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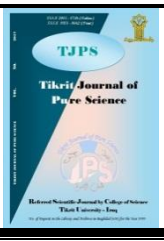

Synthesis of Mg-Ferrite nanoparticles via auto combustion method and investigation their structural, morphological and magnetic properties

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ABSTRACT

Magnesium ferrite ($MgFe_2O_4$) nanoparticles is prepared by sol-gel auto combustion method and calcinated at (200,450, 900) °C. The capping agent was urea and ($Mg(NO_3)_2.6H_2O$) and ($Fe(NO_3)_3.9H_2O$) 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 Debye-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_2O_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 [$(A)(B)_2O_4$]. 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 shows lowly magnetic properties, for Example of normal spinel; $ZnFe_2O_4$, $CdFe_2O4$, and $MgAl_2O_4$. 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 $[(B^{+3})^{tet}(A^{+2}B^{+3})^{oct}O_4]$. $NiFe_2O_4$, Fe_3O_4, and $CoFe_2O_4$ are classified under this group [4-6]. Among the known magnetic nanoparticles, magnesium ferrite, $MgFe_2O_4$, 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, $MgFe_2O_4$ 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 $MgFe_2O_4$ 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 the 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 $MgFe_2O_4$ 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 $MgFe_2O_4$ 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 ($Fe(NO_3)_3.9H_2O$, CDH, 99.9%), urea ($CO(NH_2)_2$, CDH, 99.9%), and ammonium hydroxide (NH_4OH, 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 $MgFe_2O_4$

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 $Mg(NO_3)_2.6H_2O$, and $Fe(NO_3)_3.9H_2O$ 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 manufactuerd 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 2θ 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 nano-particles, 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 oxo 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 $MgFe_2O_4$ 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 NO_3^- 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].

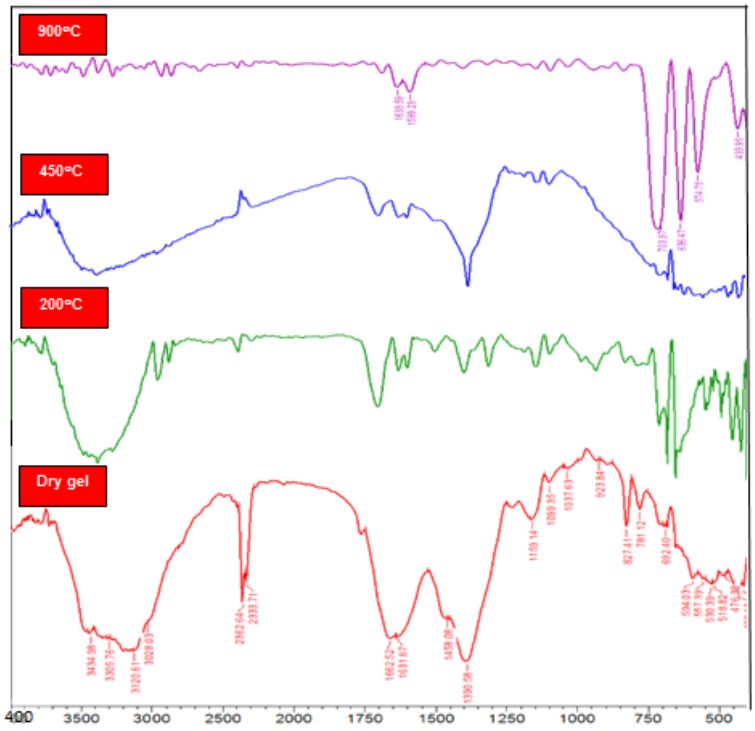

Fig. 1 : The FTIR spectra of the dry gel and annealed Mg-ferrite samples at 200 ºC, 450 ºC, and 900 ºC.

$MgFe_2O_4$ calcined at 900 °C and it can be observed only the typical peaks of $MgFe_2O_4$ 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 $MgFe_2O_4$ 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 α-Fe_2O_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 $MgFe_2O_4$ become sharp and more extreme specifying an advance in crystalline goodness for calcination temperature. All the piercing 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 $MgFe_2O_4$ is obtained using the "Scherrer's formula" [37] was 29 nm.

