Structural and Morphological Characterization of Mixed Spinel Nano-Ferrites

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ABSTRACT

Nanoparticles of mixed spinel ferrite having generic formula of $(NiZn)_xCu_{1-2x}Fe_2O_4$ were respectively prepared by sol gel auto combustion method and co-precipitation method. The prepared samples were annealed for 4 hrs at 800ºC to obtain the pure phase of Ni-Zn spinel ferrite. The X-ray diffraction confirmed the typical structure formation of spinel ferrites with no impure phase. The morphological characterization was done by using scanning and transmission electron microscopy respectively. The SEM reveals the randomly oriented cubic shape particles and TEM counter-verified the cubic shape and further confirms the as prepared sample lies within the nano meter scale

Keywords: Spinel ferrites, auto combustion, co-precipitation, X-ray diffraction, SEM, TEM etc.

I. INTRODUCTION

The ferrites with cubic structure known spinel ferrites are extensively attractive only because of its good performance and broad range of applications. The Ni-Zn nanoparticles ferrite has a unique chemical and structural behaviour that makes it the prolific material in many medical and technological applications such as magnetic delivery of drugs, ferrofluids, MRI, recording media etc. The size of spinel ferrite if confined below 25 nm then the exceptional property called as superparamagnetism occurs that offers outstanding opportunities in manipulation of nanoparticles to enhance the various applications. Numerous synthesized methods are available to prepare the spinel ferrites viz. sol-gel auto combustion, co-precipitation, hydrothermal, solid state, miscelles, microemulsion etc. The present research work aimed to prepare the Ni-Zn mixed ferrites via two different chemical routes to understand how different synthesis methods influence the structural and morphology properties.

II. EXPERIMENTAL

The chemically AR grade precursor nickel nitrate $[Ni(NO_3)_2.6H_2O]$, zinc nitrate $[Zn(NO_3)_2.6H_2O]$, copper nitrate $[Cu(NO_3)_2.4H_2O]$, ferric nitrate $[Fe(NO_3)_3.9H_2O]$ were used as starting materials and urea $[NH_2\text{-}CO\text{-}NH_2]$ used as a fuel. The lone sample of $(NiZn)_xCu_{1-2x}Fe_2O_4$ for x = 0.3 was synthesized by two most generous techniques viz. sol-gel auto combustion route and co-precipitation method.

Sol Gel Auto Combustion Technique

The metal nitrates were dissolved in minimum amount of distilled water. Urea was used as fuel and dissolved into the solution to give a molar ratio of metal ions to urea of 1:1 to form the sol. Then the sol was put on a magnetic hot plate and heated at 70^0C to obtain viscous gel. The as obtained gel was exposed to the specially designed microwave oven, followed by an instantaneous gel ignition with the formation of large amount of gas. The resulting precursor' powder was calcined at 800^0C for 4 hrs and grinded for 2 hrs to obtain pure phase of mixed ferrite [1].

1) Co precipitation Technique

The stoichiometric proportion of precursor were dissolved in deionzied water that include the slow addition of sodium hydroxide (1M) solution to form ferrite precipitate at room temperature. The aqueous solution put on the magnetic stirrer and stirred at 600 rpm for 30 mins to obtain better homogeneity. The dark brown precipitates as obtained filtered, washed off with distilled water and dried overnight. The precipitated was calcinated at 800ºC and grinded for 2 hrs to have ultrafine particles [2, 3].

III. CHARACTERIZATION TECHNIQUE

The structural characterization of samples has been studied by X- ray diffraction (Bruker Advance X-ray diffractometer). The scanning electron microscopy (SEM-JSM-7600F) and transmission electron microscopy (TEM-CM200) were used to characterize the nanostructure features of the samples.

IV. RESULT AND DISCUSSION

The as synthesized ferrites were analysed with the X-ray diffraction technique to check the structural details.Figure 1 shows the XRD patterns of spinel ferrite sample synthesized by sol- gel auto combustion and co-precipitation method. The peaks being there (1 1 1), (3 1 1), (2 2 2), (4 4 0) in the XRD clearly indicate the formation of single phase spinel ferrite structure. The XRD pattern was compared with and indexed using ICDD card no. 86-2287 that confirms the space group fd-3m [4]. The figure show XRD peaks are little broad reveals the smaller grain size within the 100 nm. Generally sol-gel and co-precipitation technique yields the nano particles of ferrites [5, 6].

