ABSTRACT

The limited magnetic properties of iron oxide nanoparticles present a challenge to the application of these particles in magnetic nanoparticle technology. Increasing the saturation magnetization of magnetic nanoparticles will permit more effective development in the field of nanotechnology. Hence the present work mainly focuses on the aspect of synthesizing novel magnetic nanoparticles.

Among spinel ferrites, cobalt ferrite CoFe$_2$O$_4$ is especially interesting because of the high cubic magnetic crystalline anisotropy, high coercivity and moderate saturation magnetization. In the case of nickel ferrite, the inverse spinel type is particularly interesting due to the high magneto crystalline anisotropy and high saturation magnetization which are attributed to the typical crystal and magnetic structure.

The substitution of copper (Cu) in these ferrites brings about a structural phase transition accompanied by a reduction in the crystal symmetry due to cooperative Jahn-Teller effect which ultimately results in some interesting electrical and magnetic properties. A novel idea of using Cu as an additive element to cobalt ferrite and nickel ferrite is proposed in this work. As the properties of the synthesized materials are influenced by
the composition and nanostructure which are sensitive to the preparation methodology, cobalt-copper ferrite $\text{Co}_{(1-x)}\text{Cu}_x\text{Fe}_2\text{O}_4$ [where $x = 0.2, 0.4, 0.6$], nickel-copper ferrite $\text{Ni}_{(1-x)}\text{Cu}_x\text{Fe}_2\text{O}_4$ [where $x = 0.2, 0.4, 0.6$], Co-Ni-Cu mixed ferrite ($\text{Co}_{0.4}\text{Ni}_{0.4}\text{Cu}_{0.2}\text{Fe}_2\text{O}_4$) and Ni-Mn-Cu mixed ferrite ($\text{Ni}_{0.4}\text{Mn}_{0.4}\text{Cu}_{0.2}\text{Fe}_2\text{O}_4$) are synthesized using co-precipitation method. The magnetic and dielectric properties of these mixed ferrites are studied and their significance is discussed in this work. The effects of sintering on these nanoparticles are also discussed.

Nanocrystalline cobalt ferrites are prepared by using stoichiometric amounts of cobaltous chloride [$\text{CoCl}_2.6\text{H}_2\text{O}$] and anhydrous ferric chloride [$\text{FeCl}_3$] dissolved in distilled water. The neutralization is carried out with sodium hydroxide solution and the reaction temperature is maintained at 60°C. The pH of the solution is maintained at 8 and it is stirred for 2 hrs. The precipitate is thoroughly washed with distilled water until it is free from impurities. The product is dried at a temperature of 100°C to remove the water contents. The dried powder is mixed homogeneously and sintered at 130°C, 600°C and 900°C. Similarly nickel ferrite, manganese ferrite, copper ferrite, copper doped cobalt ferrite, copper doped nickel ferrite, cobalt-nickel-copper mixed ferrites and nickel-manganese-copper mixed ferrites are prepared by adopting co-precipitation method.
The crystal structure of these synthesized samples is analyzed using XRD Shimadzu 6000. The FT-IR spectra of these samples are recorded using Shimadzu IRAffinity-1 that ensures the presence of the metallic compounds. The magnetic properties of these ferrite nanoparticles are studied using Lakeshore Vibrating Sample Magnetometer VSM 7410 at room temperature. The morphology and the microstructure of the samples are tested by Scanning Electron Microscopy (SEM) using a Hitachi S-3000H microscope and by High - Resolution Transmission Electron Microscopy (HR-TEM) using a JEOL JEM 2100 microscope. The elemental composition of the samples is tested by energy-dispersive X-ray spectroscopy (EDX). The dielectric properties are studied by pelletizing the samples using hydraulic press. The pellets are coated with silver paste to ensure good electrical contacts and tested using Hioki 3532-50 LCR Hi-tester.

Among the synthesized ferrites in the present study, the copper doped cobalt-nickel mixed ferrites and copper doped nickel-manganese mixed ferrites have low values of crystallite size in the range of 12 nm - 32 nm and 12 nm - 27 nm for the samples sintered at 600°C and 900°C respectively. TEM results also give the confirmation for the smaller particle size. The highest values of saturation magnetization and coercivity are found for the copper doped cobalt ferrite of composition
Co$_{0.8}$Cu$_{0.2}$Fe$_2$O$_4$ and copper doped cobalt-nickel mixed ferrites. The highest value of dielectric constant is found for Ni-Mn-Cu ferrite system.

As the co-doping of three metal ions decreases their particle size and enhances their properties, Ni-Mn-Cu mixed ferrite and Co-Ni-Cu mixed ferrite nanoparticles are subjected to the applications as humidity sensors and electrocatalysts.