Synthesis of Mg-Ferrite nanoparticles via auto combustion method and investigation their structural, morphological and magnetic properties

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ABSTRACT

Magnesium ferrite (MgFe$_2$O$_4$) nanoparticles is prepared by sol-gel auto combustion method and calcinated at (200,450, 900 °C). The capping agent was urea and (Mg(NO$_3$)$_2$.6H$_2$O) and (Fe(NO$_3$)$_2$.9H$_2$O) nitrates as sources of metal. X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FT-IR) characteristic show clear modes of the cubic Mg-ferrite structure formation. Infrared spectrum of metal-oxygen vibration at (703-636) and (574-433) cm$^{-1}$ show the tetrahedral and octahedral site of Mg-ferrite structure. Scanning Electron Microscope (SEM) images shown pure crystalline microstructure with polyhedral shapes and very small numbers of globular small particles. The crystallite size of Mg-ferrite is calculated using Debeye-Scherrer relation and was in the range of 29 nm.

1. Introduction

The ceramic powder is generally known as ferrite could afford more features over the bulk ferrite with the suggestive change of physical properties like very tiny size and increasing the surface area [1,2]. Spinel ferrites crystals have chemical formula MFe$_2$O$_4$, where M is Mn$^{2+}$, Co$^{2+}$, Ba$^{2+}$, Zn$^{2+}$, Cd$^{2+}$, Ni$^{2+}$, Mg$^{2+}$, Cu$^{2+}$, etc.) [3]. The structure of spinel is determined by bonding and arrangement of the oxygen ions (O$^{2-}$) within the lattice points. The unit cell of a spinel lattice contains eight formula units [(A)(B)$_2$O$_4$]. The large oxygen anions (32 from O$^{2-}$) form a close-packed FCC structure. Whereas the few metal cations occupy interstitial sites: tetrahedral (8) or (A) and octahedral (16) or (B) sites. Depending on how cations occupy diverse interstices, a spinel structure can be categorized into normal or inverse. In the normal spinel, the tetrahedral voids are occupied by only the divalent (A$^{2+}$) ions whereas the octahedral voids are occupied by the trivalent (B$^{3+}$) ions only. These compounds show lowly magnetic properties, for Example of normal spinel; ZnFe$_2$O$_4$, CdFe$_2$O$_4$, and MgAl$_2$O$_4$. On the other hand, inverse spinel, (A$^{2+}$) ions occupy the octahedral voids, whereas half of (B$^{3+}$) ions occupy the tetrahedral voids. It can be represented as [(B$^{3+}$)$_2$][2(A$^{2+}$B$^{3+}$)O$^4_2$]. NiFe$_2$O$_4$, Fe$_3$O$_4$, and CoFe$_2$O$_4$ are classified under this group [4-6]. Among the known magnetic nanoparticles—magnesium ferrite, MgFe$_2$O$_4$, a well-known soft magnetic, n-type semiconductor [7], So it has much application such as heterogeneous catalysis, photoelectrical, sensors, and magnetic technologies [8-16]. Conventionally, MgFe$_2$O$_4$ is usually synthesized by sol-gel auto combustion method [17-20], thermal treatment [21], coprecipitation method [22], hydrothermal synthesis [23], high energy ball milling [24] and spray pyrolysis [25], have been extensively investigated for the synthesis of well-dispersed MgFe$_2$O$_4$ nanoparticles. The methods refer to as have specific advantages dependent on required applications and properties of synthesized materials. The sol-gel auto-combustion technique has arisen as an essential prosses for preparation of high-performance ceramics, catalysts, composites, and crystalline spinel oxide materials. In this technique, the chemical reaction is an exothermicity, used to synthesis useful compounds with homogeneous mixing, regular shape, and fine stoichiometric control [26-31]. The aim of the current study graft is to consider the plain structural and magnetic belongings of MgFe$_2$O$_4$ nanoparticles prepared via the auto combustion method with urea as fuel. The structure and morphology of the nanoparticles are characterized and analyzed by X-ray diffraction patterns, scanning electron microscopy and transmission electron microscopy. The magnetic properties for the MgFe$_2$O$_4$ nanoparticles, also, are measured by using vibrating sample magnetometer at room temperature.
2. Materials and Methods

