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Effect of Molar Concentration of Zinc Ferrite ($ZnFe_2O_4$) Nano Particles Prepared by Sol-Gel on Microwaves Attenuation

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Abstract

*$NO_3)_3.9H_2O$ and $Zn(NO_3)_2.6H_2O$ by adopting molecular concentrations (0.2 M and 0.4 M). The samples were sintered at two temperatures (800 °C , 1000°C) for two hours. The spinal phase of zinc ferrite structure and crystallite size was examined by XRD spectrum, pattern show that nanoparticles structure exhibit mixed phase of α-Fe_2O_3 and $ZnFe_2O_4$. On The absorbance tests of the Zinc ferrite samples have been carried out for all samples by using the network analyzer device, at the X-band range (8-12) GHz. . The samples were sintered at 800°C, and 1000°C, and the concentration (0.2 and 0.4)M and the Dimensions of the samples is (23*10*10)mm. The absorbance greeter than (95) at frequency (8-12)GHz.*

Keywords: Molar Concentration, Zinc Ferrite, Sol-Gel, Microwaves Attenuation.

Introduction

The sol gel processes have given many advantages such as best mixing of the raw materials and excellent homogeneity, ultrafine and reproducible zinc ferrites with small size distribution. The homogeneous microstructure of zinc ferrite indicating the ability to control the electric-magnetic properties and heat treatments temperature which decrease the impurities generated during the preparation and variation in the composition [1]. The applications for sol gel-derived products are numerous. For example, scientists have used it to produce the world's lightest materials and also some of its toughest ceramics. One of the largest application areas is thin films, which can be produced on a piece of substrate by spin coating or dip coating. Protective and decorative coatings, and electro-optic components can be applied to glass, metal and other types of substrates with these methods [2] at many works before this results shows the effect of increasing and decreasing of concentrations at all compounds that used. Some researches shows the increasing of grain size by decreasing zinc concentrations [3]. And in another hand surface structural can be changed with zinc concentrations [4].

The microwaves absorbing materials were gained a much attention due to the absorb energy from microwaves and can be used in the stealth technology development of aircraft television image[5].

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Nanostructure of zinc ferrites showed interesting physical and chemical properties due to very small grain size and large surface area .The sol–gel chemical methods have been used to prepare of different mixed metal oxides, nanomaterial's, Nano scale, nanoporous oxides[6] The absorbance tests of the Zinc ferrite samples have been carried out for all samples by using the network analyzer device, at the X-band range (8-12) GHz ,Absorption of electromagnetic waves within the absorber material must be permeate the absorber surface without reflection in this case the impedance of the material must be equal the impedance of free space, the enter electromagnetic waves into radar absorbing materials, a part of energy will be scattered and other part of energy will be absorbed by absorber material[7] In our work, zinc ferrites with excellent magnetic properties were prepared by a sol-gel technique and the microwaves absorption properties of these composites were investigated and At this we will see the effect of increasing concentrations for all using materials and discussion results in any state.

Experimental Procedure

Sol-gel method was used to prepared $ZnFe_2O_4$ nanoparticles. The calculated amounts of the nitrates and Citric Acid according to their atomic/molecular weights, the raw materials used in the formation of each sample were mixed with (100 ml) of distilled water in a glass beaker, was homogenized by continuous stirring by magnetic hot plat-stirrers model (L-81).The combination consist of three solution, (0.2M) of zinc nitrate $Zn(NO_3)$, (0.2M) of iron nitrate $Fe(NO_3)$, were used as a precursor solution and were gelatin by adding 100 ml of citric acid (C_6H_8 O_7) solution with concentration (0.2M). The next

