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SYNTHESIS AND CHARACTERISATIONS OF COBALT FERRITE NANOPARTICLES

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Abstract:

The aim of the present work is to synthesize Cobalt Ferrite Nanoparticles ($CoFe_2O_4$) by co-precipitation method and to investigate its properties. A number of chemical routes have been used for the synthesis of ferrite nanoparticles which includes micro emulsion, co-precipitation, polyol, combustion, ball milling, reverse micelles, hydrothermal methods, a polymeric precursor, sol-gel, micro emulsions, laser ablation etc. Among them, co-precipitation route is a cheap and simple method, in which the control of size and size distribution is obtained by controlling the relative rates of nucleation and growth during the synthesis process. To protect the oxidation and also to reduce their agglomeration, the particles are usually coated with some surfactant like oleic acid and then dispersed in some medium like ethanol, methanol or ammonia. X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM) confirmed the formation of single phase cobalt ferrite nanoparticles in the range 11-16nm depending on the annealing temperature. Cobalt ferrites have already many applications due to low cost of production, chemical stability and bio compatibility in magnetic sensors, magnetic resonance imaging and magnetic drug delivery.

Key Words: Cobalt Ferrite Nanoparticles, Drug Delivery & Magnetic Sensors

Introduction:

Cobalt ferrite ($CoFe_2O_4$) is a well-known hard magnetic material with high coercivity and moderate magnetization. These properties, along with their great physical and chemical stability, make CoFe2O4 nanoparticles suitable for magnetic recording applications such as audio and videotape and high-density digital recording disks etc. The magnetic character of the particles used for recording media depends crucially on the size, shape and purity of these nanoparticles. In this paper, cobalt ferrite (CoFe2O4) nanoparticles are synthesized by wet chemical method (co precipitation) and its characterisations are analysed. The size distribution is varied by varying concentration and by varying temperature.

Synthesis:

0.4M (25ml) solution of iron chloride and a 0.2M (25ml) of cobalt chloride solutions were mixed in double distilled water. 3M (25ml) solution of sodium hydroxide were prepared and slowly added to the salt solution drop wise. The pH of the solution was constantly monitored as the NaOH solution was added. The reactants were constantly stirred using a magnetic stirrer until a pH level of 11-12 was reached. A specified amount of oleic acid was added to the solution as a surfactant and coating material. The liquid precipitate was then brought to a reaction temperature of 80^{o}C and stirred for one hour. The product was then cooled to room temperature. To get free particles from sodium and chlorine compounds, the precipitate was then washed twice with distilled water and then with ethanol to remove the excess surfactant from the solution. To isolate the supernatant liquid, the beaker contents were then centrifuged

for fifteen minutes at 3000 rpm. The supernatant liquid was then decanted, and then centrifuged until only thick black precipitate remained. The precipitate was then dried overnight at 100°C. The acquired substance was then grinded into a fine powder. At this stage the product ($CoFe_2O_4$) contains some associated water, which was then removed by annealed at 600°C for ten hours. The final product obtained was then grind and characterized for further studies. Four samples of cobalt ferrites Nano particles namely sample (A),sample (B), sample(C) and sample (D) are prepared. Sample A refers the sample annealed at 400 °C ,sample B refers to the sample annealed at 600 °C, sample C refers to the sample for concentration ratio 1:2 and sample D refers to the sample for concentration ratio 1:4.

Results and Discussion:

Sem Analysis:

From the SEM images of cobalt ferrite Nano particles it is clear that most of the particles appear spherical in shape. Some moderately agglomerated particles as well as separated particles are present. Agglomeration increases linearly with change in temperature. The following fig 1a, 1b ,1c ,1d corresponds to the SEM images of sample A,B,C and D respectively.

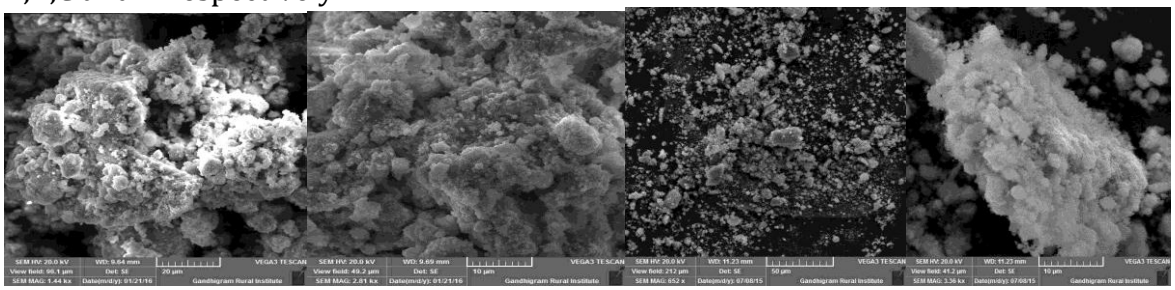
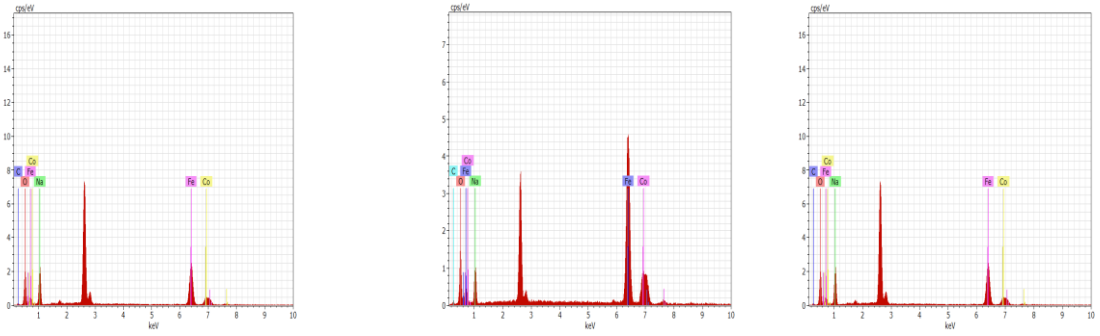


