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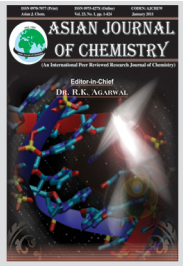

High Porosity Mn-Zn Ferrites Obtained by Using Mechano-Chemical and Wet Chemical Method

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Manganese-zinc ferrites are synthesized by two different methods namely mechano-chemical and wet chemical, using same ligand. XRD data is used to confirm formation of single phase of ferrites. Lattice constants, X-ray densities, mass densities of the ferrite samples are determined. Porosities calculated by densities method for the samples are found to have high values.

Key Words: Mechano-chemical, Lattice constants, Density, Porosity.

INTRODUCTION

Ferrites have been studied by many researchers for last several decades due to their wide applications as magnetic materials for electronic components like audio and video equipments, power transformers, telecommunications, *etc*[1]. Ferrites are also known to exhibit catalytic properties[2]. Manganese-zinc ferrites are characterized by high magnetic permeability, low core loss, high saturation magnetization and dielectric resistivity[3,4]. Mn-Zn ferrites are found to show enhanced performance with reduced particle size. During the last two to three decades researchers have reported studies on basic physical and chemical mechanism governing performance of ferrites obtained from the ceramic method, but less attention has been given to evolution of properties and the microstructure.

Insight into the porosity of ferrite materials is very important especially when these substances are to be used as catalysts or sensors. Oxide materials having high porosity exhibit better catalytic property[5,6] and are good sensors[7,8]. Convenient, simple and reliable method[9,10] of determining the porosity of materials involves using the values of lattice constants, X-ray densities and mass densities of these substances. In case of Mn-Zn ferrite it has been reported[11,12] that the lattice constant 'a' increases with Mn content.

In the present study an attempt has been made to make use of the X-ray and mass densities to evaluate the porosity of Mn-Zn ferrites prepared by a mechano-chemical method and compare the same for samples obtained by using modified wet chemical method.

EXPERIMENTAL

$Mn_xZn_{(1-x)}Fe_2O_4$ (x = 0.4, 0.5, 0.6, 0.7 and 0.8) ferrite samples were synthesized by two different methods. In the first case, calculated amounts of pure 99.9 % MnO_2, ZnO and Fe_2O_3 were taken as starting materials and ball-milled with ball to material ratio of 10 at 80 rpm speed for 10 h in Acmas Technocracy Ball Mill (model Acm-82303) to obtain a powdered mixture. This mixture was then treated with predetermined amount of aqueous hydrazinium acetate and homogenized to a thick paste. The acetate hydrazinate precursor of mixed metals was slowly dried on sand bath using conventional method of heating. The dried paste was then ignited to obtain the desired product which was used for further investigations.

In wet chemical method, aqueous solution containing manganese acetate, zinc nitrate and ferric nitrate, having metal ions in stoichiometric proportion, was treated with hydrazinium acetate to obtain the precursor. The mixture was then dried and ignited to obtain fine particle product.

The samples so obtained were characterized by using X-ray diffraction method. The d-values were used to identify the phase and to confirm the formation of ferrites. The mass density was determined from the mass and volume of the sample by using pyknometric method. From X-ray density and mass density, the porosity in each sample was determined.

RESULTS AND DISCUSSION

The X-ray diffraction patterns of $Mn_xZn_{(1-x)}Fe_2O_4$ samples, obtained by two methods, show formation of single phase of

Mn-Zn ferrite (Figs. 1 and 2). However, from the angle of diffraction 'θ' it is found that for the samples prepared by mechano-chemical method, the lattice constant values are in the range 8.4195 to 8.4519 Å while for those synthesized by wet chemical method 'a' values are on the higher side and are in the range 8.4529 to 8.4687 Å.

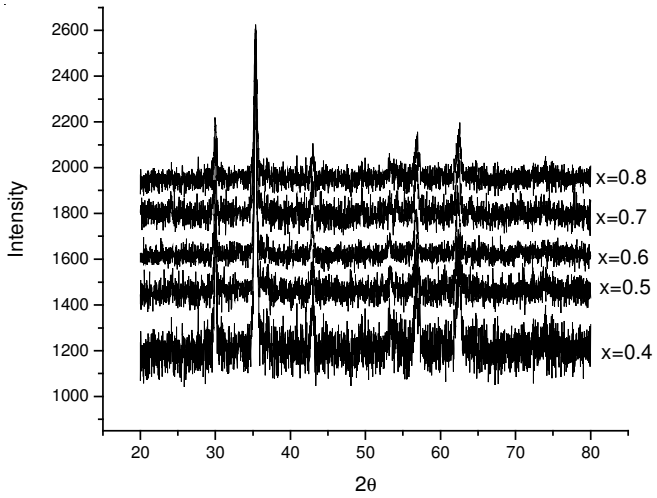

Fig. 1. XRD pattern of $Mn_xZn_{(1-x)}Fe_2O_4$ samples from mechano chemical method

