

SYNTHESIS AND CHARACTERIZATION OF CARBON IRON OXIDE NANOCOMPOSITE

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Science in Chemistry

by

Manasa.V (39030011)

Sugashri.R (39030038)



**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 – 600 119

MAY – 2022

DEPARTMENT OF CHEMISTRY

BONAFIDE CERTIFICATE

This is to certify that this project report is the bonafide work of V.Manasa (39030011), R.Sugashri (39030038) who carried out the project entitled "Synthesis and Characterization of Carbon Iron Oxide Nanocomposite" under our supervision from _____ to _____

Internal Guide

External Guide

Dr. S. Sunitha, M.Sc., M.Phil., Ph.D.

Head of the Department

Dr. J. Karthikeyan, M.sc., Ph.D.

Submitted for Viva voice Examination held on _____

Internal Examiner

External Examiner

DECLARATION

I **MANASA** (39030011) and **SUGASHRI** (39030038) hereby declare
that the Project Report entitled

**“SYNTHESIS AND CHARACTERIZATION OF CARBON IRON
OXIDE NANOCOMPOSITE”** done by us under the guidance of **Dr. S.
SUNITHA, M.Sc., M.PHIL., PH.D. PROFESSOR, DEPARTMENT OF
CHEMISTRY** and **Dr. G.MURUGADOSS, M.Sc., Ph.D. Scientist-E at
International Research Centre**

SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY,
CHENNAI – 600 119, India has submitted in partial fulfilment of the
requirements for award of **Bachelor of Science in Chemistry** in

DATE:

PLACE:

SIGNATURE OF THE CANDIDATE

ACKNOWLEDGEMENT

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

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

I would like to express my sincere and deep sense of gratitude to my Project Guide **Dr . S. SUNITHA, M.Sc., MPhil., Ph.D. Assistant Professor, Department of Chemistry** and **Dr . G. Murugadoss M.Sc., Ph.D. Scientist-E at International Research Centre**

for her valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

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

V.MANASA

R.SUGASHRI

ABSTRACT

Iron oxide nanoparticles (NPs) have attracted much consideration due to their unique properties, such as superparamagnetism, surface-to-volume ratio, greater surface area, and easy separation methodology. Among the magnetic materials, iron oxide NP is a promising nanomaterial due to its excellent biocompatibility. Although many iron oxides are known, the term “iron oxides” generally refers to three types: Fe_3O_4 (magnetite), $\alpha\text{-Fe}_2\text{O}_3$ (hematite), and $\gamma\text{-Fe}_2\text{O}_3$ (maghemite). Among all iron oxides, Fe_3O_4 has attracted more attention due to its superior magnetic properties. Magnetite (Fe_3O_4) Nanoparticles (NP) are attractive nanomaterials in materials science, chemistry and physics due to their valuable properties, such as ductile, semi-conducting ferromagnetic properties and biocompatibility. Carbon iron oxide nanocomposite were synthesized using hydrothermal method. Additionally, we used Scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR), UV-Visible Spectroscopy for characterization of properties and structure of the nanoparticles.

CONTENT

<i>S.no</i>	<i>Chapter name</i>	<i>page no</i>
1	Introduction	1
1	Nanoparticles	1
1.1	Types of nanoparticles	1
1.1.1	Zero dimensional nanoparticle	1
1.1.2	One dimensional nanoparticle	2
1.1.3	Two dimensional nanoparticle	2
1.1.4	Three dimensional nanoparticle	2
1.2	Classification of nanoparticle	3
1.2.1	Carbon based nanoparticle	3
1.2.2	Ceramic nanoparticles	3
1.2.3	Metal nanoparticles	4
1.2.4	Semiconductor nanoparticles	4
1.2.5	Polymeric nanoparticles	4
1.2.6	Lipid-Based Nanoparticles	5
1.3	Iron oxide nanoparticles	5
1.3.1	Applications of Iron oxide nanoparticles	6
1.4	Carbon Iron oxide nanocomposite	7
1.5	Synthesis of nanoparticle	7
1.5.1	Physical method	8
1.5.2	Chemical method	11
2	Literature survey	15

3	Aim and scope of present investigation	17
4	Materials and method	17
4.1	Material	17
4.2	Experimental method	18
4.3	Characterization techniques	19
5	Result and discussion	22
6	References	26

SYNTHESIS AND CHARACTERIZATION OF CARBON-IRON OXIDE NANOCOMPOSITE

INTRODUCTION

Nanoparticles are small and have dimensions of 100 nm or less. They are microscopic materials characterized by ultrafine particle sizes (<50 nm) or sizes limited to 50 nm. They are of different types based on their dimension, existence, physicochemical properties and applications. In this article, we will focus on all types of nanoparticle classification.

1.1 Types of nanoparticles

Nanoparticles can be classified into different types according to the size, dimension, morphology, physical and chemical properties. Some of them are carbon-based nanoparticles, ceramic nanoparticles, metal nanoparticles, semiconductor nanoparticles, polymeric nanoparticles and lipid-based nanoparticles.

1.1.1 Zero-dimensional nanoparticles

Zero-dimensional nanoparticles are the most common type of nanomaterials, with dimensions within nanoscales (the dimension of no nanoparticle is larger than 100nm). These nanoparticles are point like particles i.e. as small as a point). The most common examples of these particles are quantum dots, hollow spheres, nano lenses.

1.1.2 One-Dimensional Nanoparticles

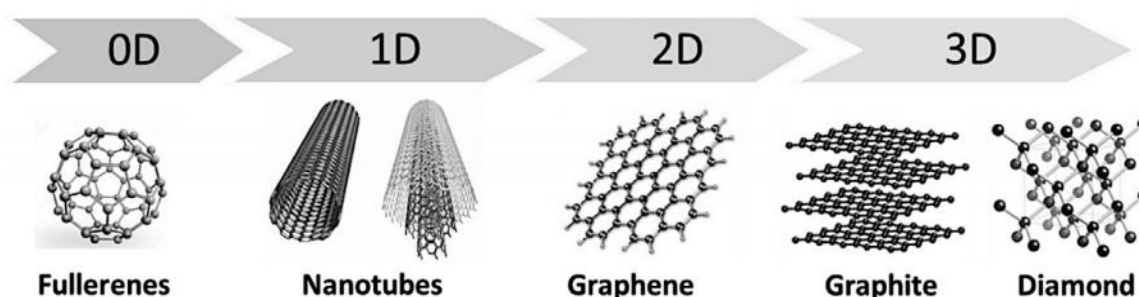
These types of nanoparticles are at least one dimension larger than nanoscales and other dimensions are within nano range. The most common examples of one-dimensional nanoparticles are nanofibers, nanotubes, and nanorods

1.1.3 Two-Dimensional Nanoparticles

These types of nanoparticles are two dimensions larger than nanoscale (100nm). The most common examples of this class are nanofilms, nanolayers, and nanocoating. This class of nanomaterials have plate-like structures.

1.1.4 Three-Dimensional Nanoparticles

Three-dimensional nanoparticles have all three dimensions larger than 100nm but their components are below 100nm in size. Nano range particles come together to form three dimension nanoparticles. These particles are generally nonporous in nature and have many applications. The most common examples of three-dimensional nanoparticles are nanocomposites, bundles of nanofibers, multilayer-type structures.



Carbon allotropes with different dimensions

1.2 Classification of nanoparticle

1.2.1 Carbon based nanoparticle

Carbon nanoparticles are composed of two types of materials. These include fullerenes and carbon nanotubes (CNTs). These two forms are pure forms of carbon, but they differ only in their molecular arrangement.

CNTs have a tube-like structure and are made from rolled sheets of graphene. These types of nanoparticles has various applications in structural reinforcement. A key feature of carbon nanoparticles is that they are 100 times stronger than steel. Single-walled carbon nanotubes consist of a uniform layer of graphite just one atom thick, in which the carbon atoms are joined by strong covalent bonds.[1] Double-walled carbon nanotubes consist of two single-walled carbon nanotubes. One carbon nanotube is inserted inside another to create a double-walled carbon nanotube.[2]

Fullerene material is one of the allotrope of carbon having a structure of hollow cage structure of C-60 is called Buckminsterfullerene. Carbon fullerenes exhibit unique physical properties such as high tensile strength, flexibility, high electrical conductivity, large surface area, unique electronic properties, and high molecular adsorption capacity. A unique property of fullerenes is their high optical activity in the UV and visible spectra. A unique property of fullerenes is their high optical activity in the UV and visible spectra. The covalent bonds between carbon atoms make fullerene C60 very strong, and the carbon atoms readily form covalent bonds with a variety of other atoms. C60 fullerenes have an interesting electrical property as very good electron acceptors, which means they accept free electrons from other materials. This feature is useful, for example, to increase the efficiency of a solar cell by converting sunlight into electricity.

