SYNTHESIS AND CHARACTERIZATION OF MIXED/DOPED FERRIMAGNETIC OXIDES

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ABSTRACT

We studied about the auto-combustion synthesis and fabrication of an oxygen-deficient cobalt iron oxide and polydimethylsiloxane composite and tested its feasibility as a magnetic composite. We prepared cobalt iron Co: Fe_2 nanoparticles using two different roots as Sol Gel and wet Chemical method. TEM images shows that the size of nano particles was prepared by Sol gel and wet chemical method which is 40% smaller than the others. The effect of synthesis process on structural and optical properties were determined by XRD and FTIR.It was found that exhibit a polycrystalline nature with polymorphic structural distributions in the structure, unusual ferri-to-diamagnetic transitional property determined by X-ray diffraction, Raman Spectroscopy and vibrating sample of magnetometer studies respectively. Field Transmission Scanning Electron Microscopy results different properties of prepared sample.

Keywords: *Synthesis, Magnetic field, Ferrimagnetic, Properties,Materials, Composites*

INTRODUCTION

Magnetic composites of fluids, foam and elastomers are a widely used as scientifically and technologically for smart magneto-responsive materials, which are dispersed in the form of non-magnetic matrix by nano or micron-sized soft-magnetic particles. These particles magnetized under an applied magnetic field**.** All such magnetic material particlesincluding in matrix in the presence of an external applied magnetic field forms column- or chain-like system in the direction of the magnetic field having dipole–dipole interaction.Such constructed materials reversed into a dispersed form in the absence of a magnetic field. Such materials in phase of fluidshows non-Newtonian behaviour. while the applied magnetic field controls the shear viscosity of the material. This phase changes a liquid to solid transformation, of magneticmaterials forms different avenues in commercial applications.Several such type of group materials behave like semi-active controllers in dampers, brakes.On the basis of this material synthesis,it is very helpful in engineering applications. The principal effect on the field- of external magnetic field responsive activity is due to the magnetic particles. The materials utilized in these systems are nano-phase soft-magnetic particles such as iron, ferrites as iron ferrites and iron alloys with variable distributions in shape. It is the particle dimension in shape and size that determine the magnetic properties of the given system. In general, soft magnetic particles are favoured materials for different magnetic properties.The low magnetic hysteresis and high saturation (magnetic) values were observed of prepared sample. From last many years, cobalt iron had been at the centre of industrial and academic research because it has been widely used in the fabrication in MR systems. Although Cobalt Iron is a pure iron powder with highly magnetic and satisfactory dimensions causes major issues with increasing sedimentation. Therefore, other iron-based low-density magnetic particles have been considered to improve the colloidalstate within the viscous and achieve carriers.But mostly in D-block

ISSN: 2278-6252
Impact Factor: 7.388

metal oxides, the negative ion-to-metal charge transfer energy is the primary reason for the unusual conductive and magnetic properties. In additional the low-density iron oxide-based compounds having considerable attention for decades owing to their satisfactory various magnetic properties, specificvalence states, magnetic interactions, structures, resistive states, and other applications. The unusual quadrivalent state of iron in the interest owing to the distinctive electronic and magnetic properties of ferrites. One such specific material is barium iron oxide and cobalt iron oxide which have different types of structure such as tetragonal, triclinic, hexagonal and rhombohedraldepending upon the available oxygen stoichiometry. It was observed that the structure and magnetism within the cobalt ferrite were strongly dependent on the oxygen content. For example, $CoFeO_2$ an oxygen-deficient form of $CoFeO_3$ demonstrates meta-magnetic transitional behaviour at low temperatures or we can say that at higher temperatures the prepared sample exhibits ferrimagnetic behaviour. However, the $CoFeO_3$ system demonstrating ferromagnetism and metallicity behaviour exhibits insulating nature in ferromagnetic behaviour. Owing to the state change due to the oxygen deficiency in the $CoFeO_3$ system because its structural variety and magnetic properties are expected to increase at room temperature for various MR applications. These metal oxides are widely carried out by the sol–gel process as well as wet chemical method involving a combination of auto-combustion routes. This process involves the use of low-cost materials and simple apparatus. The combined process of sol–gel with combustion and induces a thermal redox reaction with spontaneous reactions and release the energy in different forms which sustains the reactionin a very short period. These prepared samples are affected by the temperatureat high temperature with a release of gaseous agents are directly influenced by the fuel and the oxidant ratio. The product is crystalline, with a homogenous particle size distribution, highly porous structure, high surface area, satisfactory composition, and temperature control. Hence, it is easy to say that scale up is the most industrially viable approach to use in various applications. Thus sol–gel and wet chemical method was used for the synthesis of Cobalt iron oxide particles in this research work.

