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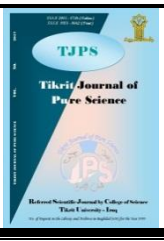

Preparation and Characterization of Zno Nano particles Prepared by Hydrothermal Method

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ABSTRACT

In this study,the nanoparticles of zinc oxide were readily prepared through Hydrothermal process by using zinc nitrate hexahydrate, and Sodium hydroxide as aprecursors. The surface topology, and crystalline structure of prepared ZnO nanoparticles were studied. X-ray diffraction (XRD) revealed that the prepared ZnO nano particles is highly crystalline, having (wurtzite) crystal structure. The optical analysis by UV–vis showed that these ZnO nano particles have considerable blue shift in the optical band gap energy (E_g = 4.9eV), and this may be to the quantum confinement effect of nano particles. The FT-IR results shows the existence of OH, COO, H2O groups the characteristic vibrational modes of Zn–O were identified. and AFM analysis showed that the diameters of the ZnO particles is in ananometer range of (70-74)nm.

1- Introduction

Zinc oxide is a material , which can have many good and unique properties. Zno has a direct band gap semiconductor, with superior thermal and piezoelectric properties[1]. The direct band gap around 3.3 eV at 300K. It has a great exciton binding energy of 60MeV. ZnO has three kinds of crystal structures, rocksalt, wurtzite ,zinc blende, Rocksalt structure will only appear at arelatively high pressure environment. Zinc blende structure can be carried out by growing ZnO on to substrates which are cubic structure. Normally at room temperature, it will stay in wurtzite structure, which is thermodynamically more stable[2,3].

ZnO nanoparticles have become familiar to researchers, and this because of the applications in different fields such as chemical sensors and gas sensors, also biosensors; and superconductors[4,5]. There are many ways to synthesize ZnO nano particles, such as asolgel method [5], solid state pyrolytic reaction process [6], and Micro mulsion Route [7]. In the present work ahydrothermal method technique can fabricating materials from low temperature aqueous solution in high vapour pressure. This method will save energy and is more environmental-friendly because the reaction is done in closed system conditions, also we can synthesis asingle crystals in low temperatures [2]. This work aim to syntheis ZnO nano particles by hydrothermal method, and characterization some properties of prepared nanoparticles.

2- Experimental

Hydrothermal method is a simple method to produce ZnO nanostructures**.** Zinc oxide nanoparticles were prepared through hydrothermal route. Zinc nitrate hexahydrate and sodium hydroxide Zn(NO**3**)2.6 H**2**O and NaOH were use as the starting chemical . AS for synthesis ZnO nano particles, 0.1 M of zinc nitrate hixahydrate dissolved in Di-ionised water to obtain aques solution then 0.3 M sodium hydroxide (NaoH) solution was added by dropwise, with vigorous stirring . The pH value of the solution was maintained to be7. Finally The resultant solution was transferred to the apparatus stainless steel autoclave, Hydrothermal processing is ahetero- geneous reaction in the presence of aqueous solvents or mineralizers under high pressure and temperature conditions to dissolve and recrystallize (recover) materials that are relatively insoluble under ordinary conditions, and it was carried out for about 150°C for 6h. The obtained precipitate was washed several times by Eathanol and then de-ionized water to remove impurities. The final product is dried at 65°C for 2 h. The

characterization of ZnO nano particles was done by X-ray technique with the (philips pw system) using CuKα as radiation source having awave length of 1.54060 Å . and the FT-IR analysis was syudied by (470infrared –spectrophotometer shimadsu). The optical properties was studied with the (T90UV Spectrometer System). AFM analysis is used to study surface of samples was (SPM-AA 3000 USA) model.

