, and two dimensions) are embedded in a ceramic, metal, or polymer material.

These composites might be a mixture of carbon-based, metal-based, or natural-based nanomaterials with any type of metal, polymer, or ceramic-based materials. Nanomaterials are synthesized in various morphologies relying upon the necessary properties for the required application [4].

1.2.4 INORGANIC BASED NANOPARTICLES

Inorganic nanoparticles are non-toxic, hydrophilic, biocompatible, and highly stable compared to organic materials. Drug delivery systems designed for enhanced drug efficacy and reduced adverse effects have evolved accompanied by the development of novel materials.

These nanomaterials incorporate metal furthermore, metal oxide nanoparticles, and nanostructured materials. These nanomaterials can be combined into metals like Au or Ag nanoparticles, metal oxides, for example, Fe_3O_4 , CeO_2 , TiO_2 , and ZnO nanoparticles, and semiconductors like silicon and ceramics.

Inorganic nanoparticles, such as quantum dots, polystyrene, magnetic, ceramic, and metallic nanoparticles, have a central core composed of inorganic materials that define their fluorescent, magnetic, electronic, and optical properties.

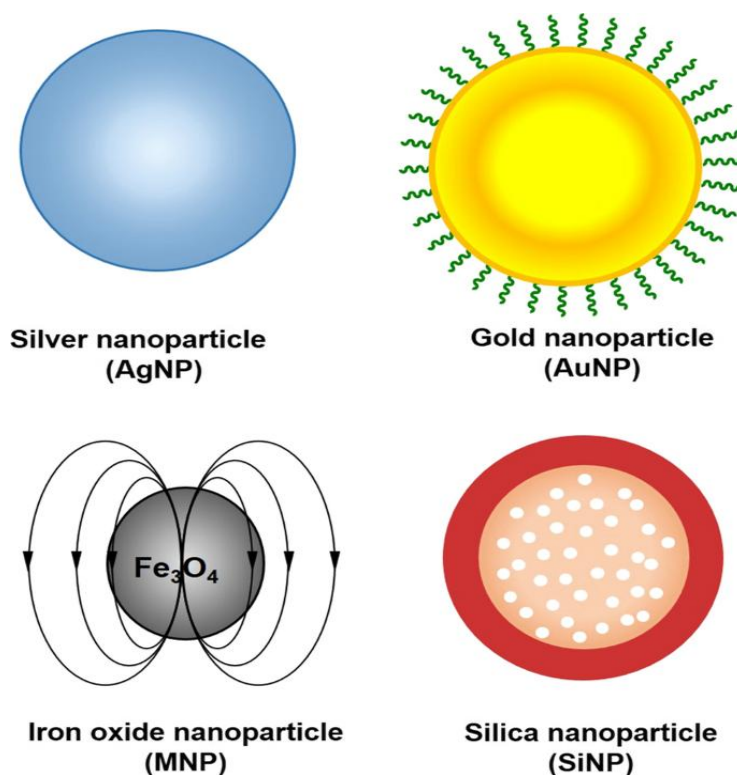


Figure 1.2.4 : Inorganic based nanoparticles(NPs)

1.3 PROPERTIES OF NANOPARTICLES

Various physicochemical properties namely large surface area, chemically reactive, optically active, and mechanically strong make the nanoparticles incredibly unique and make them suitable for numerous applications. Some of their properties are mentioned below.

1.3.1 PHYSICAL PROPERTIES

The physical properties of nanoparticles are dependent on the size, shape, composition, crystal structure, surface ligand or capping agents, and the medium in which they are dispersed. The surface area to volume ratio of nanoparticles has a significant effect on the properties of the nanoparticles. Nanoparticles have a large surface area per unit volume related to the bigger particles due to this nanoparticles are more chemically reactive. Materials are found to be inert in their bulk form and are reactive when change into nanoscale form.

1.3.2 ELECTRIC AND OPTICAL PROPERTIES

The electronic and optical properties of nanoparticles are interconnected. When the incident photons interact with electrons and show collective excitation this is known as the localized surface plasma resonance (LSPR). The peak wavelength of the LSPR spectrum is dependent upon the interparticle spacing, size, and shape of the nanoparticles. These electronic and optical properties of nanoparticles have been applied in optical filters and sensors [5].

Due to the quantum confinement effect, nanomaterials with small particle sizes exhibit better optical emission as well as nonlinear optical properties. The effect of size on nanoparticle optical properties is caused by two distinct phenomena: surface phenomena and an increase in energy level spacing due to the confinement of energy states, which is most notable in semiconductors.

1.3.3 MAGNETIC PROPERTIES

After surface cooling, magnetic nanoparticles exhibit extraordinary new phenomena including strong field irreversibility, high saturation field, super para magnetism, extra anisotropy effects, or shifted loops. The magnetic nature of individual nanoparticles is because of narrow and finite-size effects, as well as surface effects [6].

1.3.4 MECHANICAL PROPERTIES

Magnetic nanoparticles can be superparamagnetic and respond to external magnetic fields very fast with almost zero remanences. These properties provide bases for applications like biomedical imaging and information storage technology. The mechanical properties of nanoparticles such as hardness and elastic modulus, interfacial adhesion and friction, movement law, as well as its size-dependent effect.

1.4 SYNTHETIC METHODS

Approaches come under two main categories namely: top-down approach and bottom-up approach and they differ in their cost, speed, and degree of quality. The Approaches have been summarized below.

NANOTECHNOLOGY OF METAL OXIDES

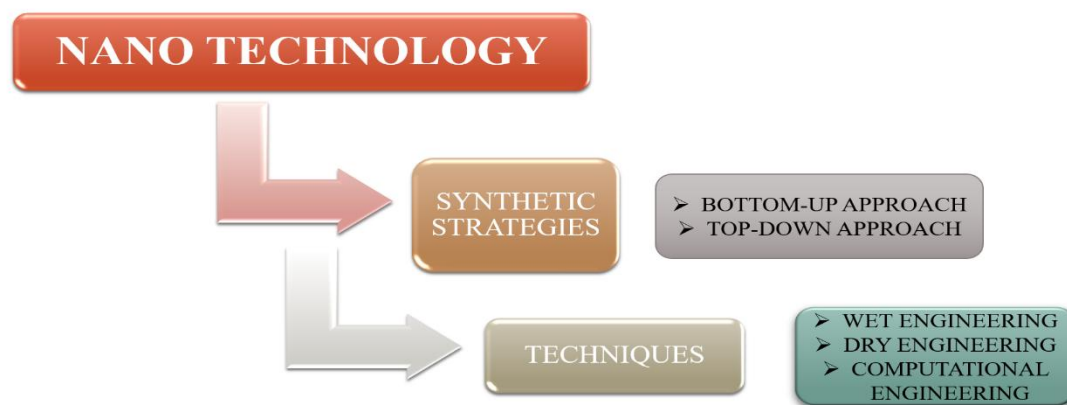


Figure 1.4(a): Nanotechnology of metal oxides

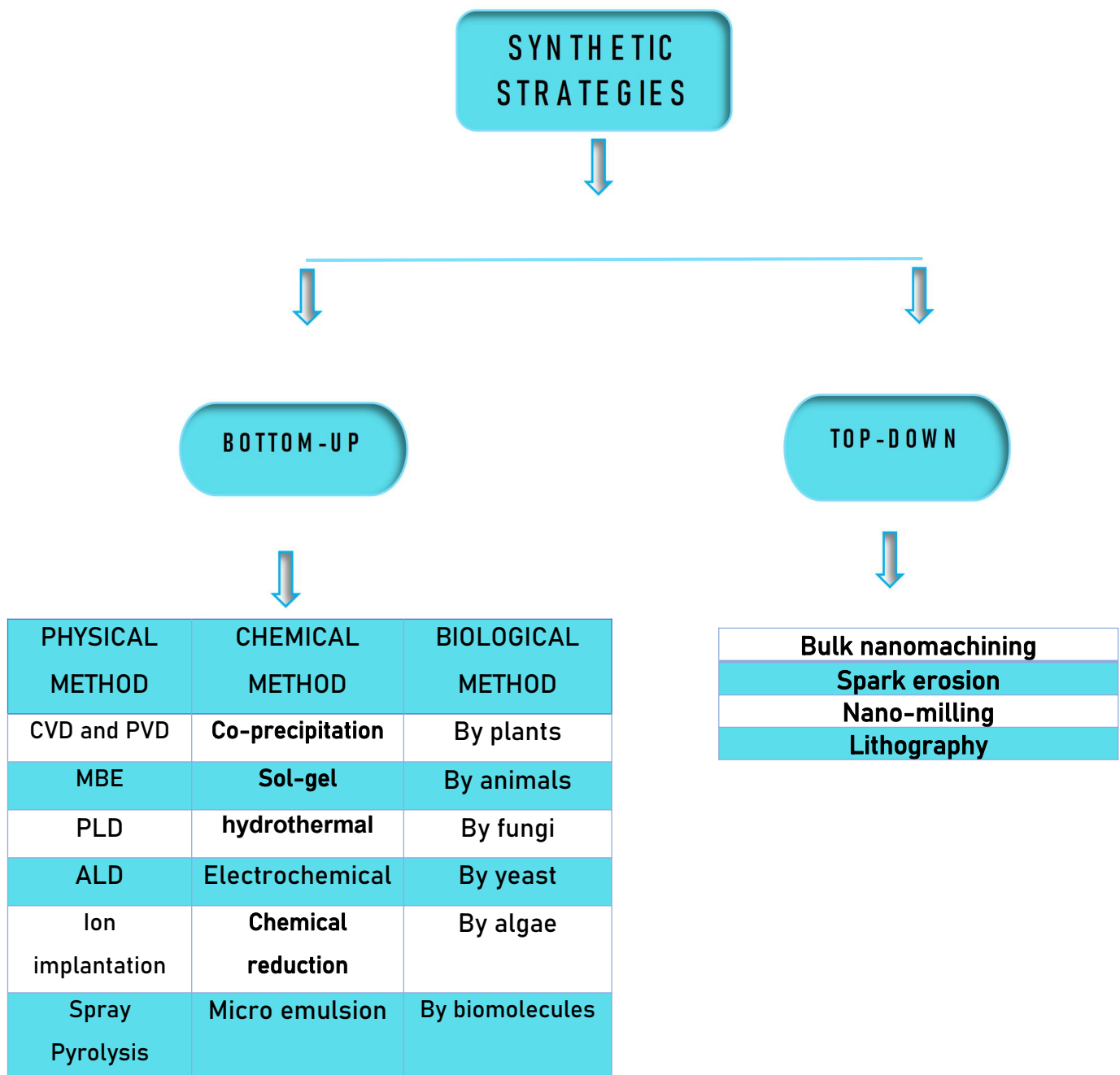


Figure 1.4(b): BOTTOM-UP and TOP-DOWN approaches

1.4.1 BOTTOM-UP APPROACH

This method is the reverse of the top-down approach. These nanoparticles are formed from simple substances and hence it is called a building-up approach. examples are physical vapor deposition, sol-gel, chemical vapor deposition, co-precipitation, solvothermal, etc.

1.4.1.1 PHYSICAL VAPOR DEPOSITION

The particle size is determined by the rate of evaporation and the pressure of the gases in the chamber. Gases such as H₂, O₂, NH₃, and N₂ are used, and the material interacts with them, forming nitrides, hydrides, and oxide particles. The gas pressure in the deposition chamber defines the size and form of the evaporated substance. Both processes were carried out in a vacuum chamber to produce a high-purity nanomaterial[7]

1.4.1.2 SOL-GEL METHOD

Two materials are involved in this method they are sol and gel. It is a low-temperature process and consumes less energy. Synthesis of nanoparticles like nanotubes and nanorods can be obtained by this method. Sol-gel synthesis involves hydrolysis, condensation, gelation, and the drying process by various routes.[8]

The sol-gel method is a proper wet route for the preparation of nanostructured metal oxides. This method is based on the hydroxylation and condensation of molecular precursors in solution, initiating a "sol" of nanometric particles. Additional condensation and inorganic polymerization lead to a three-dimensional metal oxide network, denominated as a wet gel. Extra heat treatments are needed to obtain the final crystalline state because these reactions are performed at room temperature.

The sol-gel process includes hydrolysis and condensation of metal alkoxides. Metal alkoxides are good precursors, due to their endurance in the face of hydrolysis, The hydrolysis step replaces an alkoxide with a hydroxide group from water, and free alcohol is generated. Factors that need to be considered in a sol-gel method are the solvent type, temperature, precursors, catalysts, pH, additives, and mechanical

agitation. These factors can affect the kinetics, growth, hydrolysis, and condensation reactions. For metal oxides, the sol-gel process offers some advantages compared to other methods, including good homogeneity, low cost, and high purity. Moreover, the sol-gel method has been developed for the synthesis of magnetite NPs using metal inorganic precursors.

The sol-gel method has some advantages. In this method, it is possible to prepare pure amorphous phases, with monodispersity and good control of the particle size, and also to obtain materials with a predetermined structure based on experimental conditions. Moreover, the microstructure and the homogeneity of the reaction products are controllable. The sol-gel method includes pollution from by-products of reactions, as well as the need for post-treatment of the products.

1.4.1.3 CHEMICAL VAPOUR DEPOSITION (CVD)

The deposition of a thin film of gaseous reactants onto a substrate is Chemical Vapour Deposition. At ambient temperature, gas molecules are combined and the deposition is carried out in a reaction chamber. A chemical reaction occurs when a heated substrate comes in contact with the combined gas.

A thin film of product on the substrate surface is produced and that is recovered and used. The influencing factor in CVD is substrate temperature. CVD produces nanoparticles that are strong, uniform, and hard. CVD requires special equipment and the gaseous by-products are toxic.

1.4.1.4 CHEMICAL PRECIPITATION METHOD

The chemical precipitation process mainly consists of three main steps such as chemical reaction, nucleation, and crystal growth. Solids obtained from the chemical precipitation have a wide particle size plus uncontrolled particle morphology, along with agglomeration. The nanoparticles obtain a narrow size distribution with certain requirements such as a high degree of supersaturation, a uniform spatial concentration distribution inside a reactor, and a uniform growth time for all particles or crystals.

