

The Properties of Mn-Zn Forrester Synthesis Using Nano Crystalline

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Abstract

Magnetic nano-materials are extensively studied because of their wide range of applications. The Mn-Znferrite is amongst the most important magnetic materials and has attracted a great interest in technology because of its use in memory or data storage devices, magnetic recording heads, switches and other electromagnetic gadgets. Low temperature single phase nano crystalline $Mn_xZn_{(1-x)}Fe_2O_4$ ($x = 0.4, 0.5$ and 0.6) were prepared by decomposition of precursor by sunlight. Stoichiometric amounts of manganese oxide, zinc oxide and ferric oxide are taken as starting materials. Mixture of these oxides were homogenized by using ball-mill and then treated with a ligand to produce a precursor. The samples obtained after the decomposition of precursor were characterized by frared spectral analysis (IR), X-ray diffraction (XRD) and atomic absorption spectroscopy (AAS). The lattice parameters increase with increase in Mn content (8.4386-8.4552 Å). The particle size, determined by Scherer formula, is found to be in nano range between 17nm to 26nm. The saturation magnetization values are in the range 22.47emu /g - 41.63emu /g and hysteresis loss is found to be low for all the samples. The Curie temperature (T_c) is in the range 473K –558.

Keywords

Nano Material, Magnetic Properties, Oxides, Sunlight, Self-Decomposition

1. Introduction

Magnetic iron oxide nanoparticles are of high interest due to their unique properties. Among them Mn-Znferrites are one of the most widely used magnetic materials due to their low hysteresis loss and high saturation magnetization. The applications of these versatile magnetic material ranges from actuators, electromagnetic gadgets, switches, sensors, transformers etc.[1] to biomedical applications [2-3]. Even though, it has many applications in various fields as bulk material [4] as well as their new applications as nanoparticles [5], an increase in the demand for the development of new synthesis routes for Mn-Znferrite nanoparticles, still exists and are explored by different group of researchers. Therefore, the improvement and understanding of various new synthesis routes that allow the achievement of highly crystalline materials is a constant need. As reported in the literature by different researchers [6-7]. It is very important to have the accurate control of chemical composition and particle size distribution for the preparation of high quality Mn-Zn ferrite powders. The most common method of producing Mn-Znferrite powders in the large amount in industry is via the traditional ceramic technology, involving solid phase reactions at high temperatures, of mixture of oxides [8-9], which is often faced with difficulties of providing high quality powders required for the high performance devices because of the poor phase and compositional control, in homogeneities in compound, as well as of larger and wider particle size distributions and impurities. Many novel and innovative wet-chemical methods are tried successfully in recent years to prepare high quality ultrafine powders or nanopowders, including the co-precipitation [10]-

[11], hydrothermal process [12-13], sol-gel [14-15] and sol-gel auto-combustion [16-17]. In this study a novel method was tried in which solid oxides of manganese, zinc and iron were ball milled to obtain a homogenous mixture which was then treated with a ligand hydrazinium acetate to obtain a precursor which was exposed to sunlight and on drying undergoes auto combustion- self decomposition- to give ultrafine Mn-Znferrite. 2.Experimental The stoichiometric amounts of pure 99.9% manganese dioxide (Sigma-Aldrich make), zinc oxide (Thomas Baker make) and ferric oxide (Thomas Baker make) was taken as starting materials. The mixture was then ball-milled with ball to material ratio of 10 at 80rpm speed for 10hrs in Acmas Technocracy Ball Mill (model Acme - 8203) to obtain a powdered mixture. This mixture was then treated with calculated amount of aqueous hydrazinium acetate and homogenized to a thick paste. The resulting paste containing mixed metal oxides was exposed to sunlight as shown in the fig. 1 it dried slowly by the sun rays. It was observed that, on drying the paste swells and undergoes auto combustion- self decomposition- to finely divided solid powder which was found to be magnetic in nature. The procedure was repeated for other samples and, the final product, in each case, was used for characterization as well as to study the structural, magnetic and electrical properties.