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

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

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

$$D_x = \frac{8M}{Na^3} \quad \ldots\ldots (2)$$

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 (*Dx*) 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, *Dx*, (g/cm3)
	D_{SEM}	D_{XRD}				
200	-	26	8.124	-	536.1	4.954
450	-	27	8.334	-	578.8	4.589
900	35	29	8.364	Fd-3m	585.1	4.539

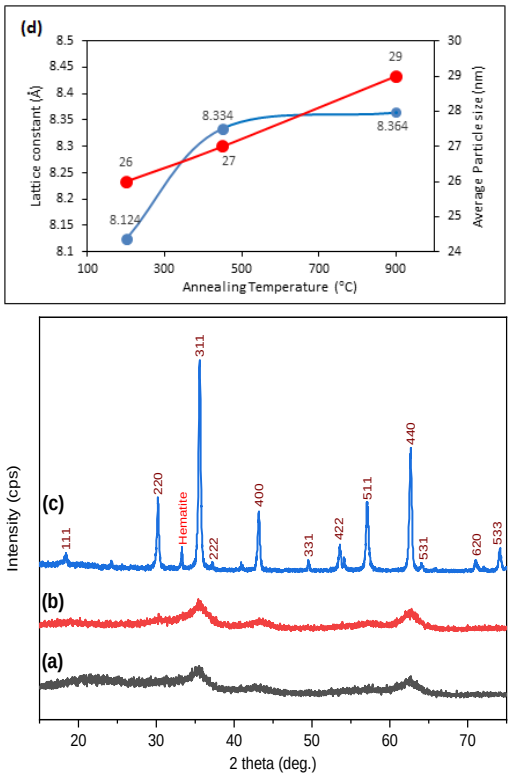

Fig. 2: XRD patterns of magnesium ferrite samples annealed at: (a)200 °C, (b) 450 °C, and (c) 900 °C. (d) The average particle size and lattice constant for $MgFe_2O_4$ nanoparticles at the annealing temperature.

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 $MgFe_2O_4$ 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 $MgFe_2O_4$ 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.

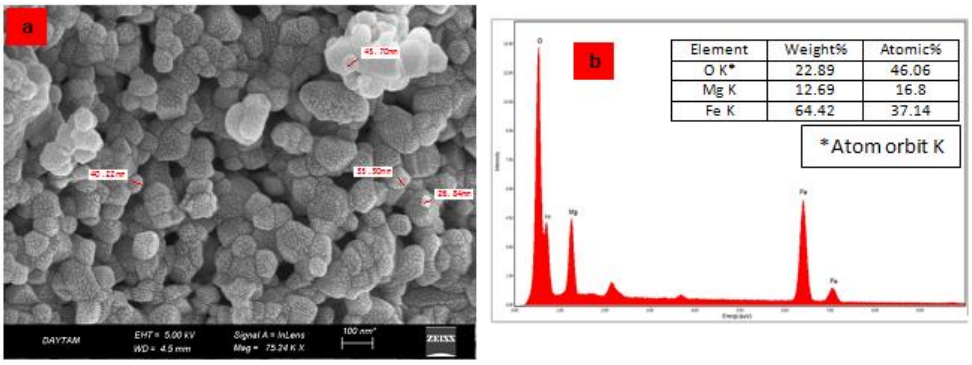

Fig. 3 (a) FE-SEM image and (b) EDX spectra of the $MgFe_2O_4$ annealed at 900 °C.