Initial materials utilized in the existent work were: magnesium nitrate, CDH, 99%, ferric nitrate 9-hydrate (Fe(NO₃)₃·9H₂O, CDH, 99.9%), urea (CO(NH₂)₂, CDH, 99.9%), and ammonium hydroxide (NH₄OH, Fluka). All chemicals in this work, were of analytical reagent grade. Deionized water is used in all the experiments.

2.1. Synthesis of magnetic nanospinel MgFe₂O₄

Mg-ferrite powder was combined affording to identified auto-combustion routes with revisions. The molar ratio of urea to metal ions (OH/M) is 2:1, while the molar ratio of Mg:Fe was specific to 1:2. In a distinctive method, known attention solutions of nitrate were equipped by dissolving Mg(NO₃)₂·6H₂O, and Fe(NO₃)₃·9H₂O in the deionized water. solutions of nitrate were then variegated via volume agreeing for identified magnitudes and apposite weight of fuel (urea) lastly additional at the variegated mixture. The pH of the mixture was attuned at 8. after that the solution was heated at about 90 °C under constant stirring, for 2h pending a gelatinous sol was attained. The gelatinous sol was animated on a hot plate about 100 °C. The gel begins swelling, and then a quick and strong auto-ignition occurs for about a minute, finishing the reaction, the sample was heated in a furnace at 200°C for 2 h. The equipped powder was annealed in 450°C-900°C for 2 h in air.

2.2. Characterization

The structural characterization of the manufactured compounds are Personalized by "Shemadzu X ray diffractometer (model 6000)". The period and crystallite size were examined using Cu-Kα radiation (λ = 1.5406Å) at 40 kV, and 30 mA. The progressive receiving slit (height 0.30 mm), with a curved graphite monochromator. The intensity data were collected by continuous scanning (step 0.02 °/s) in 20 angle range of 10–80°. The crystalline phase was identified using the International Centre for Diffraction Data (ICDD) database. The Fourier transform infrared the spectrum (FTIR) of magnesium ferrite powders (as pellets in KBr) are recorded with an IR Shimadzu 8400 spectrophotometer over the range of (400-4000) cm⁻¹. The surface morphology of the obtained samples was inspected by field emission scanning electron microscopy (FE-SEM) (Model: ZEISS Sigma 300) and “transmission electron microscopy” (TEM) micrographs were obtained utilizing a “JEOL, JEM-2100F” system, the accelerating voltage was 200 kV. To prepare the TEM specimens, powder specimens were distributed in ethanol using the ultrasonic for ten min. A little drop of the specimen solution was dropped on the top surface of a carbon film sustained Cu grid then allowed to dry in the air before the test. For analyzing the composition of the prepared compounds, energy dispersive X-ray spectroscopy (EDX) (ZEISS Sigma 300) was employed. To examine the magnetic properties of the synthesized nanoparticles, the magnetization measurements were approved up Saturation using a "vibrating scanning magnetometer (VSM)" (Cryogenic Limited PPMS) under the applied field of ±1 (T) at room temperature.

3. Results and Discussion

The physical properties of nanomaterials prepared by gel-combustion process are mainly dependent on the type of the used fuel. A wide variety of fuels is used in nanomaterials synthesis by gel-combustion. The sol is developed gradually to the produced of a gel-like network having mix solid and liquid phase at the same time [32]. The homogeneous gel is, then, heated and the auto-combustion has given a good powder. However, preparation of metal oxide includes linking the metal with o xo insides (M-O-M) or hydroxo (M-OH-M) bridges, so producing the metal-oxo or metal-hydroxo polymers in solution.