The chemical precipitation method has extreme advantages when compared to other synthetic methods. The chemical precipitation method is the simplest and the most rapid method for the synthesis of the nanoparticles. This method offers easy control of the particle size. It provides various possibilities to modify the particle surface and homogeneity and holds good control over the composition of a nanoparticle. The chemical precipitation method can be used from small scale to large scale and it is the most economical method for the synthesis of nanoparticles.

1.5 APPLICATIONS OF NANOPARTICLES

Nanoparticles are now being used in the manufacture of scratch proof 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 systems. 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've adhered to short DNA segments, they can also be used to detect genetic sequences. Nanotechnology allows damaged tissue to be repaired or replicated. The use of nanotechnology can revolutionize organ transplantation or artificial implantation[9].

The need for new antimicrobial agents has risen. Because of their inbuilt antimicrobial properties, nanoparticles (NPs) are now being considered as effective nanotechnology for regulating harmful micro-organisms.

1.6 METAL NANOPARTICLES

Metal nanoparticles do not represent a metal-metal chemical bond and it is defined as isolated particles between 1-100 nm in size.

Metal nanoparticles (NPs) have extraordinarily different properties as compared to their bulk metal which mostly contains a degenerated density of energy states and a large surface-to-volume ratio along with the sizes in the nanometer scale. Therefore

they show high chemical activity and specificity as compared to bulk metals so they are attractive to use as catalysts.

The high surface-to-volume ratio along with size effects (quantum effects) gives metal nanoparticles distinctively different properties (chemical, electronic, optical, magnetic, and mechanical) from those of bulk metal. Researchers have achieved considerable attention in nanomaterials because of their unique properties and various applications in different areas.

nanoparticles exhibit many fascinating applications in a wide variety of fields including conductors, electronics, sensors, photovoltaic devices, drug delivery, catalysis, fuel cells light-emitting diodes, industrial lithography, quantum dots, quantum wires, quantum devices, optical and biological devices, etc. due to their unique properties, which vary from molecular or bulk materials.

Incredible properties of nanoparticles intensely depend on the distinct shape and diminutive size of nanoparticles, surrounding media, and their fabrication method. So, scientists take an interest in targeting the fabrication of metal nanoparticles. Different shapes and sizes of metal nanoparticles allow exploring their applications in fields including catalysis, electronics, sensor, and optical device.

1.6.1 SILVER NANOPARTICLES

Owing to their small size, the total surface area of the nanoparticles is maximized, leading to the highest values of the activity-to-weight ratio. Due to this property being distinctly different from that of the bulk metal.[10]

Silver nanoparticles are emerging as promising agents for cancer therapy. The anticancer activities of nano-sized silver particles have been evaluated against a variety of human cancer cells, including breast cancer cells.

The AgNPs have the following advantages synthesized by various methods used as biosensor materials; optical properties are exhibited by AgNPs, ability to enhance wound healing utilized in the medical industry due to their antibacterial, antifungal, antiviral, anti-inflammatory, and osteoinductive effect.

Furthermore, currently, silver nanoparticles are widely used as agents, photochemical methods, microwave processing, and thermal decomposition of silver oxalate in water and ethylene glycol. Having widely used in bactericidal applications, burns and in wound therapy, etc.. of various shapes and sizes, including laser ablation, gamma irradiation, technique, which provides an extremely low minimum inhibitory concentration in diverse areas, including medicine, catalysis, textile engineering, biotechnology and bioengineering, water treatment, electronics against a range of microbes on the surface of paints and fabrics.

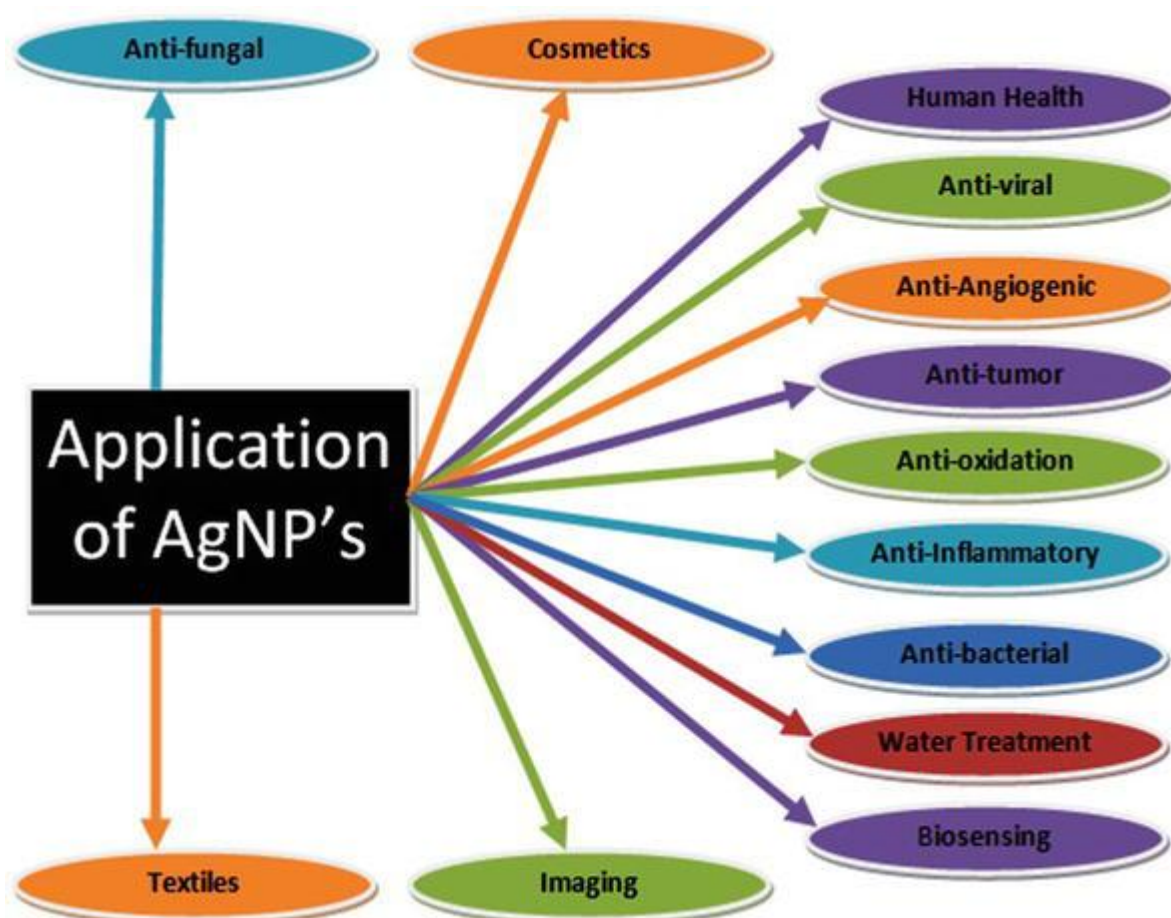


Figure 1.6.1: Applications of Silver Nanoparticles

1.7 METAL OXIDE NANOPARTICLES

Metal oxides are crystalline solids that contain a metal cation and an oxide anion. They typically react with water to form bases or with acids to form salts. Metal oxides

play an important role in numerous zones like chemistry, physics, and materials science. Metal oxides exhibit semiconductor, metallic, or insulator properties and can adopt several structural geometries and they also possess an electronic structure.

Metal oxide nanoparticles have been used in different fields due to their excellent functional properties. When metal oxides are coupled with other structures, they enhance the unique properties of the materials.

The main Metal oxides/ Mixed metal oxides have wide applications as catalysts, Adsorbents, Superconductors, semiconductors, ceramics, and antifungal agents and also have spacious applications in medicines. This review article is focused on their applications as photocatalysts in various organic reactions nanoparticles are shown in the figure.

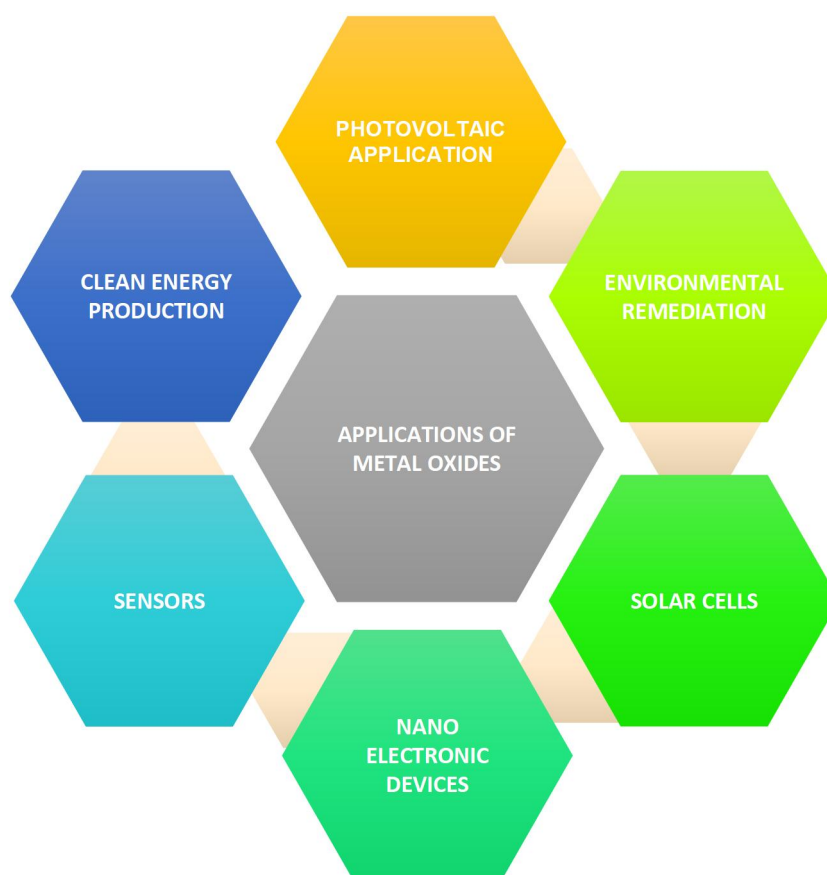


Figure 1.7: Applications of metal oxides

1.7.1 IRON OXIDE (Fe₃O₄) NANOPARTICLE

Iron oxide is a chemical compound with the formula Fe₃O₄. It occurs in nature as the mineral magnetite. It is one of a member of iron oxides.

A large number of elements are known which are plentiful in occurrence and extensively used on Earth. Iron, in the form of various oxides, is one of these known elements which has the potential for different applications.

Various types of oxides of Fe like Fe₃O₄, β-Fe₂O₃, α-Fe₂O₃, and FeO are known to exist. These polymorphs of iron oxide exhibit remarkable chemical and physical properties which are favorable in a wide range of applications.

These magnetic nanoparticles of iron oxides have many uses such as recording material, magnetic resonance imaging, magnetic drug target, environment, and catalysts.

Fe₃O₄ known as magnetite is one of the oxides of iron which plays a major role in various areas of chemistry, material sciences, physics, and medical sciences. Fe₃O₄ crystallizes in mixed oxidation state iron (Fe³⁺ and Fe²⁺) inverse cubic spinel structure.

Fe₃O₄ can be used in magnetic resonance imaging, in drug delivery systems, as a adsorbent for heavy metals, as antibacterial agents, as catalysts, as electrochemical biosensors, as shielding material in electromagnetic interference, and for energy harvesting[11].

Hence, Fe₃O₄ can be confidently tested as a photocatalyst for the remediation of aqueous contaminants under ultraviolet/visible irradiation. The photocatalytic activity of metal oxide, Fe₃O₄, can be further boosted by doping it with metal atoms.

Additionally, this type of iron oxide can easily be separated using a magnet after finishing the treatment process, can be used as a catalyst for the decomposition of adsorbed contaminants and thus reduce sludge formation, and can cost-effectively meet the environmental regulations for wastewater treatment[12].

Iron oxide nanoparticles are iron oxide particles with diameters between about 1 and 100 nanometers. The two main forms are magnetite (Fe_3O_4) and its oxidized form maghemite ($\gamma\text{-Fe}_2\text{O}_3$).

They have attracted extensive interest due to their superparamagnetic properties and their potential applications in many fields (although cobalt and nickel are also highly magnetic materials, they are toxic and easily oxidized).

Applications of iron oxide nanoparticles are magnetic storage devices, catalysis, sensors, superparamagnetic relaxometry, high-sensitivity biomolecular magnetic resonance imaging, separation of biomolecules, and targeted drug and gene delivery for medical diagnosis and therapeutics.

These applications require a coating of the nanoparticles by agents such as long-chain fatty acids, alkyl-substituted amines, and diols. They have been used in formulations for supplementation.

Magnetite has an inverse spinel ($\text{Fe}_3\text{O}_4 \rightarrow \text{FeFe}_2\text{O}_4$) system with oxygen forming a face-centered cubic crystal system. In magnetite, all tetrahedral sites are occupied by Fe^{3+} , and octahedral sites are occupied by both Fe^{3+} and Fe^{2+} .

