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SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE SPINEL MAGNESIUM FERRITE PREPARED BY SOL-GEL METHOD

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Abstract : Magnesium ferrite ($MgFe_2O_4$) nanoparticles were synthesized using a sol-gel synthesis method using citric acid as anionic surfactant and synthesized particles were calcinated at 700°C. X-ray diffraction data confirm the formation of single-phase cubic structure and the average grain sizes were evaluated. The XRD result revealed the production of a sharp single cubic spinel structure of prepared sample without any impurity peak with the crystallite size of about 33.29 nm. The high and low frequency absorption bands of $MgFe_2O_4$ were investigated using FT-IR analysis. The microstructural features were examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) and compositional analysis carried out by energy dispersive X-ray analysis (EDS).

Keywords - Nano ferrite, XRD, FT-IR, SEM, EDX, TEM.

1.INTRODUCTION

Nanocrystalline materials have attracted considerable interest due to their advanced technological progresses in nanoscience extend to different fields, such as biology, chemistry, medicine, pharmacy, agriculture, food industry, and also materials science among others. While comparing with their bulk materials, nanomaterials have particle size in the range of 1–100 nm and a high surface to volume ratio that determines different or enhanced reactivity, thermal, mechanical, optical, electrical, and magnetic properties [1]. Even though in the case of bulk materials, the chemical composition is the important factor that determines their properties, in the case of nanomaterial, besides the chemical composition, the particle size and morphology determine most of their characteristics [1, 2]. Moreover, these properties can be tuned based on the particle size and chemical composition [1, 2].

From practical as well as fundamental point of view, the ferrites are important and interesting nanomaterials. As an important member of ferrite family, spinel ferrite has unique attraction due to their vast applications. Among various types of ferrites, nanosized $CoFe_2O_4$, $MnFe_2O_4$, $ZnFe_2O_4$, $NiFe_2O_4$, and $CuFe_2O_4$ have attracted considerable attention due to their chemical and thermal stability and unique structural, optical, magnetic, electrical, and dielectric properties and also, wide potential technological applications in photoluminescence, photocatalysis, humidity-sensors, biosensors, magnetic drug delivery, catalysis, permanent magnets, magnetic refrigeration, magnetic liquids, microwave absorbers, water decontamination, ceramics pigment, corrosion protection, antimicrobial agents, and biomedicine (hyperthermia) [1, 3–8].

Among the various metal ferrites, magnesium ferrite ($MgFe_2O_4$) enjoys a special attention because of its vast applications in high density recording media, heterogeneous catalysis, adsorption, sensors and magnetic technologies. $MgFe_2O_4$ [9] is a well-known ferrite and is rarely studied. It possesses partially inverse structure which depends on synthesis method and synthesis conditions. In the literature, very few reports are available for the synthesis and characterization of $MgFe_2O_4$ [10].

Moreover, a number of chemical, physical and biological methods have been employed to create nanoparticles which are later optimized for different applications. Some of the interesting and important methods include mechanical milling [11], co-precipitation [12], aerosol route [13], hydrothermal reaction [14], oxidative precipitation [15], sonochemical decomposition [16], and sol–gel technique [17].

Among these methods, the sol-gel method is a simple, low cost, and environmentally friendly method to prepare nanomaterial as it allows good control of the microstructure, particle size, dispersion, structure, and chemical composition by carefully monitoring the preparation parameters [18-20]. The sol-gel method has been used to prepare very fine, highly dense, homogenous, and single-phase ferrite nanoparticles.

Considering the importance of $MgFe_2O_4$ in various technological applications, it was decided to study the nanocrystalline magnesium ferrite prepared by simple sol-gel method and to evaluate their structural properties. In this paper we report our results on the synthesis and structural characterizations of magnesium ferrite. Various characterization techniques have been carried out using X-ray Diffraction (XRD), (FT-IR), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX), transmission electron microscopy (TEM) and the results are presented herein.

2. Experimental

Nanocrystalline spinel structured Mg-ferrite ($MgFe_2O_4$) has been prepared by sol-gel method using citric acid. All chemicals were of AR grade and were used without further purification. Ferric nitrate [$Fe(NO_3)_3.9H_2O$], Magnesium nitrate [$Mg(NO_3)_2.6H_2O$], Zinc nitrate [$Zn(NO_3)_2.6H_2O$] and Citric acid [$C_6H_8O_7.H_2O$] were used for the synthesis.

In this sol-gel method, all the reagents were weighed separately and the metal nitrate to citric acid ratio was taken as 0.5M:0.25M:0.5M. These reagents were dissolved in distilled water and volume made up to 100 ml. The solution mixture was stirred and heated at 60ºC for 3 h and followed by 80 ºC, until the mixture changed to gel form. The gel was then dried in an oven at 100ºC for 24 h and followed by calcined in a muffle furnace at 700ºC for 2 h. Taken solid phase sample was grinded in a mortar to make it powder.