FTIR spectral analysis

Fig. 1 shows FT-IR spectra for the MgFe₂O₄ samples calcined at 900 °C. The dry gel shows an absorption band around 3434 cm⁻¹ which corresponding to the bending vibration of N–H. An absorption band around 3305-3120 cm⁻¹ shown by sample tallies to the presence of O–H group". The strong peaks at 1662 and 1390 cm⁻¹ are correspond to bending vibration of carbonyl group and NO₃⁻ ions, respectively. The absenteeism of absorption bands corresponding to tetrahedral and octahedral complexes specifies that the creation of spinel structure is not complete in the dried gel [33,34].
MgFe$_2$O$_4$ calcined at 900 °C and it can be observed only the typical peaks of MgFe$_2$O$_4$ appear at about 574 and 433 cm$^{-1}$, these bands own Fe$^{3+}$-O$^{2-}$ extending shakings on tetrahedral and octahedral location, respectively, characteristic of inverse spinel Mg-ferrite [35,36].

**X-ray Diffraction**

The XRD spectra of MgFe$_2$O$_4$ synthesized by a sol–gel method, which calcined for 3h in the air at various temperatures (200, 450 and 900 °C) are shown in Fig.2. It's noted that the forerunner is amorphous in nature. When the forerunner was calcined about 900 °C, the position of the peaks agrees with the characteristic peaks of the standard spinel phase, but the negligible amount of $\alpha$-Fe$_2$O$_3$ is found in the samples but no other phases were detected. The XRD patterns of the calcined samples show that as the calcination temperature increases, peaks Accompanying to MgFe$_2$O$_4$ become sharp and more extreme specifying an advance in crystalline goodness for calcination temperature. All the piecing diffusion peak is paralleled with typical diffraction ranks of Mg-ferrite ICDD pdf no. 00-033-0664 (space group Fd3m). The average crystallite size of the MgFe$_2$O$_4$ is obtained using the "Scherrer's formula" [37] was 29 nm.

The parameter of the lattice has been studied from the subsequent equation [38]:

\[
a = d_{hkl}\sqrt{h^2+k^2+l^2} \quad \ldots (1)
\]

where \(d\) the interplanar space for the planes (hkl). The density of X-ray (\(D_x\)) is studied utilizing the behind equation [39]:

\[
D_x = \frac{8M}{\pi N a^3} \quad \ldots (2)
\]

Where M is the molecular weight of the spinel (g/mole), N Avogadro number (6.023×10$^{23}$ atom/mol), and \(a\) is the lattice parameter (cm). The gotten "crystallite size (\(D_{XRD}\)), lattice parameter (\(a\)) and X-ray density (\(D_x\)) from the XRD spectra" are detailed in Table 1.

**Table 1. Some structural parameters for the crystalline phases involved in the magnesium ferrite.**

| Annealing temp. (°C) | Average Particle size (nm) | Lattice constant, \(a\) (Å) (XRD) | Space group | Unit cell volume, \(V\), (Å$^3$) | X-ray density, \(D_x\), (g/cm$^3$) |
|----------------------|---------------------------|-------------------------------------|-------------|---------------------------------|----------------------------------|
| 200                  | -                         | 8.124                               | -           | 536.1                           | 4.954                            |
| 450                  | -                         | 8.334                               | -           | 578.8                           | 4.589                            |
| 900                  | 35                        | 8.364                               | Fd-3m       | 585.1                           | 4.539                            |
Morphological analysis
The morphology of surface and the fundamental structure are investigated by FE-SEM combined with TEM photograph and energy dispersive X-ray analyzes. SEM micrograph and EDX spectrum of MgFe₂O₄ sample calcined at 900°C are shown in Fig. 3a and 3b respectively. It can be perceived that the calcined specimens involve of very tiny particles with a mean particles size nearly 35nm and each particle has a regular shape with a clear glassy nanostructure own polyhedral shapes and very little numbers of circular tiny particles. On the other side, the holes in the micro image are owing to the exit of additional amount of gases throughout the burning method. It is absorbed that the difference in the particle size for the present samples calculated by FE-SEM and the crystallite size obtained using Scherrer’s formula from XRD analysis, may be due to the molecular structural disorder and lattice strain, which results from the different ionic radii and/or clustering of the nanoparticles. Hence, the XRD method has a more stringent criterion and leads to smaller sizes [40]. EDX analysis of the MgFe₂O₄ sample calcined at 900°C is carried out for essential investigation. In the EDX analysis (Fig.3) no pollution element is noticed, excepting magnesium, iron, and oxygen. The atomic fraction of magnesium and iron are about 1:2.

