SYNTHESIS AND CHARACTERIZATION OF COBALT OXIDE NANOPARTICLES

Submitted in partial fulfillment of the requirements for the award of Bachelor of Science in Chemistry

by

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **A. GOKULAPRIYA(Reg.No. 40030010) and R. SRUTHI SHERIN (Reg.No. 40030036)** who carried out the project entitled **"SYNTHESIS AND CHARACTERIZATION OF COBALT OXIDE NANOPARTICLES"** under my supervision from December 2022 to April 2023.

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DECLARATION

We **A.GOKULAPRIYA (40030010)** and **R.SRUTHI SHERIN** (40030036) hereby declare that the project Report entitled **Synthesis and characterization of Cobalt oxide NanoParticles COBALT OXIDE NANO PARTICLE"** done by us under the guidance of **Dr.Y.SASIKUMAR** (Internal) and **Dr.G.MURUGADOSS** (External) **centre of nanotechnology, Sathyabama Institute of science and Technology** is submitted in partial fulfilment of the requirements for the award of Bachelor of Science degree in **Chemistry**

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CONTENTS

LIST OF ABBREVATION

1) nm – nanometre
2) NPs – nanoparticles
3) XRD - x-ray diffraction
4) UV – ultra violet
5) SEM – scanning electron microscopy
6) FTIR - fourier transform infrared
7) Co_3O_4 – cobalt oxide
8) 1D - one dimensional
9) 2D - two dimensional
10) 3D – three dimensional
11) NaOH -Sodium hydroxide
12) DI - De-ionised water
13) PVP - polyvinylpyrrolidone

Chapter 1

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 nanoparticles 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 threedimension 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, multi nanolayer-type structures.

1.2 CLASSIFICATION OF NANOPARTICLES

1.2.1 Carbon based nanoparticles

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. 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 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 method 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 application 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.

1.3 COBALT OXIDE NANOPARTICLES

Cobalt oxide nanoparticles appear as a white powder with spinel crystal structure. They are an important magnetic material, and are P-type semiconductors. When nano-cobalt oxide is exposed to the hydrogen flame and heated to 900°C (1652°F), it changes into metal cobalt.Cobalt oxide nanoparticles are graded as harmful to humans and dangerous for the environment. They can be harmful if swallowed, and cause allergic skin reactions. They are also very toxic to aquatic life with long lasting effects.