Experimental Setup of materials and methods

The hydrothermal method or Wet Chemical Method was used to synthesize cobalt ferrite. This method provides different of nanostructured materials and has a lot of benefits such as clean product with high degree of crystallization with a temperature up to 200 ^{0}C. All reagents used in this synthesis are commercially available and used as without further purification. An appropriate amount of analytical ferrous sulphate and sodium citrate was dissolved in the ratio of 1:2 in pure water. This mixture placed at 120 ^{0}C for 24 hours. We obtained a blackish precipitate which washed many times in pure water and ethanol. All reagents were utilized for synthesis as-received without any purification. We can used deionized water for the synthesis of prepared sample. Cobalt nitrate, $Co(NO_3)_2$, with a high purity iron(III) nitrate nona hydrate $Fe(NO_3)_3$, with a purity of 98% and citric acid, $C_6H_8O_7$, with purity of 99.9% were used with a purity of 98% was synthesis of Barium $BaFeO_{3-}$ powder. We obtained nanocomposites of cobalt ferrites and carbon which was heated at 900 ^{0}C for 3 hours.
During this process a chemical reduction took place as
$CoFe_2O_4 + 2C – CoFe_2 + 2CO_2$

This similar process was used to obtained the same nanocomposites of cobalt ferrite inspecific ratio. The activated carbon and cobalt ferrite should be in molar ratio of 2:1 and 10:1 respectively.

In this research work, we studied mainly on the synthesis and fabrication of magnetic composite-based materials. which consist of their mechanical properties in the presence of an applied magnetic field. Most magnetic composites are prepared by mixing magnetic particles, and Magnetic composites in the presence of a magnetic field shows various a magnetic effect. A magnetic material enables the material to reclaim its natural and original shape and property. These composites may be prospective solid-state analogy of magnetorheological fluids. This magnetic-field effects the major challenges. Which include sealing, deposition and coating of iron particles. These magnetic composites have immense potential for designing the smart devices in various commercial and non-commercial purposes in various fields like engineering areas like sensing mechanical signals, controllable signals, defined structures, absorption, and isolation of the prepared materials. A major additive for these composites is the magnetic particle having higher saturation, magnetization, permeability, and lower remanence. These properties of magnetic nano composites are highly active for various applications. The main application of prepared magnetic particles in the absence of an applied magnetic field and these can be controlled for higher or lower grades. Most of the magnetic composite materials are fabricated under various conditions such as vulcanization based on the magnetic field applied during this process. To obtained prepared nano composites can be either anisotropic or isotropic, respectively. The primary aim of this study was to evaluate the influence of different loadings of $CoFeO_3$ particles in the magnetic and mechanical behaviour of the prepared nano composite. Thus, we have to obtained various properties of magnetic and mechanical properties of the composite and molecular structure.

Structural and Magnetic Measurements-

The crystalline structure and magnetic properties of prepared sample obtained by XRD, SEM, TEM and FTIR are discussed as below

X-Ray diffraction (XRD)

XRD pattern of pure iron oxides nanoparticles and co-doped iron oxide nanoparticles were shown in Figure below. All peaks in the XRD patterns confirmed the phase and inverse spinel structure of the nanoparticles. The peaks observed at 30°, 35°, 43°, 56° and 62° corresponding to the characteristic crystallographic are (2 2 0), (3 1 1), (4 0 0), (5 1 1), and (4 4 0) planes and peaks were matched.

