Synthesis and Characterization of Composite of NiO and $ZnFe_2O_4$ as Cancer Treatment

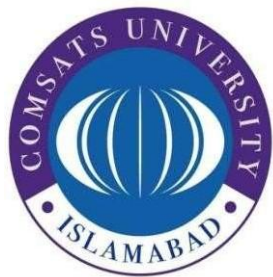Final Year Project
Report BS Physics

Submitted by:

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Supervised by

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COMSATS University Islamabad, Lahore Campus

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A Final Report / Project Presented to

COMSATS University Islamabad, Lahore Campus

In partial fulfillment of
requirement for the degree of

BS (Physics)

By

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An Undergraduate project report

submitted to the Department of Physics

As partial fulfillment for the award of Degree

of BS Physics

Supervisor:

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Synthesis and Characterization of Composite of NiO and ZnFe₂O₄ as Cancer Treatment

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DECLARATION OF ACADEMIC INTEGRITY

We, hereby declare that this project neither as a whole nor as a part there of has been copied out from any source. It is further declared that we have developed this project thesis and the accompanied report entirely based on our personal efforts made under the sincere guidance of our supervisors. No portion of the work presented in this report has been submitted in support of any other degree of qualification of this or any other University or Institute of learning, if found we shall stand responsible.

Date:10-Jan-2022   **Signature of the Student:**_____

Certificate

It is certified that **Hafiz Abdul Haseeb (CIIT/SP18-BPH-011/LHR)** and **Ubaid Ur Rehman (CIIT/SP18-BPH-029/LHR)** has carried out all the work related this thesis under supervision at the Depart of Physics COMSATS Institute of Information Technology, Lahore and the work fulfills the requirement for award of BS degree.

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DEDICATED TO

Dr Muhammad Hammad Aziz

&

Our Parents

بسم الله الرحمن الرحيم

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Last but not least we would like to acknowledge our parents for their continuous, generous, moral and financial support.

The seed which is not willing to let it`s shell root, cannot bear fruits.

Ibn e Arabi

Table of content

Chapter 1
Introduction

1. Introduction

1.1 Nanotechnology:

Nano sciences is the study of extremely small things which are observed at Nano scale in all sciences. The basic concept of Nano-sciences and nanotechnology started with a talk entitled "There's Plenty of Room at the Bottom" by physicist Richard Feynman at an American Physical Society meeting held in California Institute of Technology (CalTech) on December 29, 1959. Feynman describes that scientists are able to control and manipulate individual atoms and molecules. Practically Nano level study and experiments are carried out after the invention of tunneling microscope. Everything in this world is made up of atoms, our vehicles, our planet, and our houses even our body. So when we need to look on an individual atom and try to control it, we are unable to do so before the invention of scanning tunneling microscope (STM) and atomic force microscope The Nano scale deals with dimensions between 1 and 100 nanometers. Nanoparticles and nanomaterial's in used in the research from a long time. The reason for the importanceofnanoparticlesandnanomaterial'sisduetotheiruniqueeffectsinsurfacechemistry.

Nanoparticle contains brilliant magnetic, electric and structural properties as compared to bulk form magnetic and structural properties can be groomed if the samples are prepared in Nano form by increase in surface to volume ratio. Due to the novelty of nanoparticles, families of magnetic material ferrites are dominant. Their applications are in multiple fields like sensors, drug delivery, hyperthermia, memories storage, magnetic hyperthermia etc. The Nano scale deals with the dimensions between 1 and 100 nanometers. One nanometer is a billionth of a meter or 10^{-9} of a meter. When any particle is taken at Nanoscale, material changes its properties and you can control the properties more effectively. Basically the nanotechnology increases the surface area of a material. So due to increase in surface area, properties of a material become stronger, durable, controllable andstronger.

**1.2 Methodology of nanotechnology**:

The following approaches are used in nanotechnology:

1. Bottom-up approaches: To arrange smaller components into more complexassemblies.

2. Top-down approaches: To create smaller devices using larger ones to direct theirassembly.

3. Functional approaches: To develop components of a desired functionality without regard to how they might be assembled.

1.3 Applications of nanotechnology in medicalsciences

As the need for the development of new medicines is pressing, and given the inherent nanoscale functions of the biological components of living cells, nanotechnology has been applied to diverse medical fields such as oncology and cardiovascular medicine. Indeed, nanotechnology is being used to refine discovery of biomarkers, molecular diagnostics, and drug discovery and drug delivery, which could be applicable to management of these patients.To achieve these aims, nanotechnology strives to develop and combine new materials by precisely engineering atoms and molecules to yield new molecular assemblies on the scale of individual cells, organelles or even smaller components, providing a personalized medicine.[2] Personalized medicineis
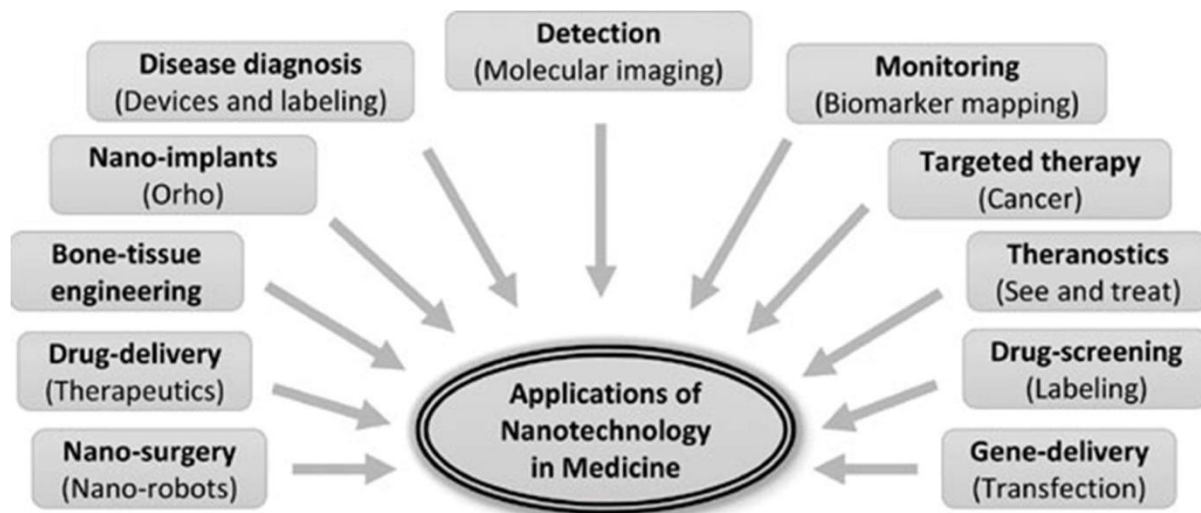
Fig 1: Application of nanotechnology in medical science

individualized or individual-based therapy, which allows the prescription of precise treatments best suited for a single patient. [3]

1.4 Nano diagnostics and Personalized CancerTherapy

Nano biotechnology has the potential to improve early detection of cancer and facilitate the personalization of cancer therapy. The most important component of personalized medicine is molecular diagnostics and Nano biotechnology will play an import role in refining it and has led to use of the term Nano diagnostics [4, 5]. Nano biotechnologies will also improve detection of cancer biomarkers as basis for devising diagnostics as well as therapeutics.

### 1.5 Role of Nanotechnology in Detection of CancerBiomarkers

Detection of cancer biomarker is important for diagnostics as well as development of new therapies for cancer. Nanotechnology can provide new platforms for high throughput, highly sensitive, functional assays to complement genomic technologies for the identification of new biomarkers of cancer risk. Micro fluids in biochips are now being further miniaturized to nano arrays and handle nano fluids. Optical nanosensors can be used for in vivo analysis of proteins and biomarkers in individual living cells[6].

