Preparation of Nano Particle $Mg_{0.2}Fe_{0.8}O$ by Solution Combustion Method and Their Characterization

Mohsen Ahmadipour, K.Venkateswara Rao

Abstract*— The present paper investigated a preparation of Nano sized $Mg_{0.2}Fe_{0.8}O$ by eco-friendly chemical combustion synthesis using magnesium and iron nitrates as oxidizers and the glycine as a fuel to drive the reaction. Powder characterization was carried out by X-ray diffraction (XRD) to estimate crystallite size, Particle Size Analyzer (PSA), Thermal gravimetric analyzer (TGA) for determining sample purity, weight loss percentage and decomposition reaction, Transmission electron microscope and Scanning electron microscope(SEM, TEM) were done to evaluation the morphology and average particle size. In this study are useful for establishing a simple method for the preparation of $Mg_{0.2}Fe_{0.8}O$ Nano powders.

***Index Terms*— Solution Combustion Synthesis, $Mg_{0.2}Fe_{0.8}O$, XRD, SEM, TEM**

I. INTRODUCTION

Magnesium ferrite ($Mg_{0.2}Fe_{0.8}O$) is one of the most important ferrites. It has a cubic structure which finds a number of applications in heterogeneous catalysis, adsorption, sensors, and in magnetic technologies [1]. Recently, nanostructures of magnetic materials have received more and more attention due to their novel material properties.

Several physical and chemical methods are used to synthesize $Mg_{0.2}Fe_{0.8}O$ Nano-powder, such as co-precipitation, sol–gel, ball milling, solid-state reaction, hydrothermal, polymerization, spray drying and freeze drying, micro emulsion and reverse micelle method [2–9]. Different synthesis methods will show different properties in product.

All these fuels serve two purposes:(a).They are the source of C and H, which on combustion form CO and H O and liberate heat.(b).They form complexes with the metal ions facilitating homogeneous mixing of the cations in solution.

The purpose of this study was to investigate the crystallite size, particle size and morphology of $Mg_{0.2}Fe_{0.8}O$ obtained by simple synthesis method. The rest of the paper has been structured as follows: Feature extraction of the test cases is discussed in section 2. In section 3, not only was analysis of data investigated, but also morphology of sample has been proposed. The last part concludes the paper.

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Mohsen Ahmadipour, M.tech Department of Nanotechnology, Center for Nano Science and Technology, Institute of Science and Technology Jawaharlal Nehru University, Hyderabad, India.

K.Venkateswara Rao, Head of Department of Nanotechnology, Center for Nano Science and Technology, Institute of Science and Technology Jawaharlal Nehru University, Hyderabad, India.

II. EXPERIMENTAL DETAILS

In this study, samples with the formula $Mg_{0.2}Fe_{0.8}O$ were synthesized via the solution combustion synthesis (SCS) under different molar compositions of fuel Ψ=1 and Ψ=1.25. The materials used as precursors were (i) iron nitrate nonahydrate p. a., $Fe(NO_3)_3.\ 9H_2O$; `(ii) magnesium nitrate hexahydrate p. a., $Mg(NO_3)_2.\ 6H_2O$ (both were procured from MERCK Ltd .India); and (iii) glycine p. a., NH_2CH_2COOH (Nuclear brand). All of them were of high purity (98%, 98%, and 99%, respectively). The water used was deionized water.

The iron and magnesium nitrates, According to Table [1], were dissolved in a beaker with sufficient deionized water, and the solution was placed under thermal stirring. The mixture was put on the hot plate. As the temperature reached 100°C, water started to boil and evaporate from the solution, which increased solution viscosity substantially, during which the compound caught fire. Finally, the light weight Brown powder which is the precursor were remain. The obtained precursor and precursor annealed for 2 h at 300°C were both characterized.

Table.1. Iron, Magnesium and fuel concentration table

Sample	X	Formula	Fe	Mg	Fuel	Ψ
M1	0.2	$Mg_{0.2}Fe_{0.8}O$	4.315	0.685	2.777	1
M2	0.2	$Mg_{0.2}Fe_{0.8}O$	4.315	0.685	3.471	1.25

The mixture ignited as soon as the gases were eliminated producing a typical flame as shown in Fig. 1. The reaction was quite fast (less than 30 min) and produced dark brown free-flowing powder that responded easily to a magnet.