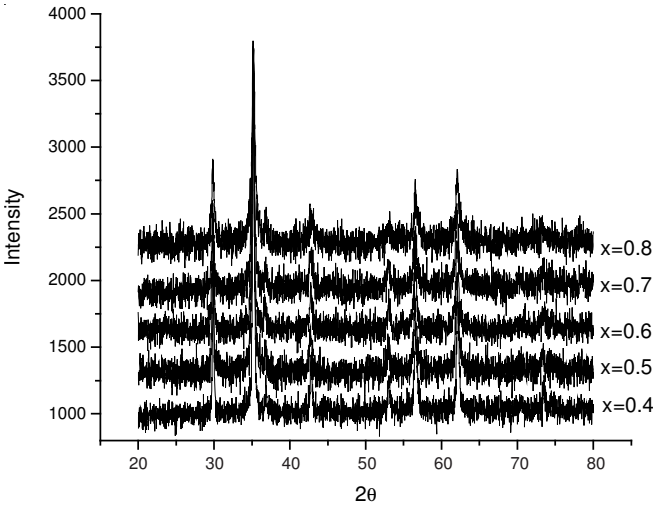

Fig. 2. XRD pattern of $Mn_xZn_{(1-x)}Fe_2O_4$ samples from wet chemical method

The lattice constants 'a' values, obtained[13] from X- ray data, were subsequently used to calculate X-ray density by using the relation $\rho_x = 8M/Na^3$, where 8 is the number of atoms in a unit cell of spinel lattice, M the molecular weight of the ferrite, 'a' is the lattice constant and N is Avogadro's number. X-ray densities for the samples obtained from mechano-chemical method (Fig. 3) are found to be lower than those of samples prepared by wet chemical method (Fig. 4). The X-ray densities depend on molecular weight and lattice constant of the ferrite sample. As expected, in both the cases, the X-ray densities are found to decrease with increase in the value of lattice constant, since 'a' is inversely proportional to the X-ray density. Also, the X-ray density decreases with increase in Mn content. Lattice constant 'a' for the samples is directly proportional to Mn content in the samples as reported earlier[11,12].

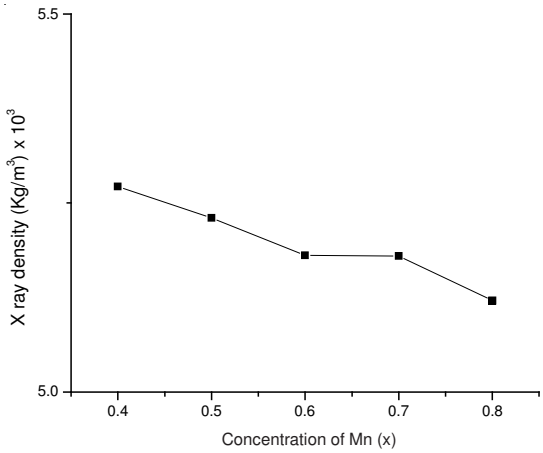

Fig. 3. X-ray density of $Mn_xZn_{(1-x)}Fe_2O_4$ samples from mechano chemical method

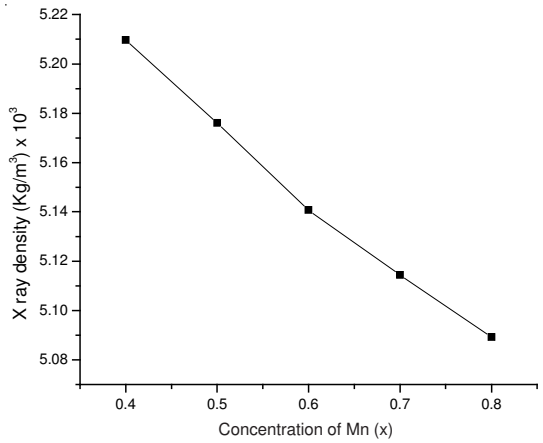

Fig. 4. X-ray density of $Mn_xZn_{(1-x)}Fe_2O_4$ of wet chemical method

The mass densities of ferrites are found to be lower for the samples prepared by the mechano-chemical method (Fig. 5) as compared to the samples obtained from wet chemical method (Fig. 6). Obviously it is due to the fact that in wet chemical method the formation of precursor occurs at ionic level and hence the compactness of the final ferrite material. This is not so in case of samples obtained from mechano-chemical method. The mass densities of ferrite samples increase with increase in Mn content which is due to the higher specific gravity of Mn (7.21 g/cm^3) as compared[14] to Zn (7.133 g/cm^3).