3. The Results and discussion

3.1 Structural Properties:

The spectrum of X Ray diffraction of prepared ZnO nanoparticles shown in figure 1 and table 1, and this is prove the hexagonal wurtzite structure of ZnO nanoparticles which is [a=b=3.25Å and C=5.2 Å] , ratio c/ a ~ 1.60 is match with the specification value for hexagonal cell [c/a=1.633] as in most II-VI materials[1,2]. The average of grain size (D) of the nanoparticles has been calculated for the peaks using the Debye – sherrer s formula[8,9]:

$$D = \frac{k\lambda}{\beta cos\theta} \ldots\ldots 1$$

Where λ the X-ray wave length (CuKα =1.54060Å), k= 0.94, β the peak width of half maximum, and θ is the braggs diffraction angle. The space displacement (d**hkl**) can be found from braggs formula[10]:

$$n\lambda = 2dsin\theta \ldots\ldots\ldots 2$$

$n = 1$, λ =1.54060 Å for CuKα. Standard d**hkl** was found from (JCPDS File, Card number 800075ICDSD) .

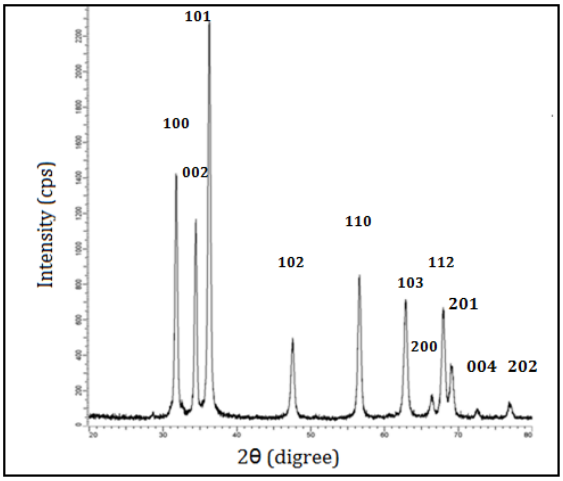

Fig 1: shows X-ray diffraction of ZnO nano particles .

Table1: shows information of X- ray with ZnO nano particles.

2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	G.S (nm)	d_{hkl} Std.(Å)	Phase	Hkl
31.7776	0.388	2.8137	21.2883	2.8137	Hex. ZnO	(100)
34.4528	0.363	2.6011	22.9129	2.6035	Hex. ZnO	(002)
36.2594	0.401	2.4755	20.8461	2.4754	Hex. ZnO	(101)
47.5507	0.505	1.9107	17.1901	1.911	Hex. ZnO	(102)
56.5837	0.486	1.6252	18.5638	1.6245	Hex. ZnO	(110)
62.872	0.523	1.4770	17.8029	1.4772	Hex. ZnO	(103)
66.4157	0.597	1.4065	15.9044	1.4069	Hex. ZnO	(200)
67.9097	0.547	1.3791	17.5091	1.3782	Hex. ZnO	(112)
69.0562	0.610	1.3590	15.8080	1.3582	Hex. ZnO	(201)
72.5651	0.500	1.3017	19.7102	1.3017	Hex. ZnO	(004)
76.8732	0.499	1.2391	20.3249	1.2377	Hex. ZnO	(202)

3-2 The (FT- IR) analysis

The FT-IR transmission spectra of prepared nanoparticles are shown in fig2:

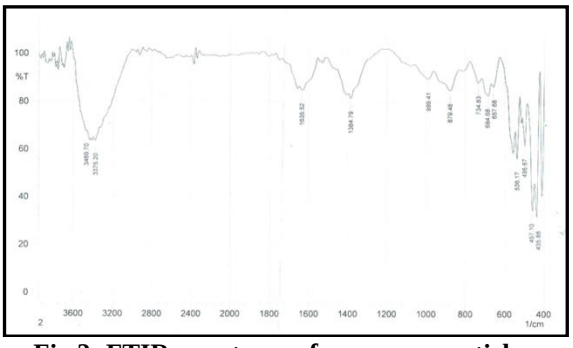

Fig 2: FTIR spectrum of zno nano particles.