Magnetic measurement

The magnetic properties of $MgFe_2O_4$ can be specified by factors like the grounding process, particles size, cation delivery, and annealing temperature and O^{-2} occupancy, etc. The hysteresis curve of the $MgFe_2O_4$ sample is shown in Fig. 4. The $MgFe_2O_4$ nanoparticles exhibit the soft ferrimagnetic behavior of the synthesized $MgFe_2O_4$ and values of the saturation magnetization (*Ms*), remnant magnetization (*Mr*), coercive field (H_C) and loop squareness ratio (*Mr*/*Ms*) are obtained and listed in Table 2. The experimental magnetic moment is meant from the next formulation [34]:

$$\mu_p = MW.Ms /5585 \quad \ldots..(3)$$

where MW is the molecular weight of the specimen and M_S is the saturation magnetization in emu/g.

Saturation magnetization (*Ms*) of $MgFe_2O_4$ was 22.4 $emug^{-1}$ and the high value of the powerful field for the as-prepared powder of $MgFe_2O_4$ 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 H_C 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 $MgFe_2O_4$ annealed at 900 °C

Annealing temp.(°C)	*Ms* (emu/g)	*Mr* (emu/g)	H_c (Oe)	*Mr/Ms*	μ_p
900	22.4	5.87	104.4	0.262	0.802

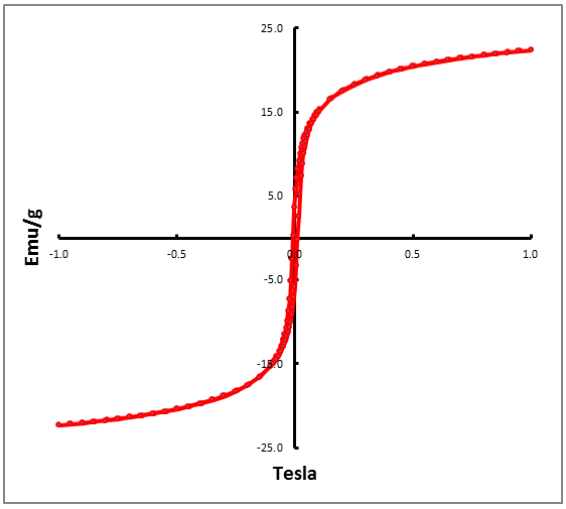

Fig. 4 Hysteresis loop of $MgFe_2O_4$ at room temperature.

4. Conclusion

Nanocrystalline and magnetic spinel $MgFe_2O_4$ powders are prepared by the sol-gel auto combustion technique using urea as chelating/fuel agents. $MgFe_2O_4$ showed superparamagnetic behavior after calcination at 900°C for 3h. Analysis of X-ray diffraction pattern confirmed the formation of $MgFe_2O_4$ as major and minor phases with a small amount of unreacted α- Fe_2O_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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تحضير جسيمات المغنيسيوم فرايت النانوية عن طريق الاحتراق التلقائي وبحث خصائصها الهيكلية والمورفولوجية والمغناطيسية

ابراهيم فهد وحيد ، فائز محسن حامد ، بيداء محمود علي

قسم الكيمياء ، كلية العلوم ، جامعة تكريت ، تكريت ، العراق

الملخص

تم تحضير دقائق المغنيسيوم فرايت النانوية ($MgFe_2O_4$) عن طريق الاحتراق الذاتي للسول–جل والمكلسنة عند 900 درجة مئوية. حيث كانت اليوريا هي العامل الشابك ونترات المغنيسيوم والحديد كمصادر للفلز. أظهر حيود الأشعة السينية (XRD) وطيف (FTIR) الأنماط المميزة لتكوين بنية المكعب. كما أظهر طيف الأشعة تحت الحمراء حزمة ارتباط الأوكسجين بالفلز عند (433–574) و (636–703) سم$^{-1}$ والتي تشير لمواقع رباعي السطوح وثماني السطوح لنظام المغنيسيوم فرايت. كما تم فحص الصور المأخوذة بمجهر المسح الإلكتروني (SEM) حيث كانت البلورات ذات بنية تركيبية دقيقة واضحة لها أشكال متعددة السطوح وأعداد صغيرة جدًا من الجزيئات الكروية الصغيرة. كما تم حساب الحجم البلوري لدقائق المغنيسيوم فرايت النانوية باستخدام معادلة Debye-Scherrer حيث كانت بحدود 29 نانومتر.