concentrations is (0.4M) of iron nitrate, (0.4M) of zinc nitrate also 100 ml of citric acid. The control on pH of the solution was fined at (7.5) by using many drops of ammonia and the solution was heated on the hot plate at 60Cº for 40 min. The temperature of solution increased to 80Cº for (8) hours, the solution was turned into gel. The gel material dried by Leave it several hours. every gel compounds annealed at (800 Cº and 1000 Cº) for three hours, to made tow samples can by studied before grinding process. The fluffy material was ground to get ferrite powder using the Handmade grinder for five minutes, Powders are mixed with glycerin material 7 drop with7gm of zinc ferrite powders, The powder was pushed at (3 Tons) by the piston oil to obtain samples as Parallelogram of ($23\times10\times10$) mm in dimensions, where template was used for this purpose, The samples were sintered, for 2 hr at tow sintering temperatures, (800ºC, 1000 ºC) , then were left to be spontaneously cooling inside the furnace. The structure and the average grain size of the spinal zinc ferrites nanoparticles were measured by X-Ray diffractometer (6000-Shimadzu) by using CuKa (α) radiation source with a wavelength, λ=1.54060 Å. The absorbance tests of the Zinc ferrite samples have been carried out for all samples by using the network analyzer device, at the X-band range (8-12) GHz.

Results and Discussion

X-Ray diffraction

The results of the tests showed the X-ray diffraction technique of the samples prepared at different temperatures as having a poly -crystallization and cube and The results shows increasing of crystalline grain size with increasing of annealing temperature , also the samples that have low concentrations more larger grain size then samples have high concentrations, and that returned to zinc concentrations level [8]. Results will illustrate in a fig.(1) for samples have concentration 0.2 M and fig.(2) for samples have concentration 0.4 M .

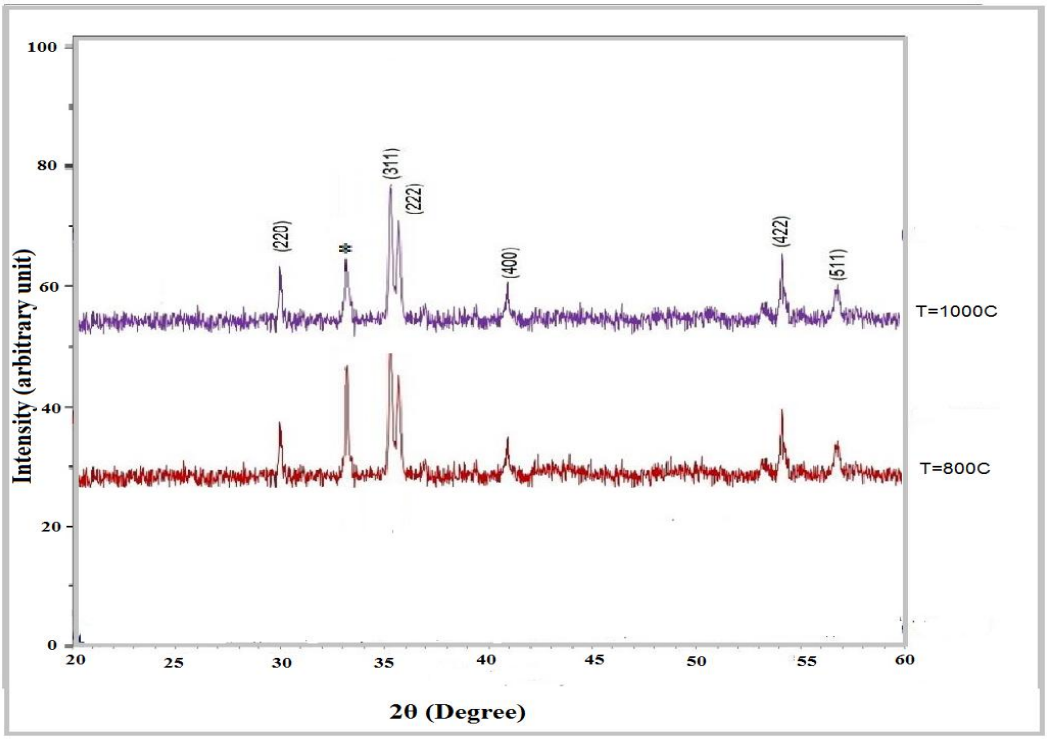

Figure I

Show X-ray diffraction curves for zinc-ferrite models at temperatures (800, 1000) C and concentrations of 0.2M.

