# Synthesis And Characterization Of Nano Particles Using Co-Precipitation Method

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**Abstract—**

*Nanotechnology is the manipulation of matter on an atomic and molecular scale. The earliest, widespread description of nanotechnology referred to the particular technological goal of precisely manipulating atoms and molecules for fabrication of macro scale products, also now referred to as molecular nanotechnology. Synthesis of nanomaterials by a simple, low cost and in high yield has been a great challenge since the very early development of nano-science. Various bottom and top down approaches have been developed so far, for the commercial production of nanomaterials. Among all top down approaches, high energy ball milling has been widely exploited for the synthesis of various nanomaterials, nano-grains, nano-alloy, and nano-composites materials. The recent developments and trends in combustion science towards the synthesis of nanomaterials are discussed. Different modifications made to conventional combustion approaches for preparation of nanomaterials are critically analysed. Special attention is paid to various applications of combustion synthesizednano-sized products.*

*In this study, Nano crystalline Nickel Ferrite (NiFe2O4), Copper Ferrite (CuFe2O4)&manganese (MnFe2O4)particles were successfully synthesized using chemical co-precipitation method.*

*Various characterization methods are used to investigate structural properties of above three particles. The main characterizations XRD, UV-Visible Spectroscopy, FTIR, and SEM analysis was used for structural investigations. The corresponding characterization frequency data for the respective sites are also presented in this project report. Nanocrystalline Copper Ferrite (CuFe2O4), Nickel Ferrite (NiFe2O4),and manganese (MnFe2O4)particles were successfully synthesized at room temperature using chemical co-precipitation method. These nanoparticles are synthesized without annealing.*

*In this study, the higher frequency band and lower frequency band are assigned to the tetrahedral and octahedral complex. X-ray diffraction pattern confirms the formation of single-phase cubic structure. Their corresponding reflection planes are also determined. The lattice constant calculated from XRD peaks is 8.398 Å. Crystallite size calculated from XRD peak broadening confirms an average particle size of 20 nm. Particle size measured using SEM show good agreement with the calculated Value.*

***Keywords – XRD, SEM, FTIR, UV-Visible Spectroscopy.***

## I. Introduction

Most definitions revolve around the study and control of phenomena and materials at length scales below 100 nm and quite often they make a comparison with a human hair, which is about 80,000 nm wide. Some definitions include a reference to molecular systems and devices and nanotechnology 'purists' argue that any definition of nanotechnology needs to include a reference to "functional systems".

Nanotechnology is being heralded as the next enabling technology that will redesign the future of several technologies, products, and markets. Nations are focusing on this emerging technology in particular and serious research as well industry efforts is being made. Recent developments, current treads and industry progress are very interesting. Nanotechnology has become one of the important sectors which are drawing intense interest and it will replace most of the existing technology in use today. The term nanotechnology itself has been variously defined. By one definition, it is the ability to do many things; measure, see, predict and make on the scale of atoms and molecules.

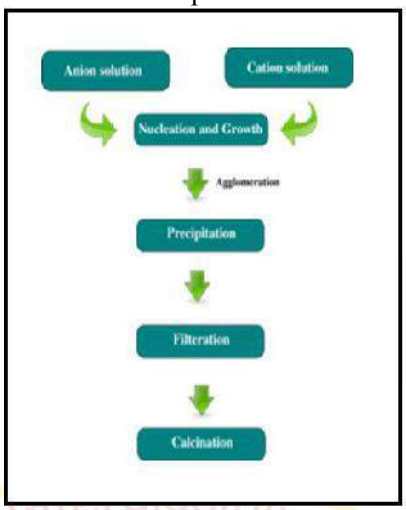
'Nanotechnology has also been defined to be dealing with materials in the range of 0.1 to 100nm'.

It is also referred to as the term for the construction and utilization of functional structures with at least one characteristic dimension measured in square names. The term nanoparticle is generally used to indicate particles with dimensions less than 100 names (nanometer). A nanometer is one billionth of a meter. For comparison, a human hair is about 50000nm in diameter. The term nanotechnology was introduced in physics by author Eric Drexler through his 1986 book engines of creation. Since then it was never looked back and has assumed such importance that today all the research institutes are sanctioning larger budgets for research work in nanotechnology.