1.6 Synthesis Techniques:

Particles having size in the range of 1-100nm are nanoparticles. The following two main approaches for the synthesis of nanoparticles are:

1- Top down approach

2- Bottom up approach
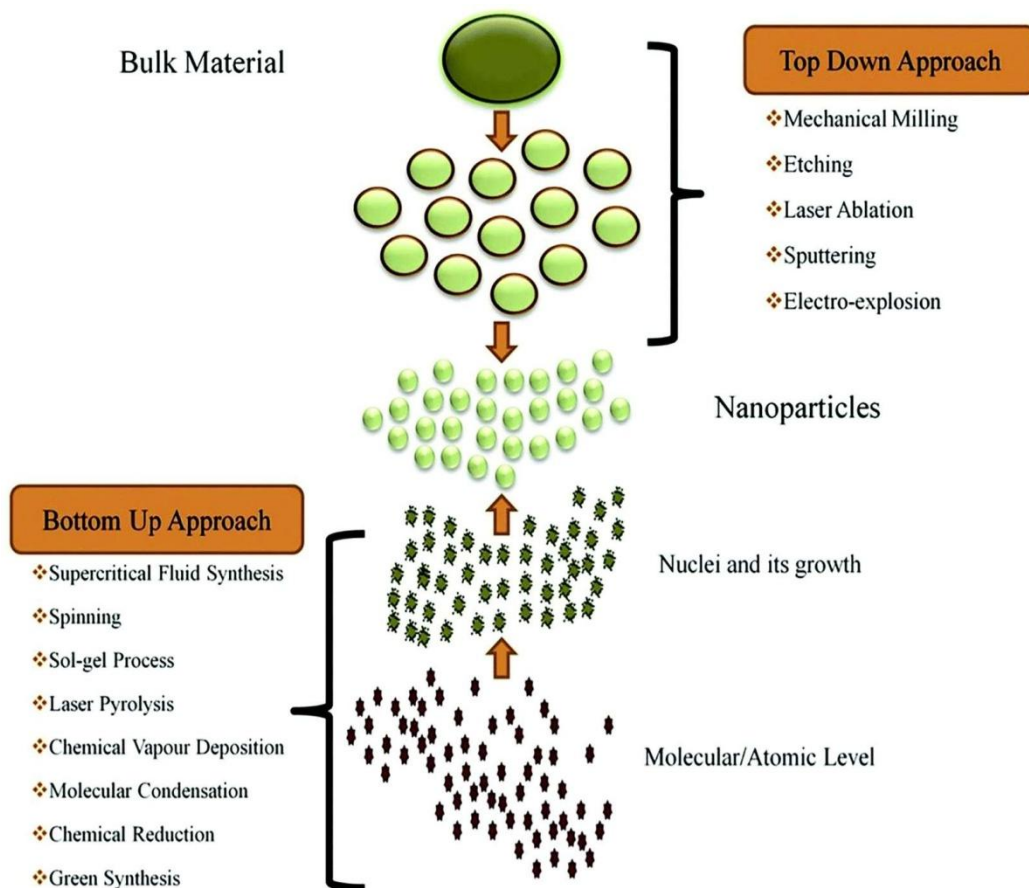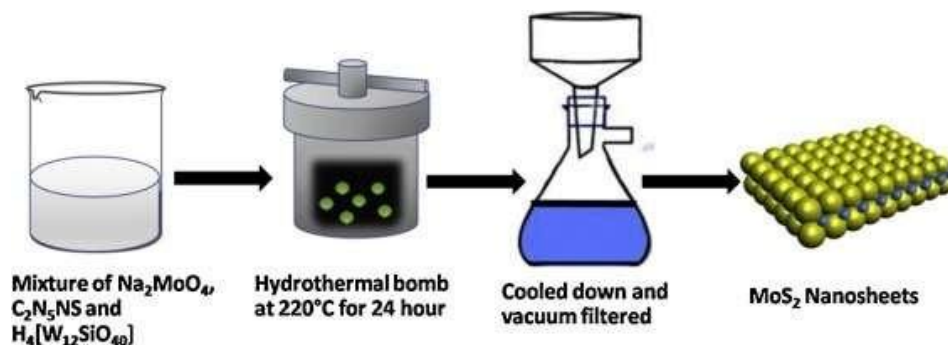
1.7 Top downapproach:

In top-down approaches, bulk materials are divided to produce nanostructured materials. Top- down methods include mechanical milling, laser ablation, etching, sputtering, and electro- explosion.

1.8 Bottom-up approach leads to development of the nanoparticle by assembling group the atoms and molecules at Nanoscale. This method is most suitable for the obtaining the uniform shape and size distribution of nanoparticles. This essentially involves chemical methods and control sized growth of the particle.

 There are many methods for the synthesis of the perovskites materials such as:

1.8.1 Chemical vapor deposition (CVD). Chemical vapor deposition methods have great significance in the generation of carbon-based nanomaterials. In CVD, a thin film is formed on the substrate surface *via* the chemical reaction of vapor-phase precursors. A precursor is considered suitable for CVD if it has adequate volatility, high chemical purity, good stability during evaporation, low cost, a non-hazardous nature, and a long shelf-life. Moreover, its decomposition should not result in residual impurities [7]. For instance, in the generation of carbon nanotubes *via* CVD, a substrate is placed in an oven and heated to high temperatures. Subsequently, a carbon-containing (such as hydrocarbons) gas is slowly introduced to thesystem as a precursor. At high temperatures, the decomposition of the gas releases carbon atoms, which recombine to form carbon nanotubes on the substrate [8]. However, the choice of catalyst plays a significant role in the morphology and type of nanomaterial obtained. In the CVD-based preparation of graphene, Ni and Co catalysts provide multilayer graphene, whereas a Cu catalyst provides monolayer graphene [9]. Overall, CVD is an excellent method for producing high-quality nanomaterials, [10] and it is well-known for the production of two-dimensional nanomaterials (Fig.)[11].

1.8.2 Solvothermal and Hydrothermal Methods. The hydrothermal process is one of the most well-known and extensively used methods used to produce nanostructured materials [12]. In the hydrothermal method, nanostructured materials are attained through a heterogeneous reaction carried out in an aqueous medium at high pressure and temperature around the critical point ina sealed vessel [13]. The solvothermal method is like the hydrothermal method. The only difference is that it is carried out in a non-aqueous medium. Hydrothermal and solvothermal methods are generally carried out in closed systems [14]. The microwave-assisted hydrothermal method has recently received significant attention for engineering nano materials, combining the merits of both hydrothermal and microwave methods [15] Hydrothermal and solvothermal methods are exciting and useful methods for producing various nano-geometries of materials, such as nano wires, nano rods, nano sheets, and nano spheres[16].

Mixture of Na_2MoO_4, C_2N_5NS and $H_4[W_{12}SiO_{40}]$ → Hydrothermal bomb at 220°C for 24 hour → Cooled down and vacuum filtered → MoS_2 Nanosheets

1.8.3 The Sol–Gel Method. The sol–gel method is a wet-chemical technique that is extensively used for the development of nanomaterials. This method is used for the development of various kinds of high-quality metal-oxide-based nanomaterials. This method is called a sol–gel method as during the synthesis of the metal-oxide nanoparticles, the liquid precursor is transformed toa sol, and the sol is ultimately converted into a network structure that is called agel

[17]. Conventional precursors for the generation of nanomaterials using the sol–gel method are metal alkoxides. The synthesis process of nanoparticles *via* the sol–gel method can be completed in several steps. In the first step, the hydrolysis of the metal oxide takes place in water or with the assistance of alcohol to form a sol. In the next step, condensation takes place, resulting in an increase in the solvent viscosity to form porous structures that are left to age. During the condensation or polycondensation process, hydroxo- (M–OH–M) or oxo- (M–O–M) bridges form, resulting in metal–hydroxo- or metal–oxo-polymer formation in solution [18]. During the aging process, polycondensation continues, with changes to the structure, properties, and porosity.


