

# Synthesis of Nano-Magnesium Ferrite Spinel and its Characterization

Joseph Govha ,T. Bala Narasaiah, Pramod Kumar , Ch. Shilpa Chakra  
Institute of Science and Technology,  
Jawaharlal Nehru Technological University Hyderabad  
Hyderabad, India

**Abstract**— Magnesium ferrite spinel has unique physical and chemical properties. They find applications in catalysis, adsorption, bio-processing, medicine, magnetic recording devices etc. The Nanoparticles synthesized were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Thermo Gravimetric-Differential Thermal Analyzer (TGA-DTA). Studies showed that, spherical nano-crystalline magnesium ferrite with cubic structure was synthesized by co-precipitation method. SEM images showed that particles size range was from 86 to 161nm. The nanoparticles were prepared from hydrated iron sulphate and hydrated magnesium sulphate as precursors at room temperature.

**Keywords**— *co-precipitation; magnesium ferrite; nanoparticles; nano-crystallin.*

## I. INTRODUCTION

The materials classified as ferrite spinel are very important because they have wide technological applications due to their interesting magnetic, electrical and chemical properties as well as thermal stability. The general formula for ferrite spinel is  $(M_{1-x}Fe_x)[M_xFe_{2-x}]O_4$ . The metal element M stands for divalent element which can be Mg, Zn, Mn, Fe, Co, Ni, or mixture of them [1, 2]. In the case where M is Mg we will have a Magnesium ferrite. The x in general formula stands for degree of inversion and the two extremes are normal where  $(x=0)$  and inverse were  $(x=1)$ , there are also intermediate mixed states [1]. The normal spinel magnesium ferrite ( $MgFe_2O_4$ ) is one of the important ferrites, it is realised when  $(x=0)$ , it has a cubic structure and it is soft magnetic n-type semiconducting material [3, 5]. The magnesium ferrite spinel find use in microwave devices, magnetic recording media, transformer cores, noise filters, rod antennas and in addition, they are very important in heterogeneous catalysis, adsorption, sensors and cancer treatment [1-5,11]. The following variables: thermal treatment, type of precursors, molar ratio and synthesis route affect cation distribution in the lattice of magnesium ferrite hence great care should be taken during synthesis. The cation distribution affects the properties of magnesium ferrite and its areas of applications [4].

The synthesis of nanoparticles as compared to their bulk counter parts have attracted wide attention in research, due to the unique properties that are exhibited by nanomaterials because of their limited size. There are a number of synthesis routes that are aimed at producing magnesium ferrite spinel with specific properties. The methods are gas condensation,

rapid solidification, Electro-deposition, wet chemical methods like co-precipitation, hydrothermal, sonochemical reactions, sol-gel method, combustion, micro-emulsion, and high energy ball milling [1-7]. The co-precipitation process is pH sensitive but it's simple and cost effective, sol-gel technique is sophisticated and requires expensive alkoxide precursors, combustion method has low processing time and low operating temperature, micro-emulsion has easy control of particle size and overall homogeneity. Furthermore, wet-chemical routes require high temperature to obtain the final product [4, 5].

The synthesis route plays an important role on the physical, chemical, structural and magnetic properties of spinel ferrite [5]. In the present work co-precipitation at room temperature was employed to produce magnesium ferrite spinel ( $MgFe_2O_4$ ) because it is simple and cost effective. The resulting properties, size, structure and morphology, of the synthesised magnesium ferrite spinel were characterized using X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Thermal Gravimetric-Differential Thermal Analyzer (TGA-DTA).

## II. EXPERIMENTAL SECTION

### A. Materials

All reagents were of analytical grade and they were used without further purification: Iron sulphate ( $FeSO_4 \cdot 7H_2O$ ); Magnesium sulphate ( $MgSO_4 \cdot 7H_2O$ ); distilled water; Sodium Hydroxide (NaOH).