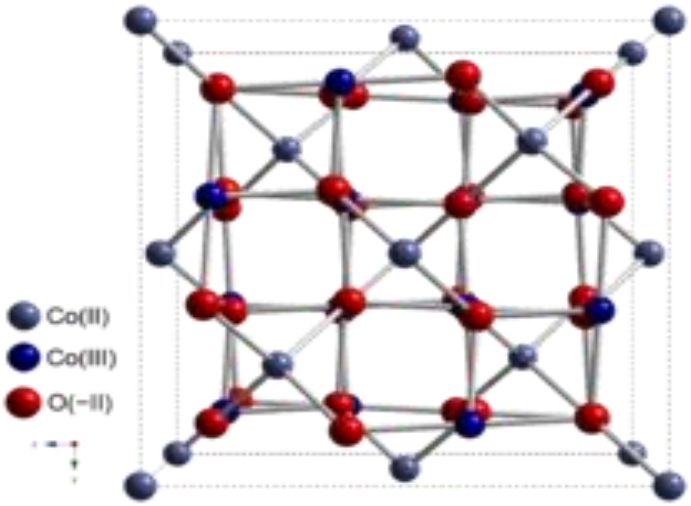


Fig 1.3 Cobalt(II,III)Oxide structure

1.4 APPLICATIONS OF COBALT OXIDE NANOPARTICLES

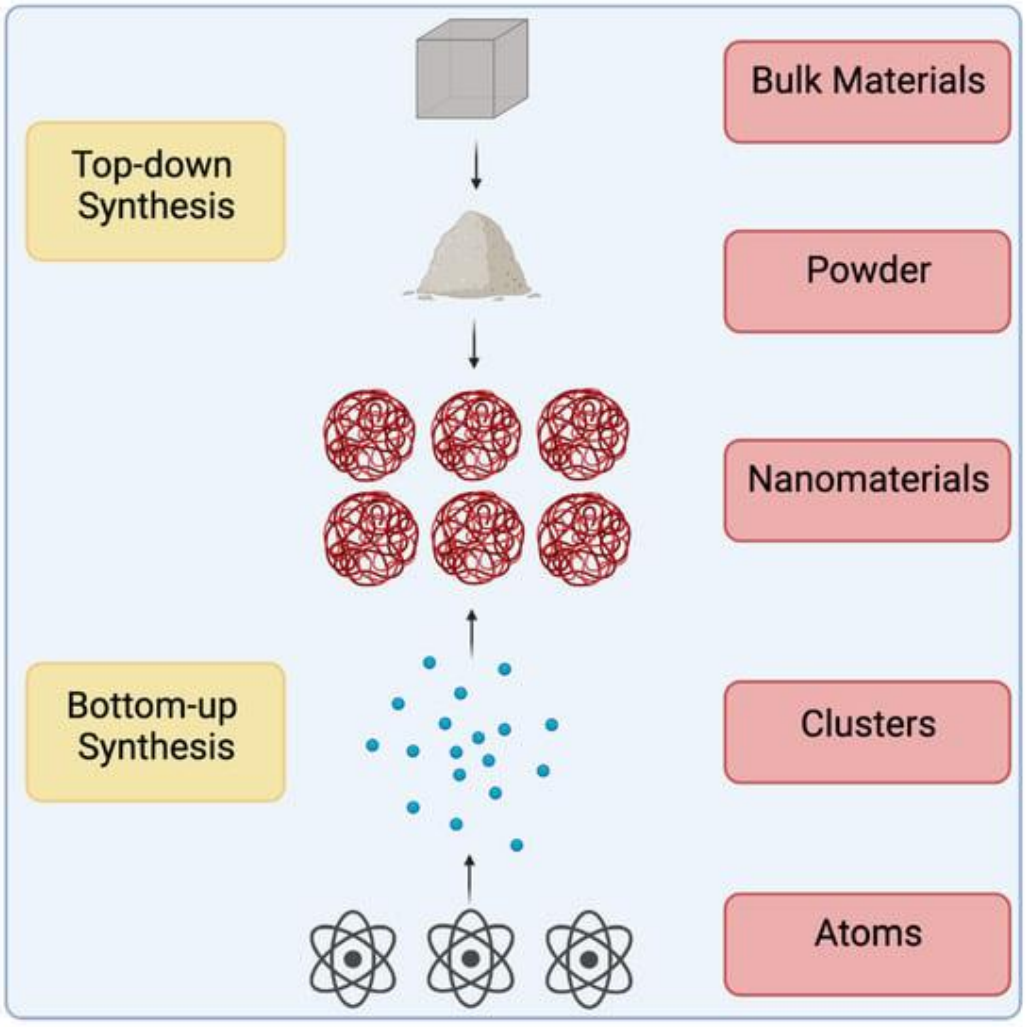
The key applications of cobalt oxide nanoparticles are as follows:

- In micro-electronics.
- As a magnetic nanoparticles with numerous uses in micro batteries, nanowires, and specific alloy and catalyst applications.
- In catalysis, superconductors, electronic ceramics and other fields as an important inorganic material.
- As catalyst and catalyst carriers.
- As an electrode active materials.
- For glass, porcelain colorants and pigments.
- Chemical industry oxidants.
- Senior goggles and other filter materials.
- As carbides.
- In temperature and gas sensors.

1.5 SYNTHESIS OF NANOPARTICLES

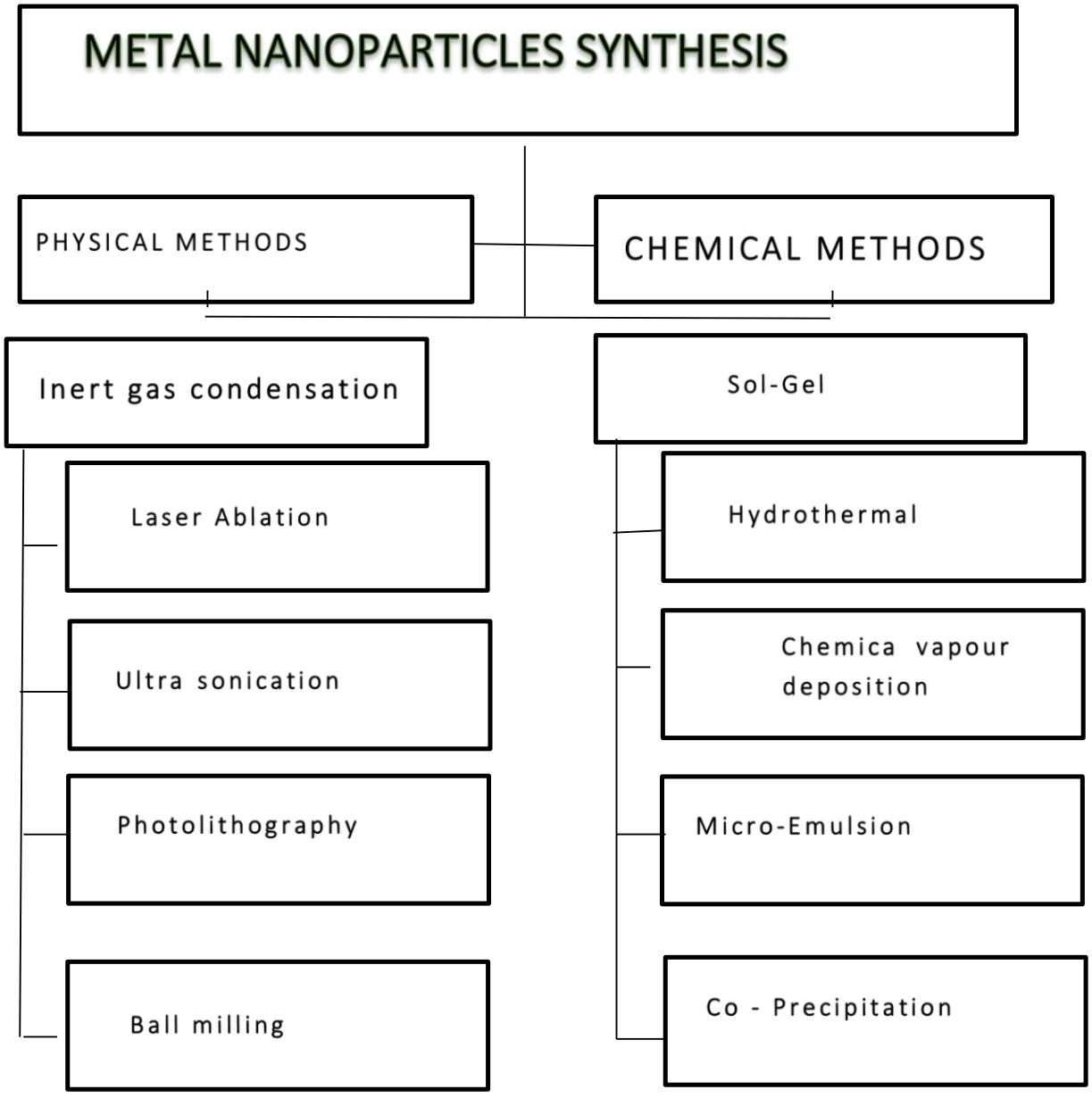
The synthesis of nanomaterials of uniform size is important because their properties include optical, magnetic, electrical and biological properties depending on size and dimension (4).