Fig 1a Fig 1b Fig 1c Fig 1d

EDAX Analysis:

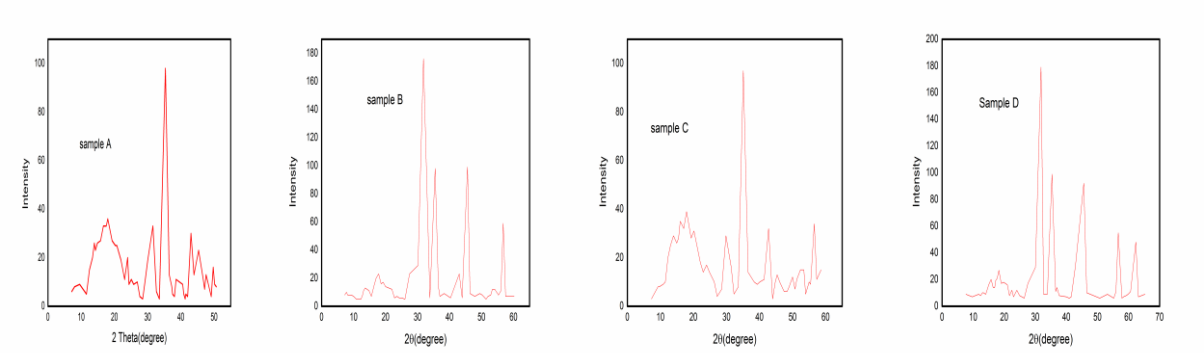
The chemical composition of the elements present in the synthesised sample is estimated using EDAX analysis and reveals the presence of O,Fe,Na,C,Co.

SAMPLE A SAMPLE B SAMPLE C

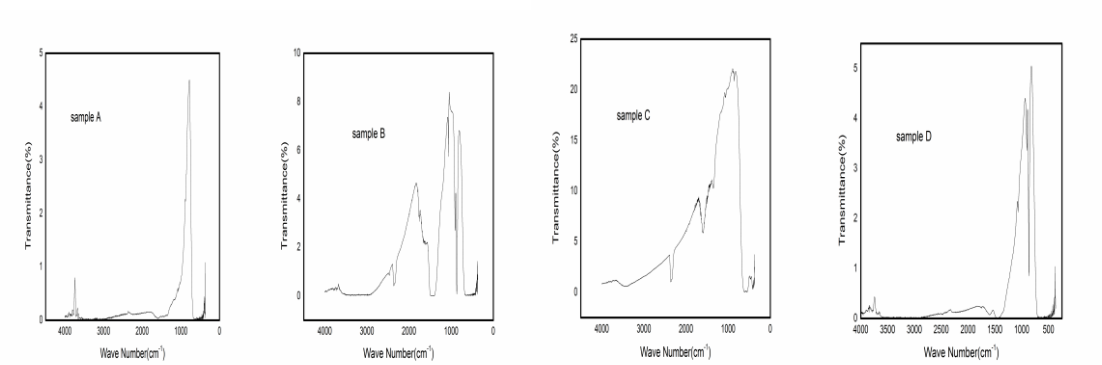


XRD Analysis:

The X-Ray diffraction pattern of the calcined powder synthesised using the wet chemical route shows that the final product is $CoFe_2O_4$ with the expected inverse spinel structure. The size of the particle was determined by scherrer formula whih results in sample A=12.57nm, Sample B = 16.041nm, Sample C= 11.84 nm and Sample D= 16.82 nm.



FTIR Analysis:



Using KBR pellets the Fourier Transform Infrared Spectra of the pure and doped $CoFe_2O_4$ powder was recorded range of 4000 cm^{-1} to 400 cm^{-1} as shown above. The peak at 564 cm^{-1} corresponds to Fe in the tetrahedral sites while the peak at 656 cm-1 is due to the stretching vibration mode associated with the metal-oxygen absorption band. The large absorption band centered at 3432 cm^{-1} can be assigned to the stretches of hydroxyl groups of gallery water molecules and hydrogen-bonded hydroxyl groups in cobalt and iron hydroxide. The peaks at about 2918, 1113 cm^{-1} are due to C-H stretching, C-O bending vibrations. The appearance of a peak at broad band at 562, 654 cm^{-1} is attributed to stretching vibrations of Fe2+-O4- which are observed in all ferrite samples. The broad band at 3424 cm^{-1} can be assigned to vibration mode of chemically bonded hydroxyl groups. The peaks at about 2928, 1107 cm^{-1} are due to C-H stretching, C-O bending vibrations.

Conclusion:

The characterization results reveals that the SEM images of cobalt ferrite nano particles appears spherical in shape. Compounds present in the synthesised samples are O,Fe,Na,C,Co which was confirmed by EDAX analysis. From XRD analysis the size of the particle was determined as sample A=12.57nm, Sample B = 16.041nm, Sample C= 11.84 nm and Sample D= 16.82 nm and the different functional group present are monitered by the vibration of the samples in FTIR.

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