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Synthesis and Study of Calcination Effect of Zinc Ferrite on the Structure and Morphology of Nanoparticles

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Abstract

Three precursor solutions of spinel zinc ferrite were successfully fabricated via a rapid and simple route (microwave combustion), followed by calcination at a temperature of 500 °C for three hours. The raw materials used in the preparation these spinel samples were ferric and zinc nitrate salts, while glycine was used as fuel. The phase purity and crystal lattice parameters were estimated via XRD. The particle size and morphologies for synthesized powdered samples are shown by field emission-scanning electron microscopy images (FE-SEM), Fourier transform infrared spectroscopy (FT-IR) were us to study the vibration mode in synthesized spinel ferrite. The results for synthesized samples showed that there is a remarkable change in phase purity, crystal lattice parameter, particle size, and morphologies with calcination. Results of XRD refer to cubic spinel - structure with space group Fd3m. The average crystallite size increases when Glycine –to-nitrate ratio is decreased. It is increase from 15.8 nm to 26.53 nm and from 23.03 nm to 28.16 nm for the as–synthesized samples and calcinated at 500 °C, respectively. As well, the results of XRD show that the average lattice constants are changed from (8.407 to 8.391) ˚A and then they increase from (8.391 to 8.427) ˚A for the synthesized samples. However, the calcination results in an increase in average crystallite size and average lattice constant. FE-SEM image indicated that the particles of zinc ferrite possess shape symmetry and uniformity.

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1. Introduction

The compounds that formed by iron oxides and transition metal oxides in order to obtained as powder or ceramic bodies with ferromagnetic-type properties known as ferrites [1]. Various factors can be influence of Structural, magnetic, and optical properties of ferrites of which, size of particle, preparation method, type of dopant ions [2], chemical compositions, as well as sintering temperature [3]. Among various ferrites spinel – structure ferrite with the general formula (MFe_2O_4) became an interesting categories of technological materials due to their an important properties of which, excellence chemical stability, high saturation magnetization, So them suitable for magnetic, mechanical, and catalytic applications [4] In spinel- structure metal cations are located between A –site (tetrahedral) and B- site (octahedral) formed through the oxygen ions in a unit cell. This distribution influence on the magnetic properties of MFe_2O_4, that could be controlled by crystallite size and thermal treatment [5] Zinc ferrite is a soft magnetic spinel structure that possesses high resistivity ,excellent magnetic properties, as well as high thermal and chemical stabilities [6] [7]. It is one of the iron based cubic spinel space group S.G. Fd3m, [8] Recently, Zinc ferrite has also received great interest because of the magnetic, adsorption, and photo-catalysis applications [9] Spinel structure was synthesized with various methods among these, chemical precipitation method [10] hydrothermal methods [11] conventional heating (CHM) method [12] in here zinc ferrite was synthesized via microwave-combustion routs. Several features of this route among those clean, morphology 0f particles are in control, short time reactions [13]; cost effectiveness of this route is lower than the other wet chemical routes for synthesis ferrites [14].

2. Experimental

Nitrate salts of Zinc and iron as metal sources and fuel via (Zn^{+2}/Fe^{+3})is taken as the stoichiometric ratio (1:2) respectively were mixed to each other and dissolved via stoichiometrical amount of distilled water then it stirred by a magnetic stirrer has a max. temperature (100 ^{0}C**)** model manufactured at China. for 20 min .Later stoichiometric amount of fuel ($C_2H_5NO_2$) was added within the mixed via continuous stirring until dark brown color was formed. The mixture was placed in crucible then transferred to a_microwave oven with microwave irradition power 1000 Watt . in the microwave combustion route. The solution via fuel boiled and subjected to dehydration accompanied by decomposition and starting released gases among those, CO_2, NO_2, and N_2 . If the solution reached the combustion point, it became solid. Finally, filter papers were used to resulting compound filtered. Product was washed via ethanol and distilled water for (5) times in order to remove the residual initial materials, dried at (70 °C) for two hours and allowed to cool in the oven itself. Finely mortar was used to ground the result powder for (3min.) in order to obtain fine powders then this fine powders were calcinated at (500 °C) for 3h.

3- Characterization techniques

After preparation of samples, Characterization for the synthesized powdered samples is an essential step In order to understand of synthesis protocol and attest the successfulness of them. In this search techniques that used for the characterization of nanoparticles are powder X-ray diffraction (XRD), Field emission scanning electron microscope (FE-SEM) and Fourier transform-infrared spectroscopy (FT-IR). The phase composition and crystalline structure of ferrites was studied using Shimadzu diffract meter model XRD 6000 (manufactured at Japan) at university of Baghdad / college of education (Ibn- Al.Haitham), Iraq .Device employs Cu Kα (λ =1.1548 Å). A proportional counter with an operating current of 30 mA and voltage of 40 kV was used. The Surface morphology of the samples was recorded with Field Emission- Scanning Electron Microscopes (FE-SEM) with various magnification powers .They detectors at university of Al –Basrah /college of science, Iraq as well as university of Tehran, Iran. (FT-IR) spectrum was recorded on a (Shimadzu-8400S spectrometer) at the range of (400 cm^{-1}– 4000 cm^{-1}) at university of Al-Qadisiyah /college of chemical engineering, Iraq.