The synthesis methods are generally divided into three categories: solution synthesis, gas phase synthesis (5). Another approach is to divide these comprehensive approaches into two broad categories



a) Physical methods and
b) Bottom-up approaches including wet chemistry [6].

The choice of a synthetic approach depends on the desired properties required for the nanoparticle mixture. In terms of size, morphology, crystal structure, etc. [7], the advantage of physical method 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.

Ex: Laser ablation, Ultrasonication, Photolithography, Electron beam lithography, Ball milling.

BOTTOM-UP APPROACH (chemical method): 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.

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

1.6 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 [8]. 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 [9]. 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 [10].

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 moves. Sound waves propagate in a liquid medium, alternating between at pressure (compression) and low pressure (dilution) cycles. During dilute 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 carbides 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 nano size 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

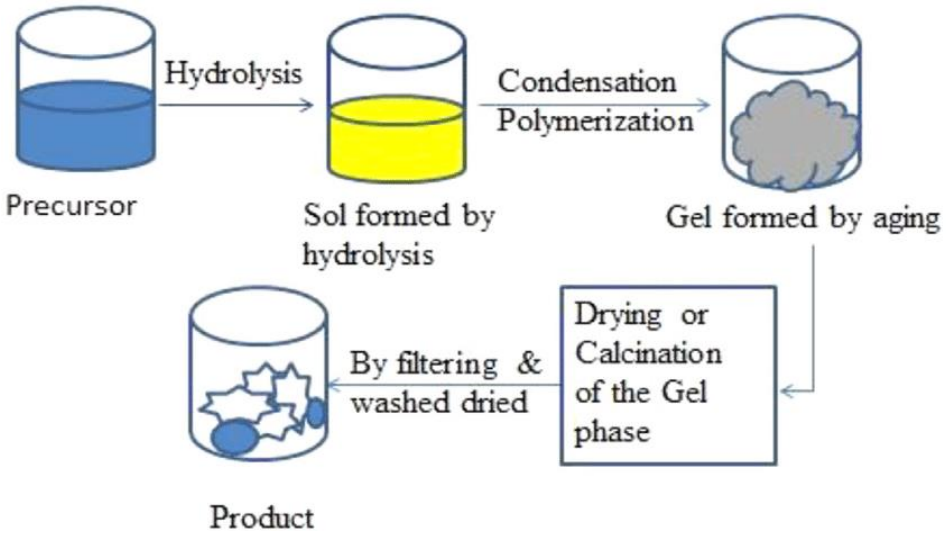
(b) Polymers (PVA, PVP)

1.7 CHEMICAL METHOD

The advantages of these methods are low cost, ease of manufacture, and high productivity [11]. 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 particles 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 [12].

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 oxides 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 hydroxo polymer 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 VAPOUR 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:

Steps in CVD method are:

1.The reactants are transported to the growth surface by a boundary layer.