1.2.2 Ceramic nanoparticles

Ceramic nanoparticles are made up of oxides, carbides, carbonates, and phosphates and are inorganic solids. These nanoparticles are chemically inert and heat resistant. They can be used for photocatalysis, dye photodegradation, medication administration, and imaging. Ceramic nanoparticles can be used as a good drug delivery agent by manipulating several of their properties, such as size,

surface area, porosity, surface to volume ratio, and so on. These nanoparticles have been successfully employed to treat a variety of ailments, including bacterial infections, glaucoma, and cancer.

1.2.3 Metal nanoparticles

Metal precursors are used to create metal nanoparticles. Chemical, electrochemical, or photochemical methods can be used to create these nanoparticles. Metal nanoparticles are created chemically by reducing metal-ion precursors in solution with chemical reducing agents. These have a high surface energy and the ability to adsorb small molecules. These nanoparticles have applications in research, biomolecule detection and imaging, and environmental and bioanalytical applications. For example, gold nanoparticles are used to coat the sample prior to SEM analysis. This is typically done to improve the electronic stream, allowing us to obtain high-quality SEM images.

1.2.4 Semiconductor nanoparticles

Semiconductor nanoparticles have similar properties to metals and nonmetals. They are found in the periodic table in groups III-V, III-V or IV-VI. These particles have a wide band gap that, when tuned, exhibit different properties. They are used in photocatalysis, electronics, optics and water separation applications. Some examples of semiconductor nanoparticles are GaN, GaP, InP, group III-V InAs, ZnO, ZnS, CdS, CdSe, CdTe which are IIIV semiconductors and group IV silicon and germanium.

1.2.5 Polymeric nanoparticles

Polymeric nanoparticles are nanoparticles made of organic materials. Depending on the method of preparation, these have nanocapsular or nanosphere shapes. A matrix-like structure characterises nanosphere particles, whereas core-shell morphology characterises nanocapsular particles. The active chemicals and polymer are uniformly diffused in the former, whereas the active compounds are contained and surrounded by a polymer shell in the later.

1.2.6 Lipid-Based Nanoparticles

Lipid nanoparticles are usually spherical in shape with diameters ranging from 10 to 100 nm. It consists of a solid core made up of lipids and a substrate containing soluble fat molecules. The outer core of these nanoparticles is stabilized by surfactants and emulsifiers. These nanoparticles have biomedical applications as drug delivery, delivery and RNA release agents in cancer therapy.

1.3 Iron oxide nanoparticles

Iron oxide particles Fe_3O_4 nanoparticles have unique magnetic properties that are influenced by their structures, including size, morphology, crystallinity, and surface properties with diameters between about 1 and 100 nanometres. Iron oxide nanoparticles are translucent to visible light and can be opaque to UV light. The two main forms are magnetite (Fe_3O_4) and its oxidized maghemite ($\gamma\text{Fe}_2\text{O}_3$). They are of great interest due to their superparamagnetic properties and potential applications in many areas (cobalt and nickel are also highly magnetic materials, but they are toxic and easily oxidized).

Fe_3O_4 can be both n-type and p-type semiconductors. Fe_3O_4 has the lowest resistivity among iron oxides due to its small bandgap (0.1 eV). Iron oxide has the ideal band gap value of 2.2 eV to serve as a photo-catalyst to dissociate the molecule of water. Fe_3O_4 NP is an ideal photocatalyst because of its high magnetization, high coercive force, excellent photon absorption capacity, and excellent intermolecular interaction and strong intermolecular bonds.[15] The Fe_3O_4 sheet-like nanoparticles exhibit good magneto chromatic properties. Iron oxide nanoparticles may display some unique paramagnetic properties because of which they may be used during magnetic hyperthermia, an experimental treatment for some types of cancers.

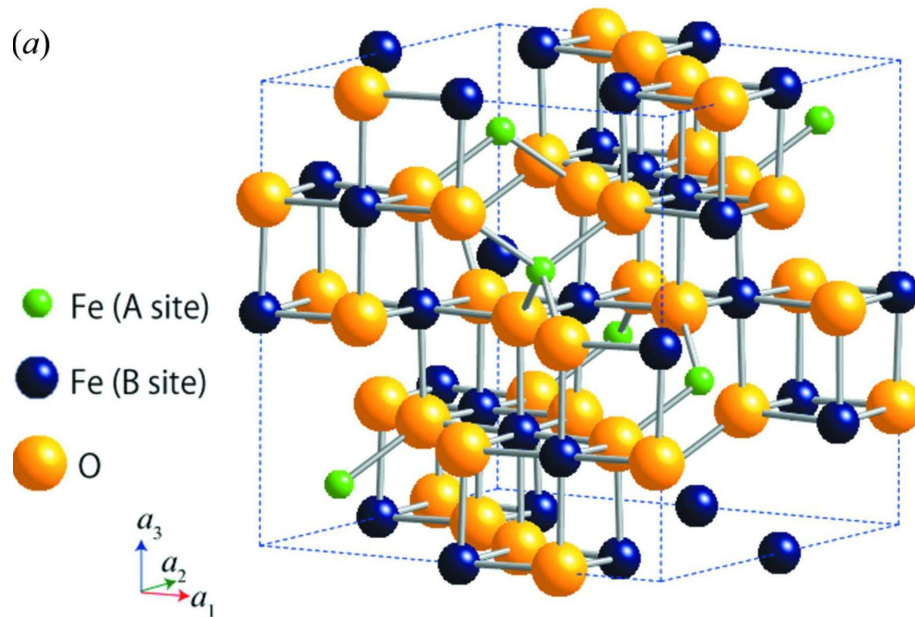


Fig Fe_3O_4 cubic inverse spinel structure

1.3.1 Applications of iron oxide nanoparticles

- In magnetic resonance imaging to provide contrast enhancement at very low concentrations in the nanoscale for the study of tumours.
- As a targeted delivery vehicle and as a drug delivery coating for nanoscale anti-cancer drugs.
- For magnetic data storage devices
- Applications for coatings, plastics, nanowires, nanofibers, textiles, and specialty alloys and catalysts.
- Used for magnetic particle imaging, catalysis, sensors separation of biomolecules, and targeted drug and gene delivery for medical diagnosis and therapeutics.

1.4 CARBON IRON OXIDE NANOCOMPOSITE

Carbon nanoparticles have extraordinary electrical conductivity, heat conductivity, and mechanical properties. They are composed of pure carbon, therefore, exhibiting high stability, good conductivity, low toxicity, environmental friendliness. Since a large part of the human body consists of carbon, it is generally thought as a biocompatible material. The good electrical conductivity, high surface area, and linear geometry make their surface highly accessible to the electrolyte.

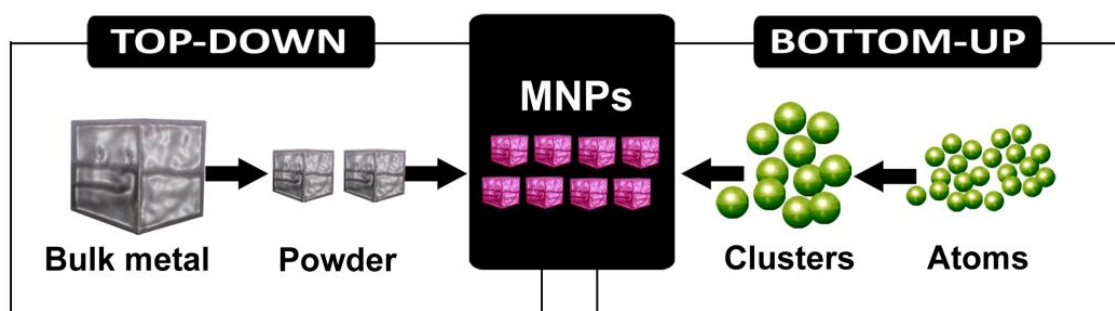
IO nanoparticles have attracted considerable interest due to their superparamagnetic properties and potential biomedical applications arising from their biocompatibility and nontoxicity.

Carbon is used with iron oxide nanoparticles to increase the conductivity of electron generated by light thus reducing the recombination rate of electron hole which is a major factor in photocatalytic activity of metal oxide and sulfide semiconductor materials.

1.5 SYNTHESIS METHODS OF NANOPARTICLES

The synthesis of nanomaterials of uniform size is important because their properties include optical, magnetic, electrical and biological properties depending on size and dimension (16).

