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Studying the Effect of the Concentration and Sintering Temperature on the Structural and Optical Properties of Zinc Ferrite ($ZnFe_2O_4$) Preparing by Sol-Gel

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Abstract

Zinc ferrite nanoparticles have been synthesized by using the sol-gel method from $Fe(NO_3)_3.9H_2O$ 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 (600 °C , 800°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 optical properties for all the films were studied by recording the transmittance and absorbance spectra in the range of (200-1000) nm. The results showed that the energy band gap for allowed direct electronic transition varies from (2.63 to 2.97)eV at sintering temperature 600 °C and concentration of 0.2 M,(2.32 to 2.70)eV at sintering tempeature 800 °C and concentration of 0.4 M.

Keywords: Zinc Ferrite, Nanoparticle , Sol-Gel Methods.

Introduction

Nano size zinc ferrite has been the particular subject of study because of its unusual structural, electrical, optical and magnetic properties, which differ from their bulk counterpart. Zinc ferrite is particularly also important because of the strong tetrahedral site preference of zinc ions in zinc ferrite. Zinc ferrite nanoparticles have extremely important in the gas sensor applications [1]. Many methods have been used to prepare magnetic ferrite nanomaterials such as sol-gel methods [2], co-precipitation[3], mechanochemical synthesis [4], hydrothermal/ solvolysis thermal [5,6], spray pyrolysis [7]. The sol–gel methods have been used to prepare of different mixed metal oxides, nanomaterials, nanoscale, nonporous oxides [8]. The sol gel processes have given numerous 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 [2]. At many works before this results shows the effect of increasing and decreasing of concentrations at all compounds that used. Some researches show the increasing of grain size by decreasing zinc concentrations [9]. And in another hand surface structural can be changed with zinc concentrations [10]. Annealing temperature affected directly at grain size and lattice constant affect by concentrations, by increasing temperature grain size increasing too [11]. At this we will see the effect of increasing concentrations for all using materials and discussion results in any case.

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Experimental Procedure

$ZnFe_2O_4$ nanoparticles were prepared by sol-gel methods. The mixture consist of three solution, (0.2M) of iron nitrate $Fe(NO_3)_3.9H_2O$, (0.2M) of zinc nitrate $Zn(NO_3)_2.6H_2O$, were used as a precursor solution and were gelatin by adding 100 ml of citric acid ($C_6H_8O_7$) 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) by using many drops of ammonia and the solution was heated on the hot plate at 60 Cº for 30 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 sintering at (600 Cº and 800 Cº) for two hours, to prepare two samples can by studied before grinding process. 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. Optical properties studied by using (UV-300 Nano) .

Results and Discussion
Structural properties
XRD tests

Results of X-Ray diffraction shows increasing of crystalline grain size with increasing of sintering temperature , also the samples that have low concentrations more larger grain size then samples have high concentrations, and that returned to zinc concentrations level [10]. Results will illustrate in a fig.(1) for samples have concentration 0.2 M and fig.(2) for samples have concentration 0.4 M .