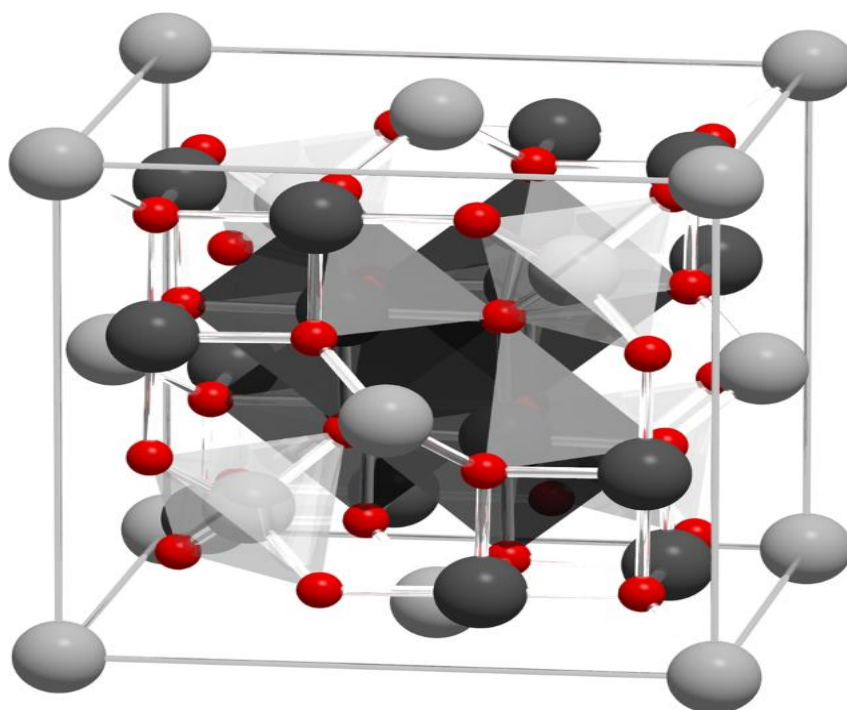


Figure 1.7.1 :Crystal structure of iron oxide(Fe_3O_4)-Magnetite

1.8 MIXED METAL OXIDE NANOPARTICLES

Mixed metal oxide (MMO) nanoparticles (also called heterometal oxide nanoparticles) can play an appreciable role in many areas of chemistry and physics. The unique electronic and magnetic properties obtained when combining two metals in an oxide matrix have been well studied[13]. However, the most common use for MMOs has been in the area of catalysis, and here they have found useful both as catalysts and as catalyst supports. Mixed metal oxides are also used in many applications in the electronic industry as passive or active components in devices. These exhibit high dielectric, and ferro or pyroelectric properties, e.g., BaTiO_3 , LiNbO_3 , KTaO_3 etc. Owing to their refractoriness, these MMOs are difficult to produce by conventional melting techniques; thus, the chemical precipitation technique is used for their preparation. we have covered the most important and recent methods of preparation of nanocrystalline MMOs[13].

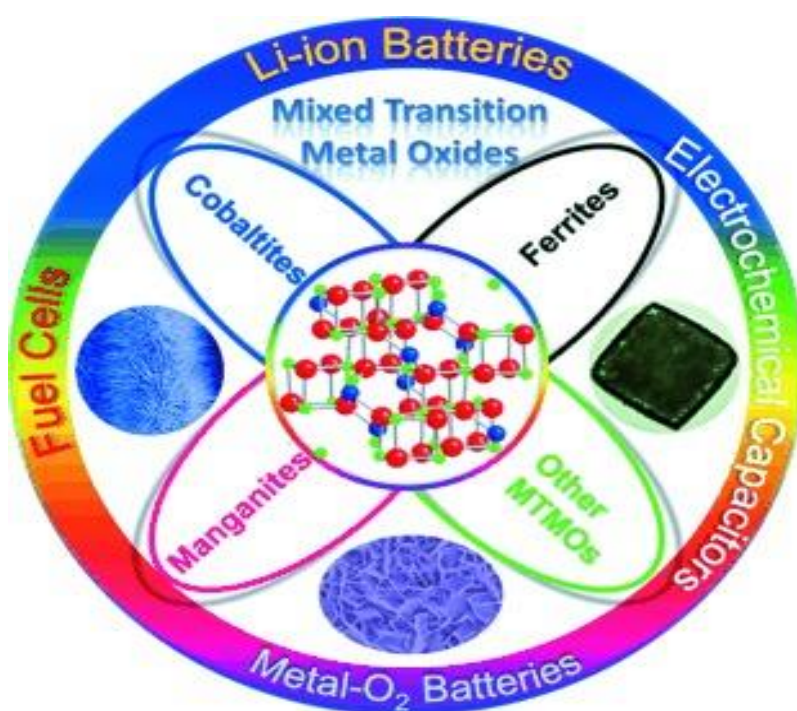


Figure 1.8: Applications of mixed metal oxides

1.8.1 SILVER DECORATED IRON OXIDE NANOCOMPOSITES

Silver decorated Iron oxide is a chemical compound with the formula (Ag- Fe_3O_4). A combination of magnetic iron oxide with silver NPs allows us to obtain nanomaterial with combined properties of both components: magnetic, optical, and antimicrobial[14]. Silver NPs, besides their antimicrobial, anti-fungal, and antiviral activity, enhance optical signals.

Therefore such a combination of silver and iron oxide is expected to exhibit high potential for the photodegradation of textile dyes, in particular as a multi-modal material with therapeutic and optical imaging abilities.

In metal-metal oxide photocatalyst, the photoexcited electron easily shifts to the Fermi level of the doped metal atom via Schottky contact which prevents the de-excitation of electrons and hence it improves the catalytic performance of metal oxide. Magnetic iron-oxide nanoparticles exhibit high efficiency in wastewater treatment for many important reasons, including that they can remove contaminants from wastewater rapidly owing to their high external surface area/unit mass and interstice reactivity.

Additionally, this type of silver-decorated iron oxide can easily be separated using a magnet after finishing the treatment process, can be used as a catalyst for the decomposition of adsorbed contaminants and thus reduce sludge formation, and can cost-effectively meet the environmental regulations for wastewater treatment.

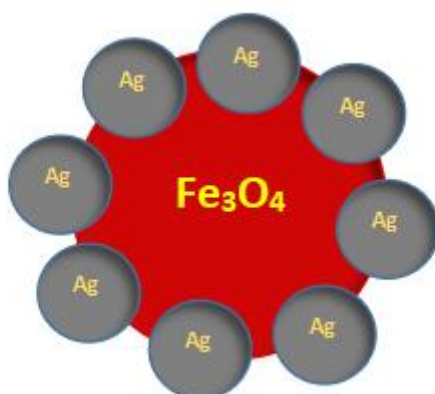


Figure 1.8.1: Structure of silver decorated iron oxide

1.9 PHOTOCATALYST

Materials that cause photo-catalytic reactions when exposed to light are called photo-catalyst. A chemical reaction caused by photo absorption of a solid substance, or "photocatalyst," which stays unchanged throughout the reaction is a general description of photocatalysis. As a result, a "photocatalyst" must act catalytically, that is, without modification, when exposed to light[15].

1.9.1 PHOTOCATALYSIS

Photocatalysis is a combination of the Greek words photo (light) and catalysis (to break apart or decompose). In general, photocatalysis is used to describe a mechanism in which light is used in the excitation of photocatalysts. Photocatalyst is not involved in accelerating the rate of the chemical reaction.

It is the acceleration of a photoreaction in the presence of a catalyst. In catalyzed photolysis, light is absorbed by an adsorbed substrate. In photogenerated catalysis, the photocatalytic activity (PCA) depends on the ability of the catalyst to create electron–hole pairs, which generate free radicals (e.g. hydroxyl radicals: $\bullet\text{OH}$) able to undergo secondary reactions. Its practical application was made possible by the discovery of water electrolysis by means of titanium dioxide (TiO_2).

In other words, photocatalysis is a photoinduced reaction that is stimulated in the presence of a catalyst. Photocatalysis is broadly classified into two categories: homogeneous and heterogeneous[16].

- Homogeneous catalysis:

When both the photocatalyst and the reactant are in the same phase then the reaction is called as homogeneous catalysis. Most common examples of homogeneous catalysis are dyes, coordination compounds, natural pigments and so on.

- Heterogeneous catalysis:

In this type of reaction, the photocatalyst and the reactant exist in different phases. An example of heterogeneous catalysis is transition metal chalcogenides which exhibit unique characteristics.

1.9.2 MECHANISM OF PHOTOCATALYSIS

When the photocatalyst is irradiated with light of a specific wavelength, an electron is excited to the conduction band (CB), leaving a positive hole in the valence band (VB). The hole in the VB can be utilized for oxidizing some compounds whereas the electron in the CB is used to reduce any substrate. This is shown in the figure given below[16].

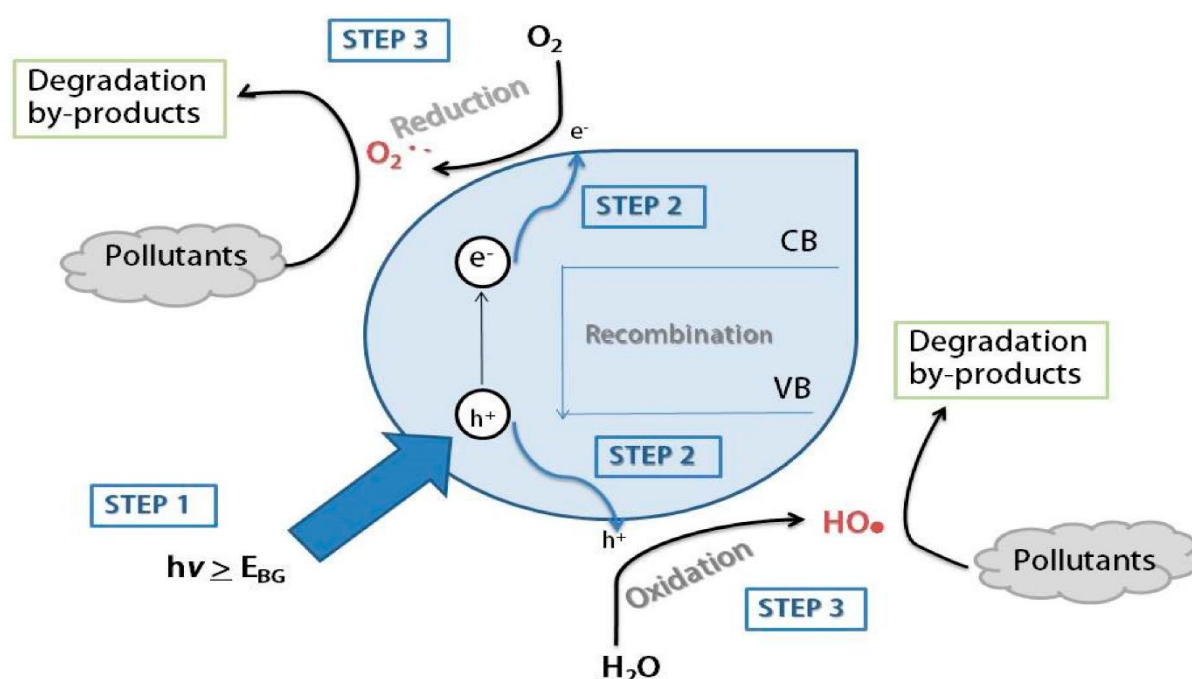


Figure 1.9.2 : Mechanism of photocatalysis

1.9.3 APPLICATIONS OF PHOTOCATALYST

The Photocatalytic technique has been proven to be one of the most “green” and successful environmental remediation techniques. Some semi-conducting materials, such as 'photocatalysts,' cause or accelerate chemical reactions when exposed to light, resulting in the decomposition of organic molecules, for example, Nanosized catalyst particles have a slightly higher reactivity than larger particles or bulk content owing to their large surface area. Charge separation happens when a photocatalyst is exposed to light with an energy greater than the photocatalyst's band gap energy.

The photocatalyst then initiates an oxidation-reduction reaction using the electrons and holes produced by charge separation[16]. Visible absorption spectroscopy is generally used for the photocatalytic degradation of dyes[17]. The major role of the photocatalyst is shown in the figure.

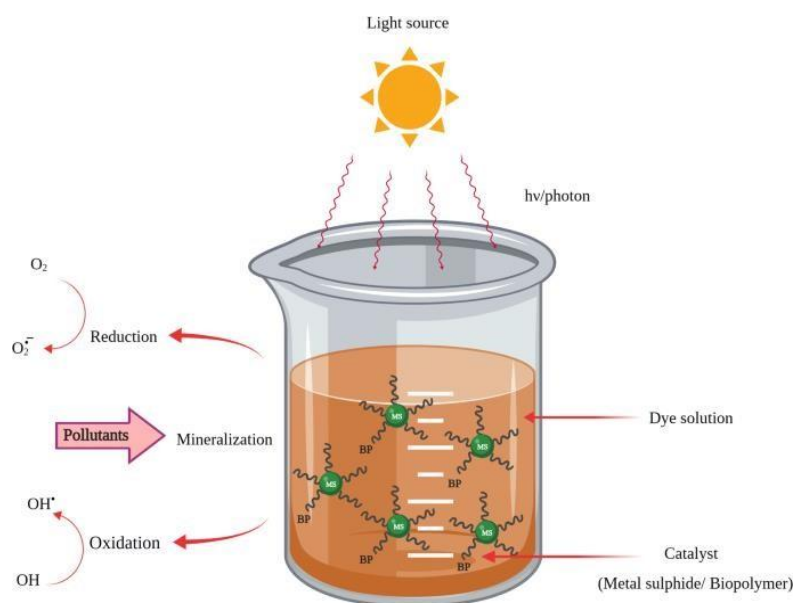


Figure 1.9.3: Role of photocatalyst

Photocatalytic degradation has gained popularity as and is used as a tool for treating dye-contaminated water among many treatment methods. Nanoparticles, nanowires, 2D compounds, planar films, 3D porous films, and complex hierarchical nanostructures are just some of the structures and morphologies found in photocatalysts. A variety of implementations have been developed to get the most out of these photocatalysts .

CHAPTER-2

LITERATURE SURVEY

1.Title: Biogenic Synthesis of Silver-Iron Oxide Nanoparticles Using Kulekhara Leaves Extract for Removing Crystal Violet and Malachite Green Dyes from Water.

Authors : Haradhan Kolya and Chun-Won Kang

Journal and year : November 28, 2022, (<https://doi.org/10.3390/su142315800>)

Inference : Elaboration of (Ag-Fe₃O₄) nanocomposite and synthesis from the leaves extract. widely discussed about the textile effluents and to provide the ways to removing those dyes using the silver-iron oxide nanoparticles.

2. Title: Synthesis of silver nanoparticles decorated on core-shell structured tannic acid-coated iron oxide nanospheres for excellent electrochemical detection and efficient catalytic reduction of hazardous 4-nitrophenol.

Authors: Arumugam Sangili, Muthaiah Annalakshmi, Shen Ming Chen, Paramasivam Balasubramanian a, Mahalingam Sundrarajan

Journal and year: April 2019, <https://doi.org/10.1016/j.compositesb.2018.10.084>

Inference: In this research, synthesis of silver nanoparticles (Ag NPs) decorated on tannic acid covered magnetite (TA@Fe₃O₄-AgNPs) is reported. TA (carbon-shell) on Fe₃O₄ nanospheres (core) acted as a green reducing and stabilizing agent for the reduction of Ag ions into Ag NPs. The as-synthesized TA@Fe₃O₄-AgNPs core-shell nanohybrid was employed as an efficient nanomaterial for the electrochemical detection and catalytic reduction of ecotoxic 4-nitrophenol (4-NP). TA@Fe₃O₄-AgNPs nanohybrid

modified glassy carbon electrode (GCE) displayed a higher cathodic current response at a very lower over potential of -0.39 V toward 4-NP detection.