FTIR spectra of ZnO samples was investigated since it is repeated. The range from (400–3469.70) cm^{-1}. These results revealed that the peak at 3469.70 , 3375 cm^{-1} respectively comes from the stretching mode vibrations of OH, but at 1635.52 $cm^{-1}$is refer to the bending vibrations of adsorbed H**2**O molecules, The small peak at 1384.79 cm^{-1} is belong to asymmetrical stretching modes COO-,which probably comes from the residues of preparation processes. It mentions the presence of small amount of organic residues absorbed on the surface of prepared nanoparticles in the samples. The peaks from 989.41 to 435.88 cm^{-1} can be ascribed to the vibration of Zn–O bond [11,12,13]. as shown in table 2:

Table (2): FTIR spectrum of ZnO nanoparticles.

ZnO cm^{-1}	**Vibrational modes**
3469.70	Vibration of OH
3375.20	Vibration of OH
1635.52	adsorbed H**2**O molecules
1384.79	COO streaching
989.41	Streaching of ZnO
879.48	Streaching of ZnO
734.83	Streaching of znO
684.68	Streaching of znO
657.68	Streaching of ZnO
536.17	Streaching of ZnO
495.67	Streaching of ZnO
457.10	Streaching of ZnO
435.88	Streaching of ZnO

3-3 UV- Visible spectroscopy: The optical properties of the prepared Nanoparticles are researched by the spectrum of the optical absorption. The absorption spectrum of ZnO nanoparticles shows a sharp absorbance onset at 250 nm; which is more small from 388 nm in the bulk size, and this is due to the deacreasing in particle size [12]. Fig 3 shows spectrum of the optical absorption of ZnO nano particles.

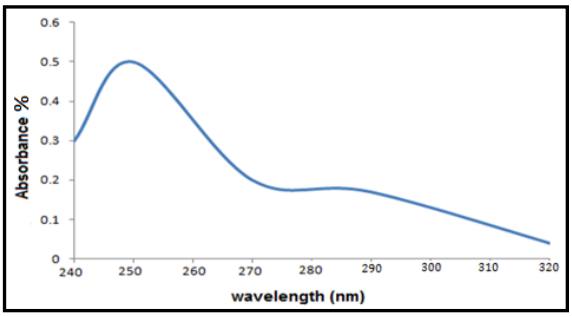

Fig3. UV absorbtion spectra of ZnO nano particles.

3-4 Determination of Band gap energy from UV absorption Spectra: Based on UV-V technique the study can use absorbance peak to find the band gap energy of synthesized ZnO nano Particles and compared with value of bulk ZnO. Energy band gap of these materials have been reported by using the absorption spectra Figure 3. By using the equation [1,14].

$E\,g = h\upsilon\; g = hc\,/\lambda$ ……..3

Where Eg is the optical band gap, (h) is plank's constant (h= 6.63×10^{-34}J.S) and, (υg) frequency of the emitted radiation. Using λ =250nm, The band gap of nano ZnO is equal to be(4.9) eV, and this mean that the band gap is higher than the value (3.3) eV of bulk ZnO . this is may due to the quantum size effect of the synthesized sample [6].

3-5 Atomic force microscope

Atomic force microscopy is used to study surface of the samples**.** Fig 4 shows typical surface AFM images (in three and two dimensional), and the particle sizes are range from (70 – 74)nm. and it is clear that the particle size is more bigger than in x-ray. also there is abrigth regions which is their grain sizes are bigger than dark regions .