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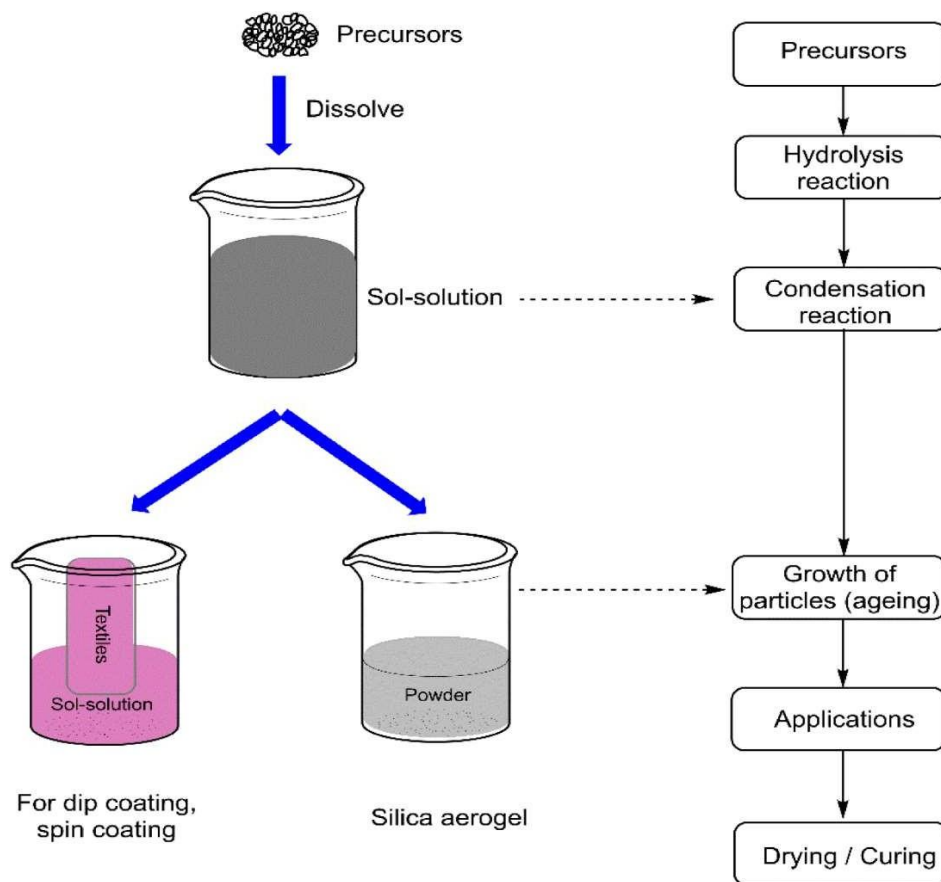
During aging, the porosity decreases, and the distance between the colloidal particles increases. After the aging process, drying takes place, in which water and organic solvents are removed from the gel. Lastly, calcination is performed to achieve nanoparticles [19]. Fig. shows film and powder formation using the sol–gel method [20]. The factors that affect the final product obtained *via* the sol–gel method are the precursor nature, hydrolysis rate, aging time, pH, and molar ratio between H_2O and the precursor [21]. The sol–gel method is economically friendly and has many other advantages, such as the produced material being homogeneous in nature, the processing temperature being low, and the method being a facile way to produce composites and complex nanostructures [22].

1.8.4 Co-precipitation Method:

Co-precipitation reactions involve the simultaneous occurrence of nucleation, growth, coarsening, and/or agglomeration processes. Coprecipitation reactions exhibit the following characteristics:

   (i) The products are generally insoluble species formed under conditions of high super saturation.
   (ii) Nucleation is a key step, and a large number of small particles will be formed.
   (iii)Secondary processes, such as Ostwald ripening and aggregation, dramatically affect the size, morphology, and properties of the products.
   (iv)The super saturation conditions necessary to induce precipitation are usually the result of a chemicalreaction.[23]

$$xAy+ (aq) + yBx- (aq) AxBy\square \ (s)$$
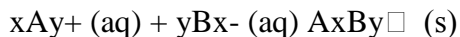
Typical Co-precipitation Synthetic Methods:

 Precipitation is the carrying down by a precipitate of soluble substances under certain conditions. Generally, when the concentration of substances reaches supersaturation, a nucleation suddenly appears in solution. The nucleation will be grown by the diffusion on to its surface which then becomes nanoparticles. During the growth, the nucleation needs to be slowed down in order to get uniform nanoparticles. Several methods can be listed as precipitation: coprecipitation, microemulsion/inverse microemulsion, polyol, etc. Coprecipitation is a convenient way to synthesize Fe_3O_4 nanoparticles.

The mixture of two chloride salts of $FeCl_2$ and $FeCl_3$ with 1:2 molar ratios of Fe^{2+} / Fe^{3+} was vigorously stirred and kept at 70°C before NH_4OH was added resulting in the black color precipitation. The Fe3O4 nanoparticles were collected after purifying through magnetic separation with ethanol and distilled water several times to decontaminate the residual chemicals. By modifying the pH and ion concentration in solution, the size of nanoparticles can be controlled [24].
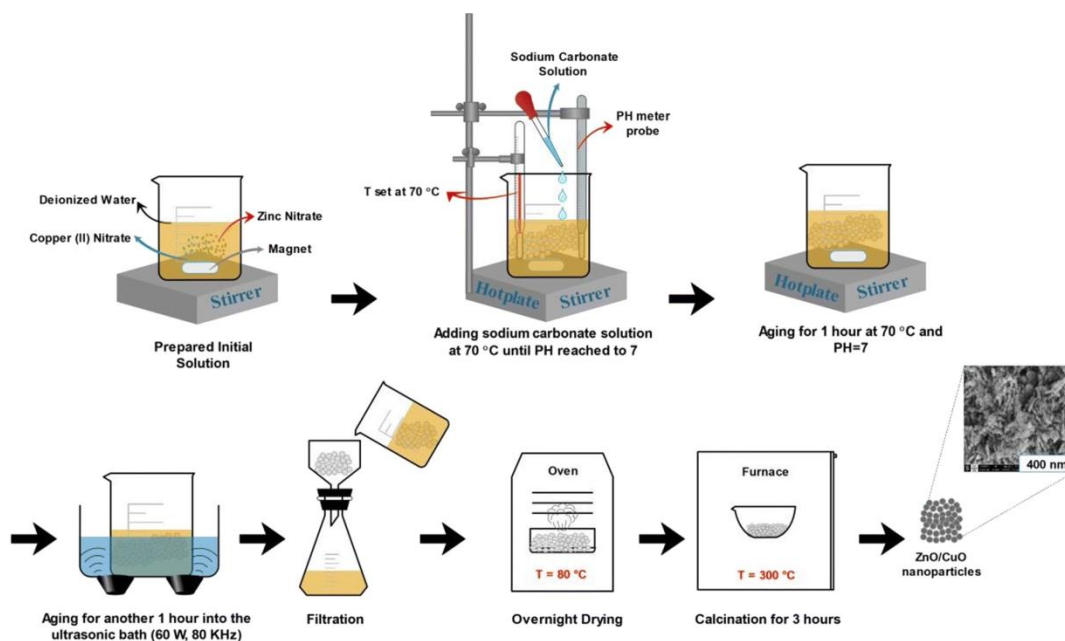
Chapter2