3.Title: High adsorptivity and visible light assisted photocatalytic activity of silver/reduced graphene oxide (Ag/rGO) nanocomposite for wastewater treatment

Authors: panelJimmy Mangalam , Manish Kumar , Mahima Sharma , Monika Josh

Journal and year: february 2019 <https://doi.org/10.1016/j.nanoso.2018.11.003>

Inference: The present study entails a simple chemical method for the preparation of high-quality silver / reduced graphene oxide (Ag/rGO) nanocomposite. The photocatalytic activity and stability of Ag/rGO nanocomposite for the degradation of methyl orange (MeO) dye was significantly increased in comparison to GO nanosheets under visible light. The adsorption capacity of Total Dissolved Solids (TDS) by Ag/rGO nanocomposite and GO nanosheets were further studied by utilizing wastewater as sample. The adsorption isothermal and kinetic studies suggest that 0.4 g/L Ag/rGO nanocomposite produce optimum separation capacity. Therefore, considering the excellent photocatalytic activity and adsorptivity nature of Ag/rGO nanocomposite, the present investigation can be effective in energy and environment related application.

4.Title: Silver nanoparticles in dye effluent treatment: A review on synthesis, treatment methods, mechanisms, photocatalytic degradation, toxic effects and mitigation of toxicity

Authors:panelSivasankari Marimuthu, ArulJayanthi Antonisamy, Sankar Malayandi, Karthikeyan Rajendran , Pei-Chien Tsai, Arivalagan Pugazhendhi , Vinoth Kumar Ponnusamy

Journal and year: April 2020 <https://doi.org/10.1016/j.jphotobiol.2020.111823>

Inference: On the contrary, studies have also recognized the mechanisms of silver nanoparticle-mediated toxicity even at deficient concentrations and their deleterious biological effects when present in treated water. Hence, the fate of the silver nanoparticles released into the treated water and sludge, contaminating the soil, aquatic environment, and underground water is of significant concern. This review summarizes the current state of knowledge regarding the use of silver nanoparticles and silver-based nanocomposites in effluent treatment and comprehends the recent research on mitigation of silver nanoparticle-induced toxicity.

5. **Title:** Synthesis of zinc oxide nanoparticles mediated by *Dictyota dichotoma* endophytic fungi and its photocatalytic degradation of fast green dye and antibacterial applications.

Authors: panel R. Vasanth Kumar, S. Vinoth, V. Baskar, M. Arun, P. Gurusaravanan

Journal and year: December 2022 <https://doi.org/10.1016/j.sajb.2022.03.016>

Inference: Biosynthesis of nanoparticles has received considerable importance because of their ecofriendly, cost effective and wide applications in biological fields. Aim of this study is to synthesis zinc oxide nanoparticle (ZnO __ NPs) using extra cellular product from isolated marine. The UV photoluminescence study exhibited the absorbance peak at the resonance of 270 nm which confirms the presence of ZnO __ NPs in the aqueous solution. The perfect sphere shape with ~ 80–100 μm in size of ZnO __ NPs has been observed under FESEM and confirmed the presence of elemental composition of nanoparticles by EDX. The synthesized nanoparticles showed potential dye degradation efficiency (~ 90%) under photo illumination. In addition, ZnO __ NPs have shown significant antibacterial effects in terms of zone of inhibition and antibiofilm activity against pathogenic bacteria.

CHAPTER- 3

AIM AND SCOPE OF PRESENT INVESTIGATION

This project work is mainly focused on the following objectives:

- To synthesis novel Ag-Fe₃O₄ nanocomposite by a chemical precipitation method.
- Characterization of the nanocomposite using XRD, UV, FT-IR, SEM.
- Determine morphology and size of the nanocomposites.
- Photocatalytic degradation of the crystal violet dye using Ag-Fe₃O₄ nanocomposite.
- Summarize the obtained results.

CHAPTER-4

4. EXPERIMENTAL SYNTHESIS TECHNIQUES

4.1 EXPERIMENTAL SYNTHESIS OF (Ag-Fe₃O₄)

Silver-decorated iron oxide(Ag-Fe₃O₄) has been synthesized in the chemical precipitation method as we discussed below.

4.2 MATERIALS AND INSTRUMENTS

The section deals with the details of all reagents employed in this study and the details regarding methods of preparation of various solutions. Doubly distilled water, Ethanol, and Acetone were used for cleaning glassware, which was used for handling chemicals in practical work and double distilled water was used for preparing all solutions.

Iron chloride

Iron(III) chloride describes the inorganic compound with the Molar Mass: 162.21 g/mol Chemical Formula: FeCl₃ also called ferric chloride, these compounds are available in anhydrous form. They are common sources of iron in its +3 oxidation state.

Silver Nitrate

Silver nitrate (Merck) Molar Mass: 169.88 g/mol Chemical Formula: AgNO₃

Silver nitrate is an inorganic compound and colorless crystalline solid. Lunar caustic is another name for silver nitrate. A solution of silver nitrate was prepared by dissolving a required amount of AgNO₃ with the addition of double distilled water. The prepared solution was used suddenly to avoid photodecomposition.

Sodium Hydroxide

Sodium hydroxide (Merck) Molar Mass: 40 g/mol Chemical Formula: NaOH

Sodium hydroxide solution was prepared by dissolving approximately weighed pellets of NaOH in double distilled water.

Ethylene glycol

Ethylene glycol is an organic compound with the formula (CH₂OH)₂ Molar Mass: 62.07 g/mol. It is an odorless, colorless, flammable, viscous liquid. Ethylene glycol has a sweet taste, but it is toxic in high concentrations.

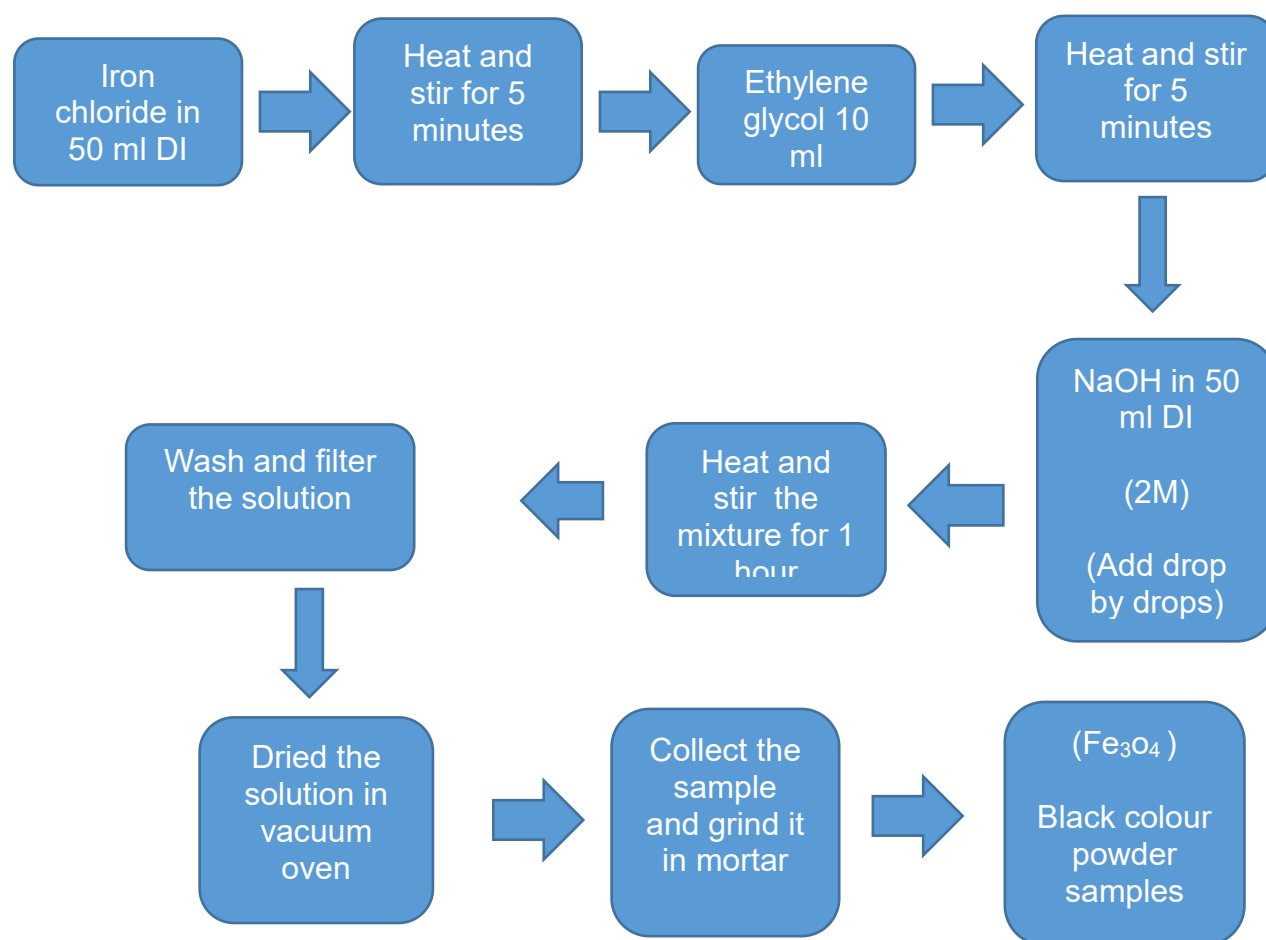
Stirrer with Hot Plate

A magnetic stirrer with the hot plate was used for the synthesis of nanoparticles at 600 rpm (range 0-1200 rpm), stirrer work on 220/230 volts AC supply, and temperature 0-200 °C. The synthesis process of nanoparticles and the size of nanoparticles are also affected by the speed of stirring and reaction temperature.

Vacuum Oven

Centrifuged samples were dried in a vacuum oven at 120 °C for 2 hours. The oven consists double wall with an inner round chamber made of stainless steel and an exterior made of mild steel duly finished in white stoving enamel/powder-coated paint with mat-finished color combinations. The oven work at the temperature range of 50 °C to 250 °C. workable on 220 VAC 50 Hz single phase and dried samples were used for further characterization.

4.3 SYNTHESIS OF Fe_3O_4 NANOCOMPOSITE



The synthesis of iron oxide nanocomposite has been synthesized in the chemical precipitation method, firstly I took 50 ml of 0.5M Iron chloride by using deionized water and heat at 120 degrees temperature.

Stir and mix the solution for five minutes in a magnetic stirrer and then add 10 ml of ethylene glycol.

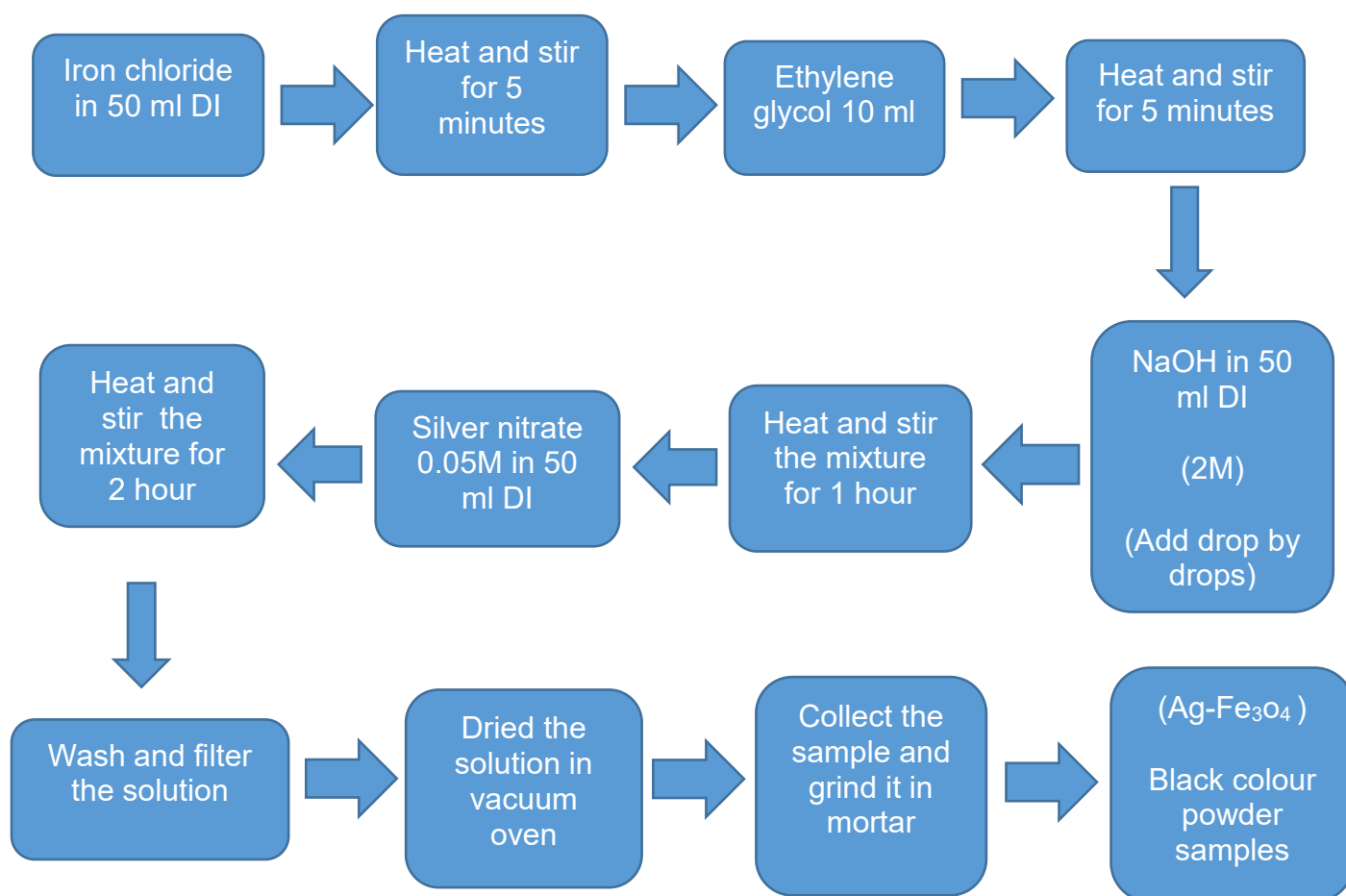
Stir this mixed solution for 5 minutes in a magnetic stirrer and then take 4g of sodium hydroxide in 50 ml water in another beaker stir the hydroxide pellets completes it gets dissolved.