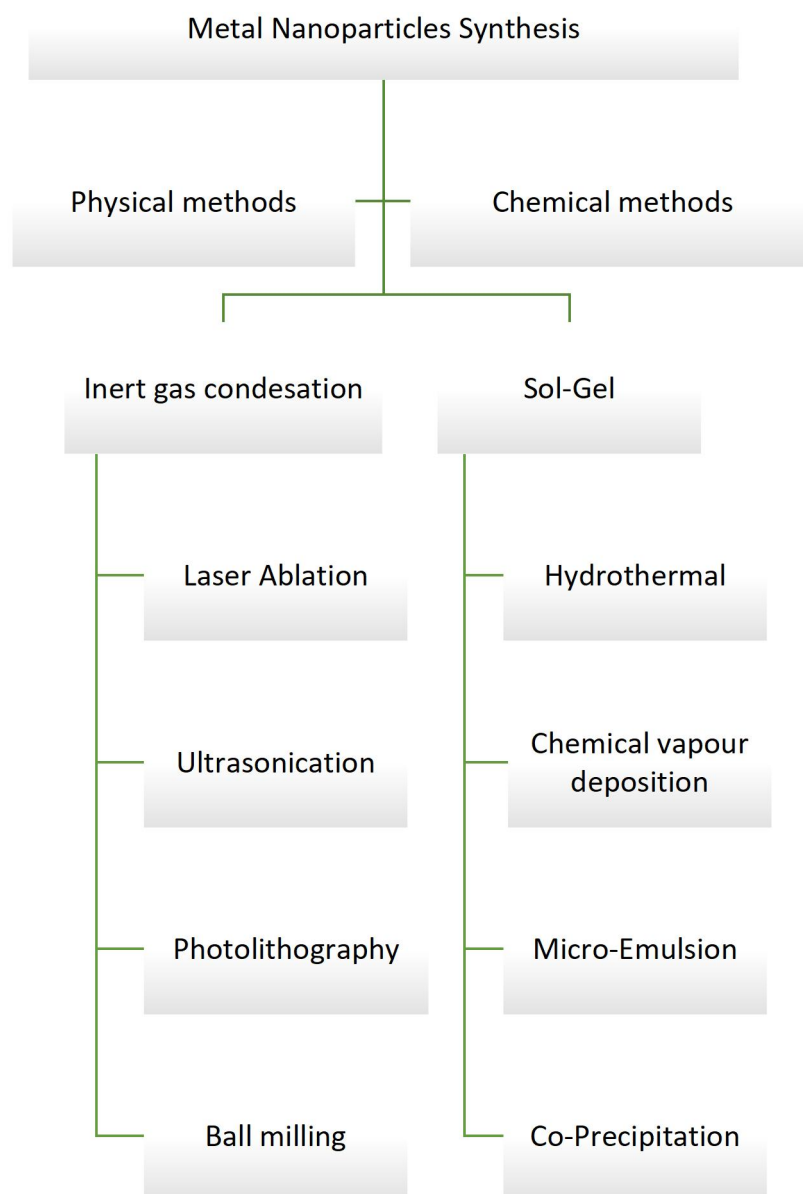
The synthesis methods are generally divided into three categories: solution synthesis, gas phase synthesis, and gas phase synthesis. (17) Another approach is to divide these comprehensive approaches into two broad categories.



a) Physical methods and

b) Bottom-up approaches including wet chemistry. [18].

The choice of a synthetic approach depends on the desired properties required for the nanoparticle mixture. In terms of size, morphology, crystal structure, etc. [19], the advantage of physical methods is that they can produce a large number of nanocomposites, but it is not easy to synthesize nanocomposite particles of the same size. Meanwhile, wet chemistry makes it easier to achieve a controlled particle size, resulting in a uniform size of the nanocomposite.



Metal nanoparticles prepared by:-

Top Down Approach (Physical methods): In this approach it involves the breaking down of the bulk material into nanosized structures or particles.

Eg: Laser ablation, Ultrasonication, Photolithography, Electron beam lithography, Ball milling,

Bottom up approach (Chemical methods): In this approach, material is synthesized from atomic or molecular species via chemical reactions, allowing precursor particles to grow in size.

which implies assembling single atoms and molecules into larger nanostructures.

Eg: Sol-gel, Hydrothermal process, Chemical vapor deposition, Colloidal precipitation , Microemulsion etc.

1.5.1 PHYSICAL METHOD

In physical methods, metal nanoparticles are usually prepared by evaporation–reduction, which could be executed by a tube heater at the atmospheric pressure [20]. The benefits of the physical methods are fast, radiation utilized as reducing factors, and no dangerous chemicals involved but, the drawbacks are high energy exhaustion, solvent pollution, and lack of uniform pattern [21]. Although the size of the nanoparticles cannot be controlled exactly, even in physical techniques, it is possible to cramp the size distribution by controlling the vaporization rate, system pressure, or by changing the system geometry [22]

Inert gas condensation

The technique is widely used to synthesize metal nanoparticles. In IGC, metal is evaporated in an extremely high vacuum chamber filled with helium or argon at typical pressures of several hundred Pascals. The vaporized metal atoms lose kinetic energy by collision with the gas and condense into small particles. These particles then grow by Brownian coagulation and agglomeration and eventually form nanocrystals.

Laser ablation

It is a process in which a laser beam is focused on the sample surface to remove material from the irradiated zone. Laser ablation has been considered and used for many technical applications, including: the production of nanomaterials, deposition of thin metallic and electric films, fabrication of superconducting materials, routine welding and bonding of metal parts, and micromachining of MEMS structure.

Ultrasonication

When a liquid sample is exposed to ultrasonic waves (> 20 kHz), the sample moves. Sound waves propagate in a liquid medium, alternating between high pressure (compression) and low pressure (dilatation) cycles. During dilatation, high-intensity sound waves create small vacuum bubbles or voids in the liquid that violently disintegrate (cavitation) during compression, resulting in very high local temperatures.

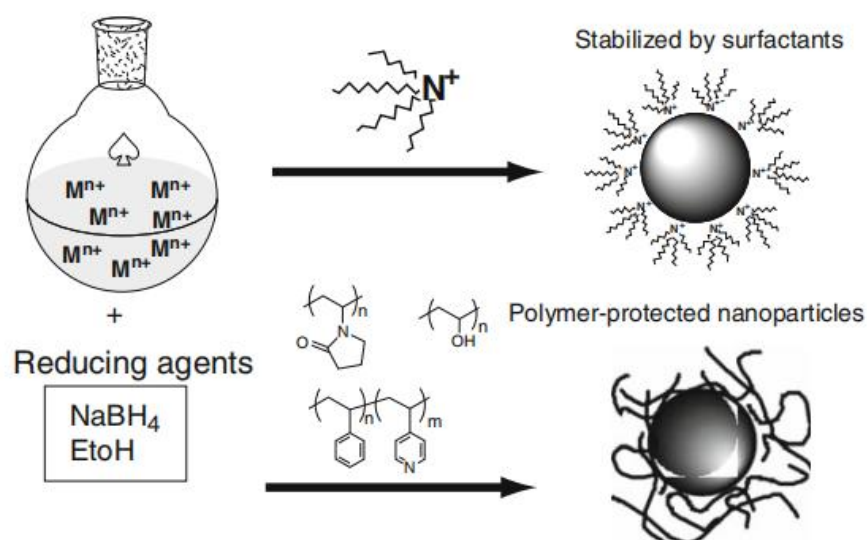
Ball milling

A ball mill contains a stainless steel container and many small iron, hardened steel, silicon carbide, or tungsten carbide balls are made to rotate inside a mill (drum). The powder of a material is taken inside the steel container. This powder will be made into nanosize using the ball milling technique. This method is used for reducing the size of nanoparticles.

