Studying the structural properties of Mg-Zn ferrite nano partical prepared by auto combustion Sol-gel method

Khalid Jubeir Jameel[1] , Sabah Mohamed[2] , Ameer Sh. Mahmoud[1]

[1] *Department of Physics, College of Education for Pure Sciences, University of Tikrit , Tikrit , Iraq*

[2] *Department of Physics, College of Education for Pure Sciences, University of Kirkuk , Kirkuk , Iraq*

Abstract

In the present work the Magnesium-zinc ferrite chain ($Mg_{1-x}Zn_xFe_2O_4$) with x=0-1,were synthesized using sol-gel auto combustion as a modern chemical methods .Nitrates, citric acid and ammonia were used in order to get ferrite nano powders at temperature 220^0C , which characterized a higher dispersion and homogeneity with particle size in the range of (27.47-68.9) nm. The obtained ferrite nano powder calcined at temperature 500^0C for a period three hours .

Samples examined by Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM). From FTIR spectral analysis the structure of the spinel ferrites shows two absorption bands, υ_1 and υ_2 , due to vibration of octahedral and tetrahedral metal oxygen bonds. SEM results show the presence of uniformly distributed and almost spherical shaped particles.

Introduction

Ferrites, both soft and hard, are used in many technological applications because of their excellent magnetic and electrical properties. Spinel ferrite (soft ferrite) with the general formula MFe_2O_4, where M = Mn, Co, Ni, Cu or Zn or a mixture of these ions, posses unique electronic or physical structure and chemical stability [1,2]. It is an ideal material for high-frequency passive components because of its high permeability, resistivity and permittivity. These materials have been used technologically in magnetic recording media and magnetic fluids for the storage and/or retrieval of information, magnetic resonance imaging (MRI) enhancement, catalysis, magnetically guided drug deliveries, sensors and pigments [3,4]. The magnetic properties of these ferrites can be changed by the substitutions of various kinds of M^{2+} among diva- lent cations (Zn^{2+}, Mn^{2+}, Ni^{2+}, Mg^{2+}, Co^{2+}, Cu^{2+}, etc.) or by introducing a relatively small amount of rare-earth ions [5]. However, the properties of these materials are determined by their chemical composition, microstructure and process mechanism. So, the interesting features are that their properties can be enhanced by controlling the chemical composition and the preparation methodology[5].

Experimental

Mg-Zn ferrites have the general formula $Mg_{1-x}Zn_xFe_2O_4$ for (X) values (where x= 0.0-1.0) . Were synthesized by the Sol-gel Auto combustion After the weight of nitrates, appropriate amount of distilled water was added to them, complete mixing of all metal nitrates and citric Acid in distiller water . The mixture placed in a glass beaker, all these are collected in a glass beaker to become a total solution and mixed well at room temperature by magnetic stirrer with high velocities and after a short period until solution becomes smooth and a slimy red colored as shown in figure(1-a). Ammonia solution was slowly added in the form of drops into the mixed solution to control its pH until reach the value of (6.9 to7) with continuous stirring and so the solution become a dark brown color as shown in the figure (1-b).

It was subsequently raise the temperature of the solution to 60°C for a period of one hour and then increase the temperature to 80°C. After that the size of the solution in the beaker glass be less with high viscosity and after 30 minutes, the solution viscosity is very high, hence the beginning of gel formation on the surface of the solution. Particularly in the middle and then all the solution turn to gel, and even this moment, the solution is still on the magnetic stirrer and temperature 80^0C as shown in the figure (1-c).

After the completion of the solution turned to gel, the temperature drops to the room temperature and this gel becomes dry and dark brown. Where the weight of the gel putting in glass beaker by a sensitive balance and then put it inside the oven at a temperature 120^0C for three hours to become dry gel and decreasing in weight as shown in figure (1-d). Then evaporation of some of the material at raise the temperature of the dried gel to 220^0C, after 15 minutes the dry gel began to change shape appear convexity in the center of the glass beaker as shown figure(1-e).