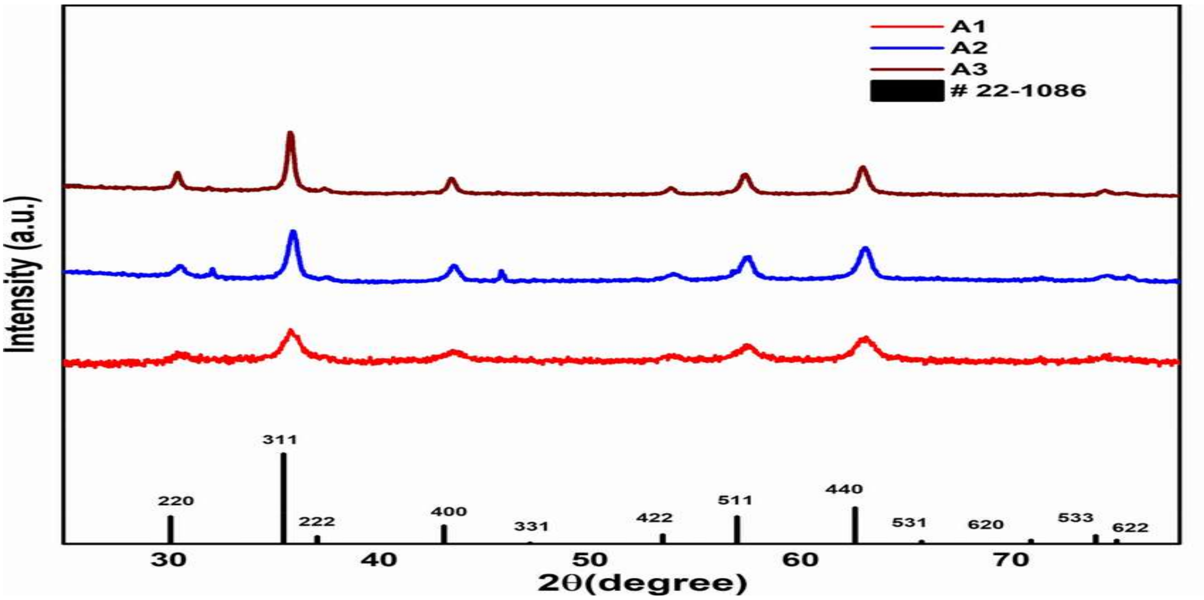

Fig- XRD pattern of Cobalt ferrite

The lattice parameters were calculated using following relations. Depicts the diffractograms for the annealed prepared powder. As illustrated, the samples exhibit high polycrystallinity, with various polymorphs of the sample in chemical phases that are also highly oxygen-deficient, few phases of iron oxide polymorphs, and cobalt ferrite.Using Scherrer's formula for the strongest peak, the crystallite sizes measured were54 nm,43 nm, and 44 nm. Listof the peak values shown in figure.

Scanning Electron Microscope (SEM)-

The XRD analysis of synthesised powder after calcination that the final product of cobalt ferrite with expected inverse spinel structure. This is very close to the expected for the cobalt ferrite. The diffraction profile obtained for the sample partially reduced obtained and perform XRD measurements. To analysis the cation distribution of the precursor compound of cobalt ferrite spectroscopy experiments at room temperature. The morphology and dimension of nanoparticle were analysed by Scanning Electron Microscope SEM measurements.