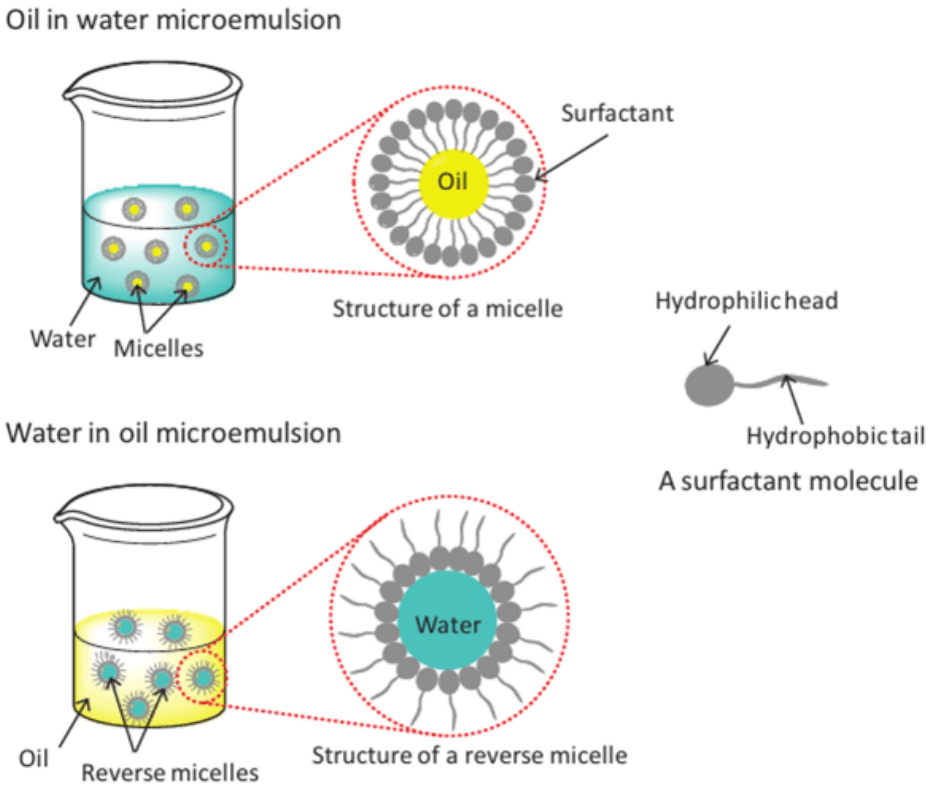
2.Chemical reactions occur on the growth surface.

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 1μm by chemical reaction taking place in the gas phase. The reaction can be controlled to produce nanoparticles with sizes between 10 and 100 nm [13,14].

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.



HYDROTHERMAL SYNTHESIS

Hydrothermal synthesis refers to the heterogeneous reaction used to synthesize inorganic materials in aqueous media above ambient temperature and pressure [15]. 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 rection 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 products yields compared to the coprecipitation method.

Chapter 2

Literature Review

There is a lot of work on the preparation of Nano scale samples in different ways and there are studies on the diagnosis and the properties of these samples; one of these properties is the magnetic property.

Karimian and his group prepared manganese oxide Nano rod and cobalt oxide nanotube by a sol-gel reaction in reverse micelles. They studied the structure, the surface morphology of the manganese oxide nanorod by means of X-ray diffraction analysis, scanning electron microscopy, and transmission microscopy (Karimian *et al*., 2012).

Magnetic $Zn_{1-x}Co_xO$ nanorods were fabricated via direct hydrothermal

\synthesis by Yang and his group. They showed that the nanorods had a ferromagnetic character and they were obtained with 98 and 36 Oe coercive fields Hc for nominal x=0.029 and 0.056, respectively (Yang *et al*., 2006). A cobalt nanoplatelet samples were synthesized with different hcp phase contents by a novel reduction process. Jiangong and his group found that the saturation magnetization of the nanoplatelet powders was lower than the corresponding bulk value and the coercivities of the nanoplatelets increased with the hcp phase content (Jiangong *et al*., 2004).

Pamela and his group, Prepared Co nano powders of 30 - 70 nm. They studied the characterization using XRD, SEM, TEM and VSM. The results of VSM showed a higher saturation magnetization at 100 K of the nano cobalt compared with that for the bulk metal (Pamela *et al*., 2011). Nickel (Ni) electrodeposited inside Cobalt (Co) nanotubes was fabricated by Narayanan and his group, using a two-step potentiostatic electro deposition method with an average diameter of 150 nm and length of ~15 µm. They found that the Ni and Co nanorods exhibited a very high longitudinal coercivity (Narayanan *et al*., 2010).

