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Eco-environmental synthesis and characterization of Co and Mg nickel ferrite nanophase powders

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Abstract – Nano crystalline spinel ferrite powders synthesized by co-precipitation method by using ferric nitrite and ethylene glycol as capping and dispersant agent. FTIR spectrum confirms presence of $CoNiFe_2O_4$ and $MgNiFe_2O_4$. The powder x ray diffraction results shows that the synthesized nano crystalline have only spinel structure without presence of impurities. The nanoparticles structure and morphology confirmed by Scanning Electron Microscopy. EDX micro analysis for verification of percentage of elements present in synthesized nanoparticles. UV-Visible spectroscopy used to identify the band gap of nanomaterials.

Key Words - $CoNiFe_2O_4$ and $MgNiFe_2O_4$ nanoparticles, FTIR, X ray diffraction, SEM, EDX, UV-Visible spectroscopy.

Introduction –

Number of various ferrite materials used in advanced electronic and magnetic applications. An even metal ferrites nanoparticle indicates substantial antibacterial activities. Spinel ferrites widely used in sensors, recording devices, magnetic cards, solar cells, magnetic drug delivery, catalysis, biomedical and biotechnology, microwave devices and super capacitors [9,11].These ferrites have found applications in water purification and waste water treatment also. Various methods such as co-precipitation, sol gel method, hydrothermal method, wet chemical co-precipitation technique, combustion method have been developed to prepare nano crystalline nickel ferrite. This paper focuses on preparation of $CoNiFe_2O_4$ and $MgNiFe_2O_4$ nanoparticles using co-precipitation method and their characterization by using FTIR, X ray diffraction, SEM, EDX, UV-Visible spectroscopy [1, 3, 4, 10, 13].

Experimental Technique –

Chemicals used –

AR grade Ferric nitrate $Fe(NO_3)_3,2H_2O$, Nickel nitrate $Ni(NO_3)_2,2H_2O$, cobalt nitrate $Co(NO_3)_2,2H_2O$, sodium hydroxide NaOH, ethylene glycol, acetone, ethanol and distilled water.

Synthesis of $CoNiFe_2O_4$ nanomaterials -

0.4M Ferric nitrate ($Fe(NO_3)_3,2H_2O$) solution, 0.2M Nickel nitrate ($Ni(NO_3)_2,2H_2O$) and 0.1M cobalt nitrate ($Co(NO_3)_2,2H_2O$) solutions mixed in each other, prepared in distilled water. Solutions temperature maintained at 80°C by drop wise addition of 3M sodium hydroxide solution (NaOH). The pH of solution maintained >12, followed by addition of 5 ml of ethylene glycol as capping agent. The mixture consistently stirred using magnetic stirrer. The precipitate was cooled to room temperature, filtered and then washed twice with distilled water and ethanol. The sample dried over night at about 100°C. Finally prepared samples of nickel ferrite ($NiFe_2O_4$) were calcinated at 500°C and then grind into the fine powder.

Synthesis of $MgNiFe_2O_4$ nanomaterials –

0.4 M Ferric nitrate ($Fe(NO_3)_3,2H_2O$) solution, 0.2M Nickel nitrate ($Ni(NO_3)_2,2H_2O$) and 0.1M magnesium nitrate ($Co(NO_3)_2,2H_2O$) solutions mixed in each other, prepared in distilled water. Solutions temperature maintained at 80°C by drop wise addition of 3M sodium hydroxide solution (NaOH). The pH of solution maintained >12, followed by addition of 5 ml of ethylene glycol as capping agent. The mixture consistently stirred using magnetic stirrer. The precipitate was cooled to room temperature, filtered and then washed twice with distilled water and ethanol. The sample dried over night at about 100°C. Finally prepared samples of nickel ferrite ($NiFe_2O_4$) were calcinated at 500°C and then grind into the fine powder.

Characterization Techniques –

The XRD pattern of sample obtained by using XRD made Bruker Model D8 advanced with Co Kα (λ = 1.79 A˚) for analysis and crystallanity and structure of both materials. By using Carl Zeiss FESEM SIGMA IV, SEM images were obtained, gives surface morphology, composition and size of formed nanomaterials. By using JASCO FTIR 61000 type A used to identify structure of both materials. JASCO UV-Visible spectrophotometer is used to obtain UV-Visible spectra for nanomaterials.