After a short period we note a flame at the top of convexity where the flame spread in all directions to burn the top layer of dried gel and turn into a grid of columns converging and intersecting at random and ascend to the top and then burn another layer of dried gel and ascend to the top also. And so until the gel flammable in complete and be the end of the flame in the bottom of the glass beaker and thus becomes all the dried gel after combustion to a fine powder with a dark gray color, which marks the beginning of formation of high purity ferrite as shown also in the figure (1-f). Briefly, we can describe the state that our saw in the following; the dried gel was placed on a hot plate at 220°C. Upon ignition, dried gel burnt in a self-propagating combustion manner until all gels were completely burnt out to form a fluffy loose structure, the fluffy material was ground to get ferrite powder..

Results and Discussion

SEM analysis.

The morphological characteristic surface of ferrite samples were studied by SEM. The SEM images of $Mg_{0.5}Zn_{0.5}Fe_2O_4$ ferrite powders calcined at temperature 200 and 500^0C are shown in figures (2) and (3) respectively, which reveal remarkable change in the microstructure .

Figure (2) contain two SEM images with different magnification degree for the same sample prepared at a temperature of 220 ^{0}C. Figure (2) shows the formation of plate like resembles spongy and fragile structure. The surface of the ferrite sample has a number of fine pores between the agglomerated particles, which are attributed to the large amount of gases liberated during the self-combustion process. Also the SEM images shows the morphology of particle were almost spherically.

Figure (3) contain two SEM images at different magnification degree for the powder ferrite calcined at temperature 500^0C. Figure (3) shows more clearly that agglomerates particle are closer to spherical and the presence of pores between particles become less than that showed in figure (2) due to the more gases liberated during the calcination process.

Fourier transform infrared analysis(FTIR).

The IR spectra of the series at room temperature for the typical samples with *X*= (0-1) Nano ferrites calcinated at 500^oC for three hours were shown in figure (4). in which the structure of the spinel ferrites shows two absorption bands , $\upsilon_1$andυ_2 , due to vibration of octahedral and tetrahedral metal oxygen bonds. The band υ_1 is for the tetrahedral position and υ_2 is for the octahedral position ,the intensity of the absorption bands depends largely on method of synthesis and substituted cation . The difference in the position of these two bands is due to the difference in bond length between metal and oxygen atoms at tetrahedral and octahedral sites.

The absorption bands for the ferrites are found to be in the expected range. The high frequency band υ_1 lies in the range 500–630 cm^{-1} while the low frequency band . Is varying in the 390–445 cm^{-1} range. This difference in the band position is expected because of the difference in the Fe^{+3}–O^{2-} distance for the octahedral and tetrahedral compounds.

Waldron [6] and Heffner [7] studied the vibrational spectra of ferrites and attributed the υ_1 band to the intrinsic vibrations of the tetrahedral groups and υ_2 the octahedral groups.

The broad peak observed at ~3415 cm^{-1} is attributed to the stretching vibration of surface hydroxyls absorbed on surface and O–H groups in absorbed water, which indicates the adsorbed moister [10]. The peaks at ~1712 cm^{-1} and ~1374 cm^{-1} are assigned to H–O–H bending vibration in water molecular or OH deformation vibration due to surface hydroxyls[8,9]. These imply good hydrophobicity that is favorable to the photo catalytic activity of the nanoparticles. The weak peak at 1222 cm^{-1} could be assigned to the C–O stretching[10] due to adsorbed CO_2. The two peaks at ~555 cm^{-1} and below 424 cm^{-1} are assigned as the vibration of ferrite groups [11], corresponding to the tetrahedral and octahedral sites of positive ions in the ferrite, respectively. The absorption peak centered at 555 cm^{-1} overall shifts to low wavenumber as increase in Zn content. This can be ascribed to the increase in distance between Fe^{3+}–O^{2-} in the tetrahedral sites. This confirms the formation of $Mg_{1-x}Zn_xFe_2O_4$ structure.

Conclusions

1. Nano crystalline Mg-Zn ferrites Nano powder was successfully synthesized by the ignition of gel precursor upon heating at 220°C.

2. The absorption bands $\upsilon_1$and υ_2 are found in the expected range and the difference in band positions is due to the difference in the Fe^{3+}-O^{2-} for the octahedral and tetrahedral complexes.