A metallic Co nanorods (about 100 nm in diameter) formed inside an array of anodized aluminum oxide AAO nanopores synthesized by Aslam and his group. They found that the increase of nanorod length led to increase the coercivity and thermal activation volume (Aslam *et al*., 2005). Shihua and his group, prepared Co nanoparticles by metal vapor synthesis technique. They studied the characterization and properties by TEM, XRD, temperature-programmed desorption, chemisorption, magnetic measurements. They concluded that the particle size of Co powder depends on the initial Co concentration in the toluene matrix, and the Co powder indicates ferromagnetic behavior (Shihua *et al*., 2003).

Cobalt nanoparticles by hydrothermal method were synthesized by Pauline and his group. The particle size decreased as the annealing temperature increases (Pauline *et al*., 2012).

Jianchun and his group, fabricated nanotubes and nanowires cobalt in the same alumina template modified with an organ amine, which was an important factor to control nanostructure shapes. They indicate that the shape anisotropy had an effect on the magnetic properties (Jianchun *et al*., 2004).

Mixture of crystalline Co_3O_4/CoO nanorods with an average length of around 80 nm and an average diameter of 42 nm by microwave hydrothermal technique were synthesized by Al-Tuwirqi and his group.

They characterized it by XRD, FE-SEM, energy-dispersive, EDX, and FTIR methods (Al-Tuwirqi *et al*., 2011). They found that the magnetic hysteresis loops at room temperature have soft magnetic behavior.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

SYNTHESIS AND CHARACTERIZATION OF COBALT OXIDE NANOPARTICLES

3.2 SCOPE

- Characterization of the synthesized Co_3O_4 nanoparticles is crucial for understanding their properties and potential applications.
- Determination of structural properties of Cobalt oxide nanoparticles.
- Determination of optical properties of Cobalt oxide nanoparticles.
- Morphology of Cobalt oxide.
- Techniques such as FTIR, UV-visible, X-ray diffraction, scanning electron microscopy, and spectroscopy can be used to study the size, shape, crystallinity, and surface properties of the nanoparticles.

CHAPTER- 4

MATERIALS AND METHODS

4.1 CHEMICALS

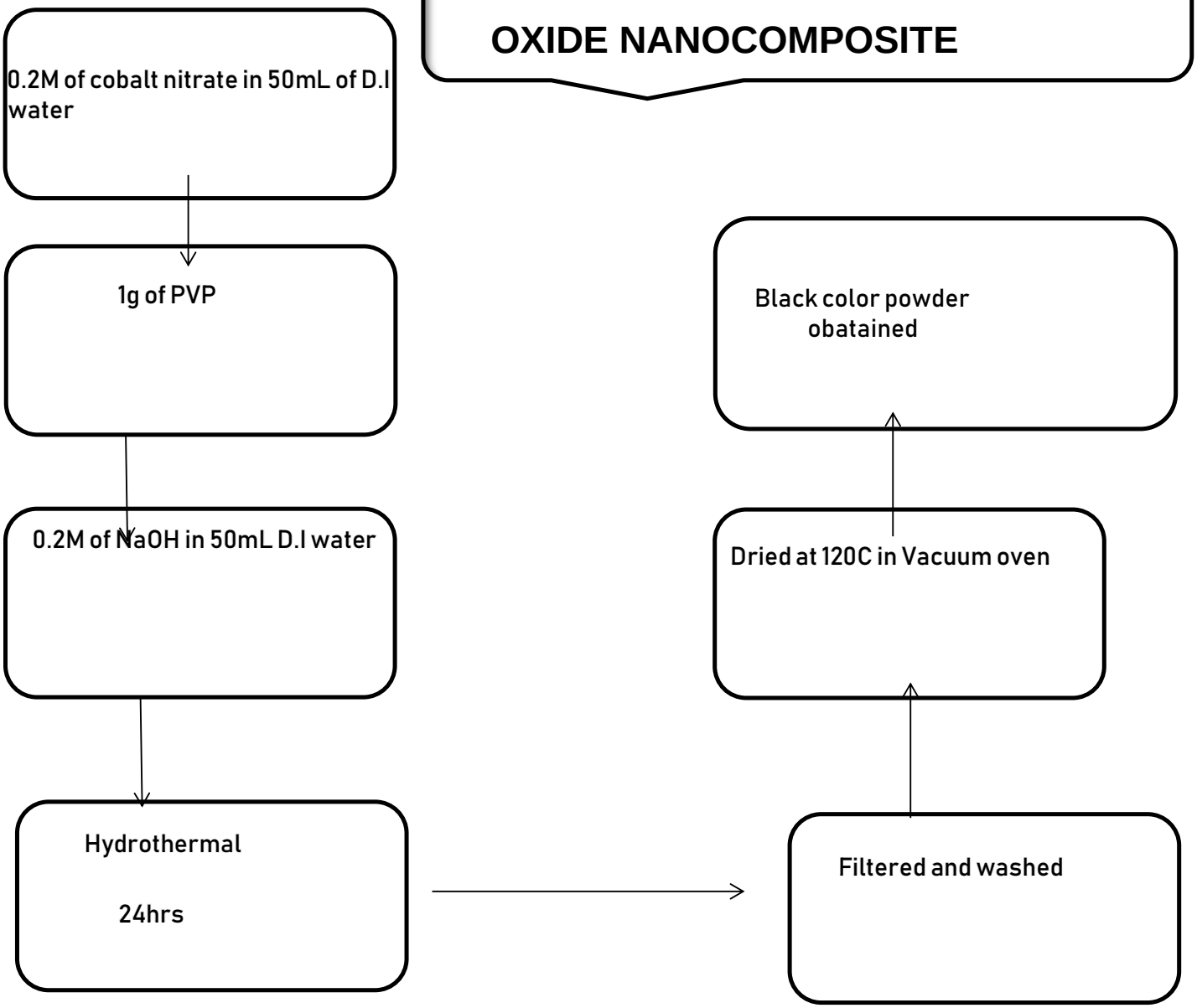
Cobalt nitrate , sodium hydroxide, polyvinyl pyrrolidone(PVP) used in this study were purchased from Sisco Research Laboratories Pvt Ltd. (SRL)- India. Deionised water. All the chemicals used in this work were of commercially available analytical grade(AR) and were used without further purification.