Results and discussion –

1. **X Ray Diffraction analysis** –

As shown in fig.1 all the XRD patterns of prepared powders show that the precursor powders were transformed into cubic spinel ferrite. In order to stabilize the functionalized nanoparticals to control their growth and to create a uniform distribution of particle size, both samples are thermally treated at 500°C. The XRD pattern shows all broad peaks due to small crystalline size of samples. The peaks of 2θ values of 21.36, 35.57, 41.56, 50.73 and 74.58 associated with (111), (220), (311), (422) and (440) for $CoNiFe_2O_4$[11,16,19].While the peaks of 2θ values of 34.16, 41.56, 50.56 and 74.58 associated with (220), (311), (422) and (440) for $MgNiFe_2O_4$ [14,15,17].

Obtained XRD peaks matched with standard characteristic peaks of cubic lattice of $CoNiFe_2O_4$ reported in ICDD reference code 22-1086 and for $MgNiFe_2O_4$ reported in ICDD reference code 45-0946.

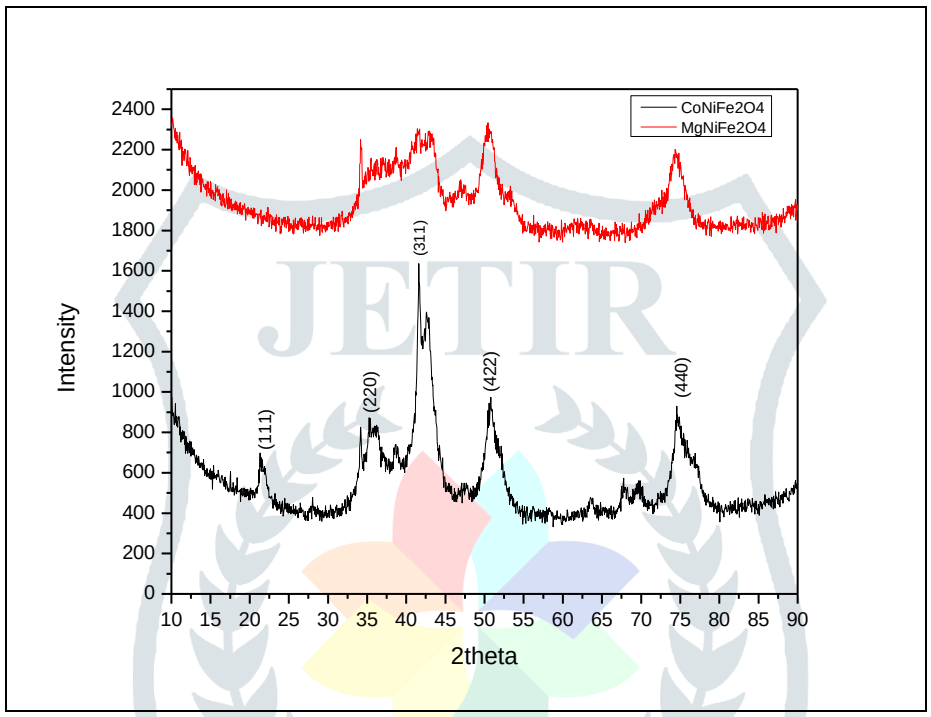

Fig. 1 XRD of $MgNiFe_2O_4$ and $CoNiFe_2O_4$

2. **Scanning Electron Microscope analysis –**