## II.Methodology

In chemistry, co precipitation (CPT) or co-precipitation is the carrying down by precipitate of substances normally soluble under the conditions employed. Analogously, in medicine, co precipitation is specifically the precipitation of an unbound "antigen along with an antigen-antibody complex". Coprecipitation is an important issue in chemical analysis, where it is often undesirable, but in some cases it can be exploited. In gravimetric analysis, which consists on precipitating the analyte and measuring its mass to

determine its concentration or purity, co precipitation is a problem because undesired impurities often co-precipitate with the analyte, resulting in excess mass. This problem can often be mitigated by "digestion".

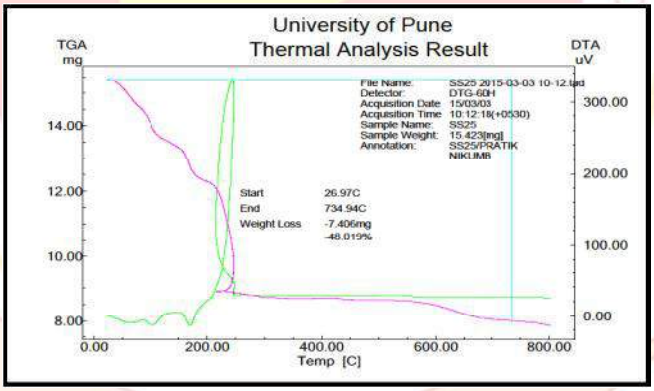



Typical co-precipitation method for micro and nano particle synthesis On the other hand, in the analysis of trace elements, as is often the case in radiochemistry, co precipitation is often the only way of separating an element. Since the trace element is too dilute (sometimes less than a part per trillion) to precipitate by conventional means, it is typically co precipitated with a carrier, a substance that has a similar crystalline structure that can incorporate the desired element.

**III. Results and discussion:**

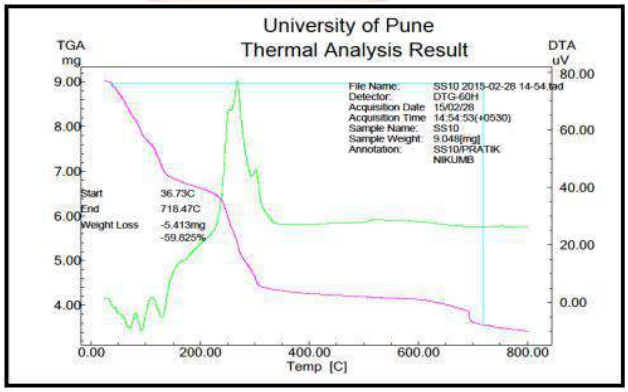
Thermo-Gravimetric Analysis:

1) TGA OF NiFe2O4




Dehydration take place at 35$_0$C to 195$_0$C then from 195$_0$C to 640$_0$C may be decomposition take place and form oxide.

TGA for NiFe2O4:




Decompositiontake place at 350C to 2100Cand from 1200C to 6300C may be carbonate formation and from this oxide is form.

| Precursor | TGA | | | DTApeak temp. (oC) | Predicted intermediates and final products |
|---|---|---|---|---|---|
| | % mass loss | | Temp. range (oC) | | |
| | Observed | Calculated | | | |
| NiFe2 (C2O4)3 .7H2O | 10.14<br>53.18 | 10.44<br>53.64 | 35-195<br>195-640 | 132<br>236 | NiFe2(C2O4)3<br>NiFe2O4 |

2) Fourier transforms infrared radiation (FTIR)