Experimental Work and Characterization Technique

2. ExperimentalWork

List of chemicals/reagents for synthesis

- Nickle chloride hexahydrate   ($NiCl_2.6H_2O$)
- Sodium hydroxide   ($NaOH$)
- Iron chloride hexahydrate   ($FeCl_36H_2O$)
- Zinc chloride   ($ZnCl_2$)
- Nickle sulphate   ($NiSO_46H_2O$)
- Deionizedwater

List of equipment and glassware for synthesis

- Electronic weight balance
- Beakers
- Magnetic stirrer
- Aluminum foil
- Gloves
- Safety Glasses
- Mortar pastel
- Dry Oven
- Sample bottles and boxes

## 2.1 Experimental Work:

2.1.1 Synthesis of NiO nanoparticles

NiCk.6H;0 and NaH as starting material and double-distilled water as dispersing solvent were used to prepare NO nanoparticles. At first, 5. 9412 gr NiCh.6H20 was dissolve in 250 CC double-distilled water as solvent to get a certain molar concentration at room temperature.

Then, obtained solution was magnetically stirred for 40 min at 50°C temperature.Afterwards; the 10 cc NaOH with certain molar was added drop wise to the solution until the pH becomes 8. So nanoparticles of NiO were fabricated by chemical reaction as follow:

$$NiCh.6H0 + H.0 + 2NaOH \rightarrow NiO + 8H.0 + 2NaCI$$

(1)In the final step, the obtained green gel washed with distilled water and ethanol to remove formed byproducts during the reaction process and dried at 60 °C temperature for 14 hr. Then, dried samples were calcined (annealed) at 500 "C temperatures for 2 h to obtain NiO nanoparticles, Because of this annealation the color of sample changes from green to black.
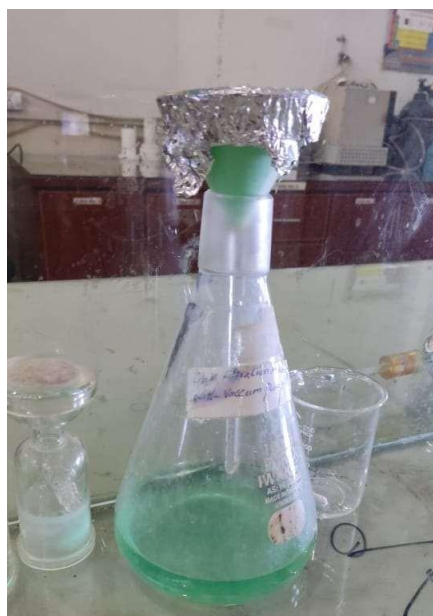
2.1.2 Synthesis of Zinc ferritesnanoparticles:

Iron (Ill) chloride hexahydrate (FeCk • 6H20), zinc (Il) chloride (ZnCh2), sodium hydroxide (NaH) and acetone were analytical grade.Zinc ferrite nanoparticles were synthesized by chemical copre-cipitation method. In this procedure, a mixed aqueous solutions prepared by dissolving required weights of iron and zinc chloride with the molar ratio of Fe to Zn as 2:1, in 100 mL distilled water. 50 mL aqueous solution of 1.5 M NaOH was used as the precipitating agent. Metal chloride and NaH solutions were added drop wise from two separate burets into a reaction vessel containing 100 mL of distilled water for obtaining uniform parti- de size distribution. The reaction vessel was heated up to the desired temperature under magnetic stirring. The resultant precipitations were collected and centrifuged at 6000 rpm and then washed with distilled water and acetone for several times and finally dried in air.


2.3 Characterization Techniques:

In this section, brief information on all of the characterization techniques will be provided. After synthesizing the anode materials, the next major step is their characterizations to analyze the properties of these synthesized anode materials (crystalline structure, morphology, presence of bonds and molecules, electrical and electrochemical properties).UV analysis is done to find

optical band gap of the materials. Raman spectroscopy is a major technique to investigate the structural and vibratory properties of nanostructures of materials. This is a suitable techniquethat detects rotational modes and vibrational modes by encountering inelastic dispersion in a system. FTIR spectroscopy technique used to find an infrared spectrum of emission or absorption. The use of X-ray diffraction (XRD) to characterize crystalline materials is a strong nondestructive method. Structures, phases, preferred crystal orientations (texture), and other structural characteristics such as average grain size, crystallinity, strain, and crystal defects are all provided. The DC conductivity of the 4-point probe was performed to analyses the conductivity of the prepared anodematerials.

- FTIRanalysis
- Ramananalysis
- UV-Visiblespectroscopy
- XRDanalysis

**2.3.1 Fourier transform infrared spectroscopy(FTIR)**

Transform of the Fourier Series Infrared Spectroscopy, commonly known as FTIR Analysis, is a technique for identifying organic, polymeric, and inorganic materials. Infrared light is used to scan test materials and examine chemical characteristics using the FTIR analysis method. The FTIR instrument passes infrared light in the range of 10,000 to 100 cm-1 through a material,with part of it being absorbed and some passing through. The sample molecules transform the absorbed radiation into rotational and/or vibrational energy. The resulting signal at the detector appears as a spectrum, ranging from 4000 cm-1 to 400 cm-1 , and represents the sample's molecular fingerprint. When assessing industrially created material, FTIR spectroscopy is a well-established technique for quality control, and it is frequently used as the initial stage in the material analysis process. A change in the absorption bands' distinctive pattern suggests a change in the material's composition or the presence of contamination. If visual inspection reveals a problem with the product, FTIR microanalysis is usually used to discover the source. This method is excellent for determining the chemical composition of tiny particles (10-50 microns) as well as larger areas on the surface. Unknown materials must be identified and characterised (e.g., films, solids, powders, orliquids).
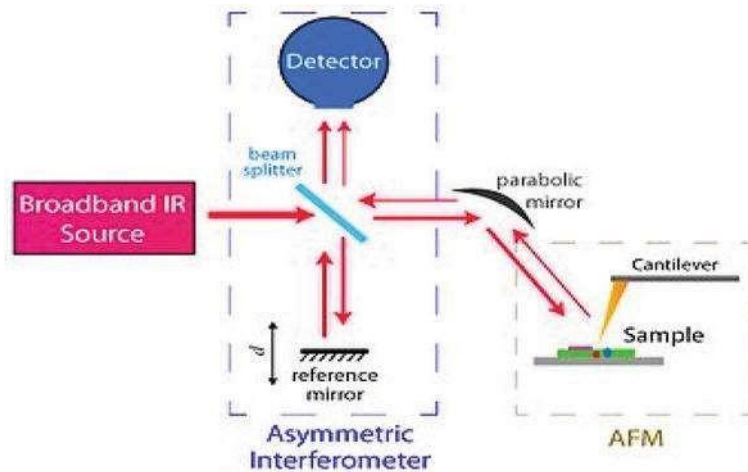
2.3.2 Scanning electron microscopy(SEM):

Scanning electron microscopy has been widely used to study fractured surfaces. The images give anideaofwhetherthefractureisductileorbrittle.Sometimes,itispossibletohaveamixedmode failuretoo.

Scanning Electron Microscopy In general, a scanning electron microscope (SEM) can make images of solid samples and can determine the elemental composition of said samples. The low end of the magnification range for an SEM is typically on the order of 20X to 50X. The maximum magnification is generally determined by the size of the electron beam and can be as high as one million (10^6). These magnification levels generally correspond to measuring features from the mm scale down to nm size. The best resolution of a high end SEM is on the order of 0.5 nm. The most common type of SEM data is the secondary electron image. The SE image is a map of secondary electron emission as a function of spatial position. SE images generally display the topography of thesample.