CHEMICAL REDUCTION OF METAL SALTS

Preparation of metal nanoparticles by chemical reduction in presence of

(a) cationic surfactant (b) polymers (PVA, PVP)

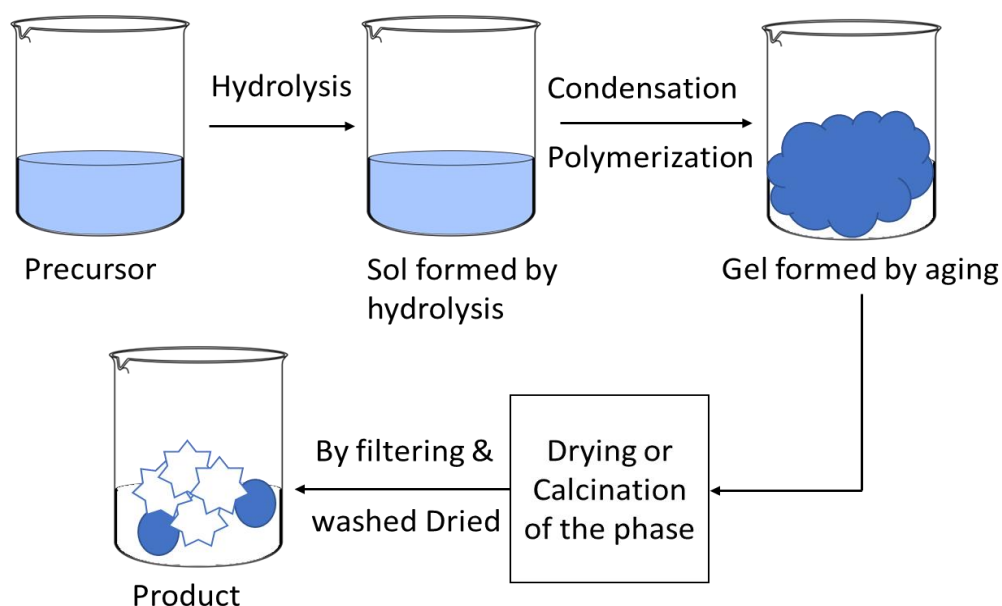


1.5.2 CHEMICAL METHOD

The advantages of these methods are low cost, ease of manufacture, and high productivity [23]. On the other hand, using the chemical methods are harmful to the living organisms, most of the chemical methods are the precise reduction control of the particle size distribution. Factors such as reaction time, component concentrations, and temperature exhibit some control over particle shape and size. In some states, the growth conditions are very sensitive to the process variables and let a little flexibility for the process changes [24].

Solgel process

The solgel process is a wet chemical technique that uses a chemical solution (abbreviated as sol for solution) or colloidal particles (sol for nanoparticles) to create an integrated network (gel). Metallic alkoxides and metal chlorides are typical precursors. They undergo hydrolysis and polycondensation reactions to form colloids. This is a system composed of nanoparticles dispersed in a solvent. The sol then evolves to form an inorganic continuous network containing the liquid phase (gel). The formation of the metal oxide involves the connection between the metal center and the oxo (M-O-M) or hydroxo (M-OH-M) bridge, thereby producing the metal oxo or metal hydroxopolymer in solution. After the drying process, the liquid phase was removed from the gel. Next, heat treatment (calcination) can be performed to promote further polycondensation and improve mechanical properties.



Chemical vapor deposition

Chemical vapor deposition (CVD) involves a chemical reaction. The CVD process is mainly used in semiconductor manufacturing to deposit thin layers of various materials. The process involves one or more volatile precursors, the substrates of which are in contact with these precursors to decompose on which the desired deposition is formed. The vaporized precursors are introduced into the CVD reactor and adsorbed on the substance placed at high temperature. The adsorbed molecules react with other molecules or break down to form crystals. The three steps in CVD method are:

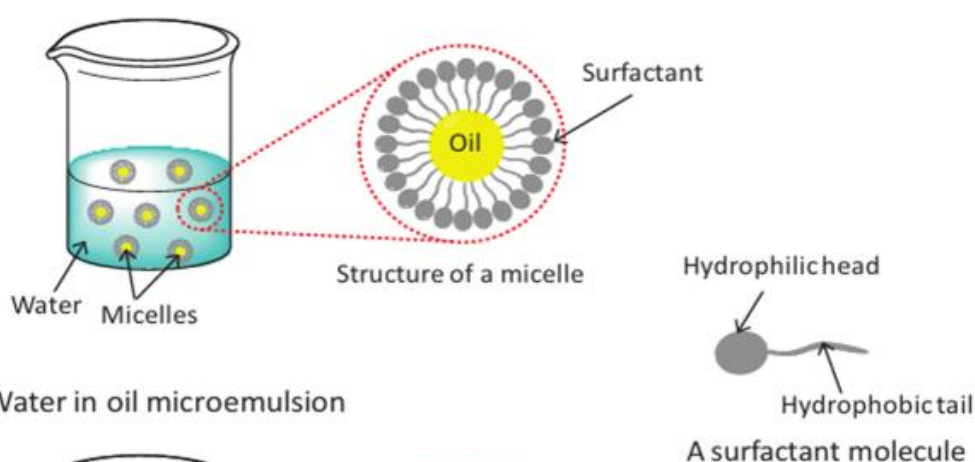
1. The reactants are transported to the growth surface by a boundary layer.
 2. Chemical reactions occur on the growth surface.
 3. The by-products from the gas phase reaction must be removed from the surface.
- Homogeneous nucleation occurs in the gas phase and heterogeneous nucleation occurs in the matrix.

The CVD method allows the synthesis of ultrafine particles with size less than 1 μm by chemical reaction taking place in the gas phase. The reaction can be controlled to produce nanoparticles with sizes between 10 and 100 nm [25, 26].

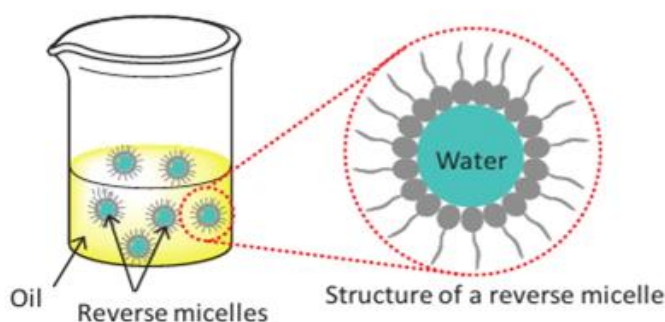
MICROEMULSION

Microemulsions are clear, stable, isotropic liquid mixtures of oils, waters and surfactants, often combined with co-surfactants. The aqueous phase may contain salts and/or other ingredients, and the "oil" may in fact be a complex mixture of various hydrocarbons and olefins. The two basic types of microemulsions are direct (oil dispersed in water, o/w) and vice versa (water dispersed in oil, w/o).

Oil in water microemulsion



Water in oil microemulsion



Structure of microemulsion

HYDROTHERMAL SYNTHESIS

Hydrothermal synthesis refers to the heterogeneous reaction used to synthesize inorganic materials in aqueous media above ambient temperature and pressure [27]. In this case, the aqueous mixture of precursors is heated above the boiling point of water in a sealed stainless steel autoclave, resulting in a dramatic

increase in pressure in the reaction autoclave above atmospheric pressure. This synergistic effect of high temperature and pressure provides a one-step process for producing highly crystalline materials without the need for post-annealing treatment.

Hydrothermal strategies for synthesizing a wide range of nanomaterials, including magnetic nanomaterials, have also been developed. Reaction parameters such as precursors, solvents, stabilizers, and reaction temperature and time properties and concentrations have significant effects on the product. Compared to the "low temperature" coprecipitation method, which usually produces low crystallinity nanoparticles, hydrothermal synthesis can result in very high crystallinity magnetic nanomaterials under high temperature and high pressure reaction conditions. .. However, the hydrothermal approach yields relatively low product yields compared to the coprecipitation method.



2. LITERATURE SURVEY

JaskiranKaur, ManpreetKaur, Manpreet Kaur Ubhia, NavneetKaur, Jean-Marc Greneche has performed Composition optimization of activated carbon-iron oxide nanocomposite for effective removal of Cr(VI)ions. Iron oxide-activated carbon nanocomposites (NCs) were synthesized using co-precipitation method. The composition of NCs has been successfully optimized for efficient Cr(VI) removal. NC was characterized by FTIR, XRD, BET, SEMEDX, TEM, and VSM analysis.[28]

Rais Ahmad* and Rajeev Kumar has performed Kinetic and Thermodynamic Studies of Brilliant Green Adsorption onto Carbon/Iron Oxide Nanocomposite. Carbon / iron oxide nanocomposites were prepared by chemical precipitation and heat treatment of the carbon with iron nitrate at 750 °C. The resulting material was carefully characterized using TEM, XRD and TGA. Thermodynamic parameters were also derived for the adsorption of BG on the nanocomposite, indicating that the adsorption is spontaneous and endothermic. [29]

Narayanan TN, Reena Mary AP, Shaijumon MM, Lijie Ci, Ajayan PM, and Anantharaman MR performed Synthesis of magnetic properties of multi-walled carbon nanotubes - Superparamagnetic iron oxide nanoparticle nanocomposites. The MWCNTSPION assembly has been investigated using low-temperature magnetization studies and zero-field cooling (ZFC) and field-cooling experiments.[30]

Hengli Xiang, Genkuan Ren, Yanjun Zhong, Xiushan Yang, Dehua Xu¹, Zhiye Zhang and Xinlong Wang have studied Characterization and synthesis of Fe₃O₄@C nanoparticles by in-situ solid-phase method. Fe₃O₄ @ C nanoparticles were successfully synthesized by the insitu solid phase method

using FeSO_4 , FeS_2 , and PVPK30 as starting materials in a nitrogen atmosphere.[31]