NiFe2O4

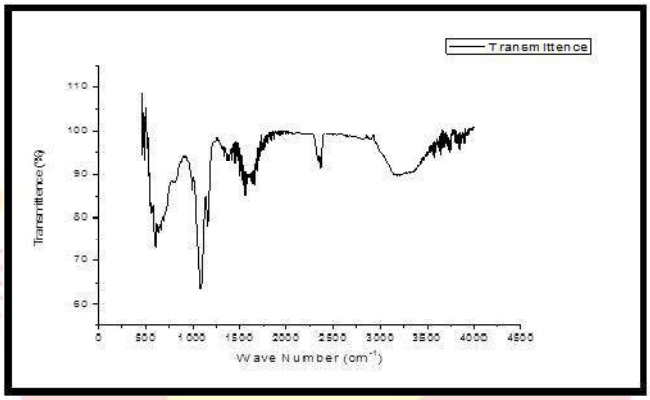



Figure: FTIR spectrum of NiFe2O4 in the range 4000-500 cm-1

The above figure shows FTIR spectra of $NiFe_2O_4$ synthesized by Co-precipitated method at 50ºC. The FTIR indicates formation of $NiFe_2O_4$ along with presence of trace quantity of organic matter which may be due Un-decomposed organic matter

3) $CuCl_2O_4$

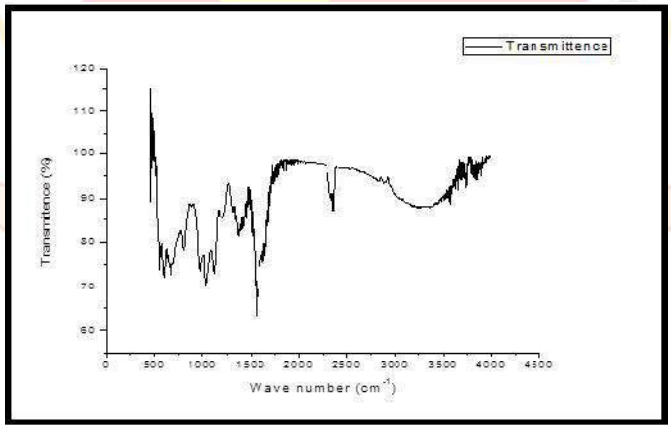



Figure: FTIR spectrum of CuCl2O4 in the range 500-4000 cm-1

The above figure shows FTIR spectra of CuCl2O4 synthesized by Co-precipitated method at 50ºC. The FTIR indicates formation of CuCl2O4 along with presence of trace quantity of organic matter which may be due Un-decomposed organic matter.

**MnFe2O**

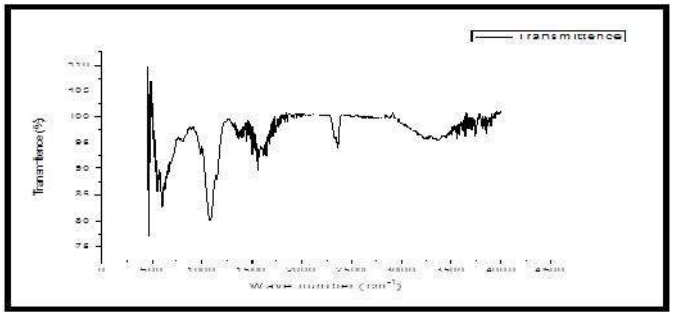



Figure: FTIR spectrum of MnFe2O4 in the range 500-4500cm-1

The above figure shows FTIR spectra of MnFe2O4 synthesized by Co-precipitated method at 50ºC. The FTIR indicates formation of MnFe2O4 along with presence of trace quantity of organic matter which may be due Un-decomposed organic matter.

**Chemical Analysis Of The Oxides:**

**For NiFe2O4**

Weight 0.20g of oxide sample and dissolve it in 10 ml of HNO3, heat for dissolution. Dilute to 50 ml then add NH4Cl +NH4OH till complete precipitation of Fe. Filter ppt. on what Mann no. 41 dry it in oven at 160˚.ignite in previously weight crucible. Filtrate contains Ni which precipitates by adding DMG. Filter ppt. on previously weighed Gooch crucible find out weight of Ni in NiFe2O4.

**X-ray diffraction (XRD)**

NiFe2O4

The NiFe2O4 nanoparticles have a degree of crystallites. The XRD pattern of Nickel ferrite was prepared by co-precipitation method. The practical size was prepared by calculated by using Scherer formula.