How Scanning Electron Microscopes Work
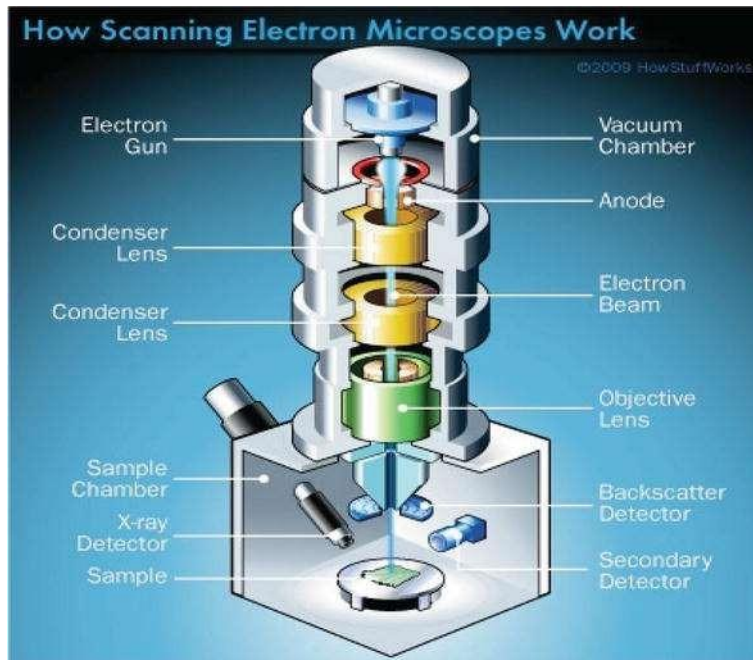
The number of secondary electrons emitted from a surface generally depends on the angle of incidence between the surface and the beam. Since the electron beam is perpendicular to the sample over the analysis area, the number of secondary electrons emitted from the sample is generally a function of the topography of the sample. Backscattered electrons (BSE) are the second most common imaging data collected with an SEM. This data is a map of backscattered electron emission as a function of spatial position. The number of BSEs that are emitted is a function of the atomic number of the sample. As the atomic number increases, so does the number of BSEs emitted. This means that images made with BSEs will generally show the composition of the sample. While it is not generally possible to observe a BSE image and know what materials are present, with some a priori knowledge of the sample's composition, it is usually possible to make a good guess about the material distribution from a high-quality BSE image. It is possible to combine BSE images with EDS to determine both the material composition and distribution in the sample. Since images are in the form of the electron emission from a sample vs. spatial position, it is not possible to determine the roughness of a sample from a plane view image of the sample as the electron emission from the sample may not depend on the height. Even if the electron emission does depend on the height, as in a secondary electron image, measuring a height from the electron emission is not generally possible. Roughness can only be determined from a cross-section of the sample. In addition to collecting imaging data to show the morphology of a sample, SEMs also generate and can collect X-rays that are

characteristic of the elements in the sample. This is usually done with an energy dispersive X-ray spectrometer or EDS system. EDS X-ray data can be used to determine the elements in the sample. If the X-ray data is mapped as a function of spatial position, an X-ray map showing the distribution of the elements in the sample can be created. The detection limit for EDS is generally on the order of 0.5% by weight within the X-ray generation volume [26].

2.3.3 TEM

Figure 3 shown above gives in brief the differences between SEM and TEM. Here, the specimens have to be prepared to thin slices of less than 100 nm thickness. Again, similar to SEM, specimen preparation is of utmost importance. Vacuum has to be maintained in the TEM, and any flaw in the maintenance of vacuum will reflect in the performance of the TEM. Usually, dislocation density and second phase precipitation can be clearly seen in TEM images.
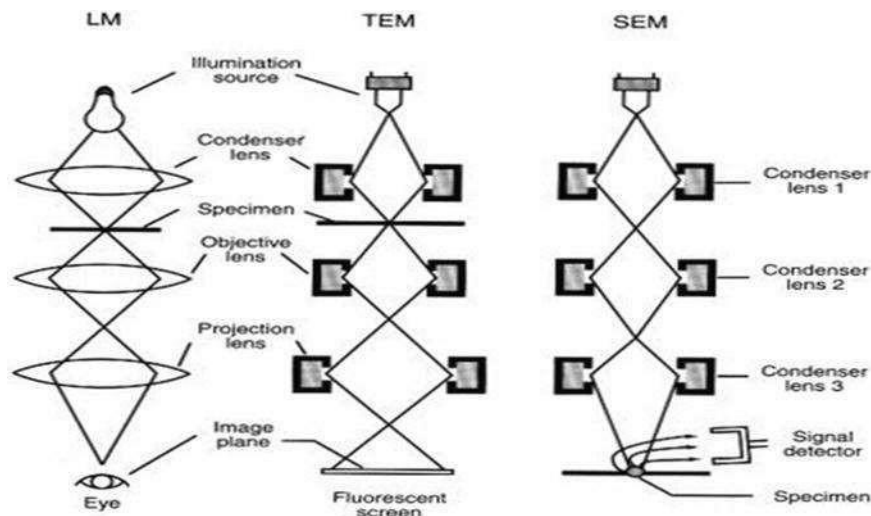
The electron gun is a source of electrons. The electrons are focused with the help of condenser lens and objective lens. There is a chamber to hold the workpiece. Care should be taken to prepare the workpiece very carefully and specialists are required for SEM sample preparation. The chamber consists of a backscatter detector and a secondary detector. So the SEM can be operated under two modes.
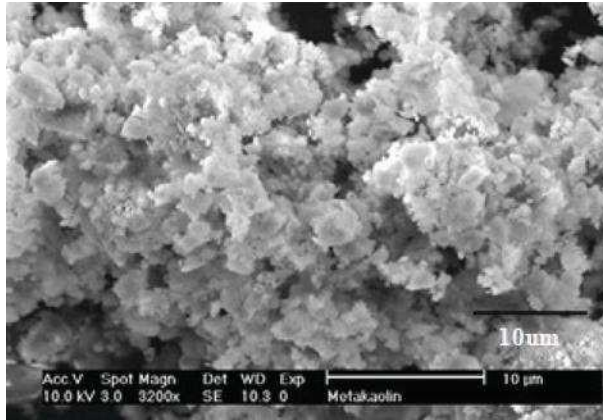
Figure 4 depicts uniform distribution of metakaolin and possibly a ductile fracture. The SEM image above shows a wide variety of shapes of fly ash, tending towards a spherical shape. There seems to be agglomeration and there also seem to be some cracks. Cracks often lead to brittleness. This SEM micrograph of a fly ash geopolymer seems to indicate mixed mode of failure [26].

2.3.4 X-Ray Diffraction (XRD):

X-ray diffraction (XRD) is one of the most important characterization tools used in solid state chemistry and materials science, which could provide most definitive structural information (e.g. interatomic distances, bond angles, crystallinity, and etc…). The extensive use of X-rays for the analysis of atomic structural arrangements is based on the fact that the wavelength of the X-rayis in the 1 x 10-10m range, which is the same order magnitude of the atomic spacing in crystalline solids. When x-rays interact with crystal lattice, a simple model called Bragg's law can be used to understand the required conditions for diffraction. The Bragg's law can be expressedas

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

where λ is the wavelength of X-ray, d is the spacing between layers of atom, θ is angle between incident X-ray beam and scattering plane, and n is integer.
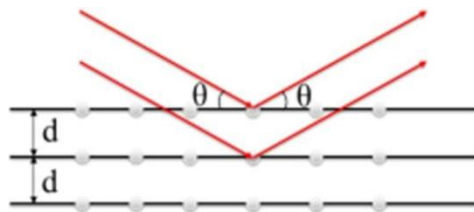
Thus, the diffracted waves will consist of sharp interference maxima (peaks) with the same symmetry as in the distribution of atoms if the atoms are arranged in a periodic fashion in crystals and the structural information of the crystals can be revealed based on the diffraction peaks. AIF X-ray Diffraction Laboratory provides access to 3 state-of-the-art X-ray diffractometers for characterizing microstructural and crystallographic properties of powders, thin films, fibers and other solid materials[27].