Xiaowang Liu*, Zhen Fang, Xiaojun Zhang, Wei Zhang, Xianwen Wei, and Baoyou Geng* has done Preparation and Characterization of $\text{Fe}_3\text{O}_4/\text{CdS}$ Nanocomposites and Their Use as Recyclable Photocatalysts. $\text{Fe}_3\text{O}_4/\text{CdS}$ nanocomposites was prepared using a sonochemical route in an aqueous solution. $\text{Fe}_3\text{O}_4/\text{CdS}$ has been reported to exhibit fluorescence, display excellent magnetic properties at room temperature, and have highly photocatalytic degradation ability hence proves it can served as effective and convenient recyclable photocatalysts.[32]

Javad Moradiganjeh and Zahra Aghajani has done Synthesis and characterization of novel magnetic $\text{Fe}_3\text{O}_4/\text{C}$ through the Co-precipitation method and investigation of its desulfurization application. The synthesized $\text{Fe}_3\text{O}_4/\text{C}$ nanocomposite was characterized by XRD, SEM, VSM, and EDS. [33]

Hamidreza Sadegh, Ramin Shahryari-ghoshekandi & Maryam Kazemi have done the Study on Synthesis and characterization of carbon nanotubes decorated by magnetic iron oxide nanoparticles. Characterization of MWCNT- Fe_3O_4 by X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscope (SEM), transmission electron microscope (TEM), analysis Thermogravimetric (TGA) and magnetic characterization were performed on a vibrating sample magnetometer (VSM).[34]

Zahra Razaghi, Zahra Aghajani², Ali Yeganeh Faal has performed Novel synthesis and characterization of $\text{Fe}_3\text{O}_4/\text{C}$ nanocomposites and investigation of its application in removal of pesticide diazinon.[35]

3. AIM AND SCOPE OF PRESENT INVESTIGATION

This project work is mainly focused on the following objectives:

- To synthesis novel C-Fe₃O₄ nanocomposite by a hydrothermal 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 Fe-Co-Ni nanocomposite
- Summarize the obtained results

4. MATERIALS AND METHOD

4.1 CHEMICALS

Urea (extra pure AR 99.5%), Ferric chloride Anhydrous (pure, 98%), Sodium Hydroxide (98%), Polyvinyl pyrrolidone (PVP) used in this study were purchased from Sisco Research Laboratories Pvt. Ltd. (SRL) - India. De-ionised water All the chemicals used in this work were of commercially available analytical grade (AR) and were used without further purification.

4.2 EXPERIMENTAL METHODS

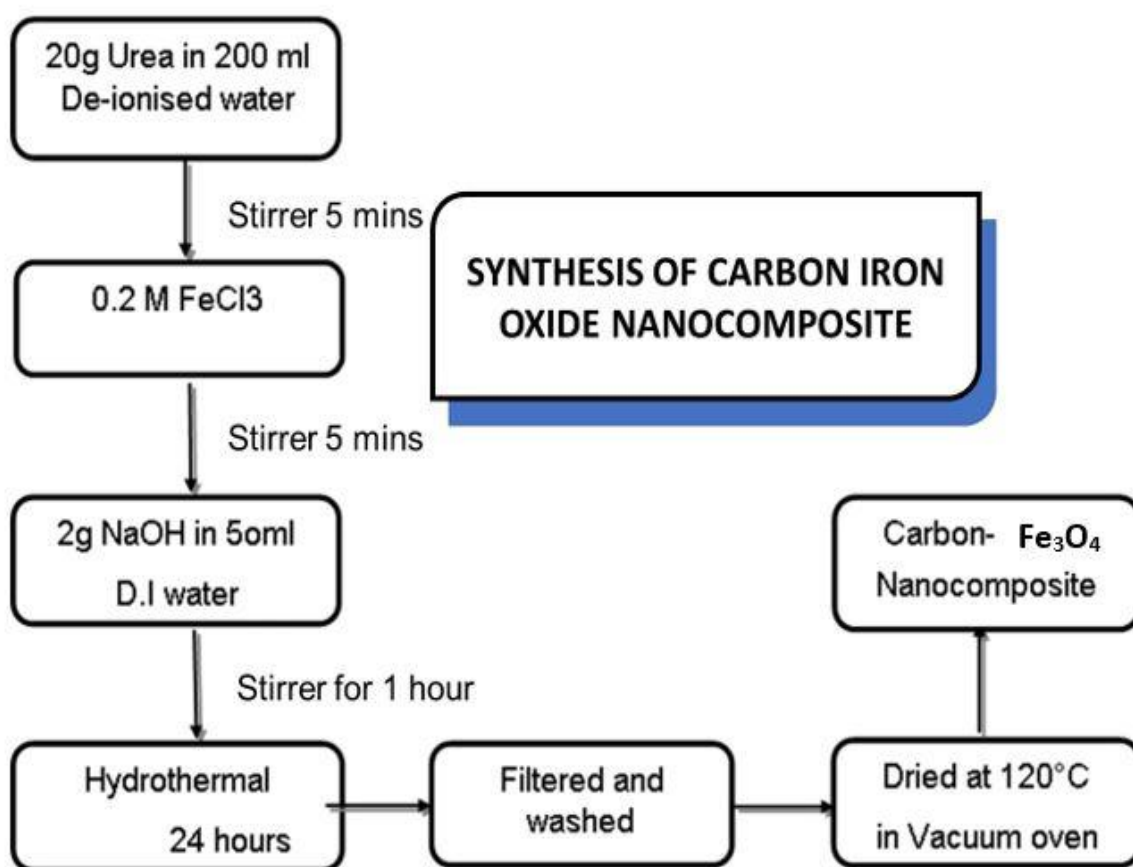
The procedure involved to prepare C-Fe₃O₄ nanocomposite is as follows

20g Urea (CH₄N₂O) and 6.4 g Ferric chloride (FeCl₃) were dissolved in 200 ml of De-ionised water and placed at the magnetic stirrer. Add 1g of Polyvinyl pyrrolidone(PVP) to reduce particle size and prevent agglomeration.

Dissolve 2 g of NaOH in 50 ml of deionized water, gradually add dropwise to the FeCl₃ solution, and kept constant stirring for 60 minutes.

After an hour, the solution was transferred into a 250 mL Teflon-tube inserted lined stainless steel autoclave and kept at 180° C for 24 h. After reaction at 180° for 24 h, the autoclave is cooled down at room temperature.

The product was filtered and washed by ethanol and deionised water then dried under Vacuum oven at 120°C.



4.3 CHARACTERIZATION TECHNIQUES

X-ray diffraction (XRD)

X-ray diffraction analysis (XRD) is a technique used in materials science to determine the crystal structure of a material. XRD works by irradiating a material with incident X-rays and measuring the intensity and scattering angle of the X-rays emitted from the material.

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. 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 [42]. 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.

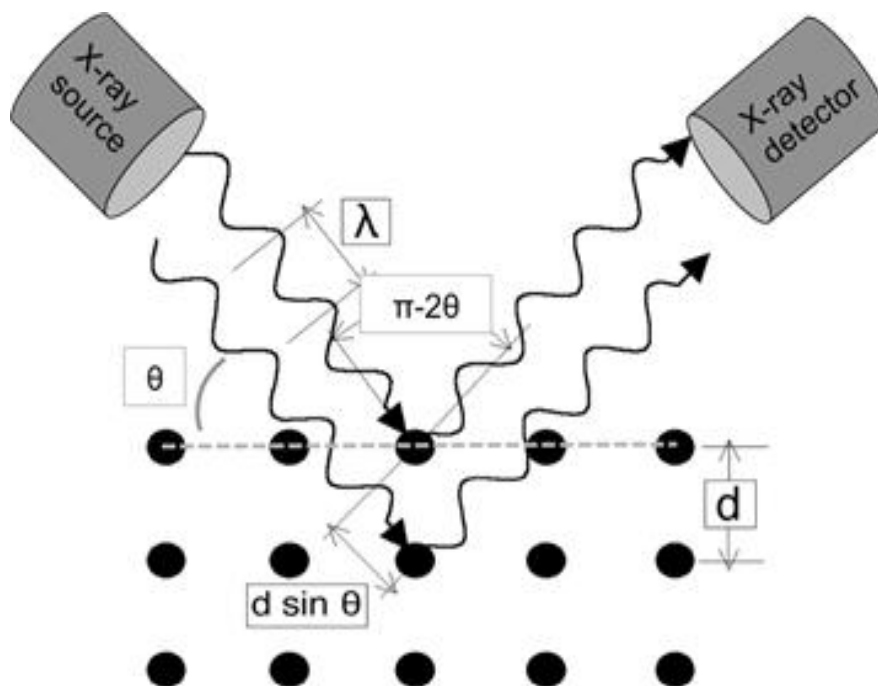
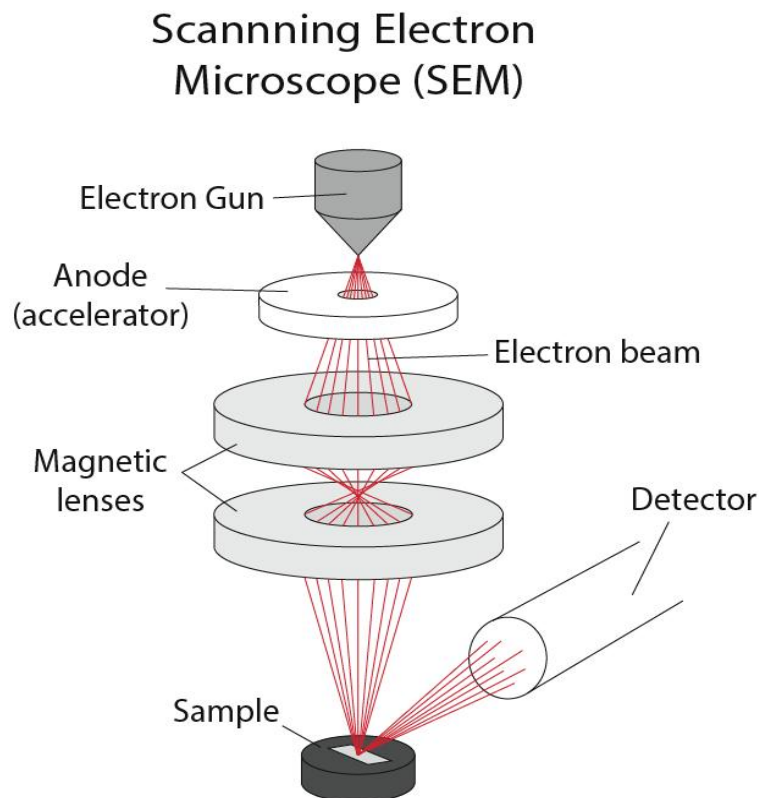