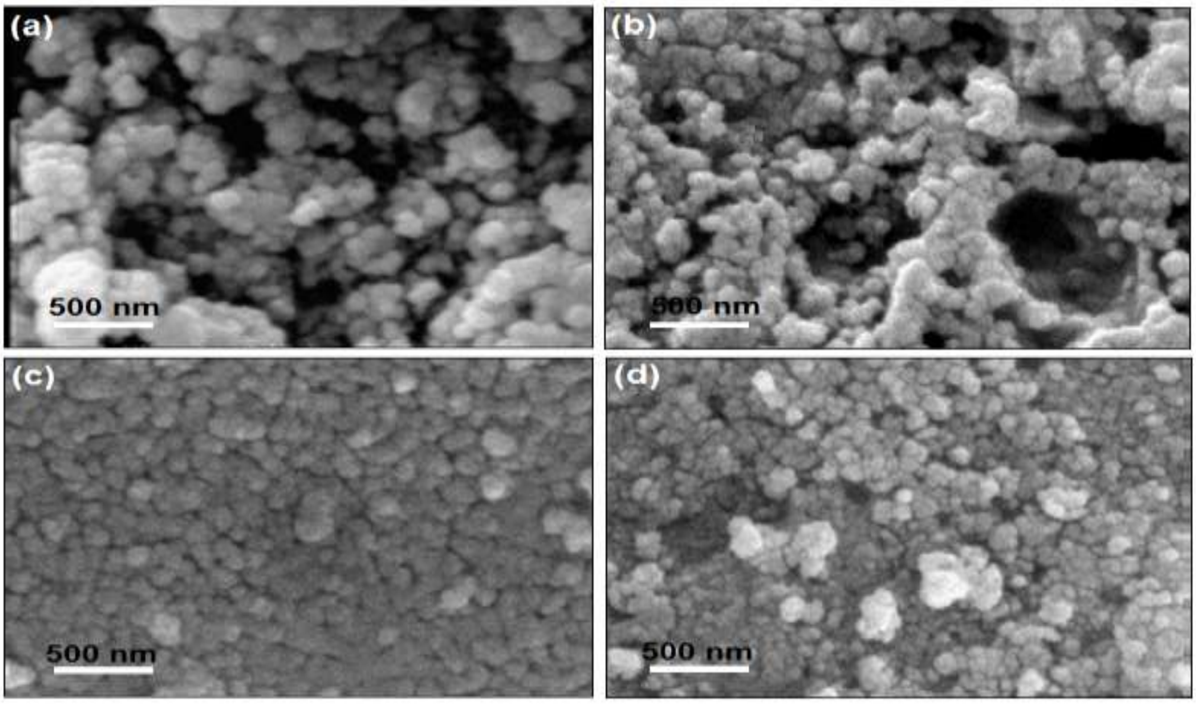

Fig- SEM of cobalt Ferrites sample

The measurements of cobalt ferrite sample read and found that the shell does not cover each nanoparticle but the particles are composed in to parts big core and thin shell about a fewnanometres.

Transmission Electron Microscope (TEM)-

The TEM measurements of nanocomposites are roughness at the surface of prepared sample but the roughness not observed at the percussor materials. The superficial materials joined with nanoparticles and most of the part of prepared sample found at interface of the particles. But due to the thickness of shell we consider necessary measurements of high-resolution TEM. TEM analysis indicates that the nanoparticles of nanocomposites are larger than the original nanoparticles show the reduction process to increase the mean size of the nanoparticle up to 98nm. TEM measurement showed that the dimension of the soft phase is larger than the critical size obtained. The cobalt ferrite sample observed the coercivity approximately 1.86kOe at normal room temperature. This results the higher than the coercivity obtained and samples treated by thermal magnetic annealing and mechanical milling respectively.