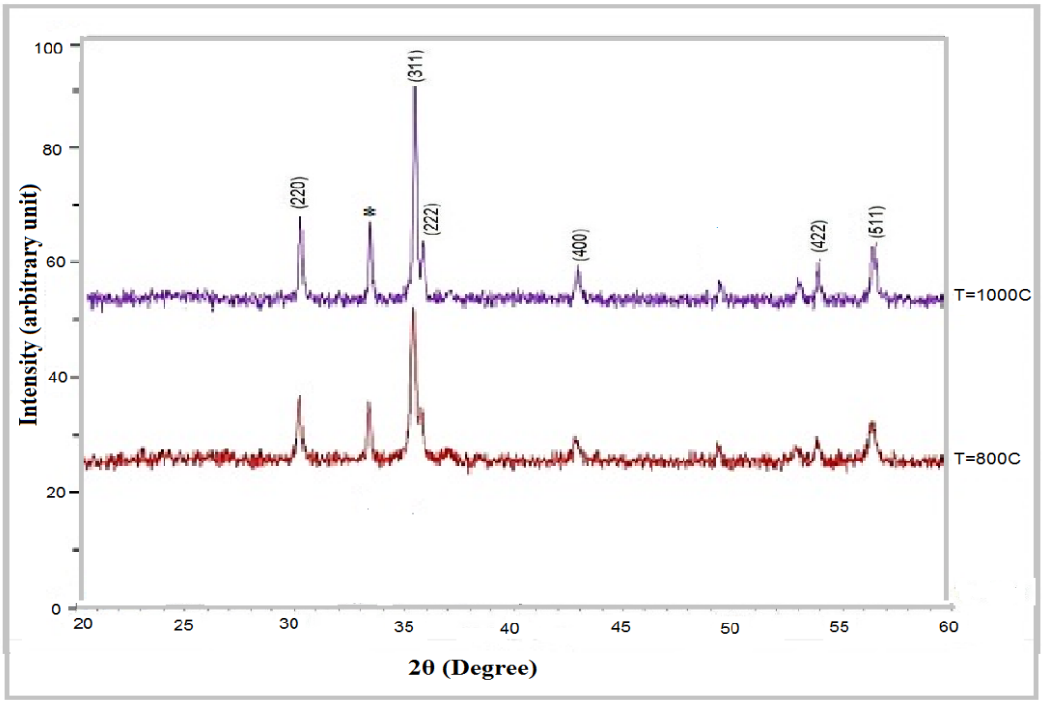

Figure II
Show X-ray diffraction curves for zinc-ferrite models at temperatures (800, 1000) C^{o} and concentrations of 0.4M.

The (*) is α- Fe_2o_3 and the planes surface is (104) a peak of α -Fe_2O_3 phase observed at 2 θ = 33.2º indicates that the single phase of cubic spinel structure $ZnFe_2O_4$ is not yet completely formed. The figure(4-1) and (4-2) shows the X-ray diffraction of all the prepared models during the tests, they were analyzed and knowledge of the sites of the peaks where the following planes surface (220), (104), (311), (222), (400), (313), (422), (511), and The corresponding angles are 2θ = 29.9, 33.2, 35.2, 35.5, 43.0, 49.5, 54.1, and 56.6, Respectively. the scan range from 20º to 60º. the trend for growth of all prepared samples is (311). When the temperature increases, Note the same planes surface Appear But When the concentration is increased, Note a relative change in intensity of the planes surface with a clear dominance and Clear growth of the (311) (and these results are largely consistent with the (Joint Committee of Powder Diffraction Standards (JCPDS 96-900-6908)). As shown in the table, (4-1)and (4-2). It is also shown that the increase in the temperature is affected on the intensity of the peaks, especially the prevailing trend (311) for the prepared zinc ferrite samples, leading to a decrease in the values of the Full width at half maximum (FWHM) with the rise of the temperature and molar concentrations .The tables can be more clear for explain the changed in structural because in above figures it too similar and the changing shows in values more differ .