Illustration of Bragg's Law.

Scanning electron microscope (SEM)

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample.

The electron beam is scanned in a raster fashion, and the position of the beam is combined with the detected signal strength to produce an image. In the most common SEM mode, secondary electrons emitted from atoms excited by the electron beam are detected using a secondary electron detector (Everhart-Thornley detector). The number of secondary electrons that can be detected, and thus the signal strength, depends on the topography of the sample. Some SEMs can achieve resolutions greater than 1 nanometer.



Fourier transform infrared (FTIR) Analysis

Fourier transform infrared (FTIR) spectroscopy identifies chemical bonds in molecules by generating infrared absorption spectra. Spectra generates a sample profile, a special molecular fingerprint that can be used to screen and scan samples for a variety of components. FTIR is an effective analytical tool for the detection of functional groups and characterization of covalent bonding information.

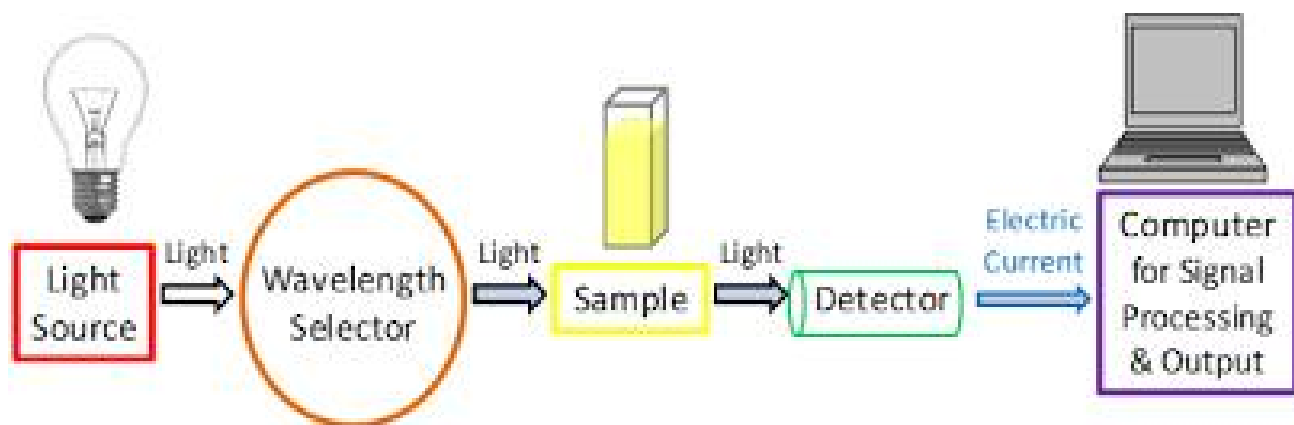
This infrared spectroscopy method is used for the determination of organic, macromolecular and in some cases inorganic materials. The FTIR assay relies on infrared light to scan the sample and observe the binding properties. The FTIR Analysis Service can identify compounds and general materials analyzed when there are unknowns. This technique is used to evaluate the purity of certain inorganic samples and is very reliable in determining the composition of polymers.

UV-Vis spectroscopy

UV-Vis spectroscopy is an analytical technique that measures the amount of discrete wavelengths of UV or visible light absorbed or transmitted through a sample compared to a standard or blank sample. When the interaction between the incident radiation and the electron cloud in the chromophore results in an electron transition involving the promotion of one or more outermost shells or the bonding of electrons from the ground state to the ground state. At higher energies, the ultraviolet visible (UVVis) spectrum is formed.

In general, the spectrum of UV and visible spectrum of substances is very wide. And may not exhibit high compound recognition accuracy. However, they are sufficient for quantitative assays and are useful as an alternative means of detection for some substances. Radiation from

a typical hot solid consists of several wavelengths and depends mainly on the temperature of the solid and can be predicted from the principle of chance, the energy released at each given wavelength.



5.RESULT AND DISCUSSION

XRD ANALYSIS

X-ray diffraction analysis is a method of structural analysis of the spatial distribution of atoms in a substance by the diffraction formed by X-ray irradiation on the crystal.

XRD used to study the crystallographic structure of the nanocomposites. X-ray diffractometer is generally used to analyze the crystal structure, lattice parameters, crystallinity and particle size of $\text{Fe}_3\text{O}_4/\text{C}$ composite materials.

The X-ray diffraction pattern of the C- Fe_3O_4 nanocomposite is synthesized via the hydrothermal route. The XRD patterns of iron oxide NPs and their NCs with carbon.

the XRD pattern of Fe_3O_4 nanoparticles shows six characteristics peak for 2θ values of 30.00 , 35.50 , 43.10 , 53.60 , 57.20 and 62.50 corresponding to the crystal planes of (220), (311), (400), (422), (511) and (440). After coating carbon on Fe_3O_4 nanoparticles, the XRD patterns of $\text{Fe}_3\text{O}_4@\text{C}$ samples also display six distinct diffraction peaks of the magnetite phase.

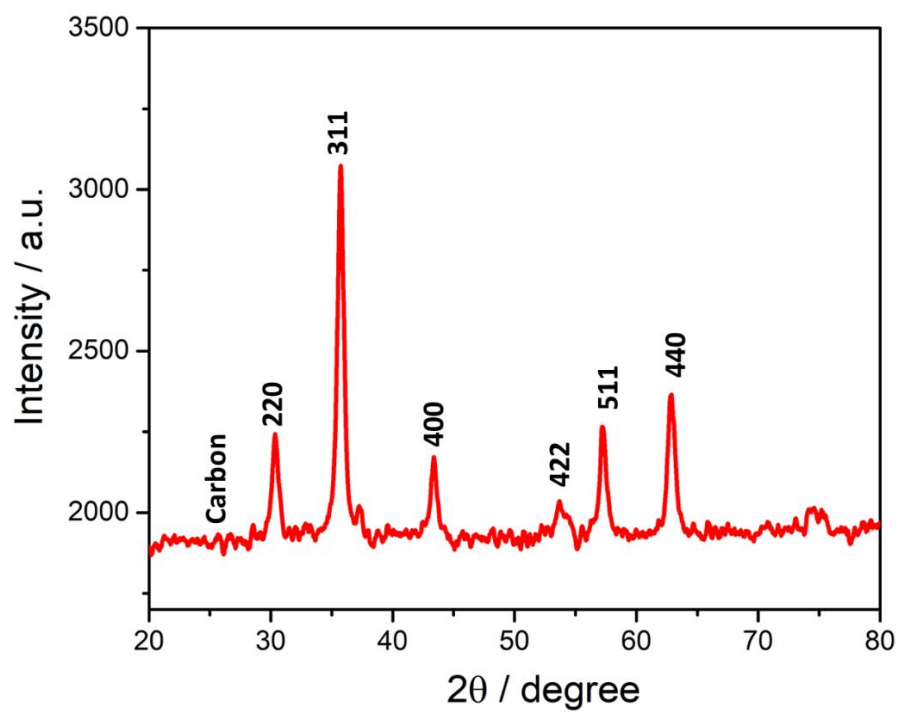


Figure 1. X-ray diffraction (XRD) spectrum of the C-Fe₃O₄ nanocomposite.