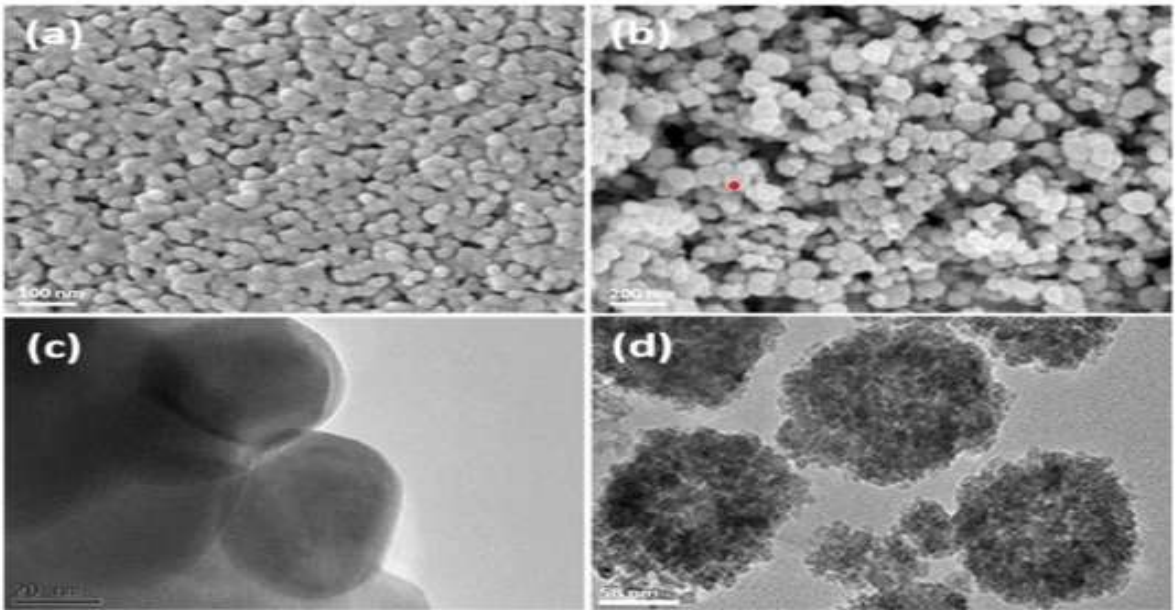

Fig- TEM of Cobalt ferrite Sample

The same behaviour observed to coercivity for the cobalt ferrite was also seen for the nanocomposites. The hysteresis curve of the nanocomposite described by single shape loop is similar to that of the single-phase indication that magnetization of both phases.

DISCUSSION

The magnetic composites are known to demonstrate a significant nonlinear contact between the matrix and magnetic filler powder. In general practice, a few additives plasticizers are introduced to enhance the strong interaction between the matrix and the filler phase. Researchers normally introduce magnetic particles, such as iron or carbon, to regulate the mechanical properties of the composites. The role played by the amount of cross-linker used is an important factor in moulding. The present work in these deals with the synthesis, characterization and fabrication of low-sedimentation magnetic composite materials. The composites were fabricated by mixing $CoFeO_3$ nano powder. Thereafter, the magnetic and mechanical behaviours of the obtained $CoFeO_3$ composites were investigated.

CONCLUSION

In conclusion, we synthesized prepared powder via auto-combustion and fabricated PDMS-based magnetic composites with different $BaFeO_3$ loadings as 0.5, 1.0 and 1.5 g. The highly polycrystalline powder demonstrated the presence of cobalt ferrite, oxygen-deficient $BaFeO_3$ and iron oxide phases, which were difficult to separate. When prepared with mixed with the powder yielded a well-dispersed composite, with very limited sedimentation. In this study, XRD demonstrated that the crystalline average size of the cobalt ferrites increased from 11.03 nm of iron oxide to Co–$Fe_2O_4$with 20% wt, 40 % and size 11.12 nm and 16.35 nm respectively. Morphology of cobalt ferrites depicted the flat surface and negligible agglomerated in SEM study. Magnetic properties of cobalt ferrites were studied Saturation of magnetisation(Ms) versus Magnetic field strength(H) it is observed that saturation of magnetization was enhanced with concentration of cobalt metal in iron oxides nanoparticles. Cobalt ferrite nanoparticle can be synthesized by combustion, coprecipitation, and precipitation methods.Average sizes of the crystals were estimated to be 68.9nm, 48.9nm and 34.7nm for combustion, coprecipitation, and precipitation methods. XRD pattern is in accordance with inverse cubic spinel structure of prepared sample with space group *fd*-3*m*. Also, can be determined the VSM data of samples showed that by decreasing particle size, saturated magnetization has decreased H_c and M_s are greatest in the combustion method.

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