4.2 EXPERIMENTAL METHODS

The procedure involved to prepare C- Co_3O_4 nanocomposite is as follows

0.2 M Cobalt nitrate were dissolved in 500 ml of deionised water and placed at the magnetic stirrer. Add 1g of polyvinyl pyrrolidone to reduce particle size and prevent aggiomeration. Dissolve 0.2M of NaOH in 50ml of deionized water, gradually add drop wise to the cobalt nitrate 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 24h. after reaction at 180 for 24h. 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.

SYNTHESIS OF COBALT OXIDE NANOCOMPOSITE



4.3 CHARACTERIZATION TECHNIQUES

4.3.1 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.

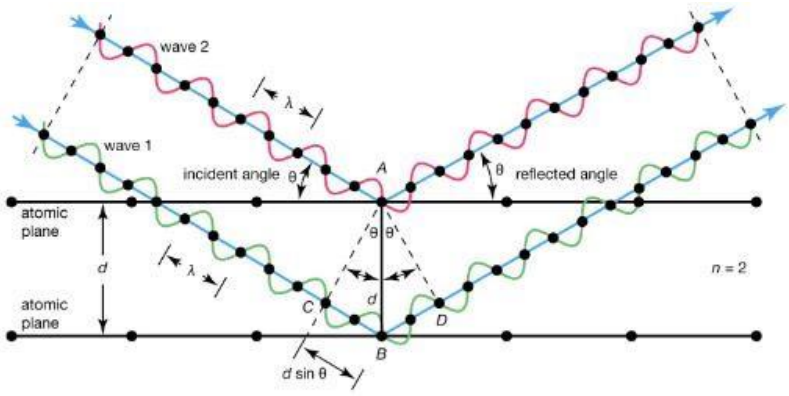
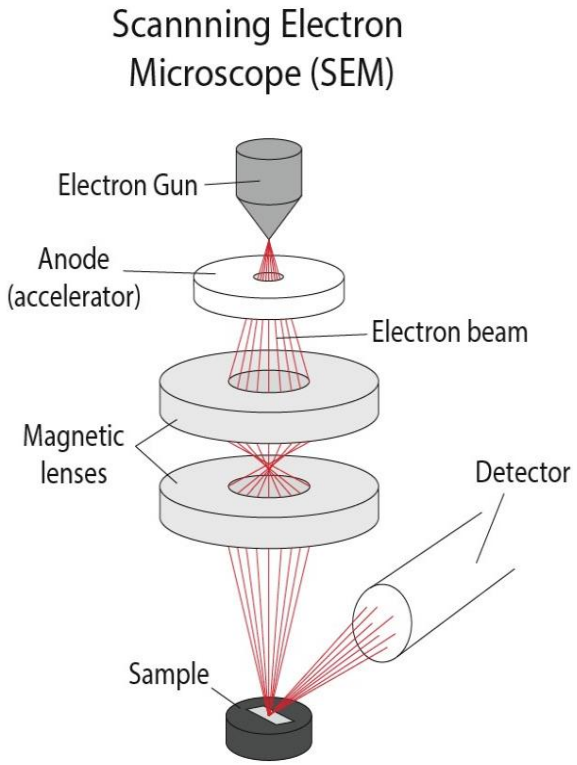


Fig 4 illustration of Bragg's Law.