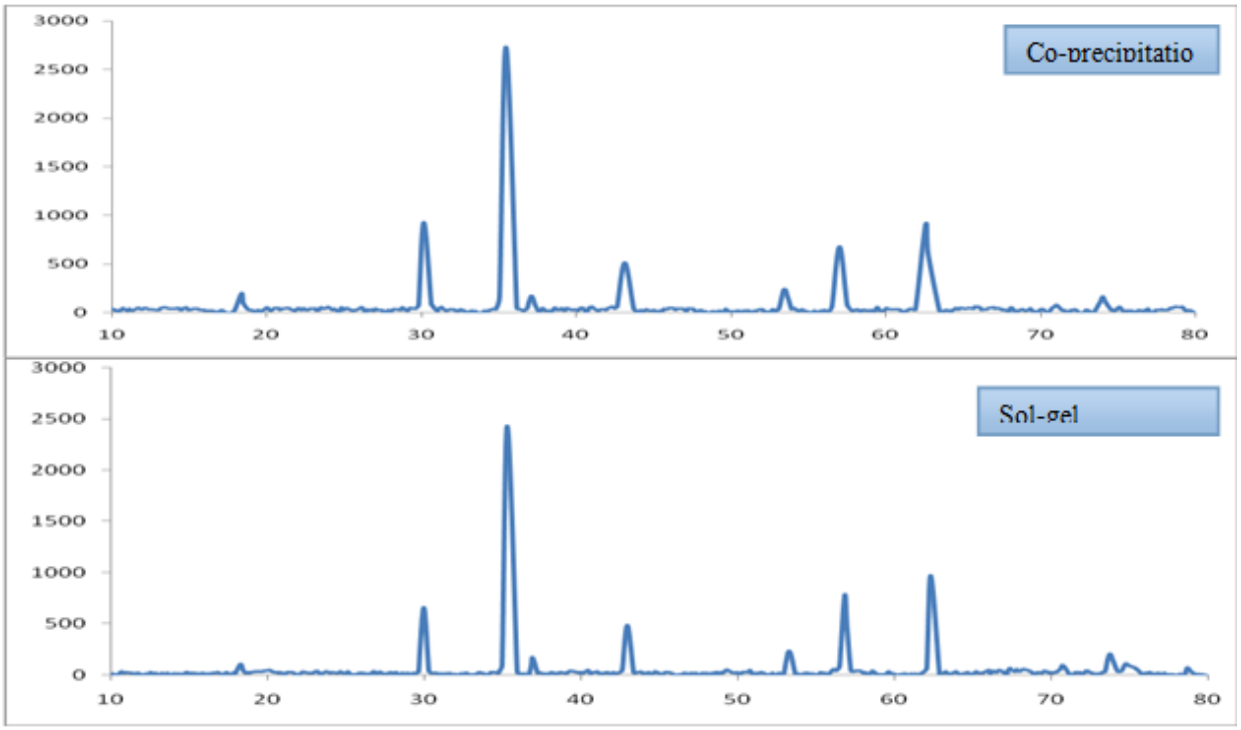

Figure 1: X-ray diffraction pattern of $(NiZn)_xCu_{1-2x}Fe_2O_4$ (x = 0.3)

The particle size obtained using De-bye scherrer formula was just about 54 nm and 37 nm for sol-gel and precipitation method respectively. The lattice constant & average particle size are depicted in the table no. 1.

Table No. 1: The values of structural parameters calculated from XRD of $(NiZn)_{0.3}Cu_{0.4}Fe_2O_4$

Sr.No.	Method	Lattice parameter (Å)	Particle Size (nm)	Porosity (%)
1	Co-precipitation	8.356	37	40
2	Sol-gel combustion	8.510	54	48