After completely dissolving the sodium hydroxide pellets, add this 50 ml sodium hydroxide solution into the solution stirring in a magnetic stirrer by using a dropper drop by drop until the solution completes

After adding the sodium hydroxide solution covers the whole solution with aluminium foil. Allow this solution to be stirred for 1 hour after adding the sodium hydroxide with temperature.

After some time drop the water where it comes from the sample, and finally, clean the sample with acetone. after washing with acetone remaining will be kept in the vacuum oven at 120 degrees for 12 hours, after drying the solution take it out from the oven and collect the sample by grinding by using mortar. After grinding the sample collect it and store it in a cleaned packing container.

4.4 SYNTHESIS OF Ag-Fe₃O₄ NANOCOMPOSITE



The synthesis of silver-decorated iron oxide nanocomposite has been synthesized in the chemical precipitation method, firstly I took 50 ml of 0.5M Iron chloride by using deionized water and heat at 120 degrees temperature.

Stir and mix the solution for five minutes in a magnetic stirrer and then add 10 ml of ethylene glycol.

Stir this mixed solution for 5 minutes in a magnetic stirrer and then take 4g of sodium hydroxide in 50 ml water in another beaker stir the hydroxide pellets completes it gets dissolved.

After completely dissolving the sodium hydroxide pellets, add this 50 ml sodium hydroxide solution into the solution stirring in a magnetic stirrer using a dropper drop by drop until the solution completes

After adding the sodium hydroxide solution covers the whole solution with aluminium foil. Allow this solution to be stirred for 1 hour after adding the sodium hydroxide with temperature.

Stir this mixed solution for 1 hour in a magnetic stirrer and then make up 50 ml of 0.05 M of silver nitrate using de-ionized water in another beaker, stir until the silver nitrate gets dissolved completely.

After completely dissolving the silver nitrate, add this 50 ml of 0.05M of silver nitrate(AgNO_3) into the solution were stirring it in a magnetic stirrer After completing the 2-hour stirring allow that solution for cooled and settle. after settling the solution drop the water and one more time add water and allow it to settle down.

After some time drop the water where it comes from the sample, and finally, clean the sample with acetone. after washing with acetone remaining will be kept in the vacuum oven at 120 degrees for 12 hours, after drying the solution take it out from the oven and collect the sample by grinding by using mortar. After grinding the sample collect it and store it in a cleaned packing container.

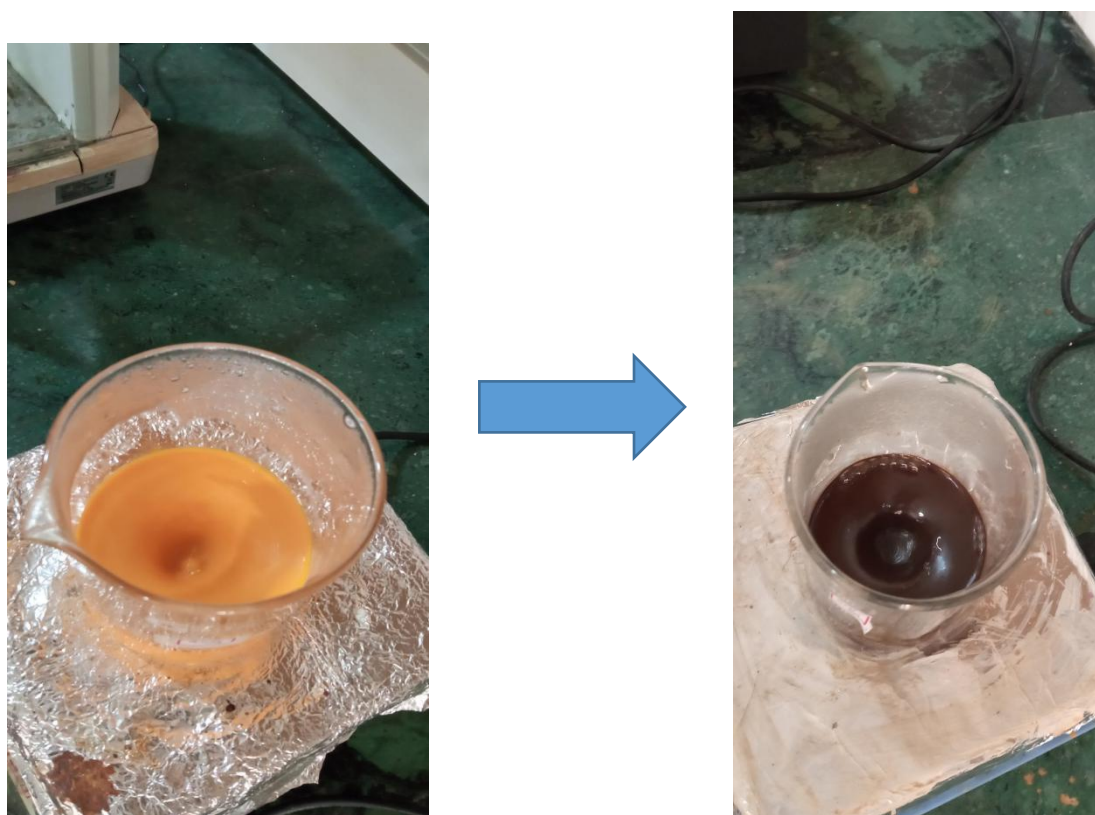


Figure 4.4(a): synthesis of Ag- Fe_3O_4 nanocomposite

Ag-Fe₃O₄ nanoparticles thus synthesized at low temperatures were analyzed and the images are displayed in the below figure.



Figure 4.4(b): synthesized samples of Fe₃O₄ and Ag- Fe₃O₄ nanocomposite

Finally, Ag-Fe₃O₄ Nanocomposite will be confirmed by characterization techniques (UV, FTIR, SEM,XRD).

CHAPTER-5

5. CHARACTERISATION 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 [18].

5.1 X-RAY DIFFRACTION (XRD)

XRD is one the characterisation 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 [19].

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 [20].

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.

BRAGG'S LAW

When a material is irradiated with a monochromatic light of wavelength around the inter atomic distance of the material, light gets dispersed by the atoms and interfere to give rise to diffracted light with a higher intensity [21]. The

arrangement of atoms in the material is related to the intensity of the scattered light. If wavelength of light is λ and the incident angle is θ and the constructive interference is obtained at an angle 2θ from the incident beam of light if Bragg's equation is satisfied. Bragg's equation is given by,

$$n\lambda = 2d \sin \theta$$

where,

n is the diffraction order,

d is the distance between the diffracting planes and

θ is the Bragg's angle.

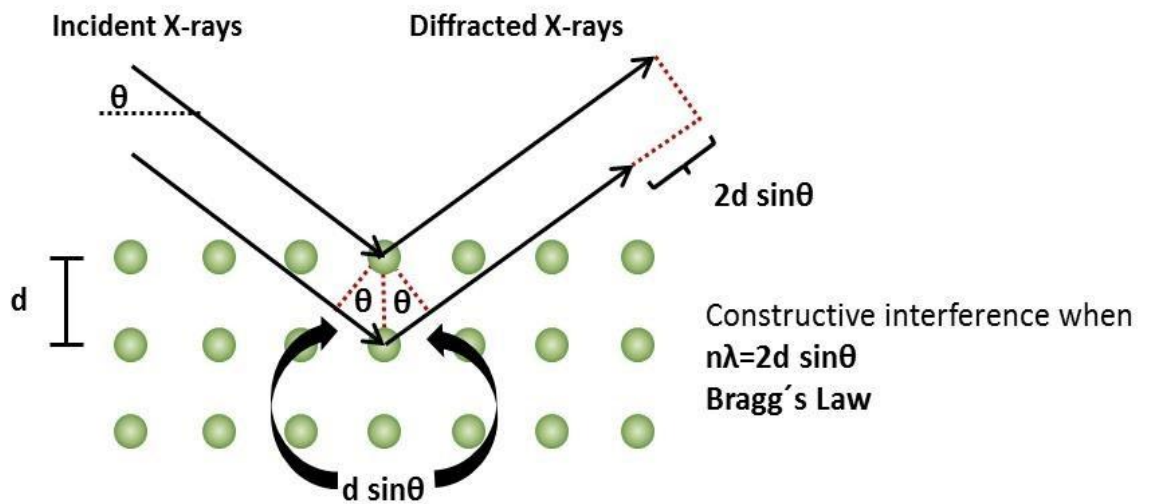


Figure 5.1: schematic representation of Bragg's law

5.2 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 [22].

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 characterise nanoparticulate materials is increasing because of the application of nanoparticles in the commercial products.

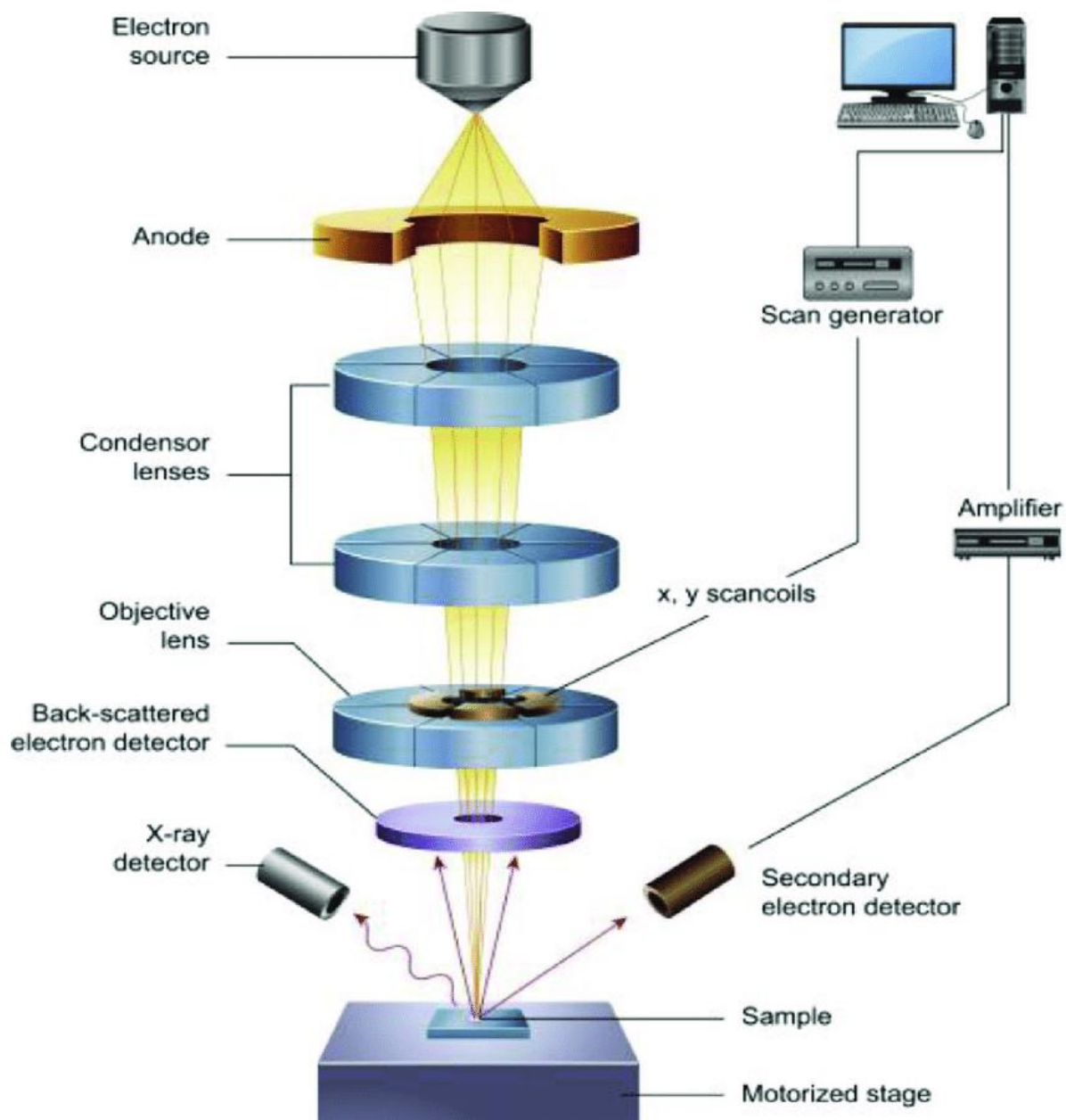


Figure 5.2 : Schematic representation of SEM

5.3 FOURIER-TRANSFORM INFRARED SPECTROSCOPY

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 [23].

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 synthesised nanoparticles, FTIR may be used to test a variety of components, including bulk or thin films, liquids, solids, pastes, powders, fibres, 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 [24].

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.