3. The positions absorption bands are compositional dependent whose dependence could be attributed to the variation in cation oxygen bond distances.

4. Particle size increases as the calcined temperature increases

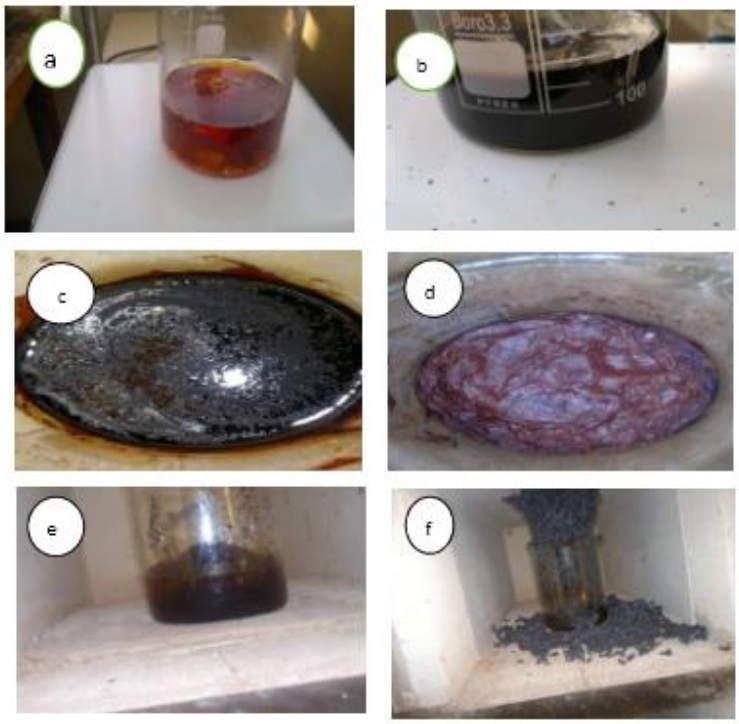
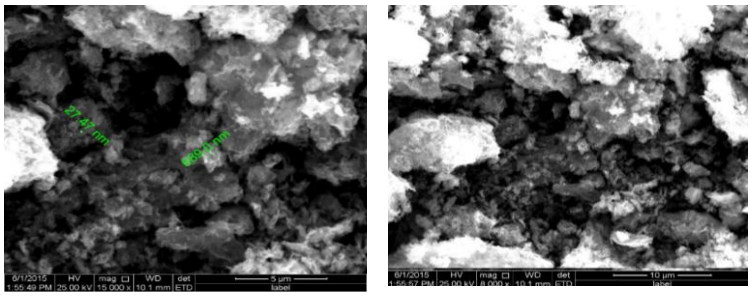
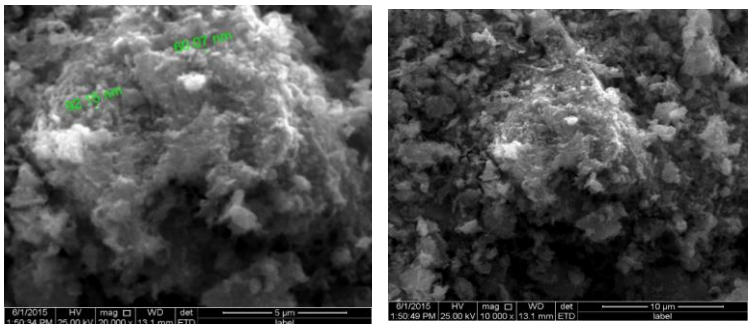


Figure (1): Photograph of (a) nitrate-citrate solution, (b) The solution after the Adding of ammonia, (c) dry gel, (d) Dry bulk temperature of 120^{0}C, (e)) Dry bulk temperature of 220^{0}C and (f) auto combustion and become Nano powder ferrite



Figure(2). SEM micrographs for $Mg_{0.5}Zn_{0.5}Fe_2o_4$ prepared at 220^{o}C.



Figure(3). SEM micrographs for $Mg_{0.5}Zn_{0.5}Fe_2o_4$ prepared at 500^{o}C.