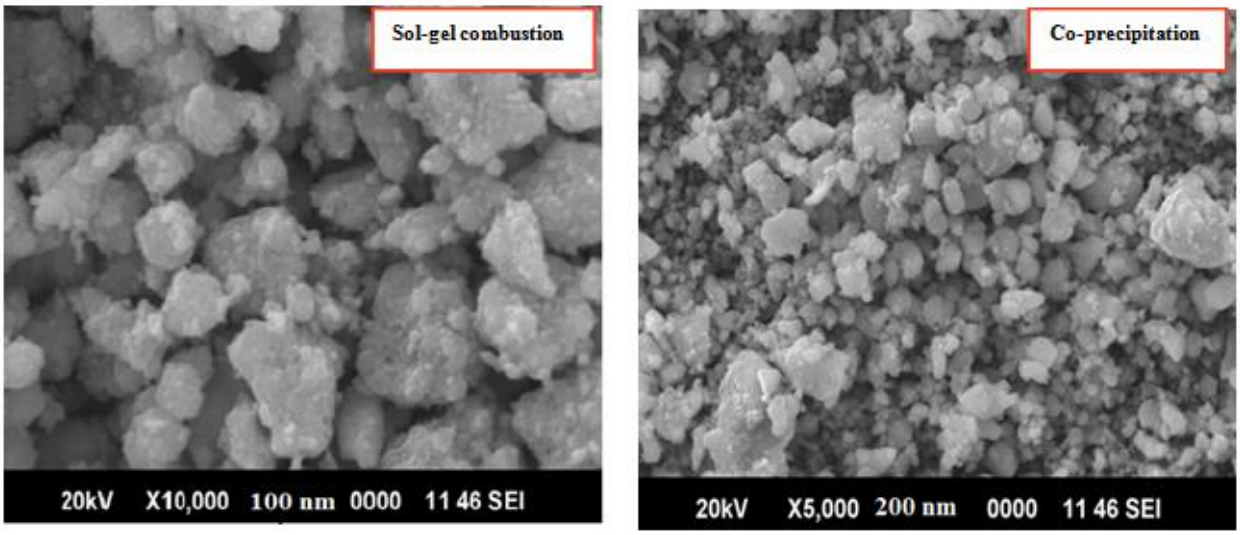

Figure 2: SEM images of $(NiZn)_xCu_{1-2x}Fe_2O_4$ (x = 0.3)

Figure 2 shows the SEM micrographs of prepared sample. Apparently the SEM image indicates the structural changes with respective synthesis method. The morphology looks non-uniform with agglomeration and grain size is somewhat affected by the respective synthesis method. Comparatively sol-gel method shows large voids indicating more porous nature of sample and is expected as large amount of gases are coming out during the synthesis.

Figure 3 shows the TEM micrographs of prepared mixed spinel ferrites by sol gel combustion and co-precipitation method respectively. TEM shows cubic morphology of synthesized ferrite with sharp edges. The grain size obtained from TEM shows good agreement with the average particle size calculated from scherrer formula. The TEM micrograph of prepared sample by sol-gel combustion method shows the agglomeration that resemblance with respective SEM image.

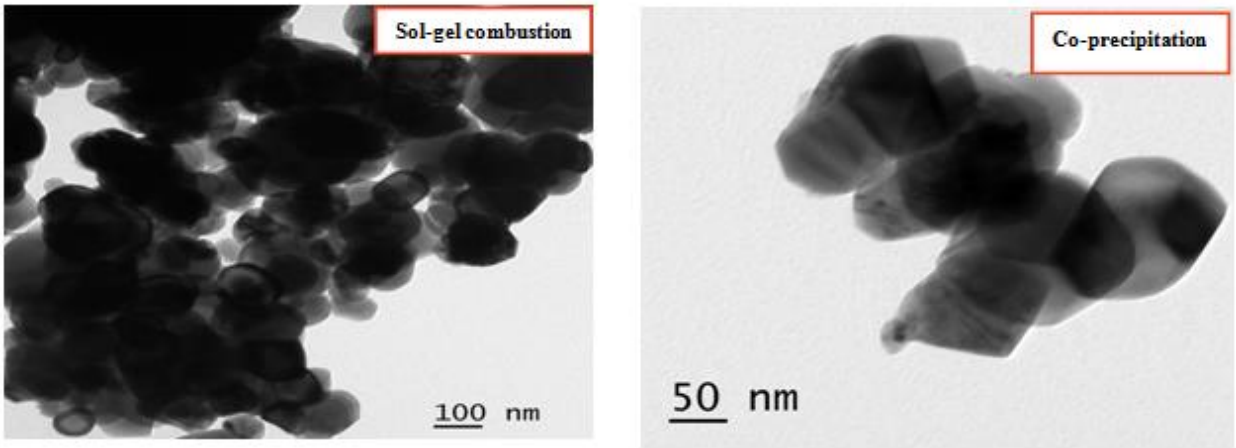

Figure 3: TEM images of $(NiZn)_xCu_{1-2x}Fe_2O_4$ (x = 0.3)

V. CONCLUSION

Nano-spinel ferrites are successfully synthesis by co-precipitation and sol-gel auto combustion techniques. Both XRD indicates crystalline nature and formation of cubic structure of synthesis material & values of lattice parameters being supported it. The SEM shows the irregular morphology with large voids indicating porous material. TEM shows cubic structure of spinel ferrites with particle size in nano meter range.

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