The synthesized powder and a solution of ethyl cellulose (a temporary binder) were mixed together with butyl cellulose, butyl carbitol acetate and turpineol in order to obtain the paste. Pastes incorporating mass percentage ratio of 80:20, with $MgFe_2O_4$ and the binder respectively were ground in an agate pestle and mortar with for 1 h. This paste was screen printed onto glass substrate surface in desired patterns. This thick films was allowed to dry for 24 h at room temperature and heat treatment was given to the film at 500°C for 1 h.

3. Result and discussions

3.1 XRD Analysis

Figure 1 exhibits X-ray Diffraction pattern of $MgFe_2O_4$ powder calcinated at 700ºC. Main peaks were found at 2θ values = 29.90º, 35.21º, 56.58º, 62.14º and 73.48º which were identified as corresponding to Miller index (220), (311), (400), (511) and (440) respectively, (JCPDS card no. 73-2211). The XRD pattern indicate the presence of sharp peaks, hence $MgFe_2O_4$ calcinated at 700ºC was crystalline in nature. The crystallite size (D) was calculated from peak broadening using the Debye Scherrer approximation,

$$D = \frac{K\lambda}{\beta\cos\theta}$$

Where, D is the average size of the crystallite, assuming that the grains are spherical, K is 0.9, λ is the wavelength of X-ray radiation, β is the peak full width at half maximum (FWHM) and θ is the angle of diffraction.

From XRD patterns of Fig. 1 indicate the formation of single phase fcc spinel structure with no extra lines corresponding to any other crystallographic phase. The crystallite size is found to be 33.29 nm. The lattice constant was reported as 8.402 (Aº) and other structural parameters are in the reported range.

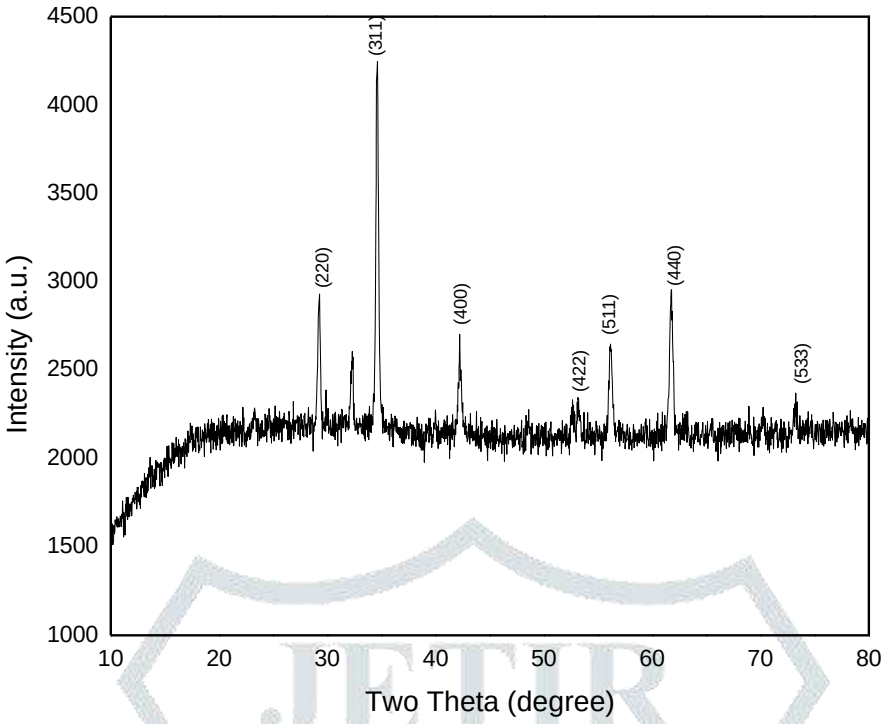

fig. 1: x-ray diffraction pattern of $mgfe_2o_4$ powder calcinated at 700°c.

3.2 FT- IR Analysis

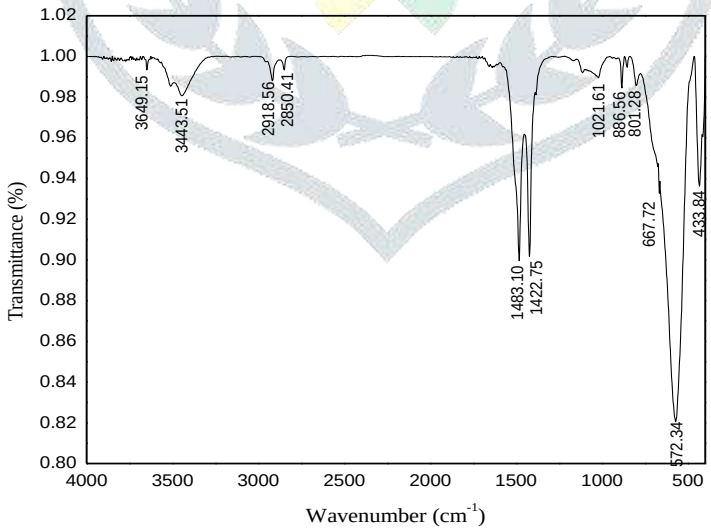

fig. 2: ft-ir spectrum of $mgfe_2o_4$ powder calcinated at 700°c.