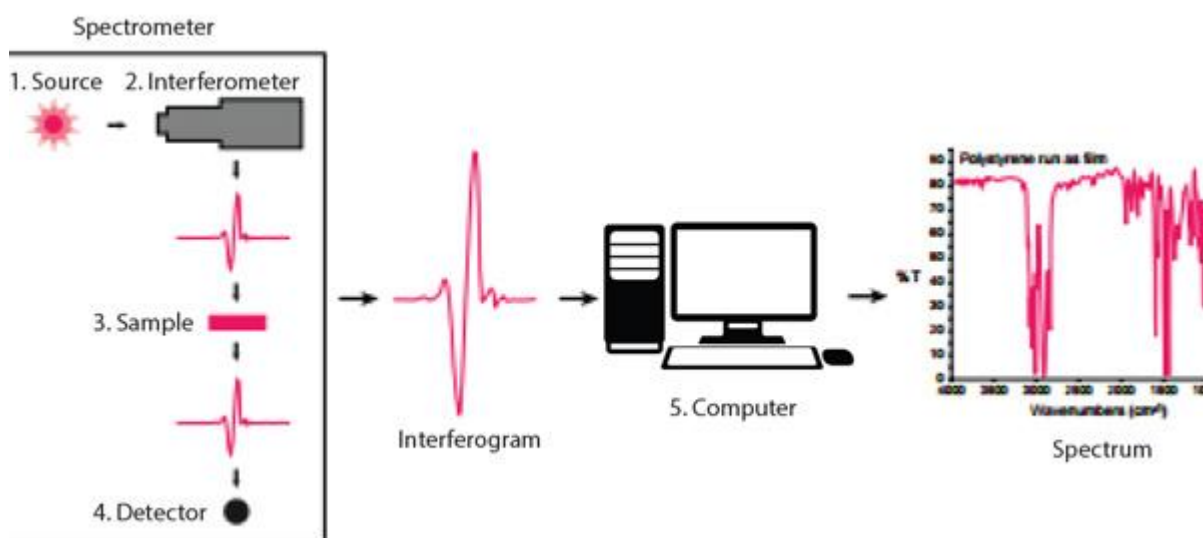


Figure 5.3 : Schematic representation of FTIR

5.4 UV-VISIBLE SPECTROSCOPY

UV-Vis spectroscopy is an analytical technique that measures the amount of discrete wavelengths of UV or visible light that are absorbed by or transmitted through a sample in comparison to a reference or blank sample.

Absorption spectroscopy is complementary to fluorescence spectroscopy, in that fluorescence deals with transitions of electrons from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state [25].

Molecules containing bonding and non-bonding electrons (n-electrons) can absorb energy in the form of ultraviolet or visible light to excite these electrons to higher anti-bonding molecular orbitals.

The more easily excited the electrons (i.e. lower energy gap between the HOMO and the LUMO), the longer the wavelength of light it can absorb. There are four possible types of transitions ($\pi-\pi^*$, $n-\pi^*$, $\sigma-\sigma^*$, and $n-\sigma^*$), and they can be ordered as follows: $\sigma-\sigma^* > n-\sigma^* > \pi-\pi^* > n-\pi^*$

UV/Vis spectroscopy is routinely used in analytical chemistry for the quantitative determination of different analytes or sample, such as transition metal ions, highly conjugated organic compounds, and biological macromolecules. Spectroscopic analysis is commonly carried out in solutions but solids and gases may also be studied [26].

- All diluted inorganic solutions are uv active
 - Solutions of transition metal ions can be colored (i.e., absorb visible light) because d electrons within the metal atoms can be excited from one electronic state to another.
 - The colour of metal ion solutions is strongly affected by the presence of other species, such as certain anions or ligands. For instance, the colour of a dilute solution of copper sulfate is a very light blue; adding ammonia intensifies the colour and changes the wavelength of maximum absorption ($\lambda_{\max}$).
 - While charge transfer complexes also give rise to colours, the colours are often too intense to be used for quantitative measurement.

The Beer–Lambert law states that the absorbance of a solution is directly proportional to the concentration of the absorbing species in the solution and the path length. Thus, for a fixed path length, UV/Vis spectroscopy can be used to determine the concentration of the absorber in a solution. It is necessary to know how quickly the absorbance changes with concentration. This can be taken from references (tables of molar extinction coefficients), or more accurately, determined from a calibration curve [27].

A UV/Vis spectrophotometer may be used as a detector for HPLC. The presence of an analyte gives a response assumed to be proportional to the concentration. For accurate results, the instrument's response to the analyte in the unknown should be compared with the response to a standard; this is very similar to the use of calibration curves. The response (e.g., peak height) for a particular concentration is known as the response factor.

The wavelengths of absorption peaks can be correlated with the types of bonds in a given molecule and are valuable in determining the functional groups within a molecule. The Woodward–Fieser rules, for instance, are a set of empirical observations used to predict λ_{max} , the wavelength of the most intense UV/Vis absorption, for conjugated organic compounds such as dienes and ketones. The spectrum alone is not, however, a specific test for any given sample.

The method is most often used in a quantitative way to determine concentrations of an absorbing species in solution, using the Beer–Lambert law:

where A = absorbance

I = intensity of the incident is the transmitted intensity,

L = the path length

c = *concentration of absorbing species*

ϵ = molar absorptivity coefficient.

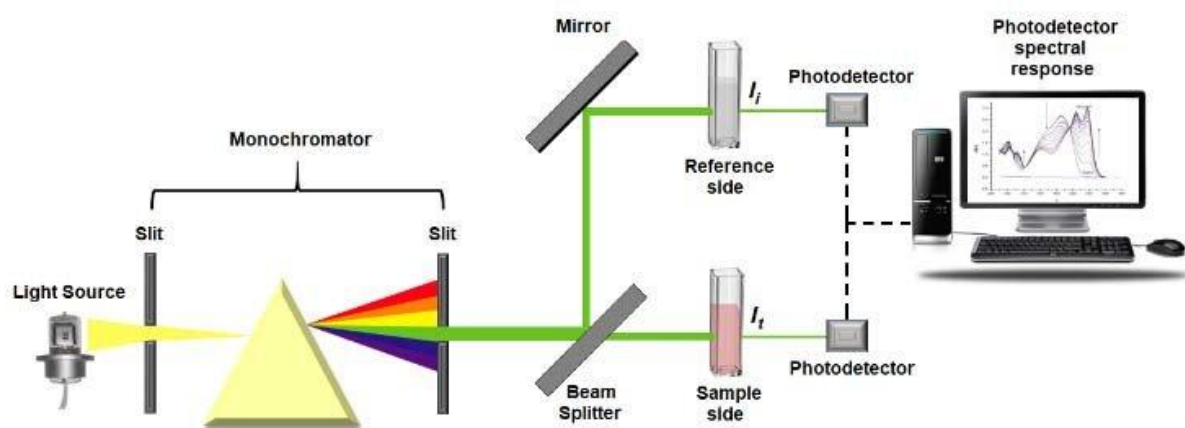


Figure 5.4 : schematic representation of uv-visible spectroscopy

CHAPTER - 6

RESULTS 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.

6.1 X-RAY DIFFRACTION (XRD)

Silver decorated iron oxide were elaborated with different ratios to form a homogeneous nanocomposite. X-ray diffraction (XRD) was used for detecting the crystalline phases represented in below figure.

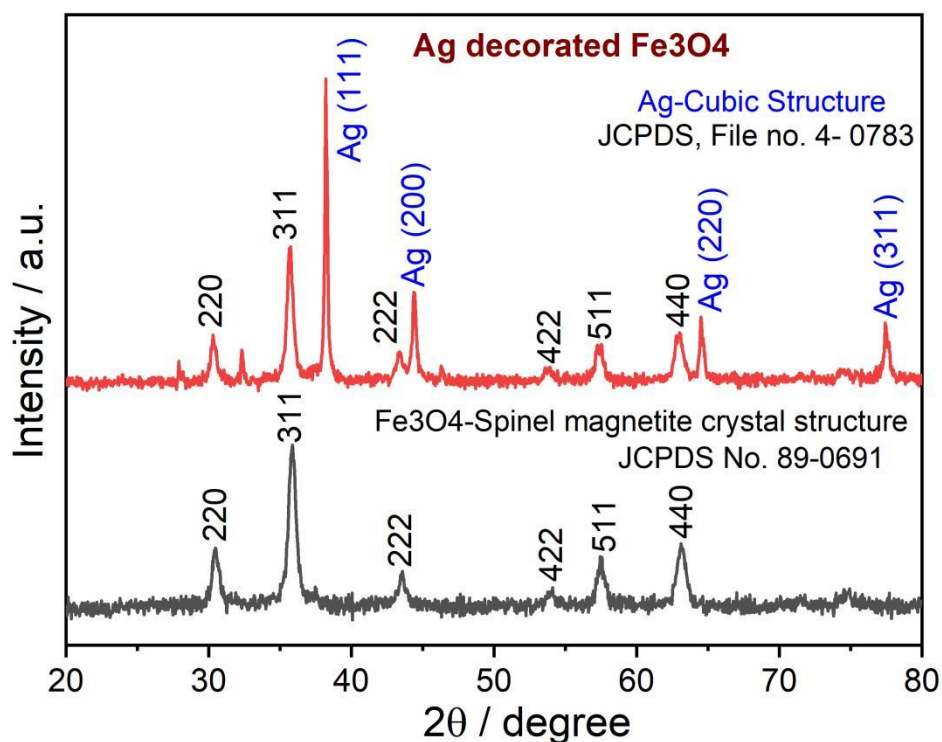


Figure 6.1 : XRD of Ag-Fe₃O₄

S.NO	PLANE	2 THETA(DEGREE)
1.	Fe ₃ O ₄ (220)	30.30
	Fe ₃ O ₄ (311)	35.85
	Fe ₃ O ₄ (222)	43.50
	Fe ₃ O ₄ (422)	54.00
	Fe ₃ O ₄ (511)	57.50
	Fe ₃ O ₄ (440)	64.10
2.	Ag-Fe ₃ O ₄ (111)	38.25
	Ag-Fe ₃ O ₄ (200)	44.40
	Ag-Fe ₃ O ₄ (220)	64.50
	Ag-Fe ₃ O ₄ (311)	77.49

The first set magnetite Fe₃O₄ phase with diffraction peaks at 30.30, 35.70, 43.50, 54.00, 57.50, and 64.10 corresponding to Miller indices (200), (311), (222), (422), (511), and (440) respectively and for Ag is well indexed to cubic structure with the planes (111), (200), (220), and (311) corresponding to 2 Θ = 38.25, 44.40, 64.5, and 77.49.

These peaks are attributed to the cubic spinal structured magnetite. No significant peaks corresponding to impurities were detected in the XRD pattern. After mixing Ag -Fe₃O₄, peaks of Silver and iron oxide are detected. In the nanocomposite, some intensities of the peaks changed due to peak interference.

Ag-Fe₃O₄ composites show the average crystallite diameter of 10.3 nm for Ag-Fe₃O₄ by using the scherrer equation ,respectively as the nanocomposites structure.

6.2 SCANNING ELECTRON MICROSCOPE (SEM)

Scanning electron microscope is especially used for size and shape characterization, because the required sample preparation and image analysis is relatively quick and simple [28].The scanning Electron Microscopy

(SEM) images allowed a detailed study on the morphological structure of the nanoparticles [29].

The scanning electron microscope operates in the same manner as an optical microscope, but instead of detecting photon, it detects electrons scattered from the sample. The wavelength of electrons can be made shorter than that of photons because electrons can be accelerated by an electric potential. As a result, the SEM will magnify pictures up to 200.000 times.

The morphological variations in the synthesised sample nanoparticles were detected and analysed. Iron oxide analysed by SEM is shown in figure.

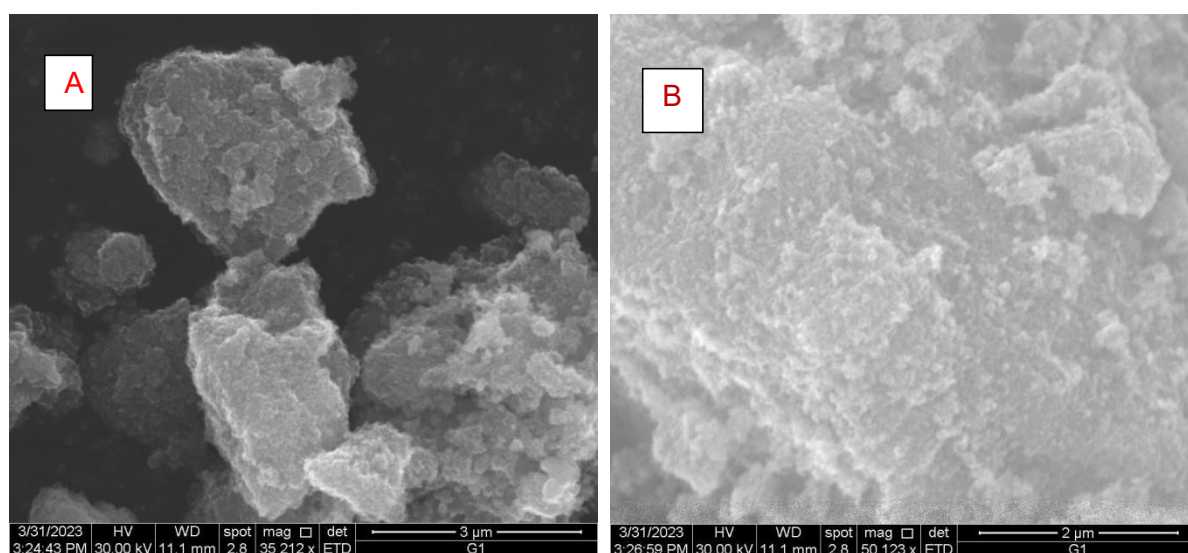


Figure 6.2 (A-B) Different magnifications of SEM images of the Fe₃O₄ nanocomposite.

The morphological variations in the synthesised sample nanoparticles were detected and analysed. Silver decorated Iron oxide analysed by SEM is shown in figure.