4 - Results and Discussion

4-1- Structural and morphological characterization

Figure 1 manifest XRD patterns for as- synthesized spine Zn Fe_2O_4 ferrite samples , while Figure 2 manifest XRD patterns for spine Zn Fe_2O_4 ferrites that followed by calcination at 500 °C for 3h

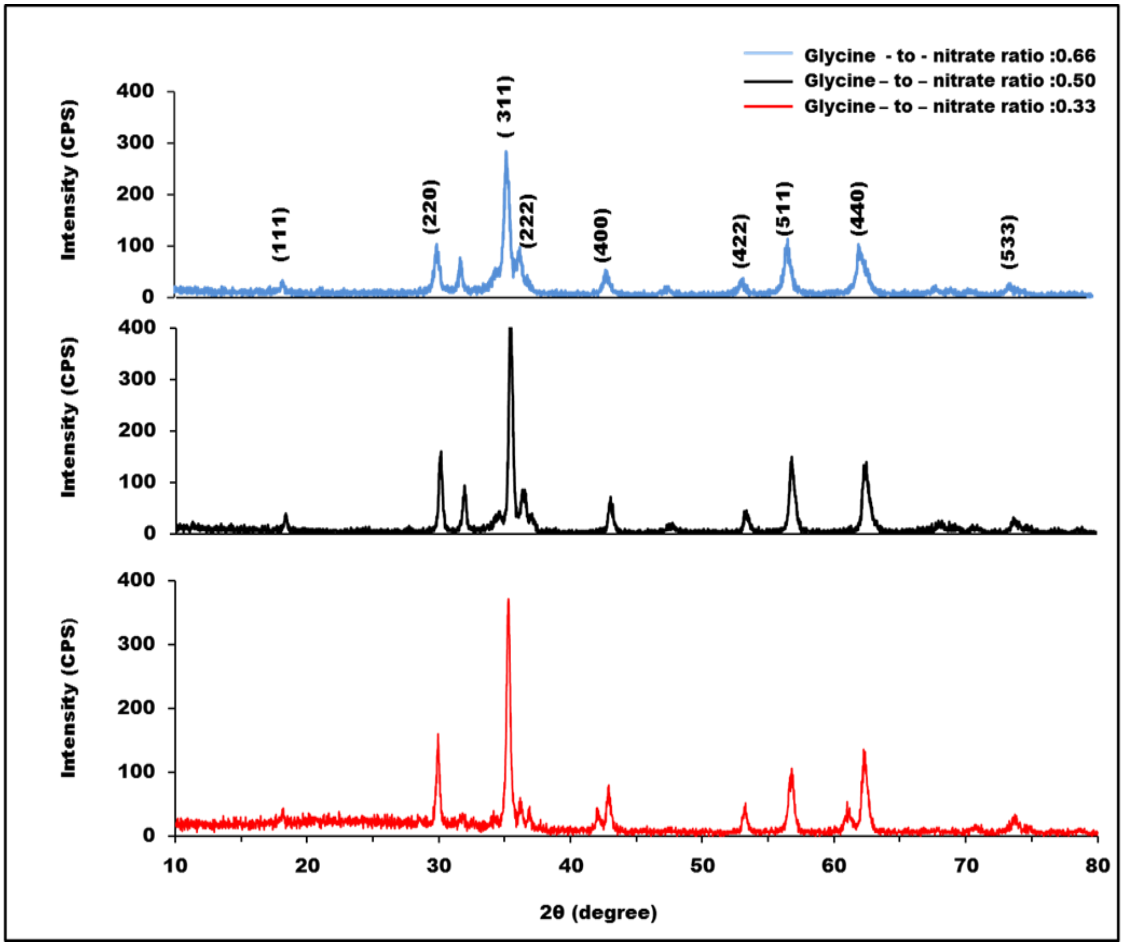

Figure 1: XRD pattern of as- synthesized normal spine $ZnFe_2O_4$ ferrite by Microwave – assisted combustion method with different glycine-to-nitrate ratio