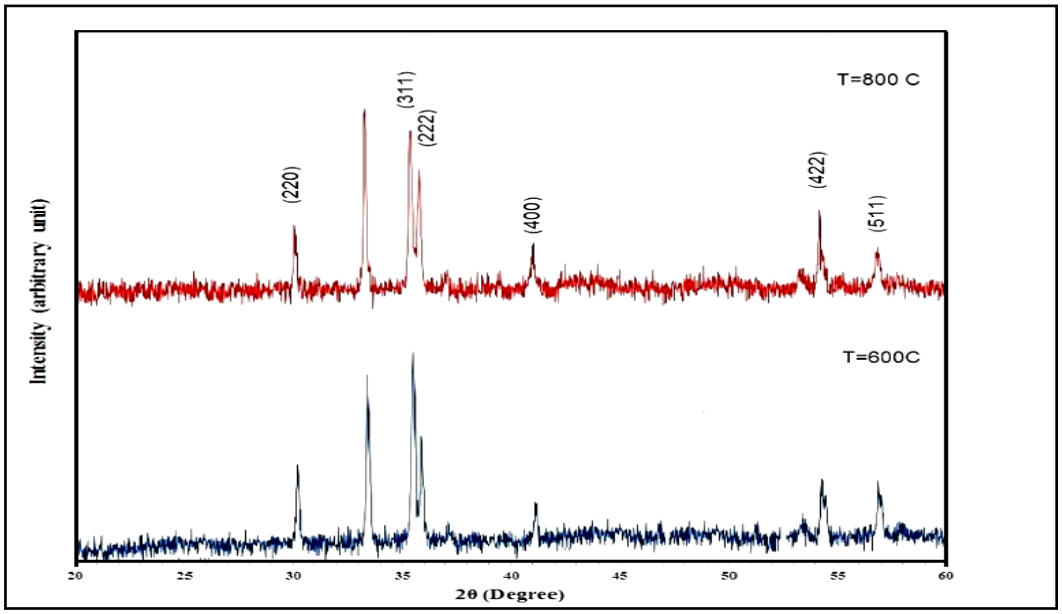

Figure I
XRD test for 0.2 M at two different sintering temperature

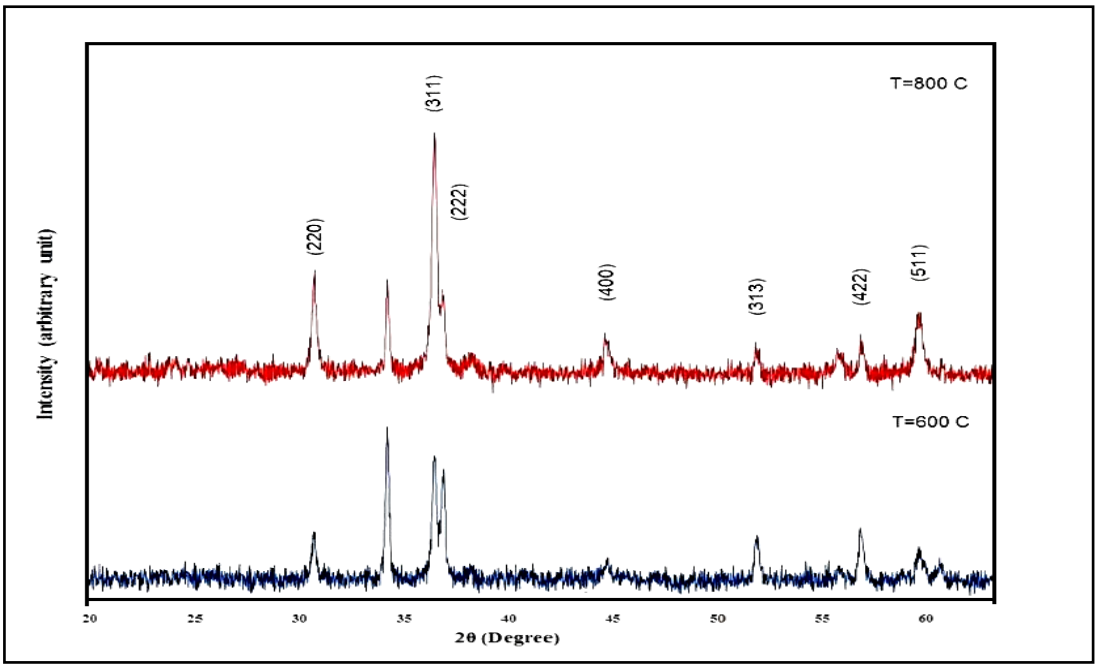

Figure II
XRD test for 0.4 M at two different sintering temperature

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°	2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	G.S (nm)	hkl	phase
600 C°	29.9	0.191363	2.982942	43	(220)	$ZnFe_2O_4$
	33.2	0.138568	2.699141	60	(104)	α-Fe_2O_3
	35.2	0.207136	2.544568	40	(311)	$ZnFe_2O_4$
	35.5	0.217136	2.525347	38	(222)	$ZnFe_2O_4$
	43.0	0.246651	2.103812	35	(400)	$ZnFe_2O_4$
	49.5	0.277136	1.839396	32	(313)	$ZnFe_2O_4$
	54.1	0.277136	1.692849	32	(422)	$ZnFe_2O_4$
	56.6	0.461894	1.624062	20	(511)	$ZnFe_2O_4$
800 C°	29.9	0.28	2.982942	29.67205559	(220)	$ZnFe_2O_4$
	33.2	0.23	2.691855	35.9000404	(104)	α-Fe_2O_3
	35.2	0.32	2.541343	25.78350033	(311)	$ZnFe_2O_4$
	35.5	0.42	2.522173	20.06933049	(222)	$ZnFe_2O_4$
	42.8	0.51	2.108132	16.79729245	(400)	$ZnFe_2O_4$
	49.5	0.46	1.839396	18.94021243	(313)	$ZnFe_2O_4$
	54.1	0.32	1.693831	27.58845165	(422)	$ZnFe_2O_4$
	56.6	0.46	1.622848	19.5409925	(511)	$ZnFe_2O_4$