Figure 2 shows the FT-IR spectra of $MgFe_2O_4$ nanoparticle. Vibrations of ions in the crystal lattice are usually observed in the range of 4000 - 400 cm^{-1} in IR analysis. Two main broad metal oxygen bands are seen in the IR spectra relative to spinel ferrite compounds. The highest one observed in the range 572.34 cm-1, corresponds to intrinsic stretching vibrations of the metal at the tetrahedral site, whereas the lowest band usually observed in the range 433.84 cm^{-1}, is assigned to octahedral-metal stretching. The Mg^{2+} ions occupy mainly the octahedral sites but fraction of these ions may be migrated into tetrahedral sites. This would explain the existence of a weak shoulder in the range of 690 –710 cm^{-1}. This confirms that the Mg ferrite has a partially inverse spinel structure.

3.3 SEM Analysis

The morphological characteristics of the obtained $MgFe_2O_4$ thick film were investigated by the scanning electron microscopy and are shown in Fig 3. Particle size of $MgFe_2O_4$ thick film was found due to the aggregation and coalescence during desiccation. It can be seen the particle morphology of high resolution the particle are most irregular in shape with a grain size is in the range of 40-170 nm.

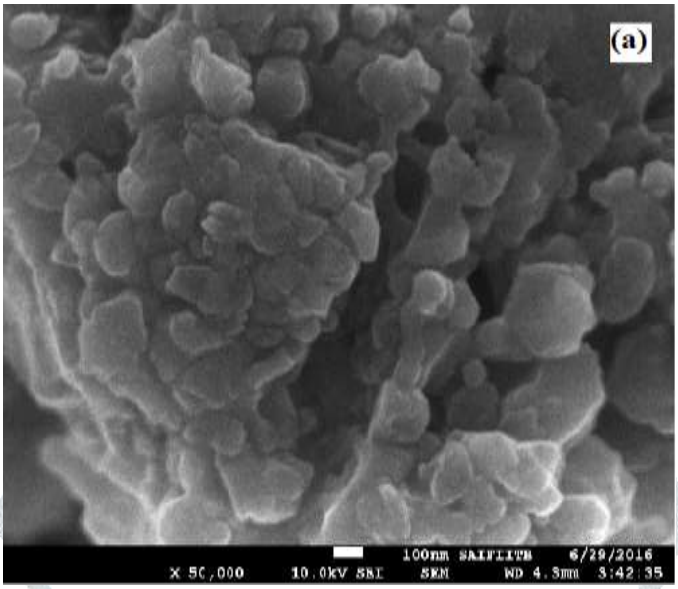

fig. 3: scanning electron micrograph of nanocrystalline $mgfe_2o_4$

3.4 Quantitative Elemental Analysis EDX

EDX spectrum of magnesium ferrite is shown in Fig. 4. Figure shows the peaks of Mg, Fe, and O elements in $MgFe_2O_4$. The observed percentage of Mg/Zn value matches well with amount of Mg/Zn used in the respective precursors. It is interesting to note that the preparation condition completely favors the formation of $MgFe_2O_4$ and allow us to study the effect on the properties of $MgFe_2O_4$. The above mentioned results confirm the formation of $MgFe_2O_4$ phase.

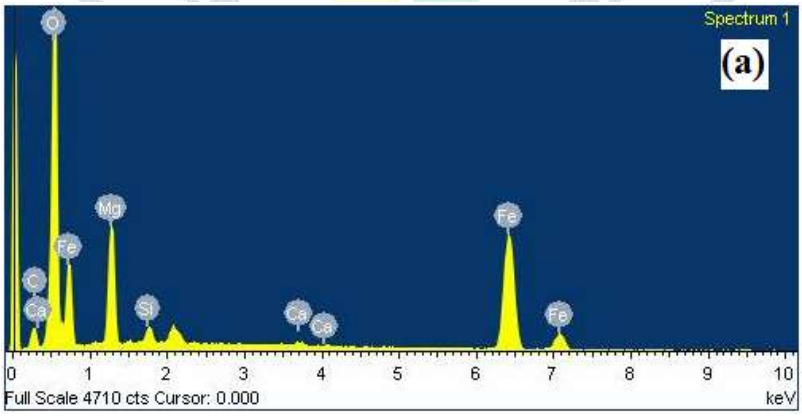

fig.4: edx of nanocrystalline $mgfe_2o_4$

3.5 TEM

The TEM image of $MgFe_2O_4$ synthesized at 700°C is presented in Fig 5. Evidently, the magnesium ferrite nanoparticles resulted from the sol- gel method was uniform in terms of morphology and crystallite size and reached the particle size of approximately 30 nm.