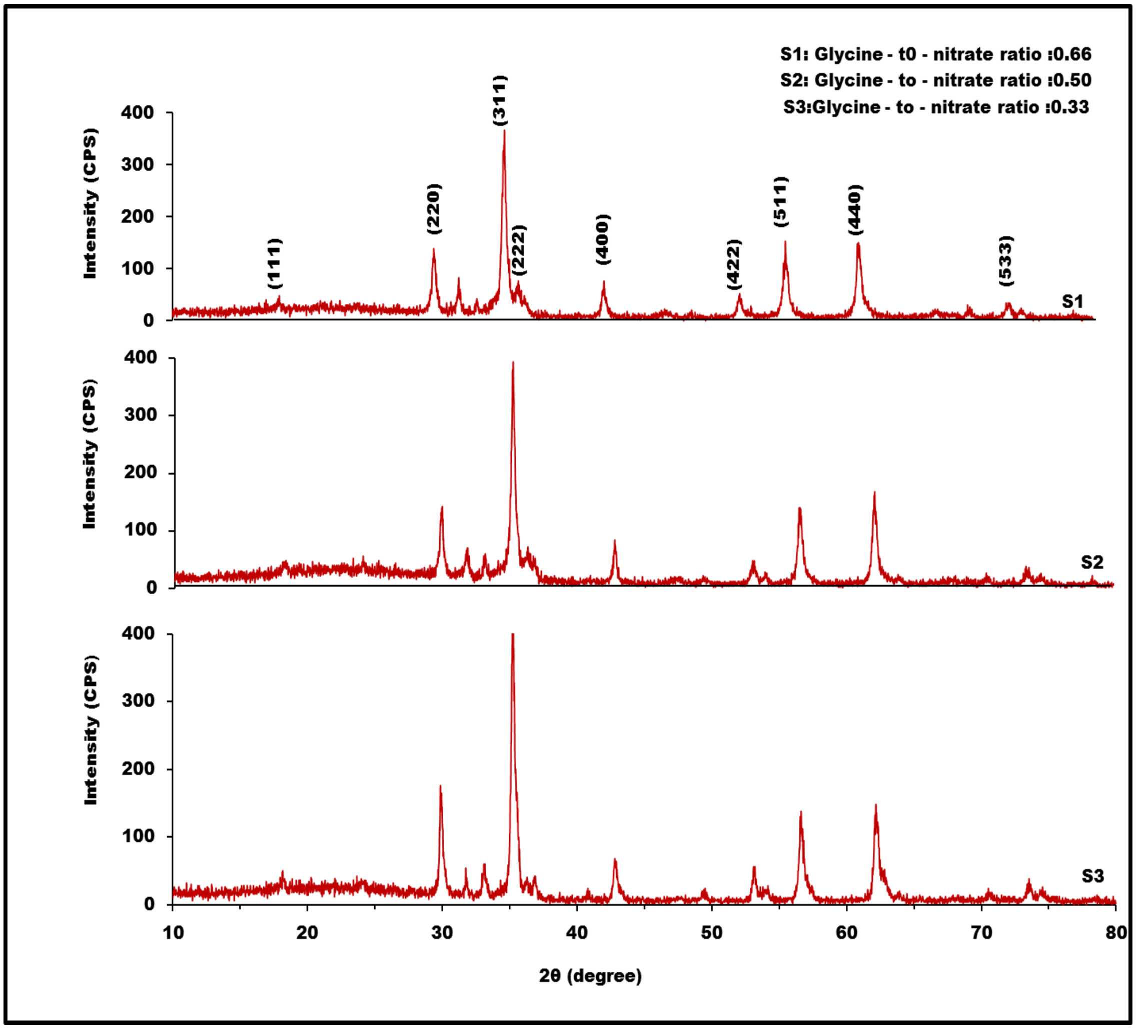

Figure 2: XRD pattern of the synthesized normal spine $ZnFe_2O_4$ ferrite by Microwave – assisted combustion route with different glycine-to-nitrate ratio followed by calcination at 500 ^{o}C for 3h.

For as-synthesized spine $ZnFe_2O_4$ ferrite in the Figure (4-1) I when the glycine-to-nitrate ratio 0.66 crystalline phase structure is observed... peaks at 2θ values of 18.31°, 30.04°, 35.37°, 36.89°, 42.94°, 53.24°, 56.72°, 62.33° and 73.61° belong to the (111), (220), (311), (222), (400), (422), (511), (440) and (533) planes of spinel zinc ferrite, respectively that can be referred to the cubic- spinel structured matches via the standard (JCPDS card No. 22-1012) of $ZnFe_2O_4$ ferrite. There are Two extra peaks at 31.84°, and 47.61°, which belong to (220) (400) plane of ZnO. Good crystalline phase structure can be observed for S2 at Glycine-to-nitrate ratio 0.5 as shown in the figure (4-1) I. As Glycine-to-nitrate ratio decrease to 0.33, regular variation in the crystalline phase structure can be observed for S3 with tiny trace in phase structure ZnO compared with the other synthesized samples S1 and S2. Two extra peaks prepared at 42.06° and 61.02.03°, that belong to, (422) and (511) plane of ZnO. Results showed that calcination at (500˚C) In Figure (4-1) II. The diffraction peaks become sharper and narrower. This refers to the increase of the crystallinity. The full width half maximum of highest peak decreases with calcination at this temperature this mean that increased in the particle size of the samples with calcination. Peaks of impurities with high crystallization were observed in the patterns in the fig. (4-1)II. The increase in the average crystallite size after calcination is because of the increase in nanoparticles size and/or small nanoparticles merge to form bigger particles [15]. The values of the average crystallite size D of the samples as well as average lattice constant a were calculated from the higher intensity of (311) plane within XRD profile, in accordance with relations (1) and (2) respectively [16].

$$D= (0.89\ \lambda)/(\beta\cos\theta)\ldots\ldots.(1)$$

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

λ- represent XRD wavelength that used (0.154 nm), β- is represent the Full Width at Half Maximum of plane (311) and θ- is represent the Bragg's angle. All these parameter were written as in Table 1 .