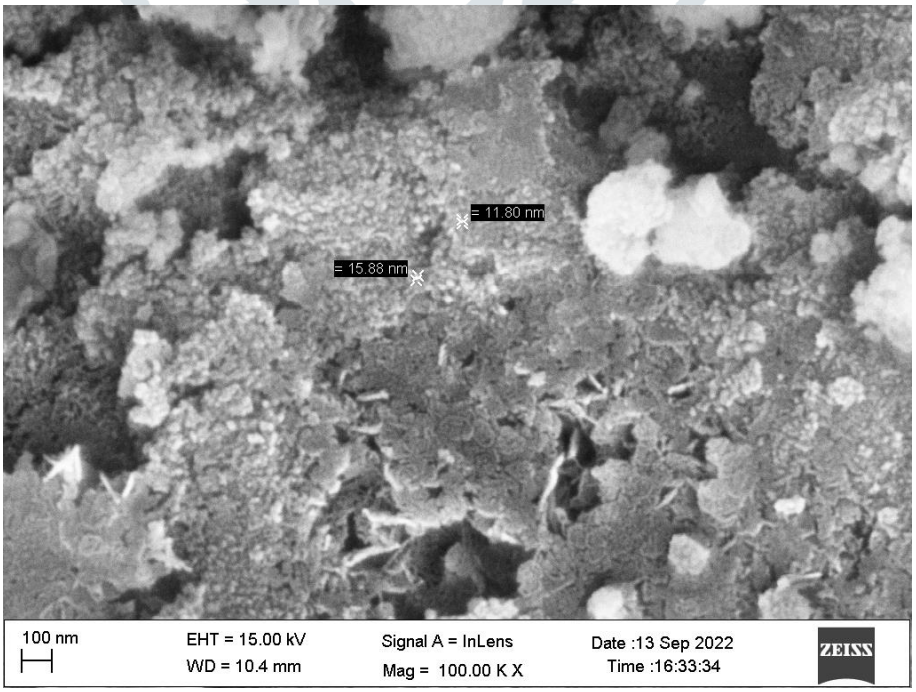

Fig.2 (a) $MgNiFe_2O_4$

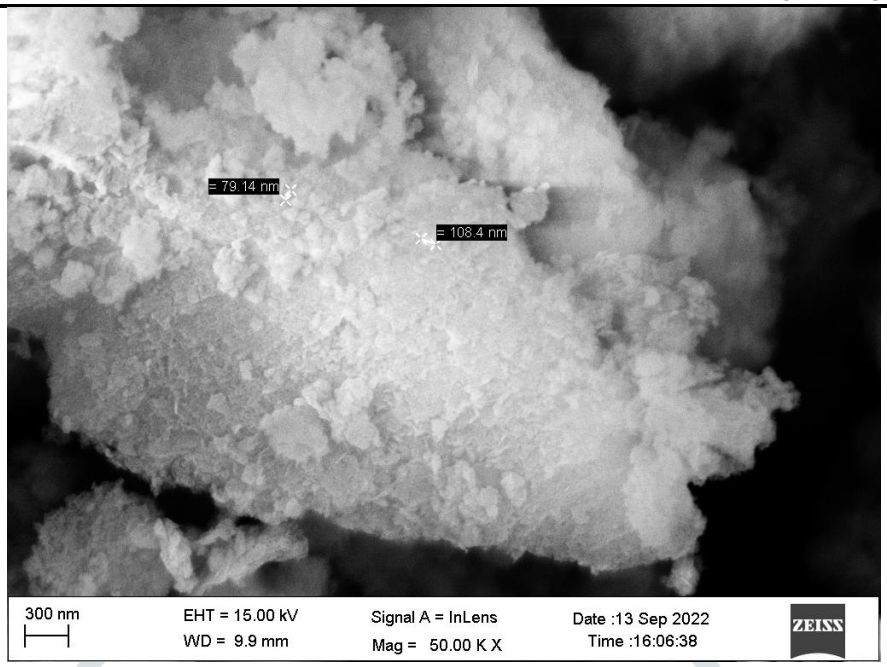

Fig. 2(b) SEM of $CoNiFe_2O_4$

Fig. 2(a & b) depict SEM micrograph shows morphology of $MgNiFe_2O_4$ and $CoNiFe_2O_4$ nano particles. It is known that morphology of any nano particles depends on concentration and molarities of solutions as well as annealing temperature. In fig 2a very tiny nano particles of MgNiFe2O4 observed of size 11.80 nm and 15.88 nm. In fig. 2b nano particles of CoNiFe2O4 observed of size 79.4 nm and 108.4 nm. From fig. 2a & b it can be concluded that formed nano particles is of zero dimensional. Nano particle size is well maintained but with agglomeration because of weak physical force. [3, 9, 12, 14, 16, 19, 20]

3. **Energy Dispersive X-ray (EDX)** :-

The EDX gives qualitative analysis of $MgNiFe_2O_4$ and $CoNiFe_2O_4$ prepared by co- precipitation method is shown in fig. 3 (a & b). The obtained percentage and constituents of elements in all investigated samples are nearly stichiometric. The obtained results supports the quality of prepared sample.[3,6,7,15]