2.3.5 Raman Spectroscopy:

Raman Spectroscopy is a non-destructive chemical analysis technique which provides detailed information about chemical structure, phase and polymorphy, crystallinity and molecular interactions. It is based upon the interaction of light with the chemical bonds within a material.

Raman is a light scattering technique, whereby a molecule scatters incident light from a high intensity laser light source. Most of the scattered light is at the same wavelength (or color) as the laser source and does not provide useful information – this is called Rayleigh Scatter. However a small amount of light (typically 0.0000001%) is scattered at different wavelengths (or colors), which depend on the chemical structure of the analyte – this is called Raman Scatter.
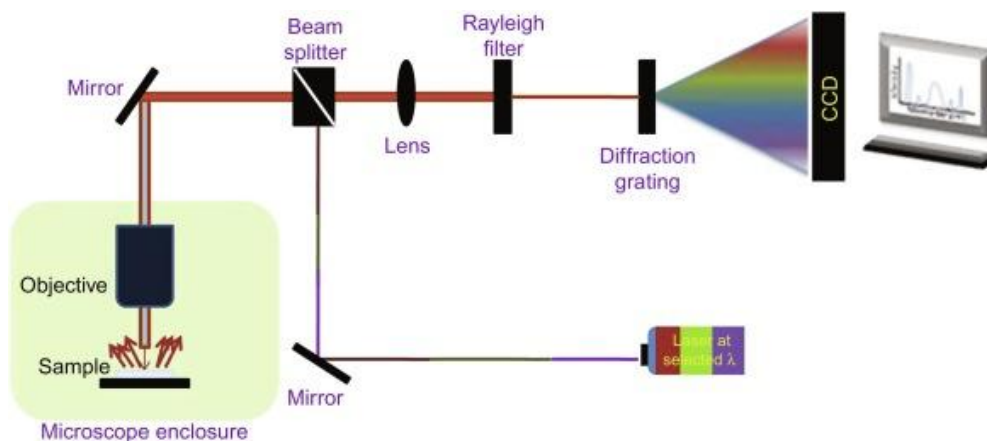
Raman spectroscopy probes the chemical structure of a material and provides information about:

- Chemical structure and identity
- Phase and polymorphism
- Intrinsic stress/strain
- Contamination and impurity

Typically, a Raman spectrum is a distinct chemical fingerprint for a particular molecule or material, and can be used to very quickly identify the material, or distinguish it from others. Raman spectral libraries are often used for identification of a material based on its Raman spectrum – libraries containing thousands of spectra are rapidly searched to find a match with the spectrum of the analyte [28].

2.3.6 Photoluminescence Spectroscopy:

Photoluminescence spectroscopy, often referred to as PL, is when light energy, or photons, stimulate the emission of a photon from any matter. It is a non-contact, nondestructive method of probing materials. In essence, light is directed onto a sample, where it is absorbed and where a process called photo-excitation can occur. The photo-excitation causes the material to jump to a higher electronic state, and will then release energy, (photons) as it relaxes and returns to back to a lower energy level. The emission of light or luminescence through this process is photoluminescence, PL.

Our PL optimized series of spectrophotometers are used in Fluorescence Spectrometers, Raman Spectrometers and our Custom Optical Solution Systems. These products are also making a major contribution to the development of nanomaterials, semiconductors, photovoltaics / solar cells.
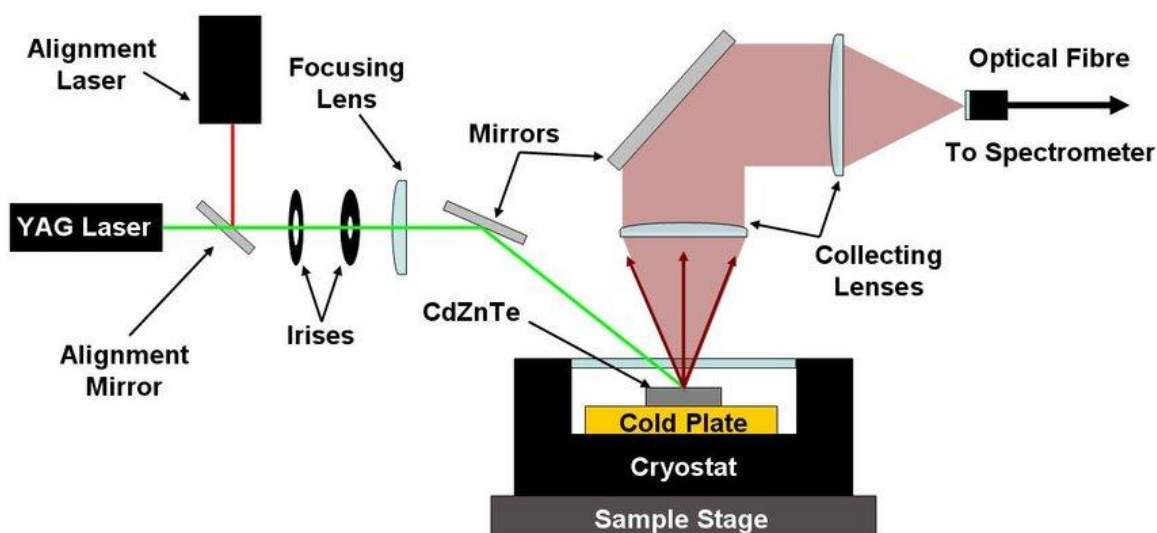
By combining Raman analysis with PL detection, it is possible to characterize both the vibrational and

electronic properties of materials on a single bench top platform. Combined Raman-PL systems allow confocal mapping capabilities with sub-micron spatial resolution. A wide range of excitation wavelengths is possible, from the UV to NIR, allowing control of the penetration depth into the material, and thus, control of the volume sampled.

Photoluminescence used in Fluorescence spectroscopy can provide two results: Fluorescence and Phosphorescence. The Photoluminescence quantum yield or PLQY of a molecule or material is defined as the number of photons emitted, as a fraction of the number of photons absorbed is one of the common techniques for Fluorescence Spectroscopy [29].

Typical applications include:

- Band Gap Determination
- Impurity Levels and Defect Detection
- Recombination Mechanisms
- Material Quality
- Molecular structure and crystallinity

Chapter 3

Result and conclusions

3.1 FTIR

FTIR is abbreviation of "Fourier transform infrared". In FTIR technique controlled (IR) radiations are passed through the sample, during passing Infrared radiations, some passes through the sample and some are absorbed the sample. Radiations which are transmitted on the sampleare recorded. As each molecule produce specific spectra, so the spectra are used to differentiate among themolecules.

The FTIR results of NiO, $ZnFe_2O_4$ and composite of NiO &$ZnFe_2O_4$ are represented in Fig. # 1.1

3.1.1 FTIR results ofNiO:

One spectrum of NiO demonstrates four unique peaks at 1638.16689 cm^{-1}, 1115.59508 cm^{-1}, 1042.40766 cm^{-1} and 975.376197 cm^{-1}. So, these peaks represent the presence of CO and CHO functional groups. This assures the presence of NiO nanoparticles. Second spectrum represents the presence of OH functional group.