Scanning electron microscopy (SEM)

SEM is a surface scanning techniques to produce a magnified image of the sample by using high-energy beam of electrons in raster scan pattern. The SEM was used to evaluate the dimension, shape, and surface morphology of the nanoparticles.

SEM technique is based on electron scanning principle, and it provides all available information about the NPs at nanoscale level. we adopt SEM to investigate the morphology of the pure PVA and prepared nanocomposite. The surface morphology of the sample after interacting with the electron beam was observed by scanning electron microscope.

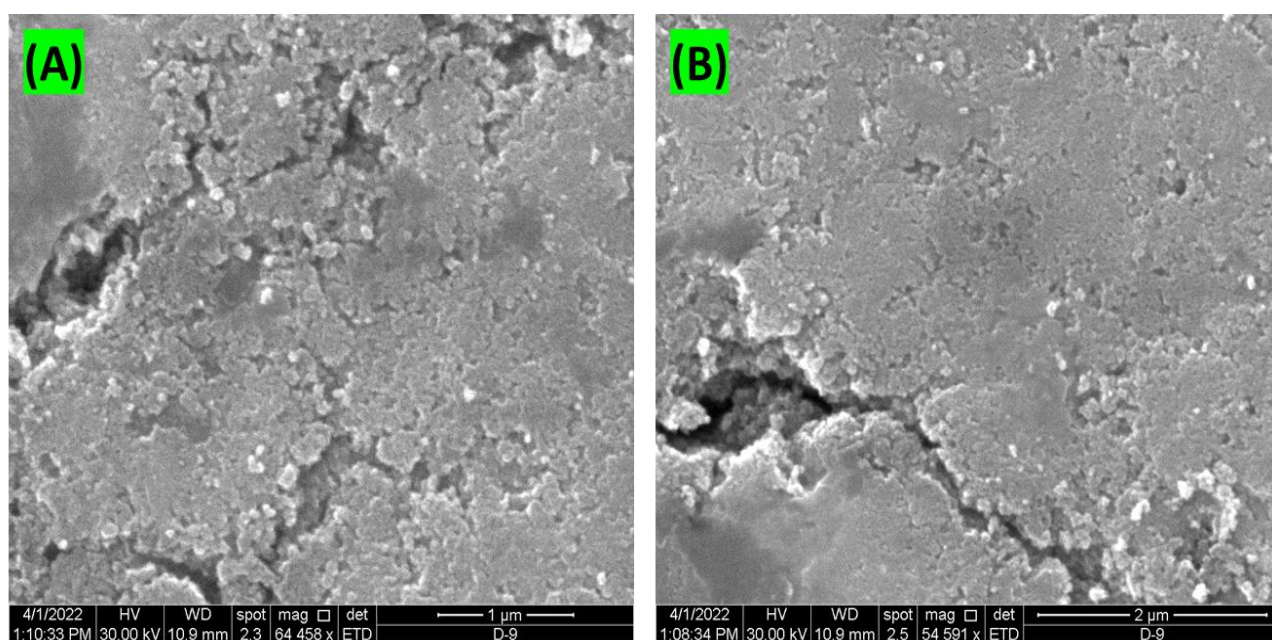


Figure 2 (A-B) Different magnifications of SEM images of the C-Fe₃O₄ nanocomposite.

FT-IR analysis

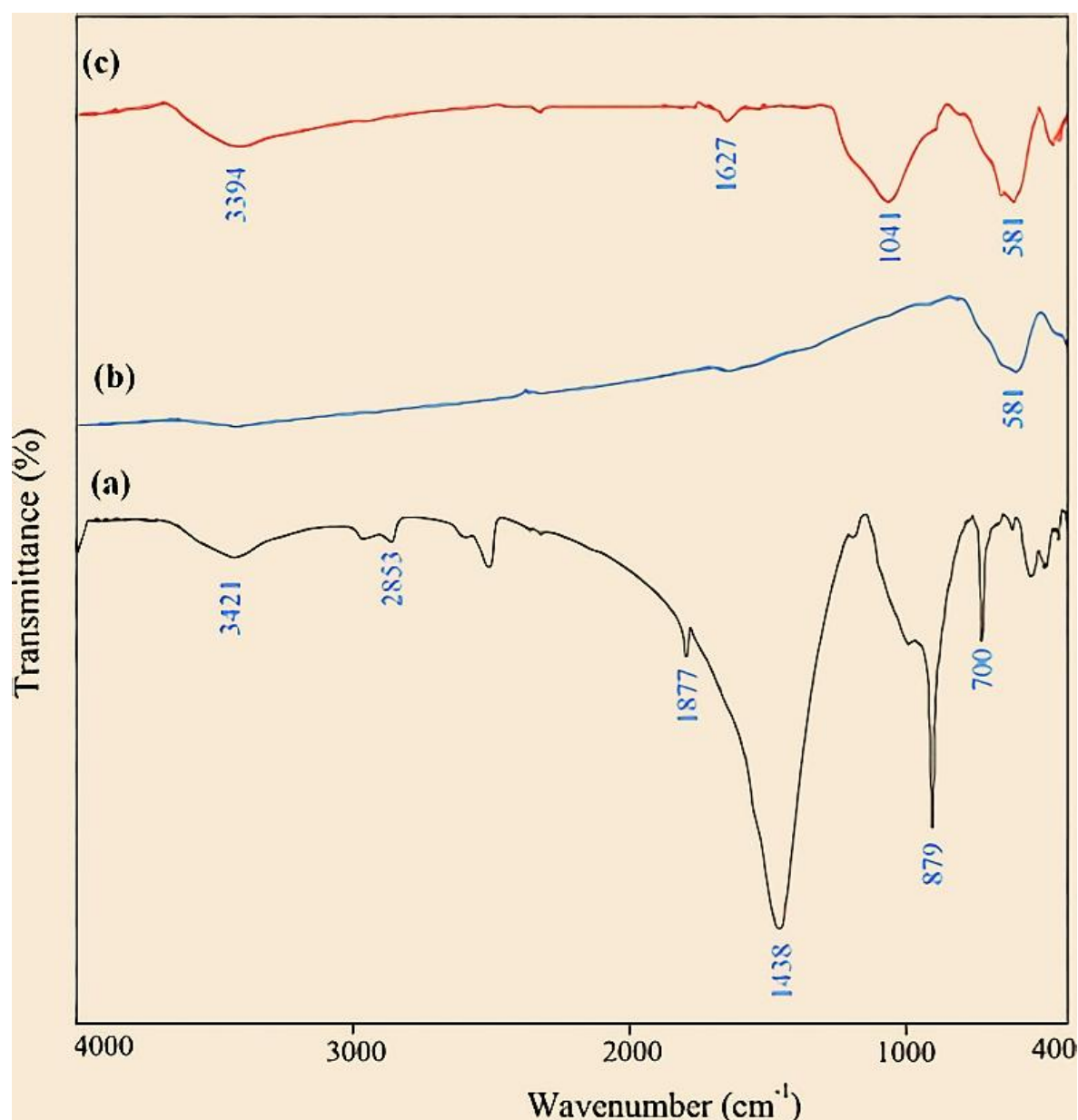


Fig. 3 FT-IR spectra of a carbon b Fe_3O_4 nanoparticles c $\text{Fe}_3\text{O}_4/\text{C}$ nanocomposite

In order to investigate the presence of functional groups in final product, Fourier transform infrared (FT-IR) spectra of carbon, Fe_3O_4 nanoparticles, and $\text{Fe}_3\text{O}_4/\text{C}$ nanocomposite were obtained in the range 400–4000 cm^{-1}

According to the Fig. 3a, the weak absorption bands centered at 3421, 2853, and 1777 cm^{-1} can be attributed to the N–H, C–H, and C=O, respectively. Besides, strong absorption peaks at 1438 and 879 cm^{-1} are assigned to C–C and C–H, respectively. In addition, a weak peak at 700 cm^{-1} is attributed to the C–H and

C:C–H. Figure 3b, c show the FT-IR spectrum of Fe₃O₄ nanoparticles and Fe₃O₄/C nanocomposite. Based on the Fig. 3b, c, absorption band at 581 cm⁻¹ is assigned to the vibration of the Fe–O. Moreover, as shown in Fig. 3c, FT-IR spectrum of Fe₃O₄/C nanocomposite, the peaks at 1041 and 1627 cm⁻¹ are assigned to the C–O and C=C, respectively. The band at 3394 cm⁻¹ is due to the O–H stretching mode of water molecules that was absorbed on the surface of the Fe₃O₄/C nanocomposite [36]