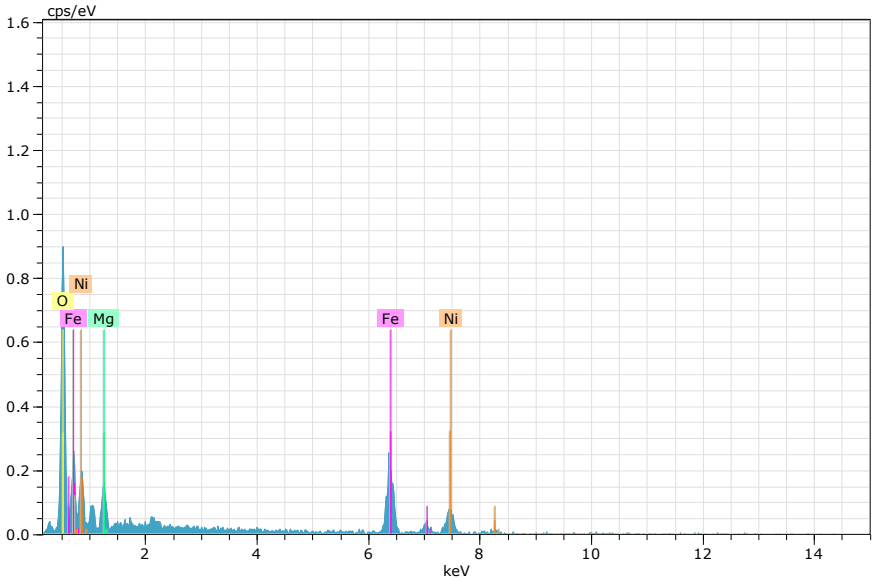

Fig. 3(a) EDS of $MgNiFe_2O_4$

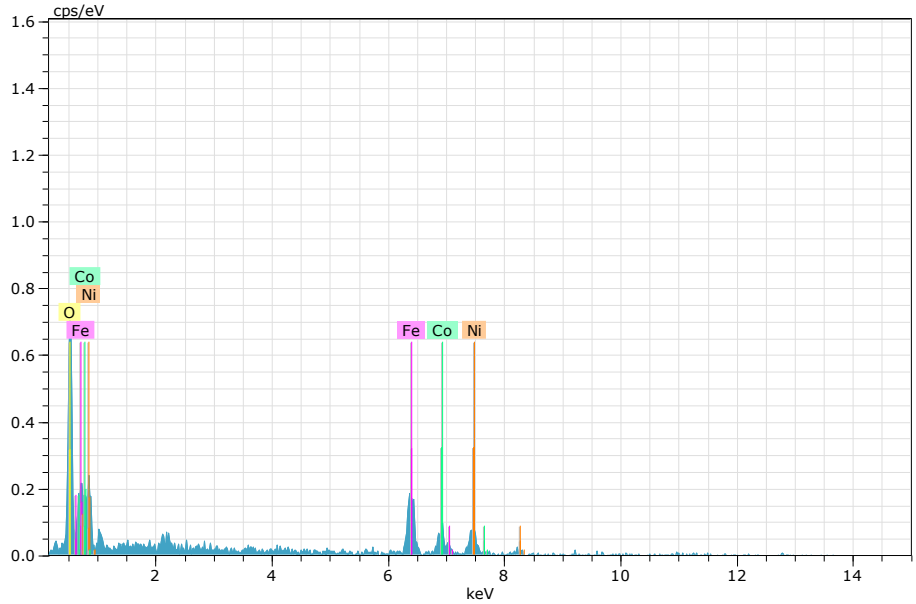

Fig. 3(b) EDS of $CoNiFe_2O_4$

4. UV-Visible Spectroscopy –

Jasco UV-Visible spectrophotometer is used to obtain UV-Visible spectra for CoNiFe2O4 and MgNiFe2O4 of nanophase powders in the absorbance mode. Wavelength between range 800 to 200 nm is used to determine λmax and energy band gap of respective nanophase powders. The UV-Visible spectrum shows exact peaks at 499 and 495 nm for $MgNiFe_2O_4$ and $CoNiFe_2O_4$ respectively (fig 4a)[5,8,].