Table 1: The values of the average crystallite size and crystal lattice parameter for synthesized samples of the spine Zn Fe_2O_4 ferrite by Microwave – assisted combustion method with different glycine-to-nitrate ratio befor and after calcination.

Sample		Glycine/nitrate ratio	Lattice Parameter a (˚A)	Crystallite Size D(nm) (XRD)
S1	as-synthesized	0.66	8.407	15.8
	Calcinate at 500˚C for 3h		8.440	23.03
S2	as-synthesized	0.50	8.391	23.70
	Calcinate at 500˚C for 3h		8.440	27.05
S3	as-synthesized	0.33	8.427	26.53
	Calcinate at 500˚C for 3h		8.434	28.16

One can be observed From above table that increase of average crystallite size when Glycine –to-nitrate ratio is decreased . It is increase from 15.8 nm to 26.53 nm and from (23.03 to 28.16) for the as–synthesized samples and calcinated at 500 °C respectively. One also can be observed From saim table that, the average lattice constants is changed from (8.407 t0 8.391)˚A and then it increase from (8.391 to 8.427)˚A for as –synthesized samples. An increase in lattice constant can be explained depending on the ionic radii of the divalent zinc cations. It is assumed that the replacement of smaller divalent iron cations (0.64Å) with the larger divalent zinc cations (0.74Å) on the B- sites causes the unit cell to expand which results in A small increase of lattice constant. This is consistent with previous work that reported by P. Sivakumar, K. Thyagarajan, and A. G. Kumarc [17]. The decrease in average lattice constant in as-synthesized sample S2 may be due to the smaller ionic radii of Fe^{3+} (0.67 Å) compared with ionic radii of divalent zinc cation (0.82 Å) . Crystal lattice parameter and avarage particle size are directly related. Literature stating that, increasing in the particle size resulting in increase or decrease in the crystal lattice parameter [18].In here we noted that if the average particle size increased the crystal lattice parameter will be decreased at Glycine-to-nitrate ratio 0.66 and 0.50 , while it is increase at Glycine-to-nitrate ratio 0.33. The results in table (4-1) indicate that the average particle size of 23nm, 27nm and 28 nm for the calcination time 3 hours at 500˚C for the calcinated samples S1, S2, and S3, respectively. These results shown that there is remarkable change in lattece parameters with the calcination.

The morphologies and particle sizes for synthesized spinel $ZnFe_2O_4$ ferrite befor and after calcination via various glycine –to-nitrate ratio were characterized via FE-SEM shown in the Fig. 3 and Fig.4, respectively. In the Fig.3 as-synthesized samples S1 and S2 their are avoids. Depending on the earlier studies average crystallite size is related to the pH value. When the pH value is too high, there is a lot of gas that products during combustion process into microwave –oven leading to an increase in porosity and a decrease in average crystallite size [19]**,** In addition to their agglomerated in particles due to the interaction between the magnetic nanoparticles[17]. In contrast to the as-synthesized sample S3 that possess no avoids may be due to the fuel- low ratio. These results indicate that a change in the fuel amount lead to significant changes in the morphology of the powders. The FESEM images show that as-synthesized Zinc ferrite possess uniform, symmetry .In Fig.4 FE-SEM images for mentioned samples which are calcinated at 500c for three hours these images showed their agglomeration. This can be illustrate based on the surface area of nanoparticles are high. Among reasons that illustrated morphologies of spherical-like particles :(i) the fuel and preparation route (ii) the lower surface-to-volume ratio for spherical-like particles. Second fact include the effect of morphology and grain boundary mobility on properties of the synthesized ferrites [20].