6. REFERENCES

1. X.-Q. Li, P.-X. Hou, C. Liu and H.-M. Cheng, *Carbon*, 2019, **147**, 187–198 [CrossRef](#) [CAS](#).
2. C. Shen, A. H. Brozena and Y. Wang, *Nanoscale*, 2011, **3**, 503–518 [RSC](#).
3. V. Popov, *Mater. Sci. Eng., R*, 2004, **43**, 61–102 [CrossRef](#).
4. Z. F. Ren, *Science*, 1998, **282**, 1105–1107 [CrossRef](#) [CAS](#).
5. A. G. Rinzler, J. Liu, H. Dai, P. Nikolaev, C. B. Huffman, F. J. Rodríguez-Macías, P. J. Boul, A. H. Lu, D. Heymann, D. T. Colbert, R. S. Lee, J. E. Fischer, A. M. Rao, P. C. Eklund and R. E. Smalley, *Appl. Phys. A: Mater. Sci. Process.*, 1998, **67**, 29–37 [CrossRef](#) [CAS](#).
6. P. Nikolaev, M. J. Bronikowski, R. K. Bradley, F. Rohmund, D. T. Colbert, K. Smith and R. E. Smalley, *Chem. Phys. Lett.*, 1999, **313**, 91–97 [CrossRef](#) [CAS](#).
7. Park HJ, Meyer J, Roth S, Skákalová V. 2010. Growth and properties of several layers of chemically vaporized graphene. *carbon*. 48 (4): 10881094. <http://dx.doi.org/10.1016/j.carbon.2009.11.030>
8. Krajewska A, Pasternak I, Sobon G, Sotor J, Przewłoka A, Ciuk T, Sobieski J, Grzonka J, Abramski KM, Strupinski W. 2017. Fabrication and applications of multilayer graphene stack on transparent polymer. *Appl. Phys. Lett.* 110 (4): 041901. <http://dx.doi.org/10.1063/1.4974457>
9. Alam SN, Sharma N, Kumar L. 2017. Synthesis of graphene oxide (GO) by modified Hummers method and its thermal reduction to obtain reduced graphene oxide (rGO) *. *graph.* 06 (01): 118. <http://dx.doi.org/10.4236/graphene.2017.61001>

10. Eda G, Chhowalla M. Chemically derived graphene oxide: towards thin film electronics and large area optoelectronics. *Forward Mat* 22 (22): 23922415. <http://dx.doi.org/10.1002/adma.200903689>
11. Pei S, Cheng H. 2012. Reduction of graphene oxide. *carbon*. 50 (9): 32103228. <http://dx.doi.org/10.1016/j.carbon.2011.11.010>
12. Du X, Skachko I, Barker A, Andrei EY. 2008. Approaches to ballistic transport in suspended graphene. *Nature of Nanotechnology*. 3 (8): 491495. <http://dx.doi.org/10.1038/nnano.2008.199>
13. Pham, S. N.; Kuether, J. E.; Gallagher, M. J.; Hernandez, R. T.; Williams, D. N.; Zhi, B.; Mensch, A. C.; Hamers, R. J.; Rosenweig, Z.; Fairbrother, H.; Krause, M. O. P.; Feng, Z. V.; Haynes, C. L. Carbon Dots: A Modular Activity to Teach Fluorescence and Nanotechnology at Multiple Levels *J. Chem. Ed.* 2017, 94, 1143-1149.
14. Schneider, E. M. ; Bartsch, A.; Stark, W. J. ; Glass, R.N. Safe OnPot Synthesis of Fluorescent Carbon Quantum Dots from Lemon Juice for hands-on experience in nanotechnology *J. Chem. Ed.* 2019, 96, 540545.
15. Mishra, P.; Patnaik, S.; Parida, K. A review of recent advances in precious metal modified magnetic Fe₃O₄ for the decomposition of photocatalytic contaminants and the evolution of H₂. *Cat science. technol.* 2019, 9, 916-941. [Cross reference]
16. Rogach AL, Talapin DV, Shevchenko EV, Kornowski A, Haase M, Weller H. Organization of materials of various size scales: monodisperse nanocrystals and their superstructures. *Advanced Functional Materials*. 2002;12(10):653-64.
17. Stankic S, Suman S, Haque F, Vidic J. Pure and multi metal oxide nanoparticles: synthesis, antibacterial and cytotoxic properties. *Journal of Nanobiotechnology*. 2016;14(1):73.13. Oliveira M, Machado A. Preparation of polymer-based nanocomposites by different routes.
18. Ajayan PM. Bulk metal and ceramic nanocomposites. *Nanocomposite science and technology*. 2003: 176. *Nanocomposites: synthesis, characterization and applications*. 2013:1-22.

19. Khodaei M, Karimzadeh F, Enayati M. Mechanically synthesized metallic ceramics Nanocomposite; Mechanism and Properties: INTECH Open Access Publisher; 2011
20. Naveed M, Khalid Sand Sajid U 2018 Drug Design: A Thousand Words of Photographs in Open Access Nanoparticle Biotechnology 7 1–8
21. XiFeng Zhang, ZhiGuo Liu, Wei Shen, and SG 2016 Silver Nanoparticles: Synthesis, characterization, characterization, application, and therapeutic approach Int JMol Sci. 17 17
22. Ceylan A., Rumaiz A.K., Shah S.I. 2007 Inert gas condensation of vaporized Ni and laser ablated CoO J.Appl. Physics 101
23. XiFeng Zhang, ZhiGuo Liu, Wei Shen and S G 2016 Silver nanoparticles: synthesis, characteristics, properties, applications and treatments Int J Mol Sci. 17
24. Ceylan A, Rumaiz AK and Shah S I 2007 Inert gas condensation of NiCoO J. Appl is evaporatively and laser-ignited. Physical. 101
25. http://shodhganga.inflibnet.ac.in/bitstream/10603/21144/10/10_chapter%203.pdf [Accessed: April 2016]
26. Khah V, Sara D, Djafar IR, Rahman N, Jafar IR. Consider methods of plasma synthesis of nanoparticles. In: Proceedings of the Sixth NanoEurope Congress and Exhibition; St. Gallen, Switzerland; 2008
27. Rabenau A. The role of hydrothermal synthesis in preparative chemistry. Angewandte. Chem. Int. Ed. Engl. 1985; 24: 1026-40.
28. JaskiranKaur, ManpreetKaur, Manpreet Kaur Ubhia, NavneetKaur, Jean-Marc Greneche has performed Composition optimization of activated carbon-iron oxide nanocomposite for effective removal of Cr(VI)ions.
29. Rais Ahmad* and Rajeev Kumar has performed Kinetic and Thermodynamic Studies of Brilliant Green Adsorption onto Carbon/Iron Oxide Nanocomposite. Carbon / iron oxide nanocomposites were prepared by chemical precipitation and heat treatment of the carbon with iron nitrate at 750 °C.
30. Narayanan TN, Reena Mary AP, Shaijumon MM, Lijie Ci, Ajayan PM, and Anantharaman MR performed Synthesis of magnetic properties of multi-

- walled carbon nanotubes - Superparamagnetic iron oxide nanoparticle nanocomposites.
31. Hengli Xiang, Genkuan Ren, Yanjun Zhong, Xiushan Yang, Dehua Xu¹, Zhiye Zhang and Xinlong Wang have studied Characterization and synthesis of Fe₃O₄@C nanoparticles by in-situ solid-phase method.
 32. Xiaowang Liu*, Zhen Fang, Xiaojun Zhang, Wei Zhang, Xianwen Wei, and Baoyou Geng* has done Preparation and Characterization of Fe₃O₄/CdS Nanocomposites and Their Use as Recyclable Photocatalysts.
 33. Javad Moradiganjeh and Zahra Aghajani has done Synthesis and characterization of novel magnetic Fe₃O₄/C through the Co-precipitation method and investigation of its desulfurization application.
 34. Hamidreza Sadegh, Ramin Shahryari-ghoshekandi & Maryam Kazemi have done the Study on Synthesis and characterization of carbon nanotubes decorated by magnetic iron oxide nanoparticles.
 35. Zahra Razaghi, Zahra Aghajani², Ali Yeganeh Faal has performed Novel synthesis and characterization of Fe₃O₄/C nanocomposites and investigation of its application in removal of pesticide diazinon.
 36. M. Salavati-Niasari, S.M. Hosseinpour-Mashkani, F. Mohandes, J. Mater. Sci. Mater. Electron. 26, 7936 (2015)