### B. Synthesis of Magnesium ferrite spinel

Magnesium ferrite spinel was synthesized by co-precipitation method at room temperature. 0.1M each of  $FeSO_4 \cdot 7H_2O$  and  $MgSO_4 \cdot 7H_2O$  was prepared and mixed. The mixed solution was stirred for 30 minutes using magnetic stirrer at 450 rpm. A burette was used to deliver NaOH to the mixed solution drop wise while stirring. Precipitant was added until a pH of 9.5 was achieved then stirring was continued for another 1hr. The supernatant liquid was left in contact with precipitate overnight for aging. The precipitant was then filtered and washed with distilled water 3 times. Drying of the filtrate was done at  $80^\circ C$  for 2hrs. The precipitate was then calcined at  $300^\circ C$  for 3hrs.

### C. Characterisation

The XRD diffraction (Shimadzu, 700N), were recorded using Cu-K $\alpha$  (1.5406Å) monochromatic radiation source, operating voltage and current maintained at 40kV and 30mA respectively in the 2 $\theta$  range 10-80 $^{\circ}$ . Thermo-gravimetric analysis and differential thermal analysis (TGA-DTASII EXSTAR 6300R, Japan) was performed using nitrogen at a heating rate of 10 $^{\circ}$ Cmin $^{-1}$ . Scanning electron microscopy (SEM) analysis (HITACHI S-3700N) was used to study morphology of the particles.

## III. RESULTS AND DISCUSSION

### A. X-Ray Analysis Magnesium ferrite spinel

The XRD for magnesium ferrite is shown in figure 1. The crystalline size (D) of the synthesized nanoparticles was measured using Scherer's equation

$$D = 0.9\lambda / \beta \cos\theta \quad (1)$$

Where,  $\lambda$  is the x-ray wavelength,  $\beta$  is equal to full width half maximum (FWHM), and  $\theta$  is Bragg angle. The average crystalline size after considering all the major peaks is 25.6nm. An analysis of the XRD revealed that synthesized material is composed of diffraction peaks that agree with the international standard diffraction data card, JCPDS (89-4924) and provides evidence for formation of MgFe $_2$ O $_4$ . The most intense reflections observed at 2 $\theta$  values are at 30.38, 35.70, 43.39, 53.92, 57.40, 63.10, and 74.6 degrees are assigned to (220), (311), (400), (422), (511), (440), and (533) planes of cubic spinel structure. The findings above indicate that synthesized material is crystalline and lattice parameter for each peak is shown in Table 1. The values of lattice parameter are almost equal an indication that the material is indeed cubic. The lattice parameter values agree with those obtained by Maensiri et al. [2]

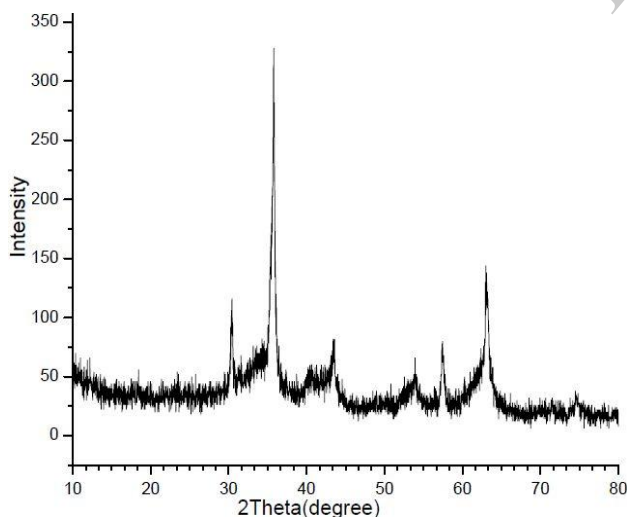


Figure1: X-ray diffraction for magnesium ferrite spinel

Table 1: Planner distances and lattice parameter

Miller indices	2( $\theta$ )	d (Planner distances) (Å)	a (lattice parameter) (Å)
220	30.38	2.9381	8.3102
311	35.70	2.5115	8.3299
400	43.39	2.0825	8.3299
422	53.92	1.6980	8.3185
511	57.40	1.6031	8.3302
440	63.10	1.4713	8.3231
533	74.60	1.2705	8.3313

### B. SEM studies Magnesium ferrite spinel

The SEM photomicrograph contains information about the synthesized materials surface topology and size [10]. The morphology of Magnesium ferrite spinel (MgFe $_2$ O $_4$ ) particles was studied using SEM under high vacuum and suspended in ethanol the image is shown in figure 2. The image shows that the synthesized particles are spherical and they agglomerates. The particle size from SEM varies from 86 to 161nm this is much bigger than crystallite size obtained from XRD using Scherer's equation (1).