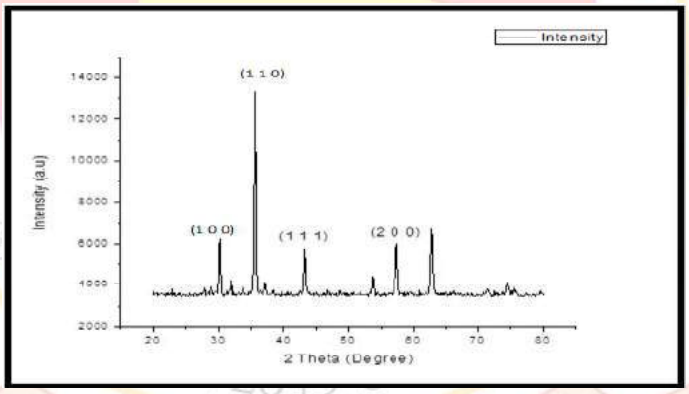



Figure: XRD pattern of NiFe2O4

| Sample | Plane | Lattice Constant (a) | $d$= λ2 Sin θ (A0) (Observed) | $d$= λ2 Sin θ (A0) (Standard) | Crystal Size (D) nm |
|---|---|---|---|---|---|
| NiFe2O4 | (1 0 0 ) | 2.9522 | 2.9522 | 2.8671 | 39.90 |
| | (1 1 0 ) | 1.47583 | 2.08715 | 2.08951 | |
| | (1 1 1 ) | 0.9829 | 1.7025 | 1.7013 | |
| | (2 0 0 ) | 0.7370 | 1.4740 | 1.4733 | |

CuCl2O4

The CuCl2O4 nanoparticles have a degree of crystallites. The XRD pattern of copper ferrite was prepared by co-precipitation method. The practical size was prepared by calculated by using Scherer formula

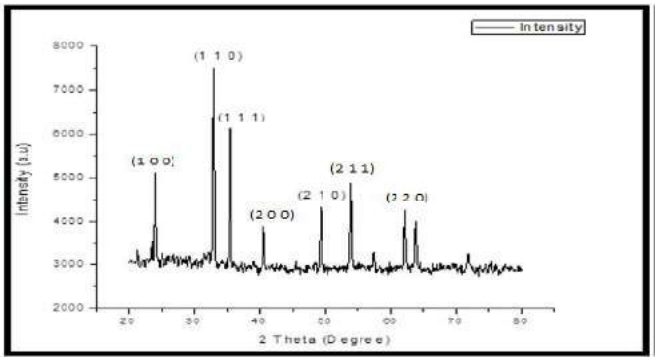


Figure: XRD pattern of CuCl2O4

| Plane | Sample | Lattice Constant (a) | $\boldsymbol{\theta 0}$ | $\boldsymbol{d = \lambda 2 \text{ Sin } \theta}$ (A0) (Observed) | Crystal Size (D) nm |
|---|---|---|---|---|---|
| 1 0 0 | CuCl2O4 | 3.68194 | 12.0713 | 3.68194 | 10.79 |
| 1 1 0 | | 1.35908 | 16.456 | 2.71816 | |
| 1 1 1 | | 0.84133 | 17.7621 | 2.5240 | |
| 2 0 0 | | 0.5521 | 20.4053 | 2.2084 | |
| 2 1 0 | | 0.36944 | 24.6346 | 1.8472 | |
| 2 1 1 | | 0.2828 | 26.9824 | 1.6970 | |
| 2 2 0 | | 0.1865 | 31.0714 | 1.4919 | |

VI) Ultraviolet-Visible (UV-Visible) spectroscopy

NiFe2O4

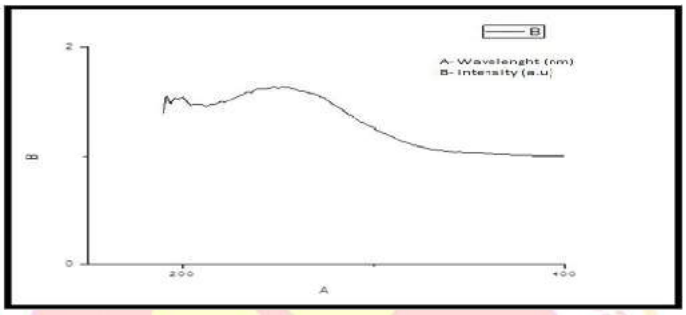



Figure: UV-Visible pattern of NiFe2O4

From the above graph we have calculated the band gap of NiFe2O4 by using the cut-off wavelength 661.613272 nm. The band gap of NiFe2O4is 1.8742 eV which was calculated by using the following formula,