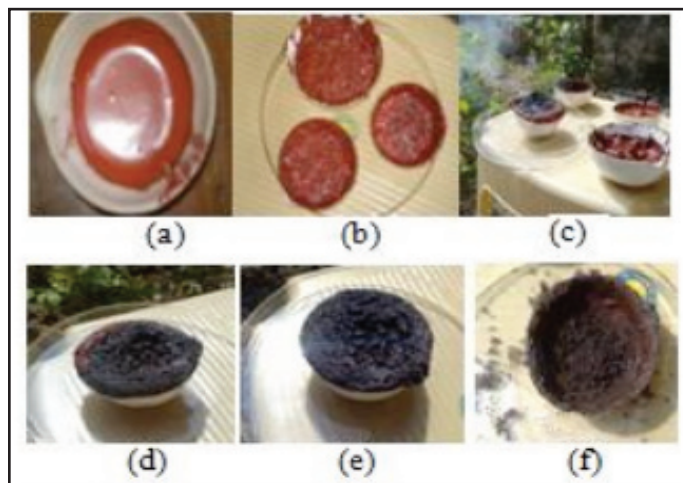


Fig. 1 (a) to (f): Photos showing different stages of Photo-catalytic auto combustion self- decomposition of a precursor

The IR spectra of Mn-Znferrite samples were recorded by using FTIR Shimadzu Model IR restige 21 series spectrophotometer. In a typical experiment, the solid ferrite sample was finely ground along with the pure and dry KBr, in the ratio 1:10. Fine grinding is required for the sample to be uniformly mixed with KBr. The mixture was then put in a sample holder and placed in the sample chamber of the IR spectrophotometer. The absorption spectrum for the sample was recorded in the wavelength range 1000cm⁻¹ to 400-1. Atomic absorption spectroscopy is a method for elemental analysis. It is also useful in detecting, both qualitatively and quantitatively, the trace metals and it is independent of the molecular form of the metal in the sample. The method is highly sensitive and can detect different metals in concentration of less than one ppm.

II. Results and Discussion

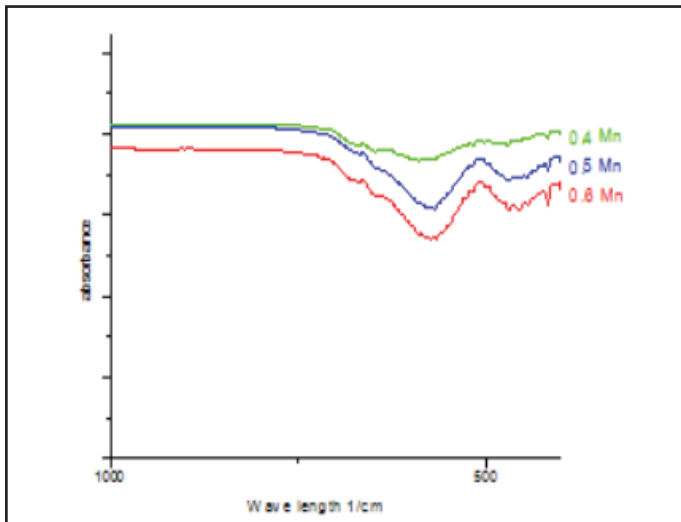


Fig. 2: IR Spectra of $\text{Mn}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$

The infrared spectra of all the ferrite samples are shown in the Fig. 2 recorded in the range of 1000-400 cm^{-1} . Infrared spectra of various ferrites have been studied by Waldron [18], reporting two bands in the region 1000 cm^{-1} to 200 cm^{-1} . He attributed the high frequency (ν_1) band to tetrahedral metal-oxygen bond and second frequency (ν_2) band to octahedral metal-oxygen bond corresponding to: (a) Me-T-O-Me stretching vibration 600-550 cm^{-1} (b) Me-OO stretching vibration 450-385 cm^{-1} where oxygen, Me is metal in the octahedral site and T is in the tetrahedral site. The metal-oxygen absorption bands (a) and (b) are pronounced for all spinel structures and essentially for ferrites, which are also seen in these samples. IR spectral data of all the ferrite samples prepared by these methods are found to show two peaks in the range 588-569 cm^{-1} and 462-447 cm^{-1} which are in agreement with the reported value [19-20].

Table 1: AAS Analysis Data for Ferrite Samples

Composition	Mn content		Zn content		Fe content	
	Theor. %	Exptl. %	Theor. %	Exptl. %	Theor. %	Exptl. %
$\text{Mn}_{0.4}\text{Zn}_{0.6}\text{Fe}_2\text{O}_4$	9.27	9.78	16.56	16.18	47.15	47.46
$\text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$	11.65	11.82	13.86	13.39	47.36	47.59
$\text{Mn}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$	14.04	14.37	11.14	11.25	47.57	46.43

The results obtained for Fe, Mn and Zn ions in each sample are given in the Table 1 and the same are in agreement with the assigned stoichiometric compositions for the ferrite samples within the permissible experimental error.