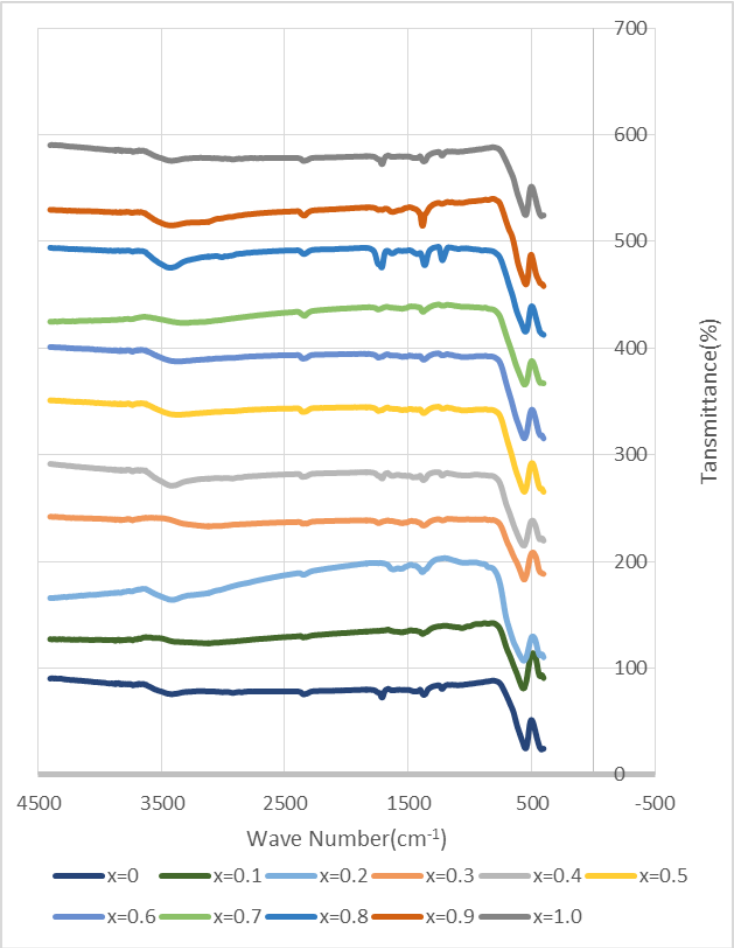


Fig (4). The IR absorption spectra of $Mg_{1-x}Zn_xFe_2o_4$ ferrites.

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دراسة الخواص التركيبية لفرايت Mg-Zn المحضرة بطريقه Sol-gel

خالد جبير جميل[1] ، صباح محمد علي[2] ، عامر شاكر محمود[1]

[1]قسم الفيزياء ، كلية التربية للعلوم الصرفة ، جامعة تكريت ، تكريت ، العراق

[2]قسم الفيزياء ، كلية التربية للعلوم الصرفة ، جامعة كركوك ، كركوك ، العراق

الملخص

تم في هذا البحث تحضير فيرايت المغنيسيوم المعوضة بأيونات الزنك ذات السلسلة الفيراتية $Mg_{1-x}Zn_xFe_2O_4$, حيث تأخذ X قيم من الصفر الى 1) بالطريقة الكيميائية الحديثة (sol-gel auto combustion) وقد استخدمت النترات وحامض الستريك والامونيا للحصول على مسحوق فيرايتي ذات دقائق نانوية عند درجة حرارة قيمتها 220^oC يتميز بالتشتت والتجانس العالي وبأحجام نانوية في المدى(27.47-68.9) نانومتر,وتم كلسنه المسحوق المنتج بدرجه حرارة 500^oC ولمدة ثلاث ساعات متواصلة.

فحصت العينات بواسطه المجهر الالكتروني الماسح وطيف الأشعة تحت الحمراء. من تحليل الأشعة تحت الحمراء تبين تركيب السبنل فرايت يتكون من آصرتين υ_1 وυ_2 ناتجه من هتزاز معادن رباعيه السطوح وثمانية السطوح المرتبطة بالأوكسجين. نتائج المجهر الالكتروني الماسح بينت وجود توزيع موحد والجسيمات ذات شكل كروي.