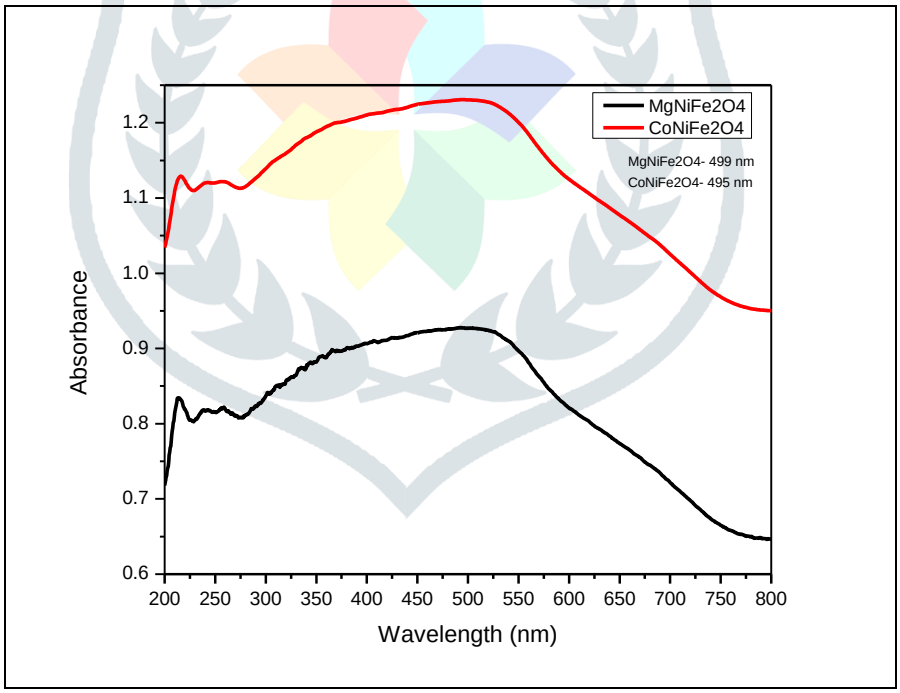

Fig. 4(a) UV-Visible spectrum of $MgNiFe2O_4$ & $CoNiFe_2O_4$

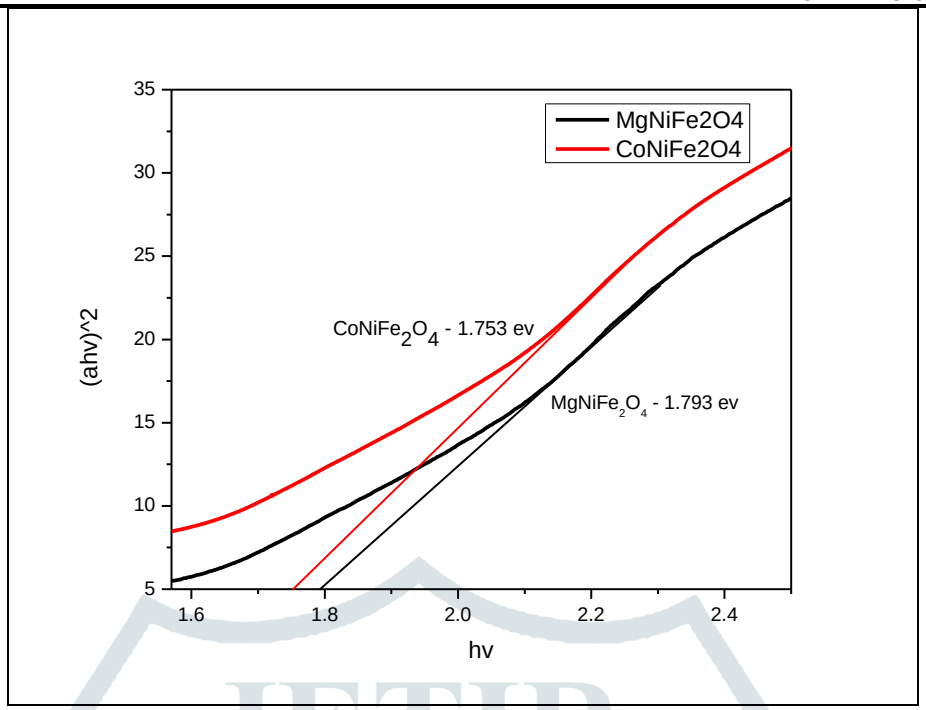

Fig. 4(b) UV-Visible spectrum of $MgNiFe2O_4$ & $CoNiFe_2O_4$ for energy band gap

From maximum wavelength absorbance band gap energy is calculated. It is 1.753 ev and 1.793 ev for $CoNiFe_2O_4$ and $MgNiFe_2O_4$ respectively. Generally semiconducting materials band gap energy lies between 0.5 to 1.5 ev. Whereas materials band gap energy is more than 2 ev termed as wide band gap materials. Thus the $CoNiFe_2O_4$ and $MgNiFe_2O_4$ materials can be acts as efficient photocatalyst because of having band gap energy smaller than 3ev [4].