<table>
<tr><th colspan="5">Table of Peak Values (FTIR results of NiO)</th></tr>
<tr><th>Peaks #</th><th>1st</th><th>2nd</th><th>3rd</th><th>4th</th></tr>
<tr><td>x-axis</td><td>975.376197</td><td>1042.40766</td><td>1115.59508</td><td>1638.16689</td></tr>
<tr><td>y-axis</td><td>0.0574816072</td><td>0.0913341822</td><td>0.071745897</td><td>0.0646137521</td></tr>
</table>

3.1.2 FTIR results ofZnFe$_2$O$_4$:

First spectrum of $ZnFe_2O_4$ demonstrates four significant peaks at 975.376197 cm^{-1}, 1054.71956 cm^{-1}, 1109.43912 cm^{-1} and 1640.90287 cm^{-1}. So, these peaks represent the presence of CO and CHO functional groups. This assures the presence of $ZnFe_2O_4$ nanoparticles. Second spectrum represents the presence of OH functional group.

<table>
<tr><th colspan="5">Table of Peak Values (FTIR results of ZnFe$_2$O$_4$)</th></tr>
<tr><th>Peaks #</th><th>1st</th><th>2nd</th><th>3rd</th><th>4th</th></tr>
<tr><td>x-axis</td><td>975.376197</td><td>1054.71956</td><td>1109.43912</td><td>1640.90287</td></tr>
<tr><td>y-axis</td><td>0.244022354</td><td>0.322576401</td><td>0.319864177</td><td>0.204443973</td></tr>
</table>

3.1.3 FTIR results of composite of NiO &ZnFe$_2$O$_4$:

FirstspectrumofcompositeofNiO&$ZnFe_2O_4$demonstratesfoursignificantpeaksat963.064295 cm^{-1}, 1060.87551 cm^{-1}, 1103.28317 cm^{-1} and 1638.16689 cm^{-1}. So, these peaks represent the presence of CO and CHO functional groups. This assures the presence of composite of NiO &$ZnFe_2O_4$ nanoparticles. Second spectrum represents the presence of OH functionalgroup.

Table of Peak Values (FTIR results of composite of NiO &ZnFe$_2$O$_4$)				
Peaks #	1st	2nd	3rd	4th
x-axis	963.064295	1060.87551	1103.28317	1638.16689
y-axis	0.173504527	0.244022354	0.268131013	0.048541313

The reason behind the presence of OH functional group in second spectrums of FTIR results of NiO, ZnFe$_2$O$_4$ and composite of NiO &ZnFe$_2$O$_4$ is that at the time of reaction and while preparing sample NaOH was used as a precursor to carried out the reaction. The appearance of such peaks suggests a humidity percentage in the sample. This may be due to the increased contact of the sample with air or due to initial preparation conditions.
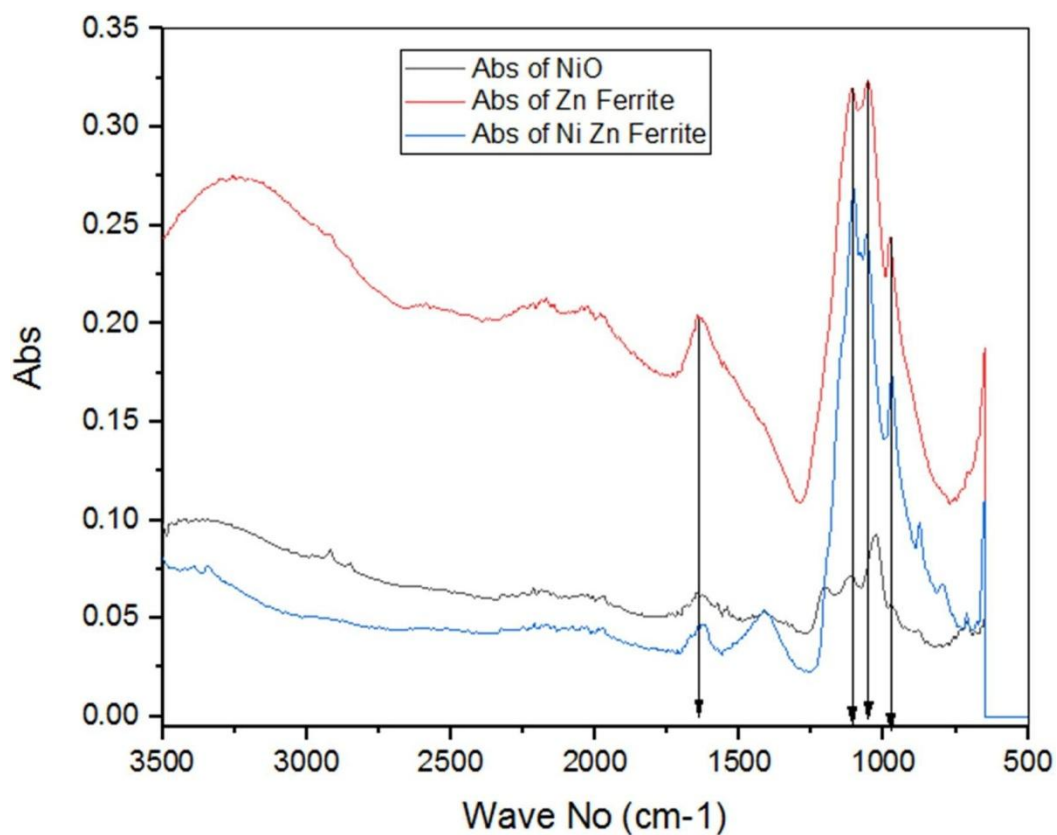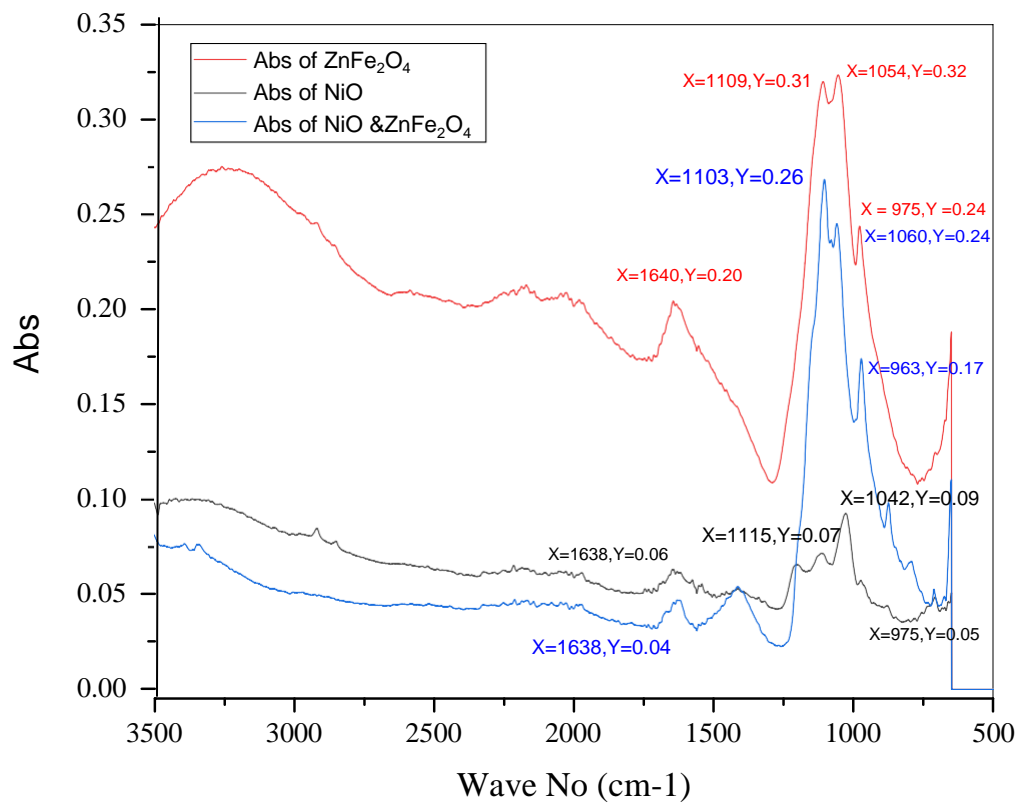