Table 2
Structural calculation of 0.4 M samples

Annealing temperature C°	2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	G.S (nm)	hkl	phase
600 C°	30.2	0.275482	2.95764	29.86857	(220)	$ZnFe_2O_4$
	33.4	0.185289	2.677216	44.76816	(104)	α-Fe_2O_3
	35.5	0.275482	2.524139	30.28233	(311)	$ZnFe_2O_4$
	35.9	0.210193	2.501629	39.7254	(222)	$ZnFe_2O_4$
	43.1	0.385675	2.095247	22.1496	(400)	$ZnFe_2O_4$
	54.3	0.220386	1.688927	40.50547	(422)	$ZnFe_2O_4$
	56.9	0.385675	1.618002	23.42321	(511)	$ZnFe_2O_4$
800 C°	30.0	0.165289	2.973543	49.76163	(220)	$ZnFe_2O_4$
	33.3	0.165289	2.685814	50.17064	(104)	α-Fe_2O_3
	35.4	0.180386	2.535554	46.22537	(311)	$ZnFe_2O_4$
	35.8	0.120386	2.505351	69.34958	(222)	$ZnFe_2O_4$
	41.0	0.230579	2.197152	36.78875	(400)	$ZnFe_2O_4$
	54.2	0.225675	1.690513	39.54638	(422)	$ZnFe_2O_4$
	56.8	0.275675	1.619441	32.76103	(511)	$ZnFe_2O_4$

AFM tests

The results tests of atomic force microscopy for zinc ferrite powder a small grain size for samples consisted of high zinc concentration, and larger in low concentrations. The next fig.(3) will illustrate 3D image of zinc ferrite compounds. And the table (3) shows all grain size values for all samples to be clearly explanation. The direction of grain's growth is upper towards and the grain had more crystallinity by increasing of sintering temperature. By increasing concentrations cracks disappear and grooves will be more tightly and almost cant seen. The values distributed from (70.35 nm) to (118.99 nm).

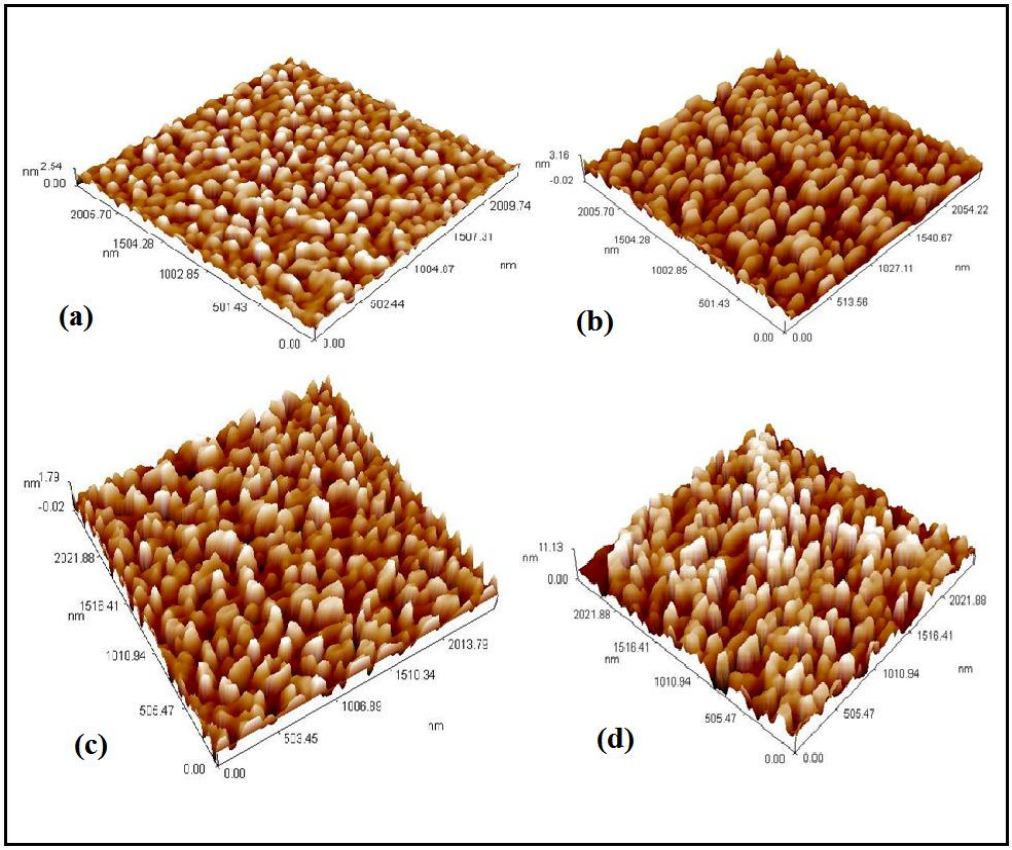

Figure III
AFM (a) & (b) 0.2 M at 600 Cº (c) & (d) 0.4 M at 800 Cº.