Magnetic measurement
The magnetic properties of MgFe₂O₄ can be specified by factors like the grounding process, particles size, cation delivery, and annealing temperature and O²⁻ occupancy, etc. The hysteresis curve of the MgFe₂O₄ sample is shown in Fig. 4. The MgFe₂O₄ nanoparticles exhibit the soft ferrimagnetic behavior of the synthesized MgFe₂O₄ and values of the saturation magnetization (Ms), remnant magnetization (Mr), coercive field (Hc) and loop squareness ratio (Mr/Ms) are obtained and listed in Table 2. The experimental magnetic moment is meant from the next formulation [34]:

\[ \mu_B = \frac{MW \cdot Ms}{5585} \]  

where MW is the molecular weight of the specimen and Ms is the saturation magnetization in emu/g.
Saturation magnetization (Ms) of MgFe₂O₄ was 22.4 emug⁻¹ and the high value of the powerful field for the as-prepared powder of MgFe₂O₄ is confirm to become 104.4 Oe for 29 nm particle size. Several researchers have reported different coercivity values than what we obtained for example, A. Pradeep et al. [34] have determined Hc value as 202.55G for 35 nm grain size. Rabanal et al. [41] have determined an Hc amount of 576.7 Oe for a grain size of 80 nanometers. A contrast of these values clarifies a straight proportional relation between coercivity and grain size, that is an influence of nano administration where the coercivity of the as-synthesized magnetic nanoparticles is negligible when the nanocrystals have single domains, that refers to much of single domains are regularly magnetized with all the spins aligned in the same direction. This is available only when the particle diameter size below 3–50 nm, depending on the materials.
### Table 2. Magnetic parameters extracted from VSM data at room temperature for MgFe$_2$O$_4$ annealed at 900°C

| Annealing temp. (°C) | Ms (emu/g) | Mr (emu/g) | Hc (Oe) | Mr/Ms | $\mu_p$ |
|---------------------|------------|------------|---------|-------|---------|
| 900                 | 22.4       | 5.87       | 104.4   | 0.262 | 0.802   |

![Hysteresis loop of MgFe$_2$O$_4$ at room temperature.](image)

**Fig. 4** Hysteresis loop of MgFe$_2$O$_4$ at room temperature.

### 4. Conclusion

Nanocrystalline and magnetic spinel MgFe$_2$O$_4$ powders are prepared by the sol-gel auto combustion technique using urea as chelating/fuel agents. MgFe$_2$O$_4$ showed superparamagnetic behavior after calcination at 900°C for 3h. Analysis of X-ray diffraction pattern confirmed the formation of MgFe$_2$O$_4$ as major and minor phases with a small amount of unreacted $\alpha$-Fe$_2$O$_3$. IR analysis presented the characteristic immersion bands and the band sites revealed that divalent cations are dispersed on both positions.

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**Abstract**

Preparation of magnesium ferrite nanoparticles through combustion and study of their structural and magnetic properties

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**Summary**

Preparation of magnesium ferrite nanoparticles (MgFe$_2$O$_4$) through combustion at a temperature of 900°C. The X-ray diffraction (XRD) pattern showed the cubic structure with a lattice constant of 8.87 Å. The Fourier transform infrared (FTIR) and X-ray diffraction (XRD) spectra showed the characteristic peaks of the samples. The average crystal size was calculated using the Debye-Scherrer equation and found to be 29 nm.