### C. TGA-DTA Analysis Magnesium ferrite spinel

Thermo-gravimetric study for magnesium ferrite spinel is shown in figure 3. The thermal study was done from room temperature to 900 $^{\circ}$ C in nitrogen atmosphere with a heating rate of 10 $^{\circ}$ C/min. The study of weight loss with temperature increase is important in determining absorption of water, sample purity, carbonate content, removal of organic impurity, change of structure and the decomposition reactions [5]. The graph in figure 3 shows a total weight loss of 16.52%. The weight loss can be divided into two categories the first which has a weight loss of 15% is from room temperature to 600 $^{\circ}$ C shows more or less a gradual weight loss pattern in that range. The weight loss can be attributed to dehydroxylation or chemisorbed water.

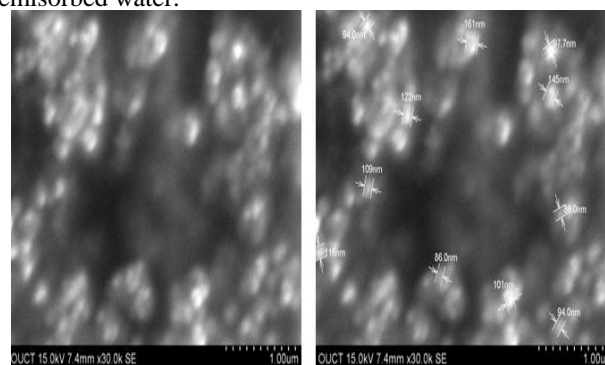


Figure 2: SEM images for magnesium ferrite spinel

The second category only shows a slight weight loss of 1.52% which could mean that in the range 600 $^{\circ}$ C. The second category only shows a slight weight loss of 1.52% which could mean that in the range 600 $^{\circ}$ C to 900 $^{\circ}$ C there could be a structure change and formation of pure spinel magnesium ferrite. The percentage weight loss for the synthesized nanomaterial could indicate the amount of hydroxyl groups present since the TGA is for calcined material hence it can be assumed that all the physically absorbed water had been driven off. The low percentage of the weight loss for the temperature range agrees with above statement. The amount

of hydroxyl groups lost can indicate the ability of a material to have high fluoride adsorption capacity because hydroxyl sites are active sites for fluoride adsorption [8, 9].