The crystal phase of the synthesized powders were determined by X-ray diffraction (XRD, BrukerD&Advance, Germany) using Cu Kα as radiation source (40 kV, step size 0.02, scan rate 0.5 min^{-1}). The particles size measured by Nano Particle Size Analyzer (SZ-100 Nanoparticle, Horiba, Japan). The thermal decomposition behaviors of the precursors were investigated by thermo gravimetric analysis (TGA/DTA, A6300R, Japan). Fourier transform infrared spectroscopy_was used as well (FTIR,PERKIN ELMER, India).The particles' size and morphology of the synthesized powder were examined by Scanning electron microscope (SEM,S-3400N-hitachi-Japan) transmission electron microscope (TEM,JEM-2100 , Jeol).

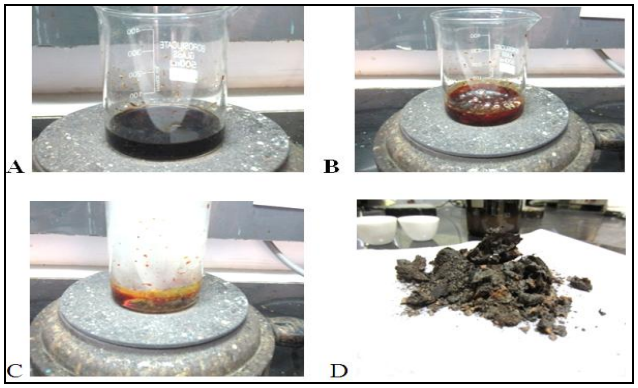

Fig. 1 Stages of combustion reaction of magnesium ferrite Liquid solution of magnesium nitrate, iron nitrate, and urea at room temperature. From (**B**) to (**D**) the hot blanked temperature is c.a. 300°C.(**B**) water started to boil and evaporating from the solution, which substantially increased solution viscosity. (**C**)The emerging intensive flame indicates that combustion reaction has been started. The reaction lasts less than 30 min. (**D**) The combustion reaction is entirely completed and the final product is powder of nanoparticles of $Mg_{0.2}Fe_{0.8}O$.

III. RESULT AND DISCUSSION

A. Crystal structure of product

The XRD pattern of the $Mg_{0.2}Fe_{0.8}O$ Nano particles obtained from solution combustion synthesis for sample (M_1), (M_2) are related to different Ψ values (Ψ equal to 1 and 1.25) respectively are as shown in Fig.2. This result shows that the structure of the $Mg_{0.2}Fe_{0.8}O$ Nano particles is in cubic phase.

The crystallite size of $Mg_{0.2}Fe_{0.8}O$, present in the investigated was based on X-ray diffraction line broadening and calculated by using full-width at half-maximum (FWHM) of the strongest diffraction peak by making use of the Scherrer formula [19],then the average were extracted.

$$D = \frac{0.9\lambda}{\beta cos\theta} \quad (1)$$

Where D is the crystallite size, λ is the wavelength of Cu-Kα radiation and β is full-width at half maximum (FWHM) of the XRD all peaks.

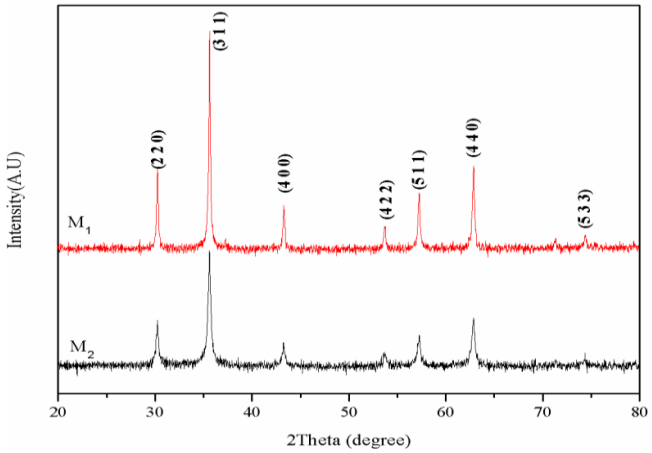

Fig.2. XRD pattern for $Mg_{0.2}Fe_{0.8}O$ nanopowder synthesized by Solution Combustion Synthesis method (a): Ψ= 1, (b): Ψ= 1.25.