4.3.2 Scanning electron microscopy (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.



4.3.3 Fourier transform infrared (FTIR) spectroscopy analysis

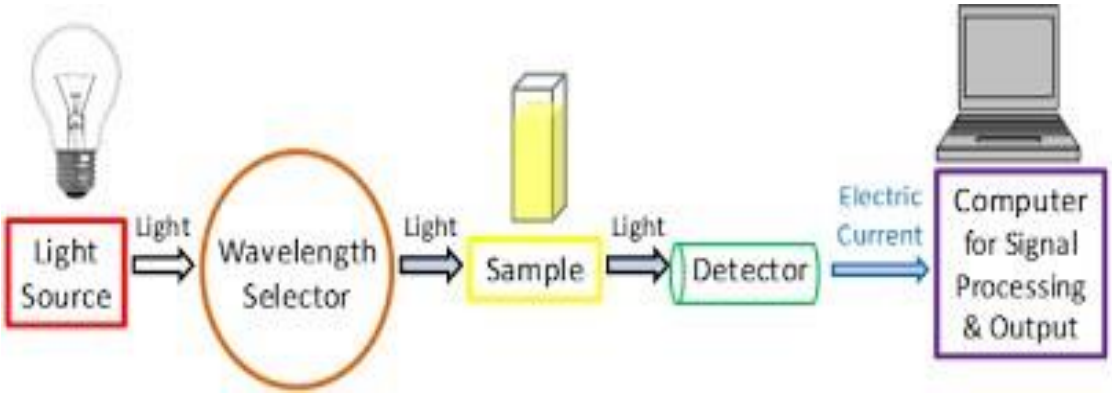
Fourier transform infrared (FTIR) spectroscopy identifies chemical bonds in molecules by generating infrared absorption spectra. Spectra generates a sample profile, a special molecular fingerprint that can be used to screen and scan sample for a variety of components. FTIR is an effective analytical 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 analysed 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.

4.3.4 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 (UV-Vis) 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.



Chapter 5

RESULTS AND DISCUSSION

Crystal structure, morphology, compositional information, organic functional groups on the surface and catalytic 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) and ultraviolet spectroscopy (UV). These highlighted techniques are presented in this project work. Brief information about techniques has been discussed in the following section.

UV-V Spectroscopy

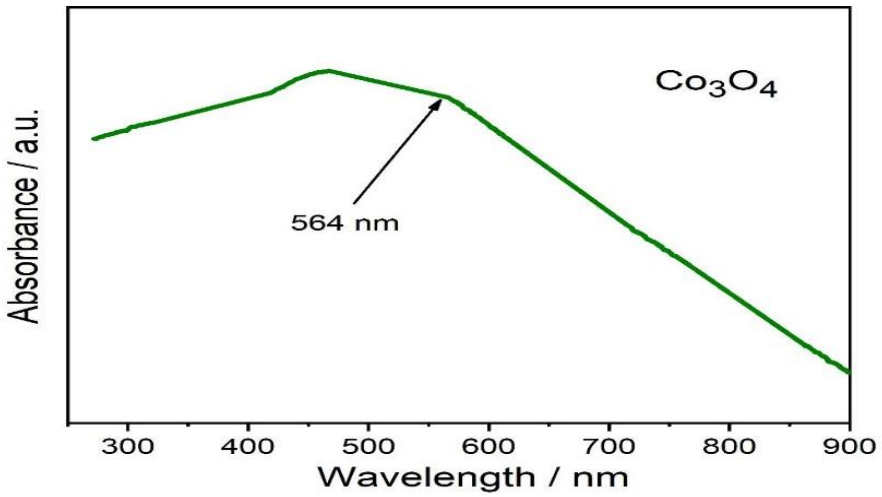


Fig 5.4 UV-visible spectra for Co_3O_4

For UV- VIS spectroscopy, the absorption spectrum of a solution of cobalt oxides nanoparticles is measured as a function of wavelength. This is shown in Figure it shown a wide absorption band around 564.0 nm in the visible range of wavelengths from 300.0 to 900.0 nm, indicating the formation of cobalt oxide nano structured materials [26]. The bandgap energy (E_{bg}) is calculated based on the maximum absorption band of the nanoparticles and is obtained as 2.2 eV according to Equation

$E_{bg} = 1240 / \lambda$ (ev)

Where E_{bg} is the bandgap energy and kmax is the wavelength of the nanoparticles (564.0 nm).

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 Co_3O_4/C composite materials.

The X-ray diffraction pattern of the C- Co_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 Co_3O_4 nanoparticles shows six characteristics peak for 2θ values of 21.00, 31.00, 38.00, 39.00, 47.00, 59.00, 62.00 and 67.00 corresponding to the crystal planes of (111), (220), (311), (222), (400),(422),(333) and (440). After coating carbon on Co_3O_4 nanoparticles, the XRD patterns of Co_3O_4@C samples also display six distinct diffraction peaks of the magnetite phase.