Table 3
AFM values according to concentrations and Annealing temperature

concentrations (M)	*annealing temperature (Cº)*	*Avg. Diameter (nm)*	*Root mean square (nm)*	*Roughness Avg. (nm)*	*size (nm)*
0.2	*600*	*88.84*	*0.678*	*0.585*	*2507-2568*
	800	*98.67*	*0.509*	*0.509*	*2507-2512*
0.4	*600*	*70.35*	*0.413*	*0.341*	*2517-2527*
	800	*82.82*	*2.82*	*2.39*	*2527*

Optical properties

Optical properties was measured by (UV-3000 Nano device) . Transmittance shows a little drop of samples that have (0.4 M) specially at 600 Cº sintering treatments , at 800 Cº in both samples didn't shows any clearly changes . fig.(4) will shows transmittance of both sintering degree (600 Cº and 800 Cº) . Absorbance results shows of samples of (0.4 M) a large grain size and so that it was more high of samples that had a concentrations (0.2 M) , with all that effects of concentrations was stronger of sintering effect because of sintering temperature was so close from each other. fig.(5) illustrate absorbance of samples at 600 Cº and 800 Cº.

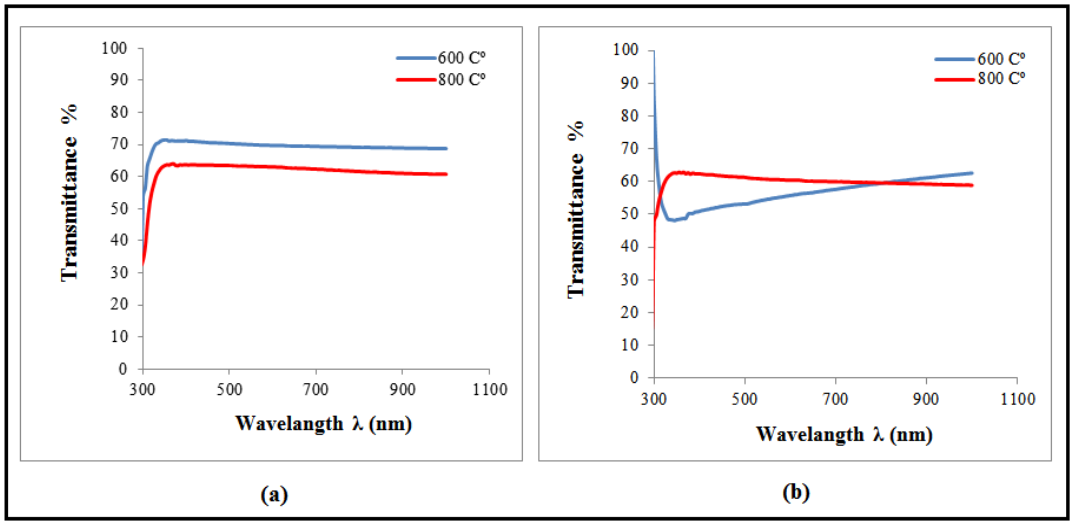

Figure IV
Transmittance (a) concentration of (0.2 M) (b) concentration of (0.4 M)