Table 1
Structural calculation of 0.2 M samples

Annealing temperature C^{o}	2θ (Deg.)	FWHM (Deg.)	dhkl Exp.(Å)	G.S (nm)	hkl	phase
800 C^{o}	29.9	0.177136259	2.985343014	46.48231912	(220)	ZnFe2O4
	33.2	0.180946882	2.697963205	45.87224653	(104)	α Fe_2o_3
	35.2	0.163256351	2.548983575	51.11681549	(311)	ZnFe2O4
	35.5	0.255704388	2.527641542	32.66377474	(222)	ZnFe2O4
	42.8	0.508083141	2.103458171	16.82594456	(400)	ZnFe2O4
	54.1	0.193325635	1.695438746	46.19322733	(422)	ZnFe2O4

	56.6	0.431893764	1.624942633	20.91999083	(511)	ZnFe2O4
1000 C°	30	0.369515012	2.985343014	22.28246173	(220)	$ZnFe_2O_4$
	33.3	0.138568129	2.697963205	59.90150859	(104)	α Fe_2o_3
	35.4	0.133325635	2.536959053	62.62212955	(311)	$ZnFe_2O_4$
	35.7	0.209515012	2.517874915	39.88066644	(222)	$ZnFe_2O_4$
	43	0.323325635	2.105505108	26.43678853	(400)	$ZnFe_2O_4$
	54.2	0.184757506	1.691543547	48.36447668	(422)	$ZnFe_2O_4$
	56.7	0.369515012	1.622940823	24.46030739	(511)	$ZnFe_2O_4$

Table 2
Structural calculation of 0.4 M samples

Annealing temperature C°	2θ (Deg.)	FWHM (Deg.)	dhkl Exp.(Å)	G.S (nm)	hkl	phase
800 C°	30.0	0.125289256	2.975936234	65.7324145	(220)	ZnFe2O4
	33.3	0.160289256	2.687975281	51.80126174	(104)	α Fe_2o_3
	35.4	0.160385675	2.537595129	52.05528826	(311)	ZnFe2O4
	35.8	0.120385675	2.507367999	69.43705162	(222)	ZnFe2O4
	41.0	0.330578512	2.198920834	25.69208294	(400)	ZnFe2O4
	54.2	0.145674931	1.69187378	61.33686385	(422)	ZnFe2O4
	56.8	0.335674931	1.620744166	26.93683692	(511)	ZnFe2O4
1000 C°	30.1	0.123482094	2.974949621	66.69599926	(220)	$ZnFe_2O_4$
	33.3	0.135289256	2.687975281	61.37357797	(104)	$ZnFe_2O_4$
	35.4	0.115289256	2.537595129	72.4171776	(311)	$ZnFe_2O_4$
	35.8	0.085289256	2.507367999	98.01030865	(222)	$ZnFe_2O_4$
	41.0	0.205867769	2.201748963	41.24844209	(400)	$ZnFe_2O_4$
	54.2	0.095674931	1.69187378	93.39168904	(422)	$ZnFe_2O_4$
	56.9	0.078586777	1.619304545	115.0876925	(511)	$ZnFe_2O_4$

Results of Absorbance for $ZnFe_2O_4$ samples prepared using Sol-Gel Methods:

The absorbance tests of the Zinc ferrite samples have been carried out for all samples by using the network analyzer device, at the X-band range (8-12) GHz. The samples were sintered at 800°C, and 1000°C, and the concentration (0.2 and 0.4)M and the Dimensions of the samples is (23*10*10)mm. However, the study calculated the parameters Γ^2, A^2 by using the equations

$$\Gamma = X \pm \sqrt{X^2 - 1} \quad (1)$$

$$\Gamma^2 + A^2 = 1 \quad (2)$$

Where:

$$X = \frac{S_{11}^2 - S_{21}^2 + 1}{2S_{11}} \quad (3)$$

A: is the absorption coefficient

S_{11} , S_{21} : is the Scattering Parameters

respectively:And the graphs which include curves of S_{11} , S_{21}, Γ^2, A^2, curves as a function of frequency at tow sintering temperatures (800and 1000)C^o and two concentration (0.2 and 0.4)M. In all Figs.(3) the first Column And the second Column represents the value of Γ^2 and A^2 ,where the blue color represents the Γ^2 and the red color represents the A^2.