Figure 3. FTIR Spectra of NiO, ZnFe2O4 and composite of NiO & ZnFe2o4

3.2 Raman

Raman of $ZnFe_2O_4$, Composite of NiO and $ZnFe_2O_4$ and NiO

Raman tells us about vibration moods as explained above. In this part investigation of Raman analysis of three samples NiO, Composite of NiO, $ZnFe_2O_4$ and has been examined. The obtained spectra have been prescribed in figure givenbelow. [30]

During examination of NiO, Composite of NiO, $ZnFe_2O_4$different peaks comes which are following in the table given below. The peak at 1059.84952 cm^{-1}shows regular vibration of NiO.

Table of Peak Values (Raman results of NiO)				
Peaks #	1st	2nd	3rd	4th
x-axis (cm^{-1})	121.067031	535.738714	680.232558	1059.84952
y-axis (au)	1895.61404	1569.29825	1545.61404	2761.40351

During examination of composite of NiO and $ZnFe_2O_4$different peaks comes which are following in the table given below. The peak at 1006.83995cm^{-1}shows regular vibration of composite.

Table of Peak Values (Raman results of Composite of NiO & $ZnFe_2O_4$)				
Peaks #	1st	2nd	3rd	4th
x-axis (cm^{-1})	125.341997	224.521204	611.833105	1006.83995
y-axis (au)	1101.75439	1202.63158	1093.85965	1218.42105

During examination of $ZnFe_2O_4$different peaks comes which are following in the table given below. The peak at 983.75513 cm^{-1}shows regular vibration of ferriate.

Table of Peak Values (Raman results of $ZnFe_2O_4$)				
Peaks #	1st	2nd	3rd	4th
x-axis (cm^{-1})	277.53078	398.939808	611.833105	983.75513
y-axis (au)	430.701754	213.157895	220.175439	735.087719
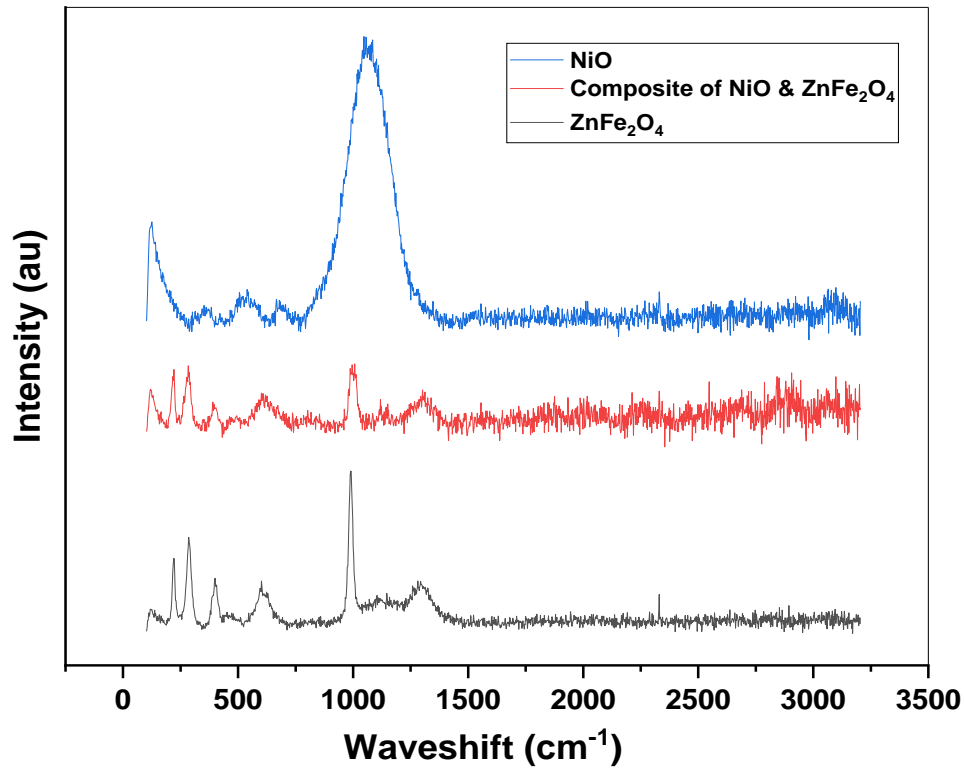
3.3 Photo Luminescence (PL)

PL of $ZnFe_2O_4$, Composite of NiO and $ZnFe_2O_4$ and NiO

In the below diagram the optical properties of prepared $ZnFe_2O_4$, Composite of NiO and $ZnFe_2O_4$ and NiOstudied with thehelp of PL spectroscopy.All prepared sample shows the strong peak at715cmwhich means that it almost shows the strong emission of light at this wavelength. Which means theywill transmit the blue color and absorb all remaining colors. [31]
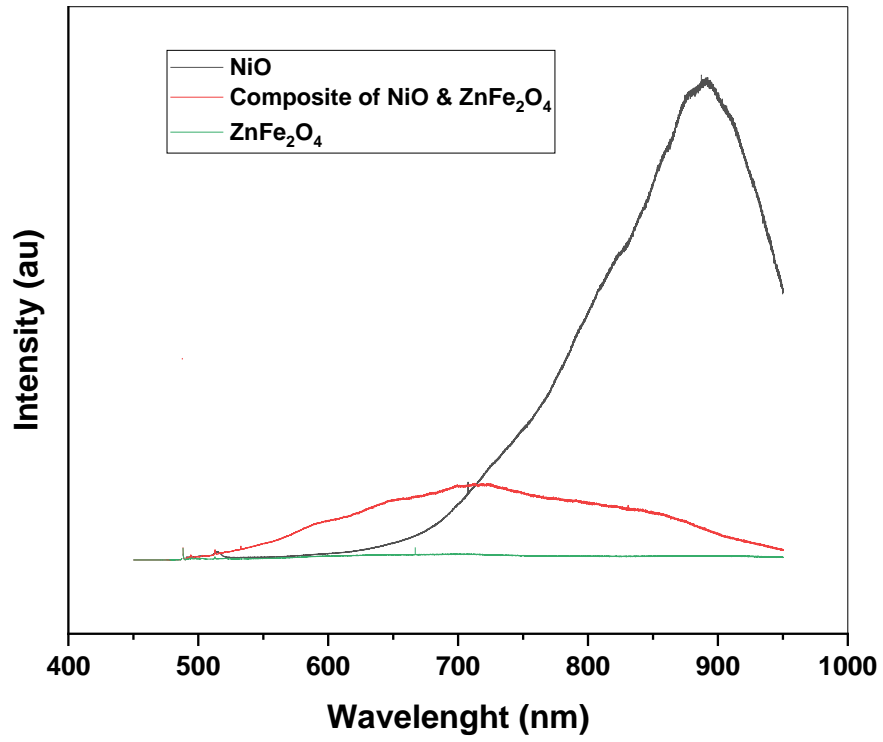
Chapter 4

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