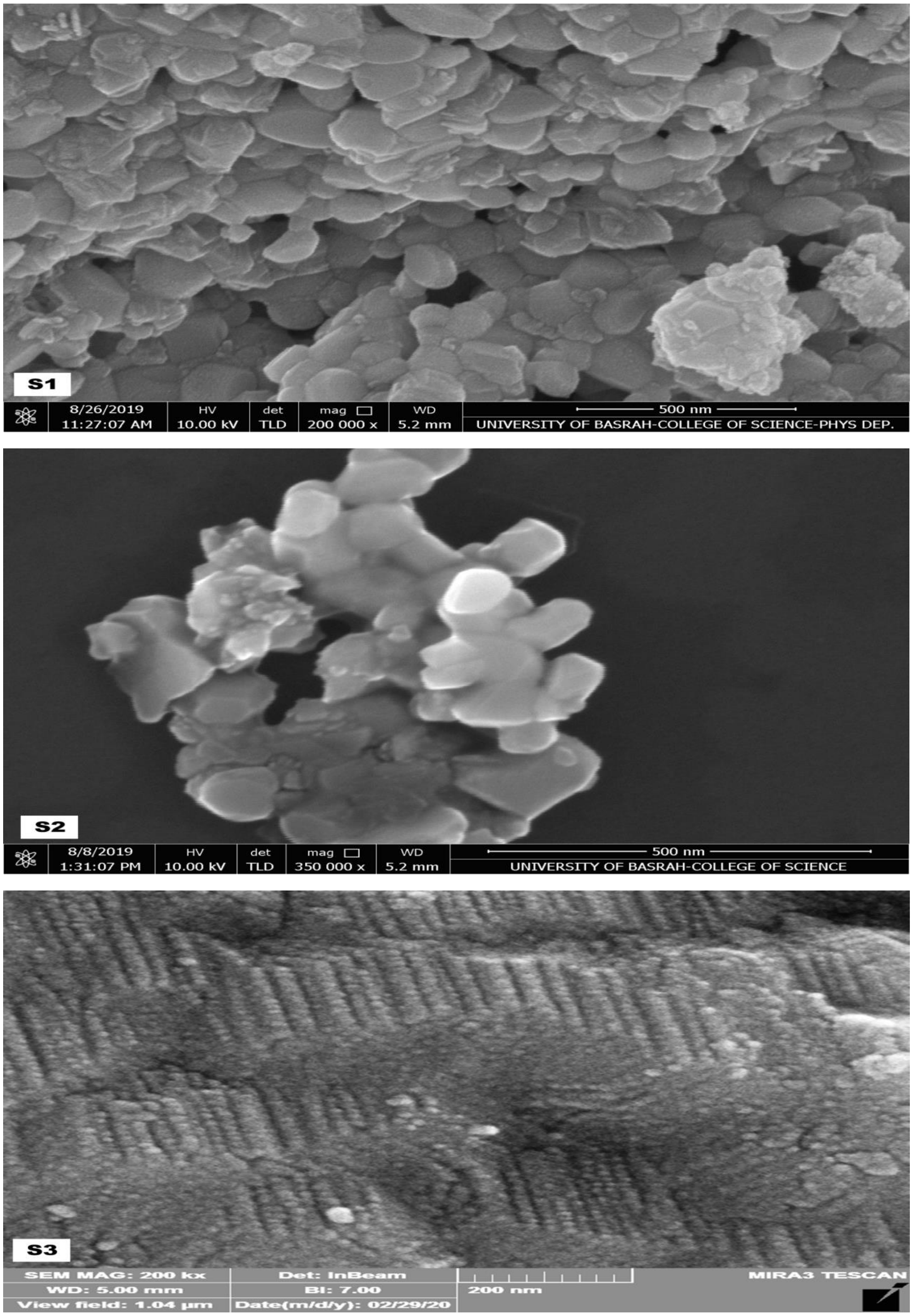

Figure 3: FE-SEM images of as- synthesezid spine $ZnFe_2O_4$ ferrite by Microwave – assisted combustion method with different glycine-to-nitrate ratio S1) glycine to nitrates ratio :0.66 S2) glycine to nitrates ratio :0.50 S3)glycine to nitrates ratio :0.33.