The average grain sizes of the samples were calculated using the Scherer's formula as 34 and 28 nm for sample (M_1, M_2) respectively. According to JCPDS#89-4924, the obtained phase has a cubic structure whose space group is Fd3m.The existence of a peak around the diffraction angle (2h) equal to 35° corresponding to (311) plane confirms the formation of spinel ferrites.

The size of the Nano powders in this analysis measured with Nano Particle Size Analyzer (SZ100) the average particle sizes for Ψ= 1 and Ψ = 1.25 were 71 nm and 70 nm, respectively. Figure 3,4 Shows those $Mg_{0.2}Fe_{0.8}O$ particles possess a narrow distribution with an average effective diameter of 40 nm. This result agreed with the measure of TEM. The size of $Mg_{0.2}Fe_{0.8}O$ in Figure 6(b) became bigger, and its size distribution ranged from 21 to 82nm with one intensive peak at 40 nm.

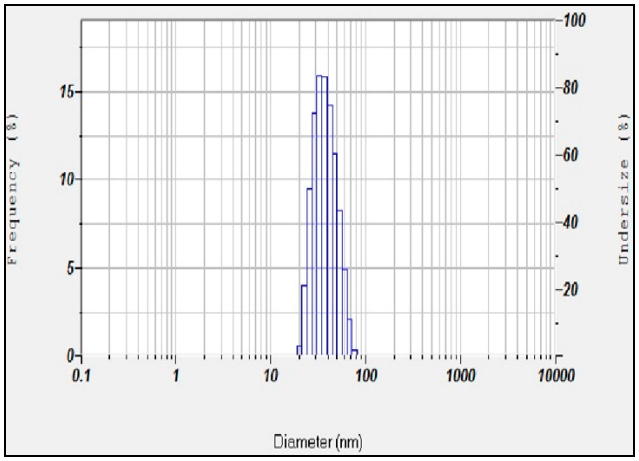

Fig.3. Results of Nano Particle Size Analysis for $Mg_{0.2}Fe_{0.8}O$ synthesized by Solution Combustion Synthesis method, Ψ= 1

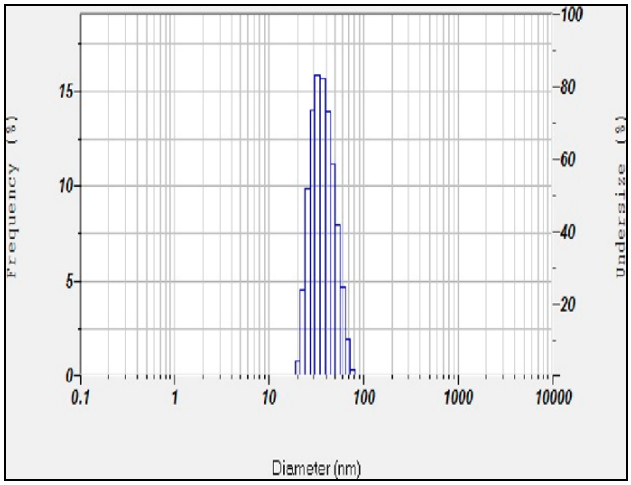

Fig.4. Results of Nano Particle Size Analysis for $Mg_{0.2}Fe_{0.8}O$ synthesized by Solution Combustion Synthesis method, (b) Ψ = 1.25.

B. Thermal properties

TGA curves of $Mg_{0.2}Fe_{0.8}O$ Nano particles for data of different Ψ value (Ψ = 1, 1.25) are as shown in Fig.5. The temperature range is 50°C to 800°C. After combustion,

some amount of water remains in sample which should be removed by calcination of product. Thermal analysis of $Mg_{0.2}Fe_{0.8}O$ samples were carried out in air atmosphere. It is well known that materials with a cubic crystal structure are prone to grow into a spherical shape [10-11] to minimize the surface tension.