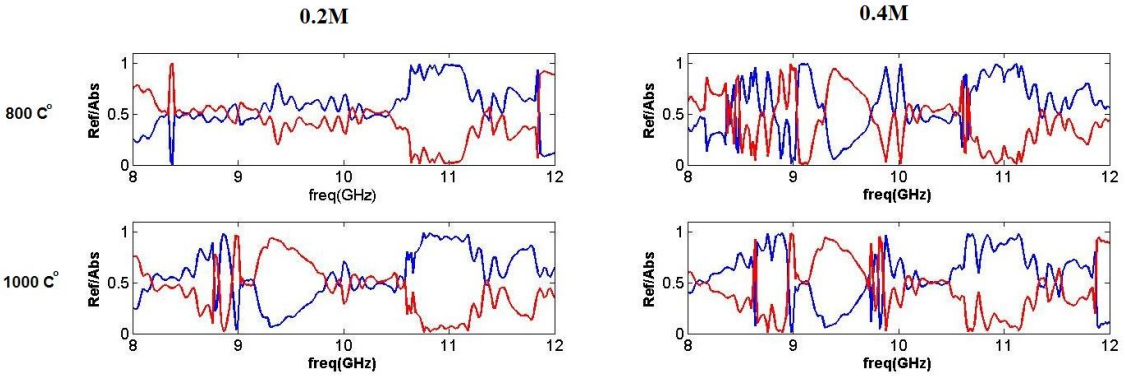

Figure III
The Ref/Abs as a function to the frequency at two different temperatures(800,1000) for (0.2 M) and (0.4 M) concentrations

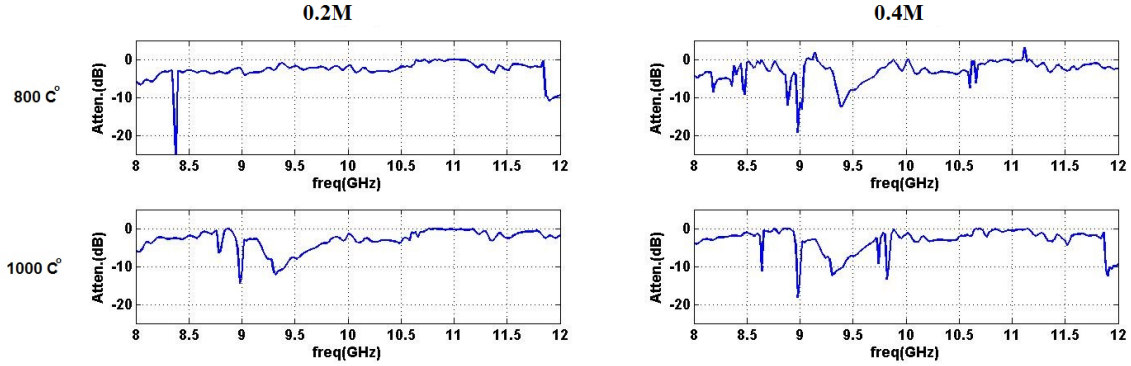

Figure IV
The attenuation coefficient as a function to the frequency at two different temperatures(800,1000) for (0.2 M) and (0.4 M) concentrations

Absorbance of $ZnFe_2O_4$ at (800C^0)and (0.2M)

From Figs.(3) and(4)The highest values of the absorption at 800°C and (0.2M) are**(**0.79, 0.90, 0.62, 0.57, 0.91**)** at frequency (8, 8.3, 9, 11.3**,** 11.8**)** GHz respectively and the attenuation coefficient values corresponding respectively are(-6.7, -24.7, -4.3, -3.5, -10.8).

Absorbance of $ZnFe_2O_4$ at (1000C^0)and (0.2M)

From Figs.(3) and(4)The highest values of the absorption at 1000°C and (0.2M) are**(**0.76, 0.90, 0.91, 0.60, 0.45**)** at frequency (8, 8.9, 9.3, 10.4, 11.9**)** GHz respectively and the attenuation coefficient values corresponding respectively are(-3.9, -14.3, -12.4, -3.5, -2.8).