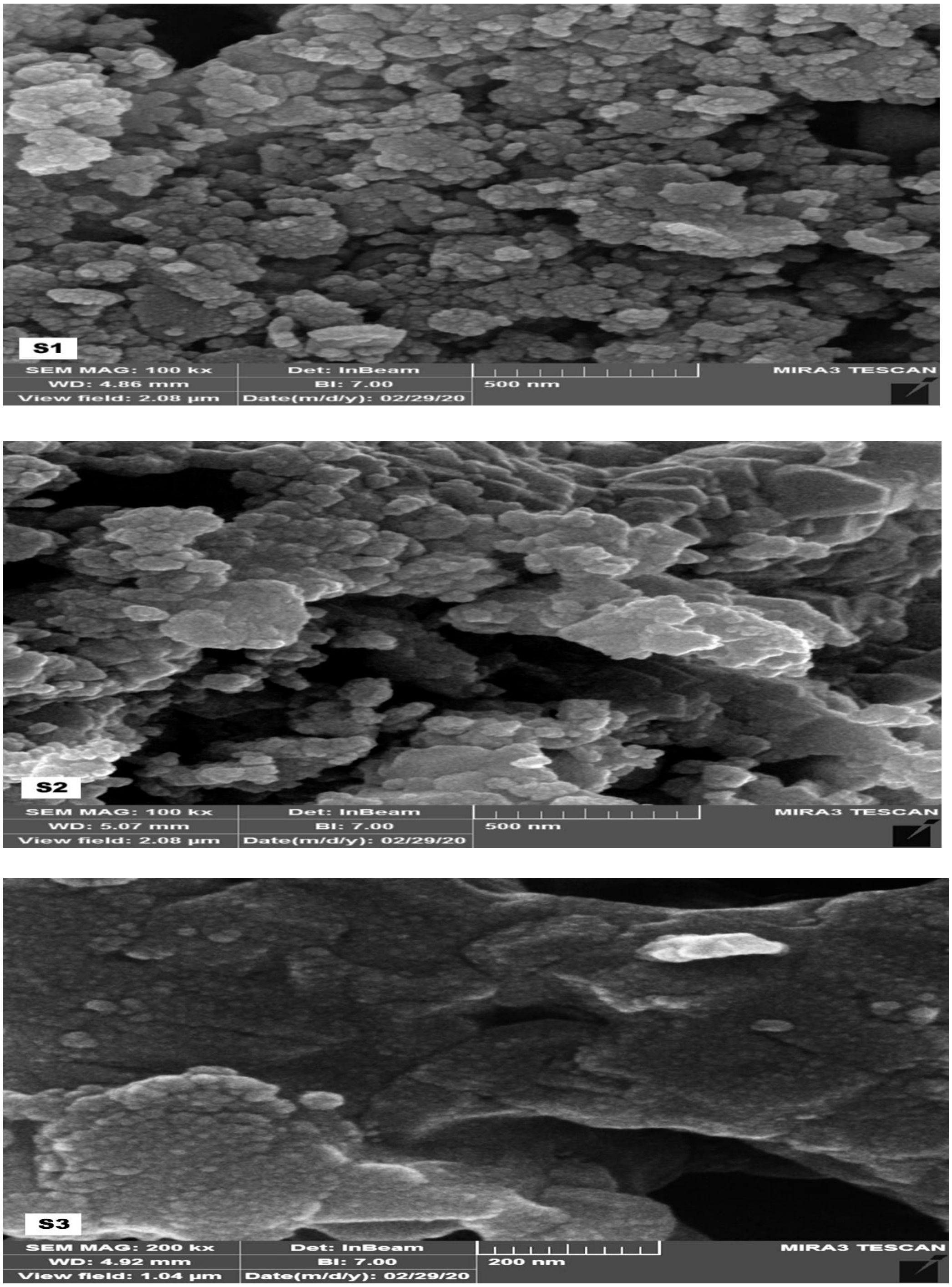

Figure 4: FE-SEM images of synthesezid spine $ZnFe_2O_4$ ferrite by Microwave – assisted combustion method followed by calcination at a temperature of 500 °C for 3h with different glycine-to-nitrate ratio D) glycine to nitrates ratio :0.66 E) glycine to nitrates ratio :0.50 F)glycine to nitrates ratio :0.33.

4.2. Fourier Transforms Infrared spectroscopy characterization

The FT- IR spectra of all synthesized spinel $ZnFe_2O_4$ ferrite before and after calcination via Microwave –assisted combustion method with different glycine to nitrates ratio presented in Fig. 5 and 6, respectively.