Band Gap of MnFe2O4: $\boldsymbol{E}$=$\mathbf{1240}/\lambda$=$\mathbf{1240/\ 661.613272}$=$\mathbf{1.8742}$ $\boldsymbol{eV}$

**2) CuFe2O4**

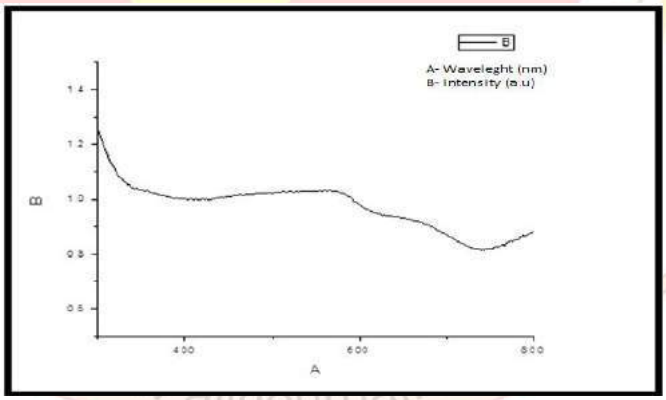



Figure: UV-Visible pattern of $CuFe_2O_4$

From the above graph we have calculated the band gap of CuFe2O4 by using the cut-off wavelength 610.755149 nm. The band gap of CuFe2O4is 2.03072 eV which was calculated by using the following formula,

Band Gap of CuFe2O4:$\boldsymbol{E}$=$\mathbf{1240}/\lambda$=$\mathbf{1240/610.755149}$=$\mathbf{2.03027}$ $\boldsymbol{eV}$

**MnFe2O4**

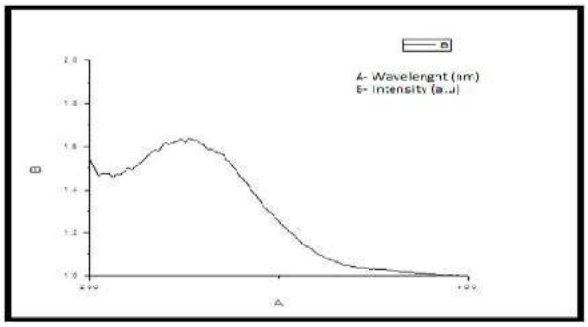



Figure: UV-Visible pattern of MnFe2O4

From the above graph we have calculated the band gap of MnFe2O4 by using the cut-off wavelength 289.130435 nm. The band gap of MnFe2O4is 4.2887 eV which was calculated by using the following formula Band Gap of

MnFe2O4: $E = 1240/\lambda = 1240/289.130435 = 4.2887\ eV$

As annealing temperature increases grain size increases. Also sample become sharper, indicating has better crystal degree.

The morphology of the crystals, particles sizes are studied by a scanning electron microscopy.

Thermo gravimetric analysis showed two steps of decomposition to formed respective ferrites.

Using Ultraviolet-Visible (UV-Visible) spectroscopy, band gaps for each ferrite are observed. (For Ni=1.8742 eV , Cu=2.030227 eV &Mn=4.2887 eV)

**Conclusion :**

We have used co-precipitate method of the three compounds (Nickel Iron Oxide (NiFe2O4), Copper Iron Oxide (CuFe2O4) & Manganese (MnFe2O4)) are characterized by using X-ray diffraction (XRD), Ultraviolet-Visible (UV-Visible) spectroscopy & Fourier transforms infrared radiation (FTIR) techniques. We have successfully synthesis of nanomaterials by using Co precipitate method. The following conclusions were drawn from the present investigation: From X-ray diffraction it is conclude that obtained film contain some impurities of Ni & Cu. The calcinated product of nickel ferrite, copper ferrite & manganese ferrite showed with cubic structure, which is confirmed byX-ray power diffraction.

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