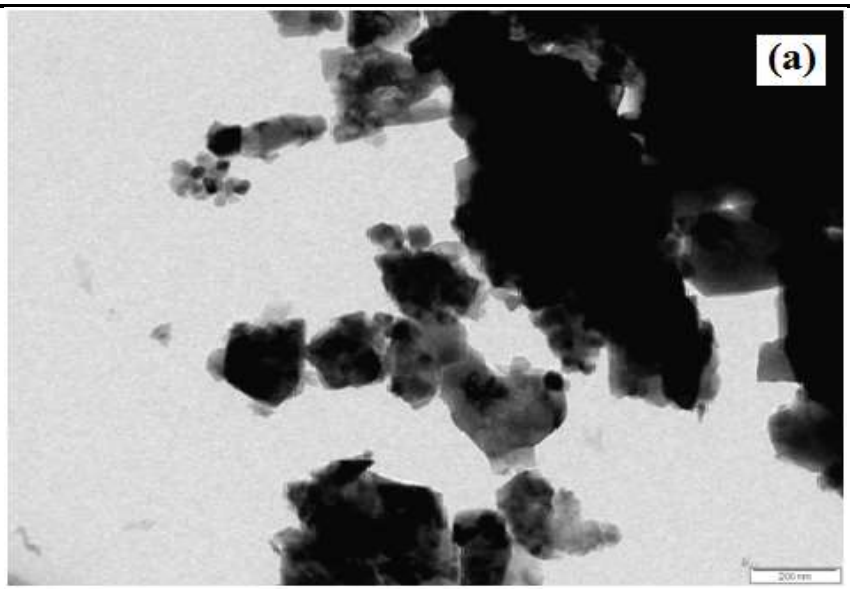

fig. 5: tem image of nanocrystalline mgfe$_2$o$_4$

3.6 SAED

The selected area electron diffraction (SAED) pattern in Fig 6 exhibits presence of bright rings corresponding to lattice planes of $MgFe_2O_4$ structure and is in good agreement with the X-ray diffraction pattern. It is observed that the spot type pattern which is indicative of the presence of single crystallite particles and no evidence was found for more than one pattern, suggesting the single-phase nature of the material.

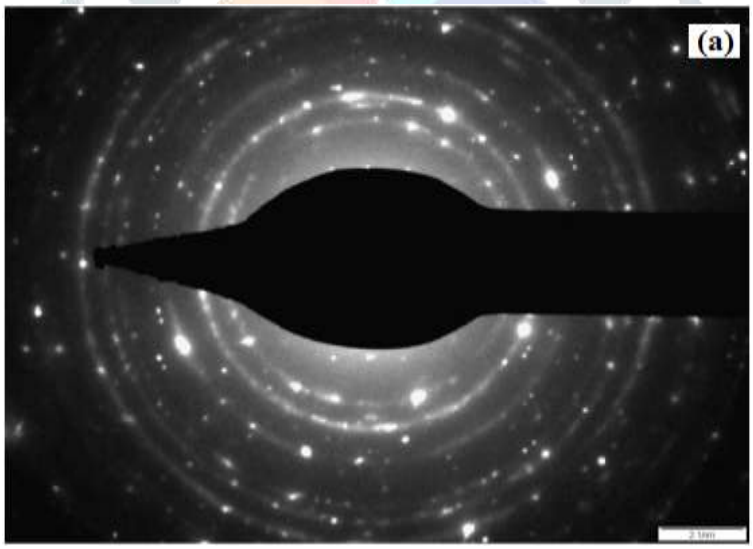

Fig. 6: saed micrograph of nanocrystalline mgfe$_2$o$_4$

Conclusions

In the present study, we have successfully synthesized nanocrystalline $MgFe_2O_4$ was synthesized using less expensive, environment-friendly and low temperature sol-gel route using citric acid as anionic surfactant occurring at a temperature 700^o C. From the XRD analysis it is cleared that, the prepared $MgFe_2O_4$ belongs to single phase cubic spinel structure. Also, it was evident from XRD pattern that Bragg's angle (2θ) reflections are in very good agreement with the reported literature. The lattice constant was reported as 8.402 A^o and other structural parameters are in the reported range. FT-IR spectra ensured the vibrational stretching frequencies analogous to the composite. SEM analysis validated the structure of final powder with the grain size 40-170 nm. EDAX analysis confirms the presence of Mg, Fe and O. The nearly cubical shaped morphology with the isolated nanoparticles shows potential of this simple method.

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