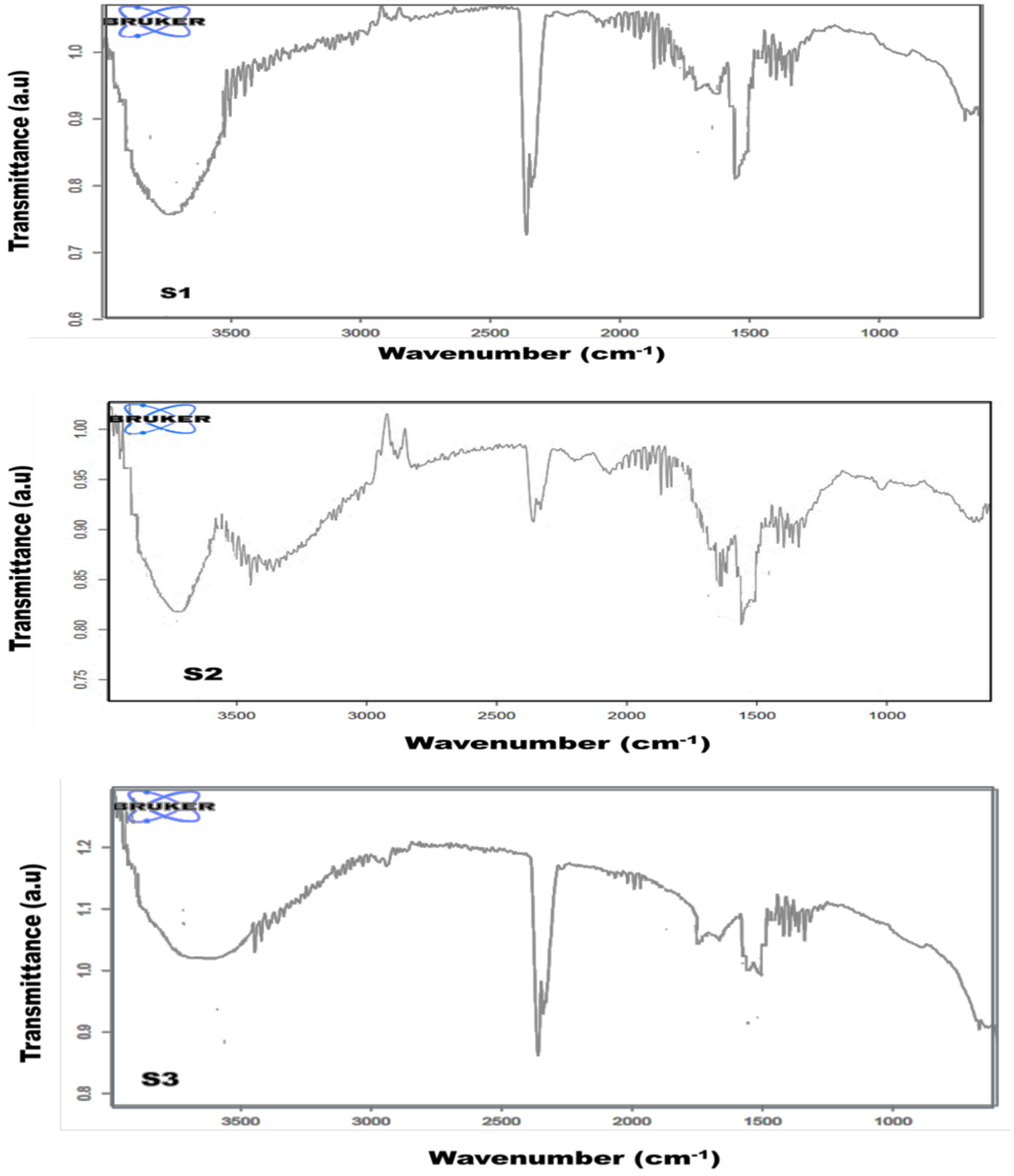

Figure 5: FT-IR spectra for as-synthesized spinel $ZnFe_2O_4$ ferrite via Microwave –assisted combustion method via various glycine-to-nitrates ratio S1: glycine-to-nitrate ratio: 066, S2: glycine-to -nitrate ratio: 050, S3: glycine- to- nitrate ratio: 033.

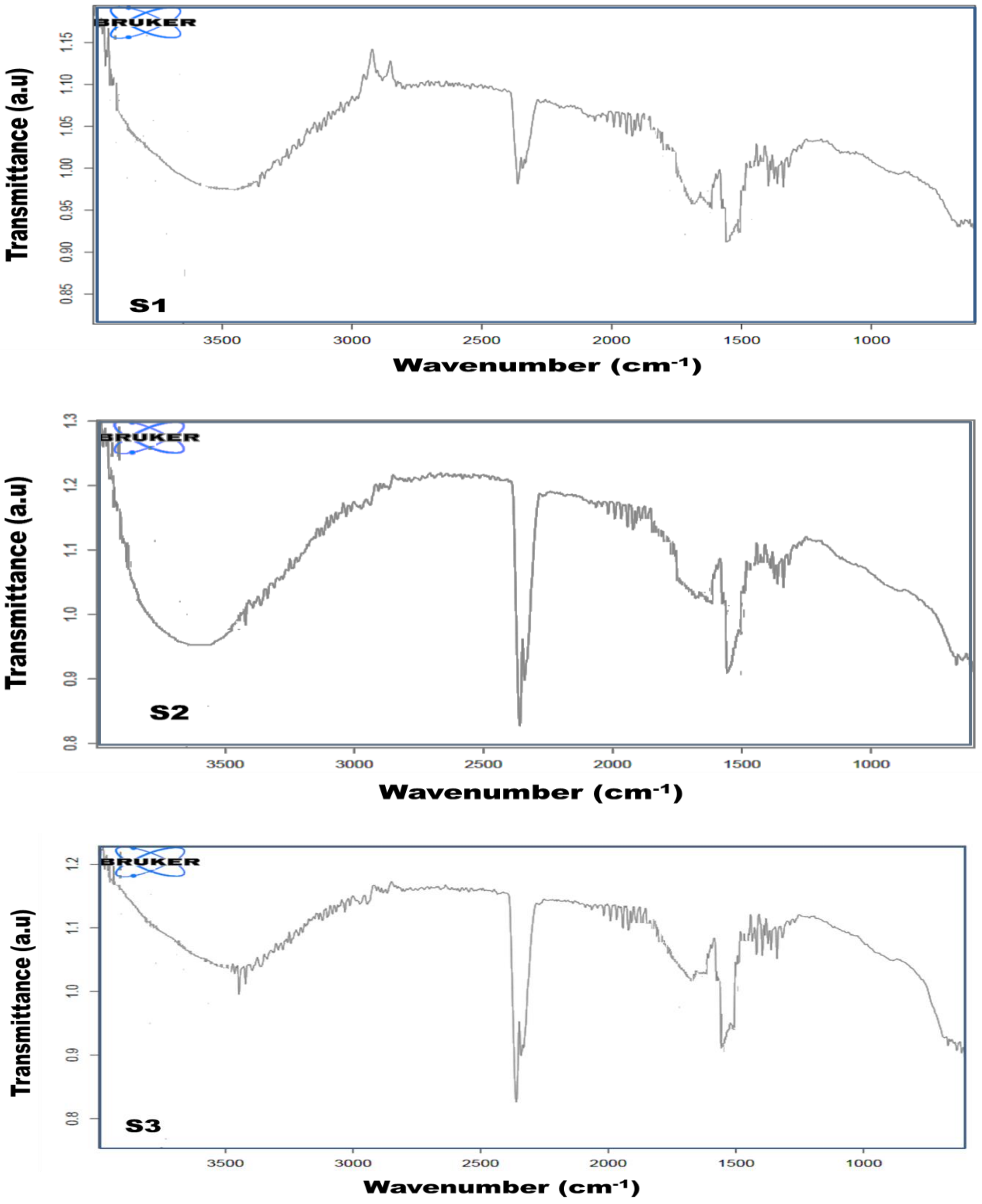

Figure 6: FT-IR spectra for synthesized spinel $ZnFe_2O_4$ ferrite via Microwave – assisted combustion route followed by calcination at 500 °C for 3h via various glycine-to-nitrates ratio S1: glycine-to-nitrate ratio :066 , S2: glycine-to -nitrate ratio :050 , S3: glycine- to- nitrate ratio :033 .