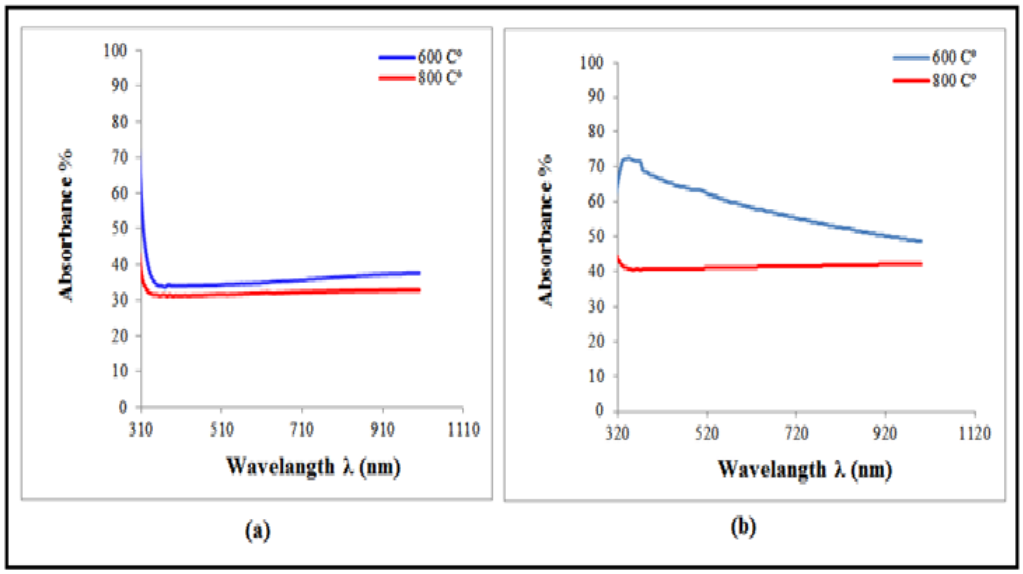

Figure V
Absorbance (a) concentration of (0.2 M) (b) Concentration of (0.4 M)

As mentioned above the height of zinc concentrations and large grain size as a results of that, sintering temperature didn't affect clearly of absorbance [9] .And for that if zinc concentrations are high the solution of zinc ferrite if deposit as a thin films will get a homogeneous surface films [12]. Energy gap of zinc ferrite decreasing by increasing sintering temperature and increasing with increasing of concentrations, and that because of large grain size and large of crystalline size. The next fig.(7) illustrate that and table (4) shows the values of energy gap.

Table 4
Energy gap values

Concentrations M	***Sintering at 600 C°***	***Sintering at 800 C°***
0.2 M	2.63	2.32
0.4 M	2.79	2.70

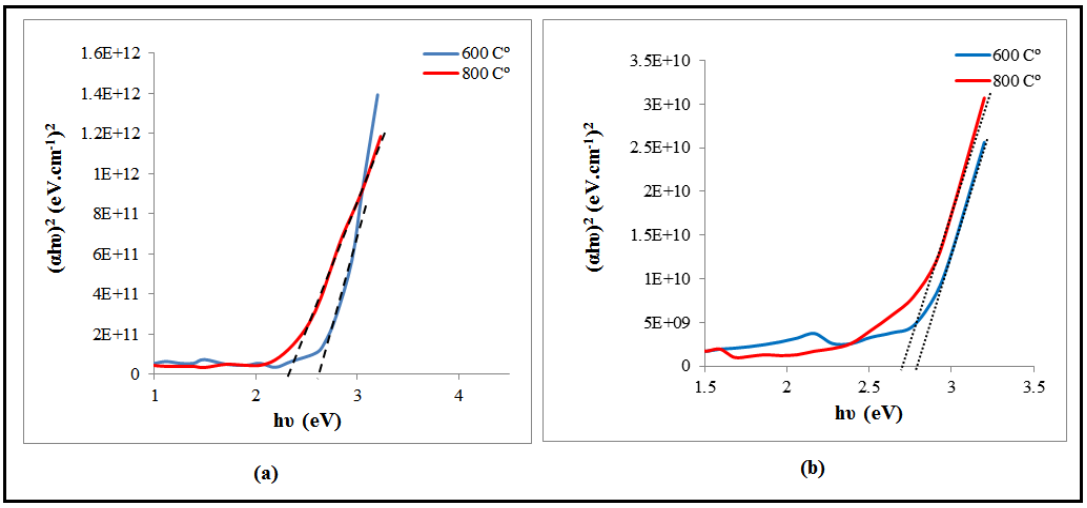

Figure VII
Energy gap (a) concentration 0.2 M (b) Concentration 0.4 M

Conclusions

$ZnFe_2O_4$ nanoparticles were successfully prepared by sol-gel methods by adopting molecular concentrations (0.2 M and 0.4 M).The powder XRD analysis revealed presence of the mixed phases of $ZnFe_2O_4$ and α-Fe_2O_3 in the synthesized nanomaterials. The $ZnFe_2O_4$ belongs to face centered regular spinel cubic structure and the crystallization increases with increasing of sintering temperature. AFM figures shows that the concentration (0.2M) has an homogeneous distribution of nanoparticle than of (0.4 M).The band gap energy of $ZnFe_2O_4$ nanoparticles was obtained to be (2.63 e V for 0.2 M , 2.79e V for 0.4 M) at 600 *C*º and be (2.32 e V for 0.2 M , 2.70 e V for 0.4 M) at 800 *C*º. The refractive index of the films increased with increase of annealing temperature.

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