Thermo gravimetric is measures the mass of a sample as the temperature increasing. This method is useful for determining sample purity of water, carbonate and organic content and for studying decomposition reaction. TG curve shows consistent weight loss as it is illustrated in Fig 5**.** Among the samples which are placed in TG-DTA after calcination less weight loss and impurity were observed.

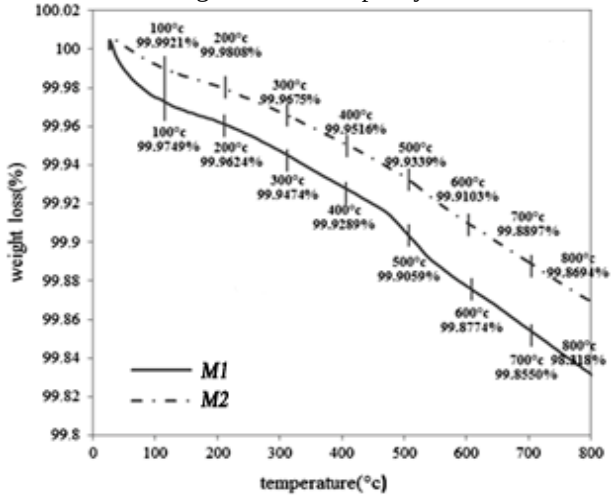

Fig.5. XRD pattern for $Mg_{0.2}Fe_{0.8}O$ Nano powder synthesized by Solution Combustion Synthesis method (a): Ψ= 1, (b): Ψ = 1.25.

C. Morphology of product

The grain size, shape and surface properties like morphology were observed by the SEM, TEM as shown in Fig. 5. Fig. 5(a, b) shows that the particles have high porous structure like structure exhibit spherical granules. TEM can be used to measure the particle size of individual particles, which is one of the most basic parameters in nanoparticle research. Transmission electron microscopy (TEM) shows the particle's morphology, distribution and its size. The particle size estimated from TEM is smaller than the crystallite size estimated from XRD using Dybe- formula. Fig. 5(c, d) shows that the particles are in spherical shape.

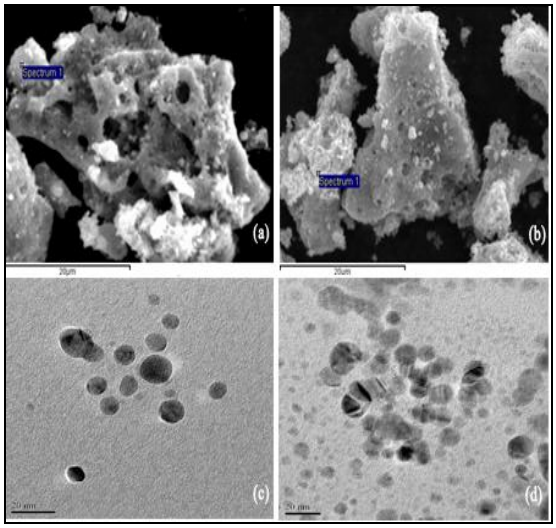

Fig. 6 (a, b)SEM micrograph of sample , scale bar 20µm, (M1) Ψ = 1, (M2) Ψ = 1.25,(c, d) TEM image of the samples, scale bar 20nm, (M1) Ψ = 1, (M2) Ψ = 1.25

IV. CONCLUSION

The porous Nano crystalline $Mg_{0.2}Fe_{0.8}O$ with average particle size of around 28nm, 34nm was have been synthesized with two different amount of Ψ value as Ψ = 1 and Ψ= 1.25 by Novel low temperature initiated, self-propagating and gas producing solution combustion process. It was observed from XRD pattern that $Mg_{0.2}Fe_{0.8}O$ nanoparticles have cubic spinel Fd-3m structure, from SEM image that has porous structure as it is shown in Fig.5(a, b) and from TEM image that has spherical morphology of particles as illustrated in Fig.5(c, d).

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