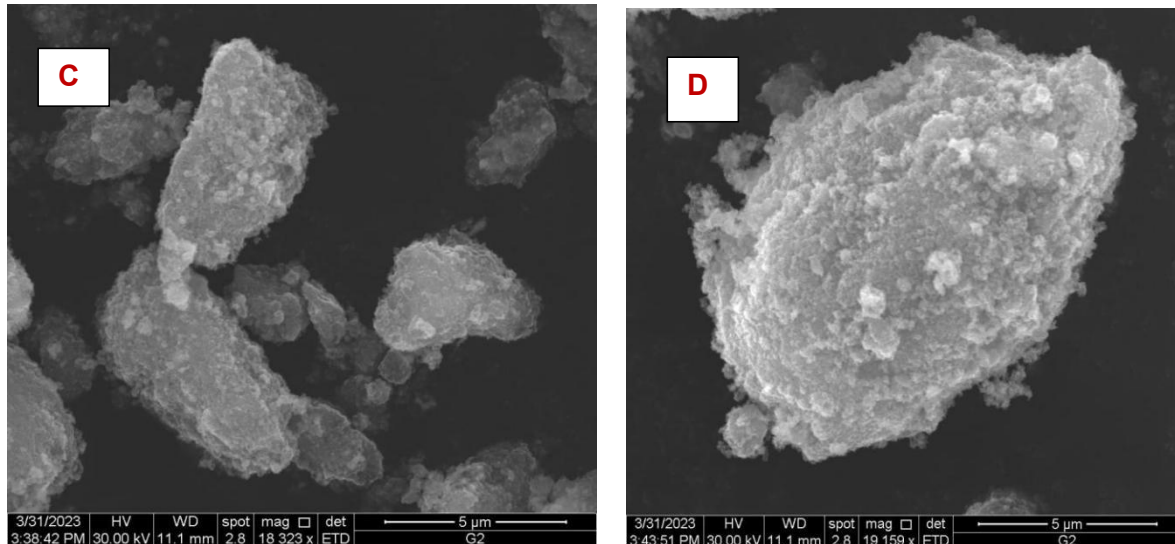


Figure 6.2 (C-D) Different magnifications of SEM images of the Ag-Fe₃O₄ nanocomposite.

Ag-Fe₃O₄ composites show the cubic and spherical morphology respectively as the nanocomposites structure.

6.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY

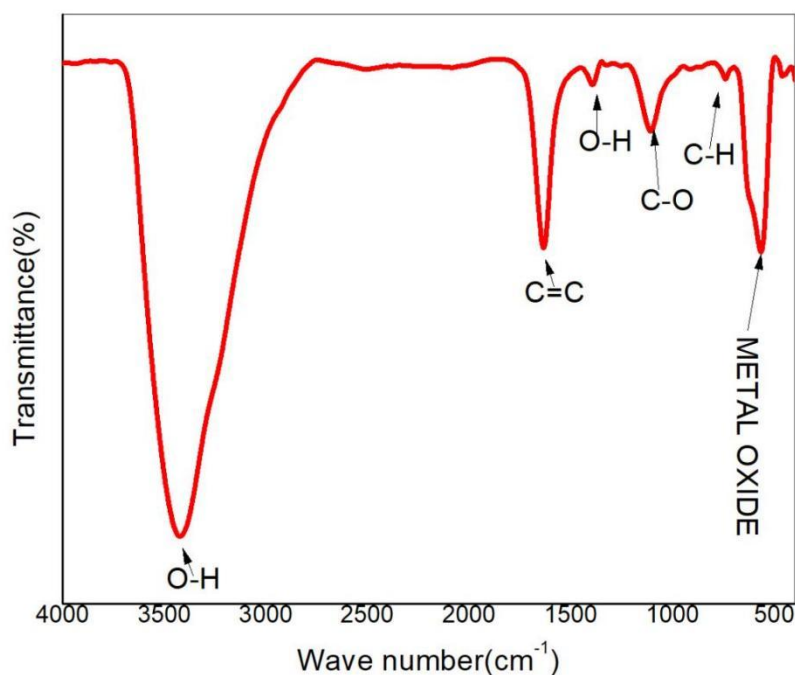


Figure 6.3(a): FTIR OF Fe₃O₄ NANOCOMPOSITE

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

S.NO	FUNCTIONAL GROUP	WAVENUMBER (CM ⁻¹)
1	O-H	3420
2	C=C	1630
3	O-H	1390
4	C=O	1107
5	C-H	748
6	METAL OXIDE	560

The above table shows that the various functional groups present at different wavenumbers, in this O-H group observed at 3420 cm⁻¹ then alkene group was observed at 1630 cm⁻¹ then again O-H group was observed at 1390 cm⁻¹

and then C=O group was observed at 1107 cm^{-1} after this C-H group was observed at 740 cm^{-1} and finally metal oxide peak was observed at 560 cm^{-1} .

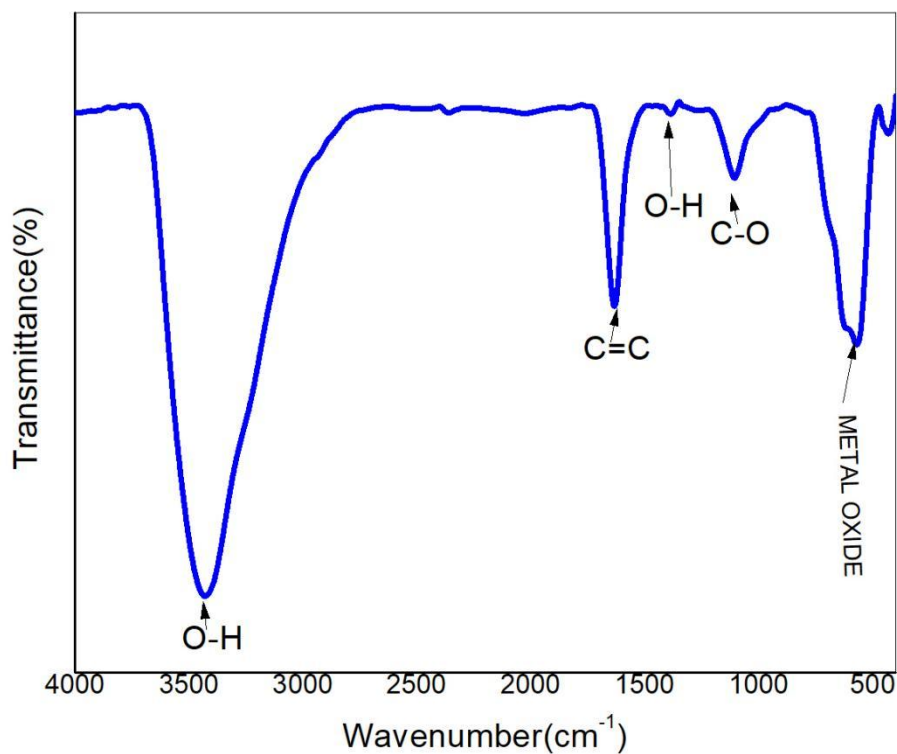


Figure 6.3(b): FTIR OF Ag-Fe₃O₄ NANOCOMPOSITE

S.NO	FUNCTIONAL GROUP	WAVENUMBER (CM^{-1})
1	O-H	3420
2	C=C	1630
3	O-H	1390
4	C=O	1107
5	METAL OXIDE	560

The above table shows that the various functional groups present at different wavenumbers, in this O-H group observed at 3430 cm^{-1} then alkene group was observed at 1630 cm^{-1} then again O-H group was observed at 1380 cm^{-1} and then C=O group was observed at 1107 cm^{-1} and finally metal oxide peak was observed at 560 cm^{-1} and the peak is slightly deviated because of Ag nanoparticles.

6.4 UV-VISIBLE SPECTROSCOPY

Ultraviolet-visible (UV-Vis) spectroscopy is a widely used technique in many areas of science ranging from bacterial culturing, drug identification and nucleic acid purity checks and quantitation, to quality control in the beverage industry and chemical research.

It is a quantitative technique used to measure how much a chemical substance absorbs light. This is done by measuring the intensity of light that passes through a sample with respect to the intensity of light through a reference sample or blank.

This spectroscopy technique is also used to determine the absorption of the given nanocomposite under suitable conditions.

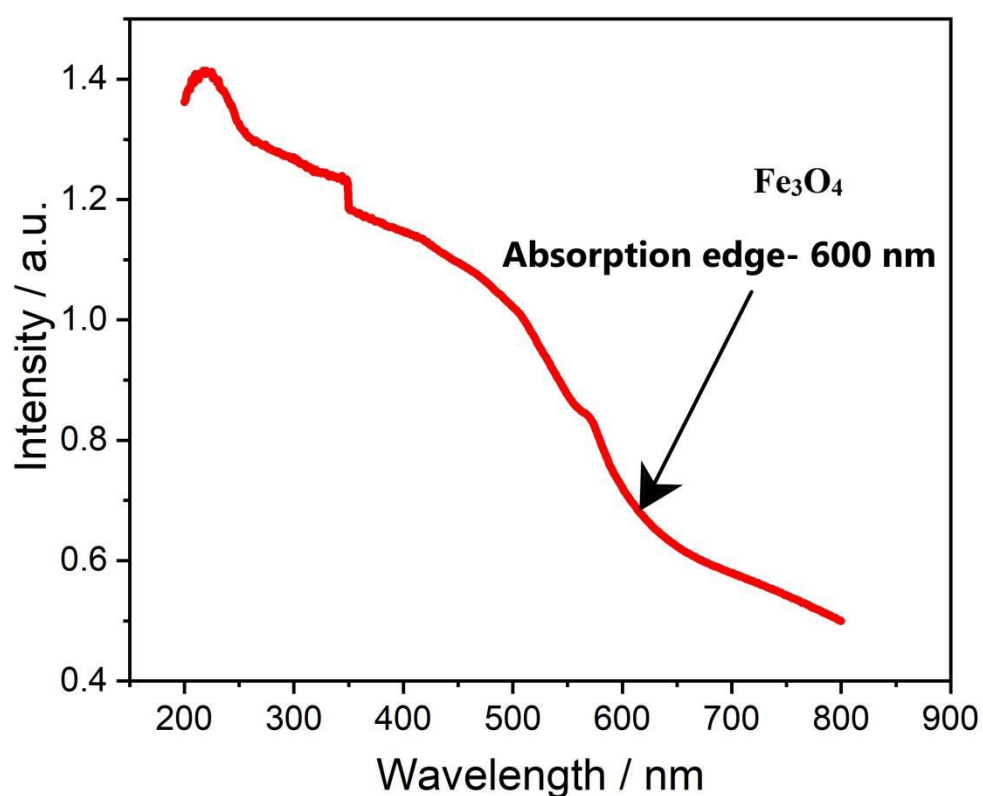


Figure 6.4(a) : UV-VISIBLE SPECTRA OF Fe₃O₄

From the graph, we come to know that the absorption edge was obtained at 600 nm. And this iron oxide nanocomposite shows the band gap at 2.06 eV. This means our compound shows that the good conductivity and good electron absorber.

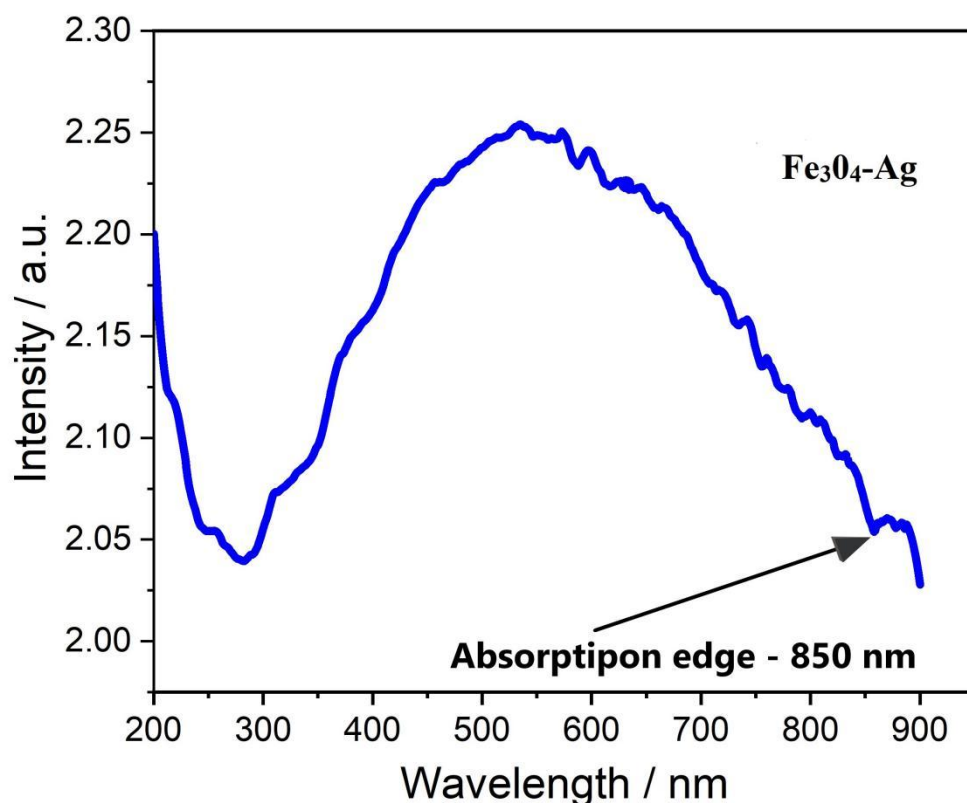


Figure 6.4(b): UV-VISIBLE SPECTRA OF Ag-Fe₃O₄

From the graph, we come to know that the absorption edge was obtained at 850 nm.

The band gap (E_G) is the gap in energy between the bound state and the free state, between the valence band and conduction band. Therefore, the band gap is the minimum change in energy required to excite the electron so that it can participate in conduction. Schematic of the energy bands for electrons in a solid.

And this Silver decorated iron oxide nanocomposite shows the band gap at 1.45 eV. This means our compound shows that the good conductivity and good electron absorber in visible region than iron oxide. The addition of silver shows high absorbance in the visible region.

6.5 PHOTO DEGRADATION STUDY

6.5.1 Photocatalytic activity test

Fast green (FG) was chosen as the model organic pollutants to test the photocatalytic dye degradation analysis for the catalyst. Typically, a catalysts (20 mg) was added into the 50 ml of aqueous dye solution (20 mg L^{-1}) then, the dye solution was kept in dark room to attain the adsorption and desorption equilibrium of dyes on surface of catalyst. Then, the dye solution was irradiated on sunlight for overall time period of 120 minutes. 2 ml of solution was taken at appropriate interval of time. The collected solution was centrifuged and filtered using a $0.2 \mu\text{m}$ syringe filter. The degradation efficiency is determined by the degradation of organic dyes on UV-Vis spectrophotometer. The degradation efficiency is calculated by the formula,

$$\text{Degradation efficiency (\%)} = \frac{C - C_0}{C} \times 100 \quad (1)$$

Where C and C_0 are initial and final concentration of dye molecules. After 1st completed the photocatalytic reaction, the catalyst was collected by centrifuge and washed with water and dried at 70°C . The regenerated catalyst was used to evaluate the stability and reusability of catalyst by degradation the dye solution.