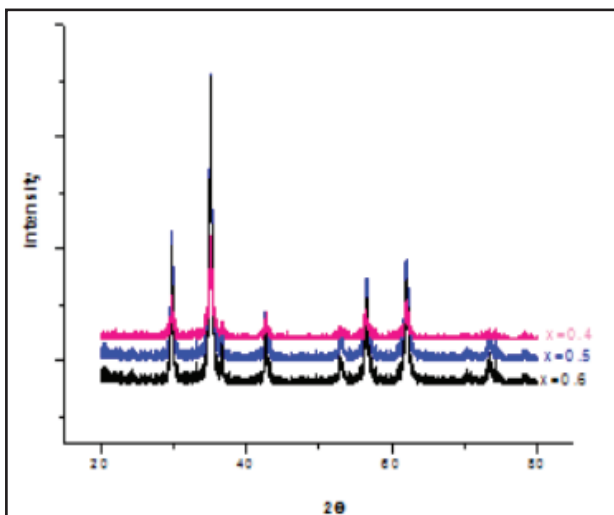


Fig. 3: X-ray Diffraction Patterns of Samples $\text{Mn}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$

Fig. 3 shows the X-ray diffraction patterns of samples $\text{Mn}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$, where $x = 0.4, 0.5$, and 0.6 nanoparticles. All the peaks correspond to cubic spinel ferrite structure for all the samples and confirm the formation of single phase ferrite. Average crystallite sizes were calculated by using XRD data by measuring the full-width at half maximum (FWHM) for most intense characteristic (311) peak for each sample with the help of the Scherrer formula as given in equation (1), and are in the range 16-26 nm for different Mn concentrations.

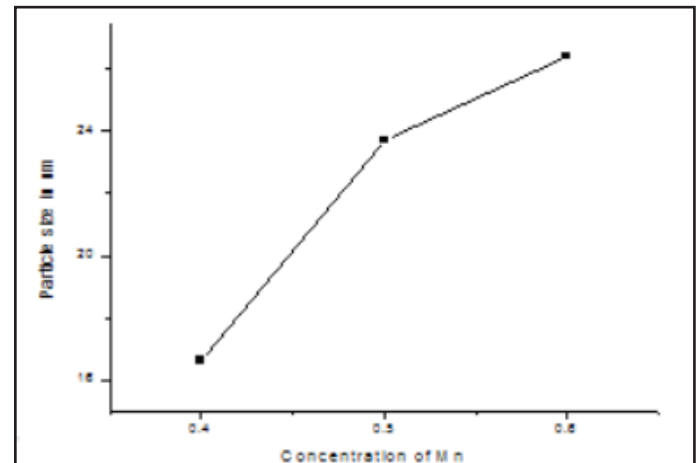


Fig. 5: Variation of Particle Size with Mn Content

Fig. 5 shows the crystallite sizes are found to increase with Mn content and in the range 16-26 nm for different compositions. It is observed that crystallite size of 16 nm is observed for the ferrite sample with Mn concentration of $x = 0.4$ which increases to 26 nm for Mn concentration of $x = 0.6$. It may be due to the larger size of ionic radius of Mn^{2+} ions as shown in Fig. 5.

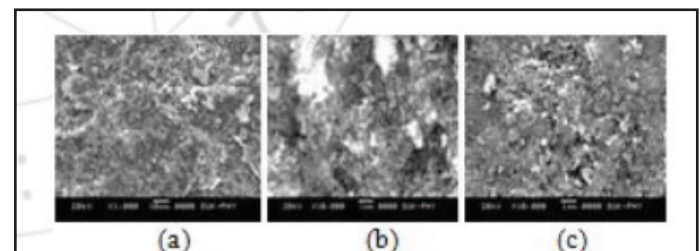


Fig. 6: SEM Images of $\text{Mn}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0.4, 0.5$, and 0.6) Nanoparticles

The morphology of $\text{Mn}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$, where $x = 0.4, 0.5$, and 0.6 nanoparticles is shown in SEM micrographs in Fig. 6(a), (b) and (c). For all compositions the Mn-Zn ferrites show similar morphology of particles. As seen in Fig. 6 particles show very strong agglomeration due to magnetic characteristics of particles and are of uniform grain size.

II. Conclusion

The simple mechano chemical method using metallic oxides, as starting material, adopting photocatalytic auto combustion-self decomposition for the synthesis of the final material was found to produce high performance nanoparticles of Mn-Zn mixed ferrite material.

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