Absorbance of $ZnFe_2O_4$ at (800C^0)and (0.4M)

From Figs.(4.11) and(4.14)The highest values of the absorption at 800°C and (0.4M) are**(**0.85, 0.90, 0.90, 0.91, 0.83**)** at frequency (8.1, 8.4, 8.9, 9.5, 10.7**)** GHz respectively and the attenuation coefficient values corresponding respectively are(-8.4, -9.1, -19.1, -12.3, -6.1).

Absorbance of $ZnFe_2O_4$ at (1000C^0)and (0.4M)

From Figs.(4.11) and(4.14)The highest values of

the absorption at 1000°C and (0.4M) are**(**0.93, 0.95, 0.95, 0.95, 0.95**)** at frequency (8.6, 8.9, 9.3, 9.8, 11.8**)** GHz respectively **,**and the attenuation coefficient values corresponding respectively are(-11.1, -18.1, -12.3, -13.2, - 12.3).

Conclusions

After examining the prepared materials and analyzing the results, we can assume the following points:

1. From (X-ray diffraction) analysis monitored crystalline structure of ZnFe2O4 has (polycrystalline) structure have the unit cell (cube type) The mainstream is (311).
2. The sol-gel method is considered a method of good results in preparing ferrite materials. This method is considered simple in steps as a comparison for the other methods, The ferrite that formed by this method is pure with big absorption .
3. The sintering temperature has a great impact in forming the ferrite materials and the absorption ability. It has an effect on the amount of grain size and the number of secondary phases.
4. The best value of sintering temperatures is at $1000C^{o}$ for spinel ferrite. This indicates that spinel ferrite misses to the sintering temperature of more than $1000C^{o}$ for the complete formation of ferrite and accepting best absorbance because of high sintering temperature cancels all secondary phases that are made up with ferrite.
5. The factors that lead to the high value of absorption for ferrite materials are the relative magnetic permeability and dielectric constant for this material, specially the imaginary parts that cause material absorption for microwave, where the absorption as caused by the domain rotation, or domain magnetic wall movement, with noticing of the incomplete hysterics magnetic loop in microwave frequencies.

Reference

1. Zahi S. Synthesis, Permeability and Microstructure of the optimal Nickel –Zinc Ferrites by Sol-Gel route. Journal. Electromagnetic Analysis & Applications. 2010; l-2:56–62.
2. J. Phalippou, " Sol-Gel: A Low temperature Process for the Materials of the New Millennium ", The Sol-Gel Gate Away, (2000).
3. Ahmed Saied, Faheim El-Saaey, Abd El Fattah Mustafa Khourshid, "Effect Investigation of Zn Substitution on the characterization of Cobalt Ferrite Nano Particles Prepared Co-precipitation method", IOSR Journal of Applied Chemistry (IOSR-JAC), Nov. 2014 .
4. Nan-Nan Jiang, Yang Yang , " Influence of zinc concentration on structure, complex permittivity and permeability of Ni–Zn ferrites at high frequency" , Journal of Magnetism and Magnetic Materials 401 (2016) 370–377 , School of Physics and Information Technology, Shaanxi Normal University, Xi'an 710119, People's Republic of China.
5. Peng C, Hwang C, Wan J, Tsai J, Chen S. Microwaves absorbing characteristic for the composites of thermal plastic polyurethane(TPU)bonded NiZn ferrites prepared by combustion synthesis method. Materials Science and engineering. 2007; B117:27–36.
6. Gatelyte A, Jasaitis D, Beganskiene A, Kareva A. Sol-Gel Synthesis and Characterization of selected transition metal Nano-Ferrites. Materials Science. 2011; 17(3):302–7.
7. Kingery W, Uhmann D, Bowen H. Introduction to ceramic. 2nd ed. New York: John wiley and son; 1976.
8. Nan-Nan Jiang, Yang Yang , " Influence of zinc concentration on structure, complex permittivity and permeability of Ni–Zn ferrites at high frequency" , Journal of Magnetism and Magnetic Materials 401 (2016) 370–377 , School of Physics and Information Technology, Shaanxi Normal University, Xi'an 710119, People's Republic of China.