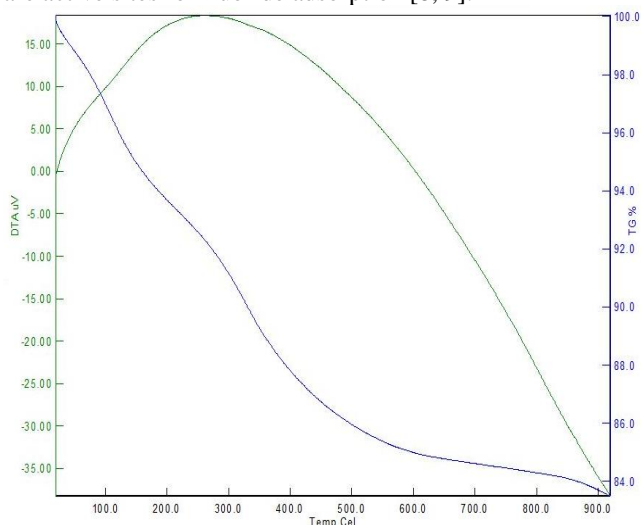


Figure 3: TGA and DTA curves for  $\text{MgFe}_2\text{O}_4$

#### IV. CONCLUSION

Magnesium ferrite spinel ( $\text{MgFe}_2\text{O}_4$ ) particles with a spherical shape and a crystalline size of 25.6 nm were successfully synthesized using co-precipitation method. Studies from TGA-DTA shows that the produced ferrite still had some hydroxyl in the structure this is supported by weight loss of 16.52%. The XRD shows that the produced spinel is not entirely pure  $\text{MgFe}_2\text{O}_4$  as it shows the existence of amorphous phase in the synthesized nanoparticles. There is need to increase calcination temperature or calcination time in order to have completely pure crystalline magnesium ferrite spinel. There is high probability that the synthesized magnetic magnesium ferrite spinel can be applied in different areas like catalysis, Catalyst support, sensing equipment, adsorption processes and biomedical application (cancer).

#### ACKNOWLEDGMENT

We wish to gratefully thank Osmania University College of Technology for the SEM AND XRD. We would also want to thank Head Centre for Chemical Sciences & Technology and Centre for Nano Science & Technology for the invaluable support in conducting this.

#### REFERENCES

- [1] Swapan Kumar Pradhan, Sumanta Sain, and Hema Dutta, Microstructure characterization of nanocrystalline magnesium ferrite annealed at elevated temperatures by rietveld method, International Scholarly Research Network ISRN Ceramics Volume 2011, Article ID 194575
- [2] Santi Maensiri, Montana Sangmanee, and Amporn Wiengmoon, Magnesium ferrite ( $\text{mgfe}_2\text{o}_4$ ) nanostructures fabricated by electrospinning, Nanoscale Res Lett. 2009; 4(3): 221–228.
- [3] M. M. Hessien, Z. I. Zaki, Q. Mohsen, Synthesis of nanocrystalline magnesium ferrites powder from mill scale, IJMS Vol.1 Iss.1 2011 PP.30-34
- [4] N. M. Deraz, and Omar H. Abd-Elkader, Effects of magnesia content on spinel magnesium ferrite formation, Int. J. Electrochem. Sci., 8 (2013) 8632 – 8644
- [5] S. Kanagesan, M. Hashim, S. Tamilselvan, N. B. Alitheen, I. Ismail and G. Bahmanrokh, Cytotoxic effect of nanocrystalline  $\text{mgfe}_2\text{o}_4$  particles for cancer cure, Hindawi Publishing Corporation Journal of Nanomaterials Volume 2013, Article ID 865024,
- [6] Mohsen Ahmadipour, Mozghan Hatami, Kalagadda Venkateswara Rao, Preparation and characterization of nano-sized  $(\text{mg}(x)\text{fe}(1-x)\text{o})/\text{siO}_2$  ( $x = 0.1$ ) core-shell nanoparticles by chemical precipitation method, *Advances in Nanoparticles*, 2012, 1, 37-43
- [7] Mohamed I. M. Omer, A.A.Elbadawi, O. A. Yassin, Synthesis and structural properties of  $\text{mgfe}_2\text{o}_4$  ferrite nano-particles, Journal of Applied and Industrial Sciences, 2013,(4): 20-23
- [8] Lin Chen, Bo-Yang He, Shuai He, Ting-Jie Wang, Chao-Li Su, Yong Jin, Fe–Ti oxide nano-adsorbent synthesized by co-precipitation for fluoride removal from drinking water and its adsorption mechanism, *Powder Technology* 227 (2012) 3–8
- [9] S.K. Swain, Tanushree Patnaik, P.C. Patnaik, Usha Jha, R.K. Dey, Development of new alginate entrapped Fe(III)–Zr(IV) binary mixed oxide for removal of fluoride from water bodies, *Chemical Engineering Journal* 215–216 (2013) 763–771
- [10] N. Minju, K. Venkatswaroop, K. Haribabu, V. Sivasubramanian and P. Senthil Kumar, Removal of fluoride from aqueous media by magnesium oxide-coated nanoparticles, *Desalination and water treatment*, (2013), 1-10, Taylor and Francis
- [11] M. Raghasudha, D. Ravinderand, P. Veerasomaiah, Characterization of nano-structured magnesium chromium ferrites synthesized citrate-gel auto combustion, *Advanced materials letters* (2013)