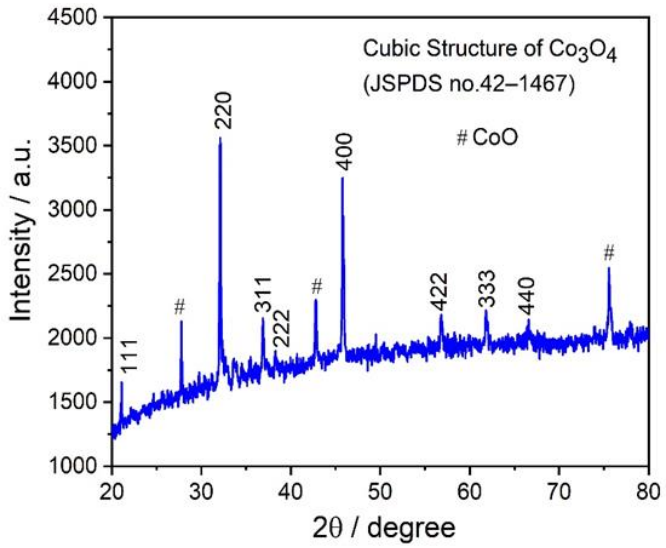


Fig 5.1. X-ray diffraction (XRD) spectrum of the C- Co_3O_4 nanocomposite.

Scanning electron microscopy (SEM)

SEM is a surface scanning technique 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.

Fig 5.2 shows that the morphology of annealed C- Co_3O_4 nanocomposite analyzed using SEM. Figure 5.2 a and b having different energy magnifications of nanocomposite at 1 micron and 2 microns respectively. As shown in SEM images, the sample exhibits spherical structure and has particle size ranging between 0.1 to 0.2 micron. The average diameter 9.9 nm for C-Co_3O_4.

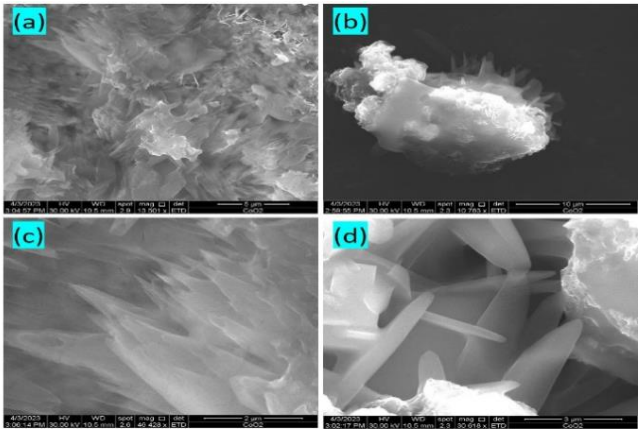


Fig 5.2 (A-B) Different magnifications of SEM images of the C- Co_3O_4 nanocomposite

FT-IR analysis

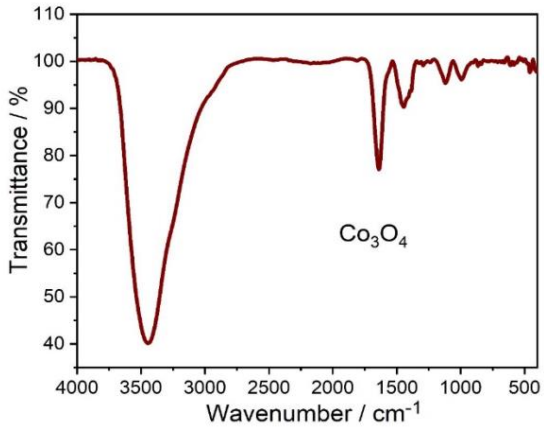


Figure 5.2 shows FT-IR spectra of cobalt oxide metal nanoparticles synthesized by co-precipitation method. FTIR spectroscopy was carried out in order to ascertain the purity and nature of cobalt oxide metal nanoparticles as synthesized by coprecipitation method.

Oxides and hydroxides of metal nanoparticles generally gives absorption peak in the finger print region i.e. below wavelength of 1000 nm arising from inter-atomic vibrations. The bands at 515 and 480 cm-1 correspond to the Co–O bond (Kang et al, 2007;Li et al, 2007; Potter and Rossman, 1979). From the above result we conclude that the synthesized nanomaterial is cobalt oxide. Absorption peak observed at 2924.09 cm-1 may be due to -CH3 stretching vibrations. The absorption peaks at 2852.72 cm-1 , 2021.40 cm -1 and 1382.96 cm-1 may be due to –CH2 stretching, =C-H stretching and –C-H stretching vibrations

CHAPTER 6

SUMMARY AND CONCLUSIONS

Co_3O_4 nanoparticles have been successfully synthesized by Co-Precipitation method. The nanoparticles are characterized by XRD, SEM, UV and FTIR techniques. Co_3O_4 nanoparticles of simple cubic structure were synthesized by co-precipitation method using green chemistry. The FT-IR spectral analysis reveals the characteristics peaks of Co-O stretching. The UV-visible absorption shows sharp absorption at 564 nm due to metal nanoparticles. There are large numbers of potential applications of Co_3O_4 metal nanoparticles such as in the field of electrode materials in different rechargeable batteries, biosensors, coatings, nanofibres, nanowires and also in specific biogenic and bioscience applications.

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