In these figures spinel ferrites structure shows two major bands, υ_A and υ_B. These two vibration bands are related to intrinsic stretching vibration for M $_{Tet}$ ↔ O and octahedral M $_{Oct}$ ↔ O sites [2] [21]. The band that appears between (υ_A,500cm^{-1}-600cm^{-1}) refers to tetrahedral -site, while the band that appears between (υ_B,400 cm^{-1}-480cm^{-1}) corresponding to the octahedral - sites [2].Those bands are features of all ferrites [22].In this visitations the bands of absorption for the spinel $ZnFe_2O_4$ ferrite are lies between (500 cm^{-1} –590 cm^{-1}) for higher wave number υ_A while it is varying between 390 cm^{-1}– 485 cm^{-1} for the lower wave number υ_B. Peak at 1222 cm^{-1} that is weak peak could be assigned to the C–O stretching [23] due to adsorbed CO_2 [24]. The band between 1400 cm^{-1}-1500cm^{-1} is ascribed to a symmetric stretching vibration appearing from the residual nitrate [25].The peaks that appeared between (1620 cm^{-1} and 1650 cm^{-1}) represent C=O vibrations mode. The appeared peaks about (1712 cm^{-1} and 1374 cm^{-1}) are referred to H–O–H vibration in H_2O or OH deformation vibration because of surface hydroxyls [26], [27]; those imply excellent hydrophobicity that is suitable to the photo-catalytic activity of ferrites. The observed peak around (2330 cm^{-1} – 2350 cm^{-1}) corresponds to (C-H) bond stretching [2] Abroad band at (3000 cm^{-1} and 3600 cm^{-1}) is observed, which corresponds to a stretching (O-H) group, representing the presence of water as moisture . [2], [23]. There appears to be a peak in S1at around at 3700cm-1 is assignable to O-H band from water, when the product powders were washed with distills water after synthesis [24].

5- Conclusions

Three precursor solutions of spinel zinc ferrite were successfully fabricated via rapid and simple route (microwave combustion) followed by calcination at temperature of 500 °C for three hours. Results of XRD refereed to cubic spinel - structure with space group Fd3m . average crystallite size is increase when Glycine –to-nitrate ratio is decreased 15.8 nm to 26.53 nm . average crystallite size is also increase with calcination at 500 °C (23.03 to 28.16) .The average lattice constants is changed from (8.407 t0 8.391)˚A and then it increase from (8.391 to 8.427)˚A for as –synthesized samples. However the calcination result in A small increase in average lattice constant. These results indicate that a change in the fuel amount lead to significant changes in the morphology of the powders. The FESEM images show that as-synthesized Zinc ferrite possess uniform, symmetry , In addition to that FE-SEM images for mentioned samples which are calcinated at 500c for three hours showed their agglomeration and like-spherical shape.

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المستخلص

تم تصنيع ثلاثة نماذج من فرايتات الزنك بطريقة بسيطة و سريعة (الاحتراق بالميكروويف) متبوعة بتحميص بدرجة حرارة 500 درجة مئوية لمدة ثلاث ساعات. تم استخدام املاح نترات الفرك والزنك كمادة اساسيه في تحضير العينات ، بينما تم استخدام الجلايسين كوقود. تم تقدير نقاء الطور ومعلمة الشبكة البلورية باستخدام حيود الاشعة السينية. ومن خلال صور جهاز المجهر الإلكتروني الماسح الضوئي (FE-SEM) تم تحديد حجم الجسيمات لعينات المسحوق المحضرة وتشكلاتها . و لدراسة انماط الاهتزاز في الفريتات تم استخدام جهاز طيف الاشعة تحت الحمراء (FT-IR) . أظهرت نتائج العينات المحضرة أن هناك تغيرًا ملحوظًا في نقاء الطور ومعلمة الشبكة البلورية وحجم الجسيمات والتشكيلات مع التحميص. نتائج فحوصات حيود الاشعة السينية اثبتت ظهور التركيب البلوري المكعب من نوع السبينل المجموعة Fd-3m. كما ان متوسط حجم البلورات يزداد عندما تنخفض نسبة الجلايسين إلى النترات حيث يتم زيادتها من 15.8 نانومتر إلى 26.53 نانومتر بالنسبة للعينات المصنعة بدون تحميص ومن (23.03 إلى 28.16) للعينات التي تم تحميصها عند 500 درجة مئوية. وكذلك أظهرت نتائج حيود الاشعة السينية أن متوسط ثوابت الشبكة يتغير من (8.407 إلى 8.391) انكستروم ثم يزيد من (8.391 إلى 8.427) انكستروم للعينات المصنعة. اضافة الى ذلك ، ينتج عن التكليس زيادة في متوسط حجم البلورات ومتوسط ثابت الشبكة. أشارت صورة جهاز المجهر الإلكتروني الماسح الضوئي إلى أن جسيمات الزنك فرايت تمتلك شكل موحد ومتناسق.