The first order kinetic reaction value was used to determine the correct degradation rate of FG as follows;

$$-\ln(C/C_0) = kt \quad (2)$$

Where, k = rate of reaction constant and t is time. The Photocatalytic dye degradation activity were performed during the well sun shining between 11:00 AM to 2:00 PM in clear sky. The average solar light intensity was measured about $1.00 \times 10^5 \text{ lux}$.

6.5.2. Photocatalytic activity

The photocatalytic degradation efficiency of as prepared catalysts of Fe_3O_4 , and $\text{Fe}_3\text{O}_4\text{-Ag}$ nanocomposites were resolute by the deprivation of FG dye under irradiation of Sun light. When irradiating the FG dye solution, the concentration of the dye was noticeably reduced, and it was confirmed by the significant color variation.

For Fe_3O_4 catalyst, the catalyst influences the organic molecule to achieve the efficiency of about 80.2% within 120 minutes. The photocatalyst experiment was further extended to nanocomposite sample. The degradation reaction of $\text{Fe}_3\text{O}_4\text{-Ag}$ sample was performed by the same process. The rate of electron-hole pair recombination was delayed by doping the metal ions into the Fe_3O_4 structure results increased the degradation efficiency. Compared to Fe_3O_4 catalyst, $\text{Fe}_3\text{O}_4\text{-Ag}$ catalysts reached high efficiency of degradation of 93.5%, respectively[1,2].

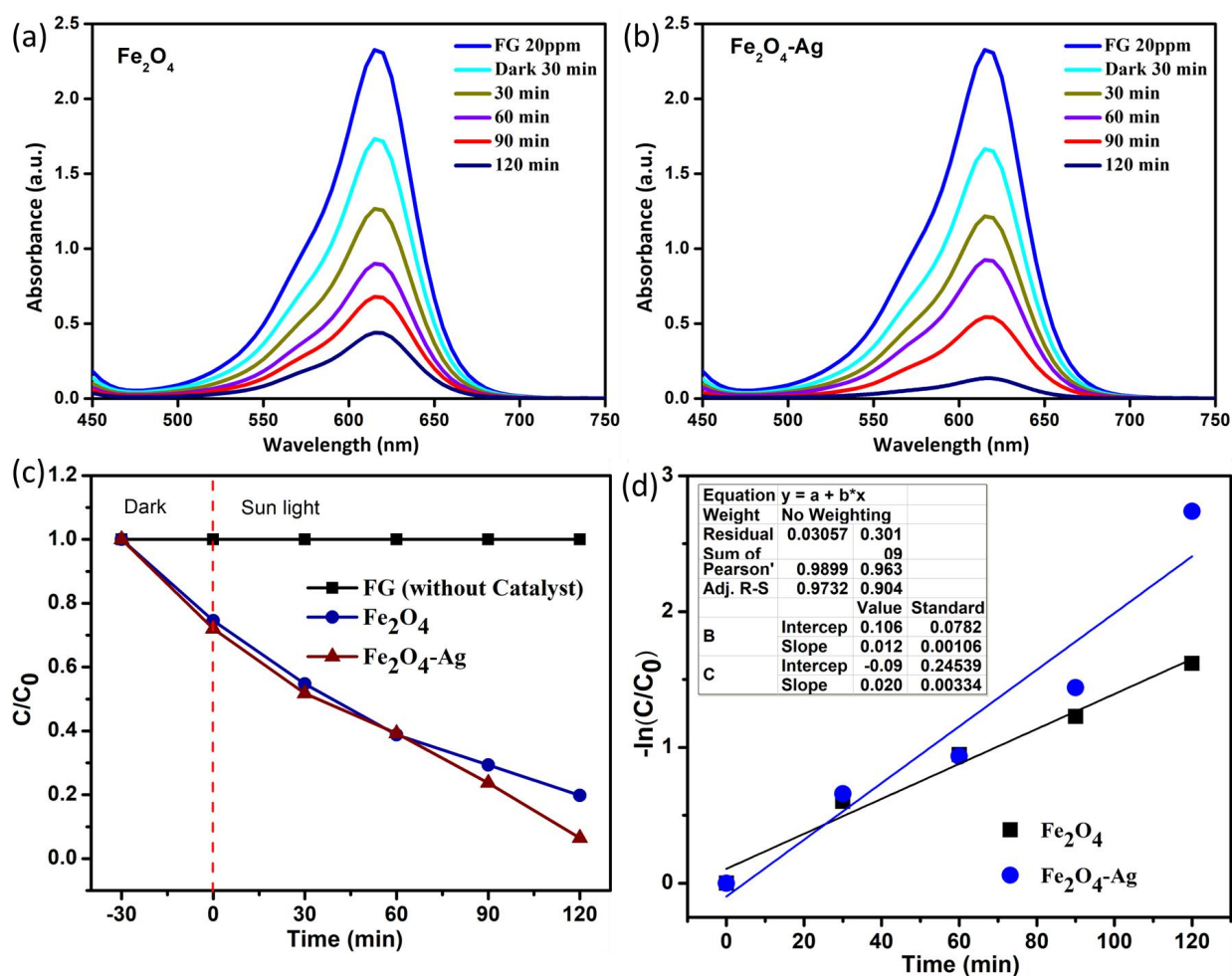


Figure.6.5.2 (A-B) represents the UV-visible spectra for the FG with occurrence of catalysts Fe_3O_4 , and $\text{Fe}_3\text{O}_4\text{-Ag}$ were irradiated by sun light for 120 minutes. (C) C/C_0 versus time interval plot of FG dye (D) $-\ln(C/C_0)$ versus time interval plot of FG dye

The significant improvement of the degradation efficiency is due to the doping effect of nanomaterial of Fe_3O_4 lattice sites, it reduce recombination of excited charge carriers (electron-hole pair). Fig.6.5.2 (c) depicts the changes in initial and final concentration of dye in the presence and absence of photocatalyst against the

light irradiation with respect to time (t) for the FG dye solution. The Fe₃O₄-Ag catalyst shows highest degradation ability due to the fact that sub energy-level act as traps for recombination. Based on the sub energy level of the conduction band helps to produce the large number of reactive oxygen species (ROS).

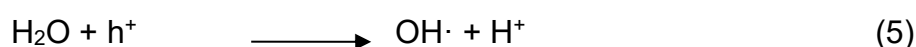
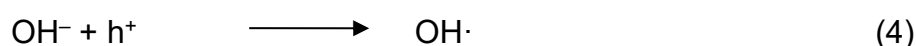
Additional investigation of pseudo-first order kinetics studies shown in Figure.6.5.2 (d) the degradation rates were 0.027 min⁻¹ and 0.046 min⁻¹ for Fe₃O₄, and Fe₃O₄-Ag nanocomposites against FG dye, respectively. From the studies rate constant value and R² values are listed in the Table 6.5.2.

The mechanism of FG dye degradation for the Fe₃O₄-Ag catalyst is described as follows,

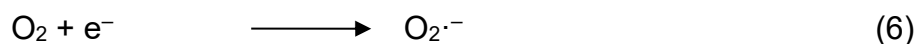
(i) Formation of electron hole pair



(ii) Formation of hydroxyl radicals



(iii) Formation of superoxide radicals



(iv) Photo degradation of FG dye



Basically, a heterogeneous photocatalytic dye degradation reaction is composed of two consecutive steps, i) Dye molecules interact with the catalyst (adsorption occurs on the surface) ii) Photodegradation of the dye begins. So, the photocatalytic system is majorly depending on adsorption and photodegradation capability of the catalyst. Subsequently, as the highest degradation efficiency achieved for the Fe₃O₄-Ag nanocomposites amongst all catalyst, it was only select for the scavenger study for further understanding the degradation mechanism. In general, the photo active catalyst produce the reactive oxygen species (h⁺, OH[•], and

$O_2^{\bullet -}$) and it is important to categorise which reactive species impacts a major role during photo degradation. Hence, the h^+ , $OH^{\bullet}$, and $O_2^{\bullet -}$ are eliminated by accumulation of related scavengers such as EDTA (h^+ scavenger), Methanol ($OH^{\bullet}$ scavenger), and p-BQ ($O_2^{\bullet -}$ scavenger) into reaction solution, respectively. Fig. 6.5.2 (a) illustrates the efficiency of with and without scavengers. Since scavenger studies, the p-BQ only shows the prominent deviations in efficiency of dyes. Therefore, the superoxide radicals play significant role the overall photodegradation efficiency[3].

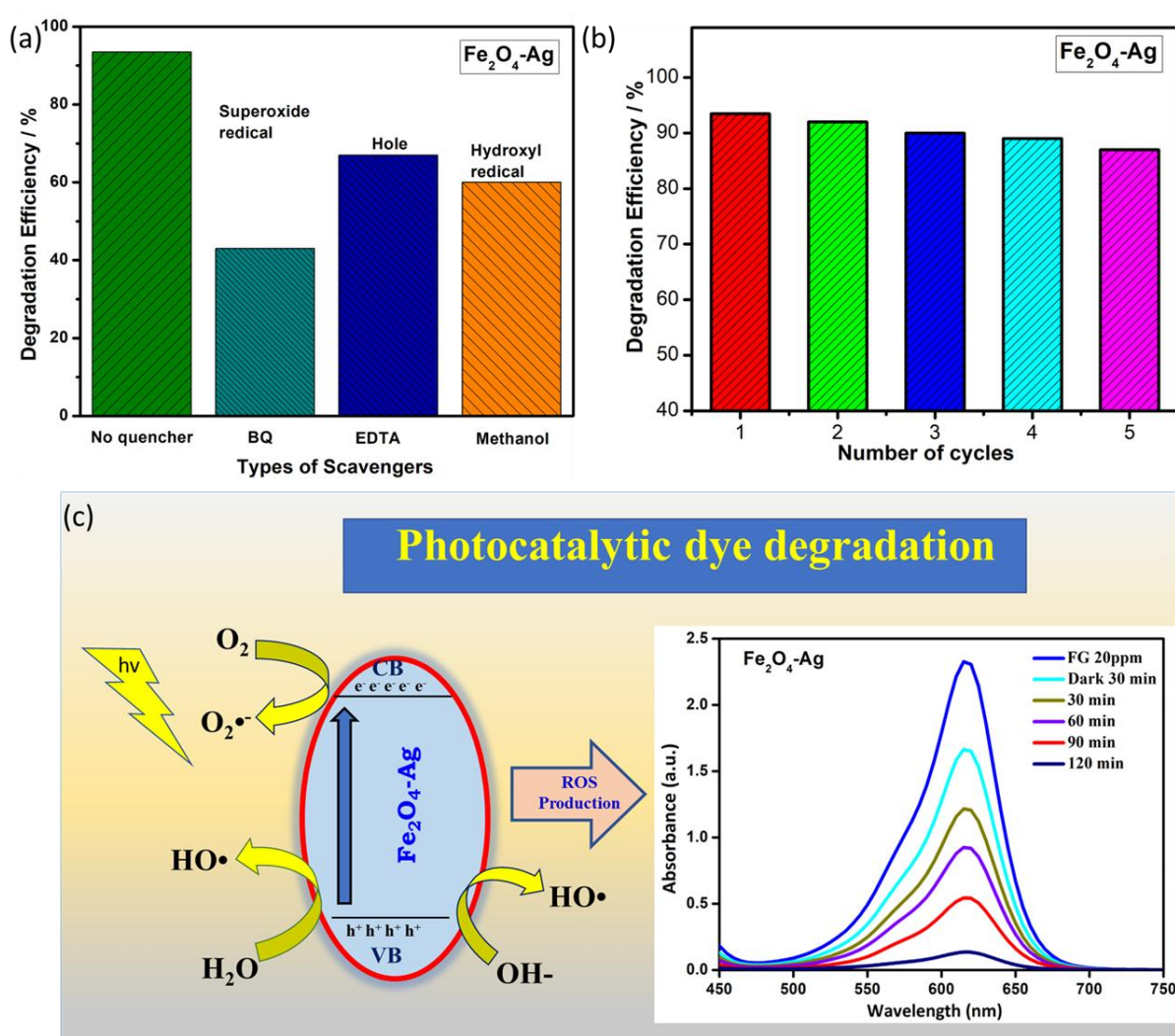


Figure. 6.5.2 (a) Scavengers study (b) reusability test graph of photocatalytic activity (c) Schematic diagram illustrating the mechanism of charge separation and photocatalytic activity of photocatalyst under the sunlight irradiation.

Table 4: photocatalytic activity result of Fe₃O₄ and Fe₃O₄-Ag nanoparticles.

S.No	Catalyst	Band gap (eV)	Degradation efficiency (%) (120 minutes)	K (min ⁻¹)	R ²
Fast green(20 ppm)					
1	Fe ₂ O ₄	2.06	80.5	0.027	0.98
2	Fe ₂ O ₄ -Ag	1.45	93.5	0.046	0.96

Additionally, a very essential study is stability and reusability of photocatalysts (Fe₂O₄-Ag nanocomposites). For reusability test, the results reveal the stability of the catalyst and there is no significant loss in degradation efficiency even after five cycles of the photocatalytic process shown in Fig. 6.5.2 (b). After 5 cycles, the efficiency of catalyst is decreased due to the loss of sample while washing process.

7.SUMMARY AND CONCLUSION

- Mixed Metal oxide nanocomposite have attracted much attention due to its applications in catalysis, dye degradation, electrode materials in battery and optoelectronic devices.
- Herein, low temperature based Ag-Fe₃O₄ nanocomposite was Synthesized by a chemical precipitation method.
- Equal concentration of precursors were used.
- Crystallite structure and size were confirmed by XRD. The size of the crystallite was calculated using Debye Scherrer formula, the size was found to be 10.3 nm
- The SEM microscopy demonstrated the synthesized nanoparticles were in nanoscale region with asymmetric surface morphology structure.
- Organic functional groups presence on the surface of the nanoparticles were identified using FT-IR spectroscopy.
- The investigated photocatalytic result demonstrated that Ag-Fe₃O₄-nanocomposite showed excellent dye degradation of FAST GREEN which is due to its effective charge carriers creation and followed by the free radical formation during the light irradiation.
- Thus, the prepared cost effective and low temperature Ag-Fe₃O₄ could be the suitable candidate for water purification, through the photocatalysis process

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