5. FT-IR Spectroscopy –

FTIR spectra used to study nature of bonding and cation distribution of $MgNiFe_2O_4$ and $CoNiFe_2O_4$. The transmission bands at two frequency regions confirm their spinel structure respectively. The lower frequency near 400 – 410 cm^{-1} is related to stretching and vibrating of metal cation oxygen bond in tetrahedral site, while the higher frequency band near 650 to 680 cm-1 indicates metal oxygen bonds stretching and vibrating in octahedral sites shown in fig.5 [2,6].

The broad band at 1658 and 1773 cm^{-1} indicates O-H-O bending vibrations of absorbed water in $CoNiFe_2O_4$ and $MgNiFe_2O_4$ respectively. The bands near 3000 to 3600 cm^{-1} assigned to stretching vibrations of hydrogen bonded OH group [19, 20].

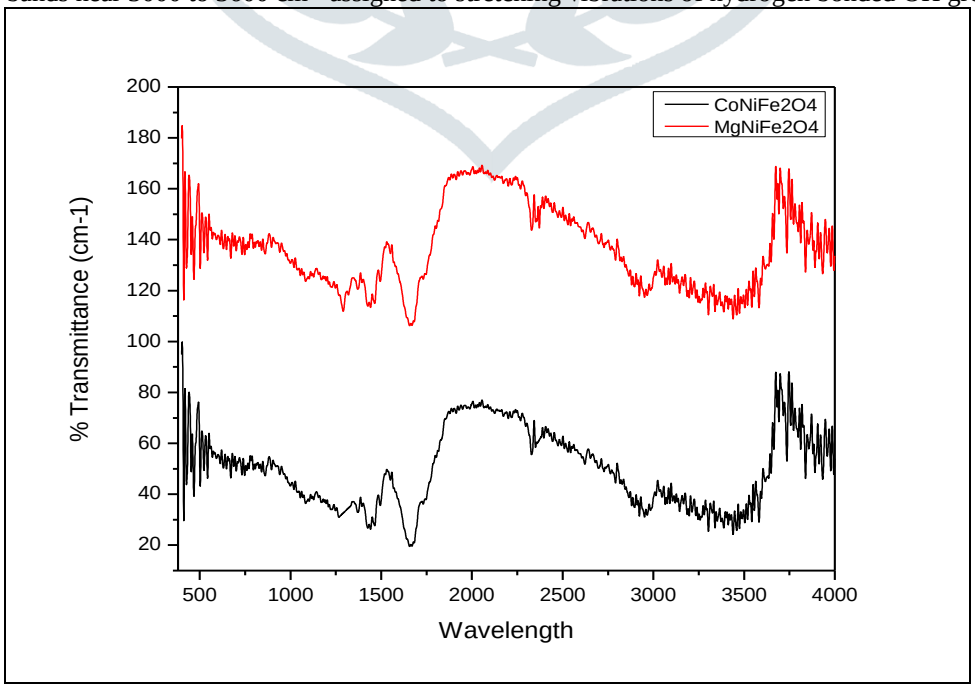

Fig. 5 FTIR spectrum of $MgNiFe2O_4$ & $CoNiFe_2O_4$

Conclusion –

The magnesium nickel ferrites and cobalt nickel ferrite have been successfully synthesized by co-precipitation method. The average particle size of $MgNiFe_2O_4$ nanoparticles was found to be 10 to 50 nm from SEM. For $CoNiFe_2O_4$ size of nanoparticles goes on increased and found to be 50 to 100 nm. XRD study for $MgNiFe_2O_4$ and $CoNiFe_2O_4$ supports and confirm formation of cubic shaped structure. FTIR studies shows formation of tetrahedral and octahedral shapes in cobalt nickel ferrite and magnesium nickel ferrite. The band gap energy for $MgNiFe_2O_4$ and $CoNiFe_2O_4$ is 1.793 and 1.753 respectively, studied from UV-Visible spectroscopy. Co-precipitation method is simple, economic and inexpensive with excellent control over size and homogeneity.

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