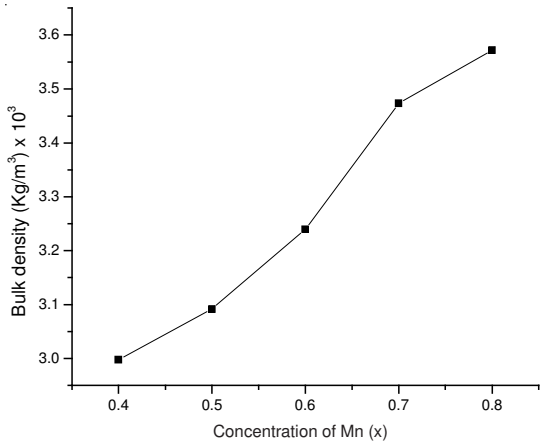

Fig. 5. Mass density of $Mn_xZn_{(1-x)}Fe_2O_4$ from mechano chemical method

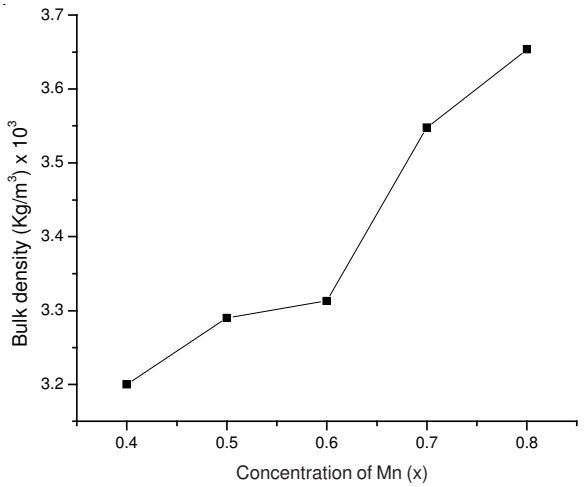

Fig. 6. Mass density of $Mn_xZn_{(1-x)}Fe_2O_4$ from wet chemical method

The particle size of the ferrite sample was calculated using Scherer's formula *i.e.* $t = 0.9\,\lambda/B\cos\theta$. The values so obtained for the samples are in the range of 16.12 nm to 28.16 nm for mechano-chemical method, where as for the wet chemical method it is in the range 16.42 to 33.54 nm. In wet chemical method involving hydrazine based ligand, additional heat is generated during the thermal decomposition of the precursor due to self-sustained exothermic auto combustion resulting in nucleation of ferrite materials. Due to this, the particle size of ferrite samples in case of the wet chemical method is larger than that of samples prepared by the mechano-chemical method.

The percentage porosity (P) was calculated using the formula: $P = 1 - (\rho/\rho_x)$, where 'ρ' is bulk density and 'ρ_x' is X-ray density[12]. Samples obtained by both methods have high porosity values. Among the two methods, ferrites obtained from the mechano-chemical method show higher porosity values ranging from 43.14 to 30.23 % (Fig. 7). And the values are in the range of 38.57 to 28.21 % for the wet chemical method (Fig. 8). The porosity is found to decrease as the ratio (ρ/ρ_x) increases with increasing Mn content.

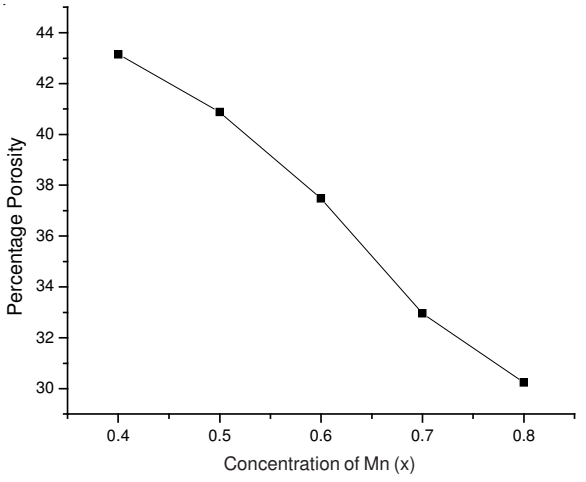

Fig. 7. Percentage porosity of $Mn_xZn_{(1-x)}Fe_2O_4$ samples prepared by mechano chemical method

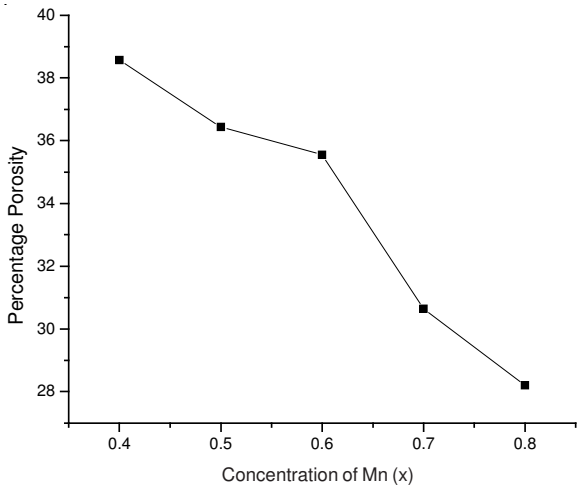

Fig. 8. Percentage porosity of $Mn_xZn_{(1-x)}Fe_2O_4$ samples prepared by wet chemical method

Conclusion

(a) The formation of single phase ferrite from precursor obtained with the same ligand in mechano-chemical method and wet chemical method. (b) Lattice constants obtained for mechano chemical method are lower as compared to wet chemical method. (c) Mass densities of ferrites obtained by the mechano-chemical method are also found to be lower in comparison to the wet chemical method. (d) Ferrites obtained by these methods have high porosity with slightly higher values in the case of mechano-chemical method.

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