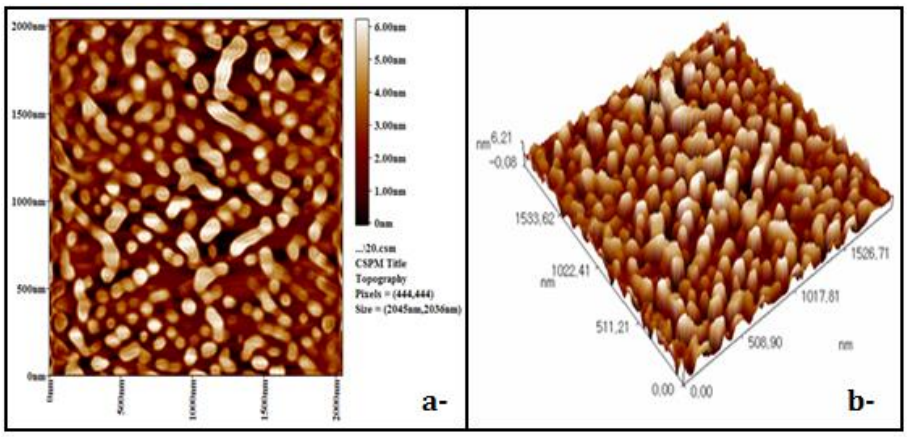

Fig 4:AFM images of ZnO nano aprticles, a- in two dimention ,b- in three dimention.

4- Conclusions

In the present study, nanosized ZnO particles were successfully prepared by hydrothermal method. In (XRD) analysis the size of the nano particles in the range of (15.8 - 22.9) nm and having wurtzite crystal structure. The band gap was lower for it prepared (ZnO nano particles) compared to the (bulk ZnO particles). AFM analysis show that the average of grain size was (70 -74)nm. Thus the synthesis of ZnO nano particles by hydrothermal method is simple, fast and no complicated in nature. These nano particles can be used in different applications; sush as anti bacterial applications , sensing of gas.

5- References

[1] Almoqayyad, S .I. (2012). Study of Synthesis and Growth of ZnO Nanoparticles. M,Sc.thesis, Al-Azhar University of Gaza.

[2] Idris, W.A. (2015). Characterization of Nanostructure ZnO synthesized through hydrothermal method, M.Sc. thesis, University of Tun Hussein Onn Malaysia.

[3] Chowska, K .Z. (2014). Methods of ZnO nanoparticles synth-esis. *Journal of Biotechnology, Computational Biology and Bio nanotechnology*, **95(2)**:150-159.

[4] Amin, G. (2012). ZnO and CuO Nanostructures; low tempera-ture growth Characterization, Their Optoelectronic and Sensing Applications. M,Sc. thesis, Linköping University.

[5] Kolekar, T.V.et al. (2013). Synthesis and characterization of ZnO nanoparticles for efficient gas sensors. *Archives of applied Science research*, **5 (6):**20-28.

[6] Teklemichael, S.T. (2012). Defects in zinc oxide nanocrystal. Ph. D. thesis, Washington state University.

[7]-Kumar, H. (2013). Structural and optical characterization of ZnO Nanoparticles synthesized by micro emulsion route. *International letters of chemistry; physics and astronomy*, **14**: 26-36

[8] Reddy, A.R. (2015). Hydrothermal synthesis and characterization of ZnO nano Crystals. *International journal of minning and metallurgy* , **3(2)**: 52-56.
[9] Vinosel, V. M. et al. (2017). Facile synthesis of CuO nano particles by hydrothermal method and their application on their antibacterial activity. *International research Journal of enginering and Technology*, **4(8):** 106-109
[10] Poole, C.p. (2003). Introduction to nanotechnology. John Wiley and sons, Inc.
[11] Kulkarni, S.S. (2015). Optical and Structural properties of zinc oxide Nanoparticles. *international journal of advanced research in physical science* , **2(1)**:14-18.
[12] Alwan, M.A. et al. (2015). Synthesis of zinc oxide Nanoparticles Sol Gel route and their charecterization. *Nanoscience and technology*, **5(1):** 1-6
[13] Munoz, M.M. et al. (2017). Shape-control of zinc oxide Nanoparticles; enhancing photocatalytic activity under UV irradiation. *International Congressof Engineering physics*, IOP pulishing. series 792/ 012068.
[14] Kannaki, K. et al. (2012). Hydrothermal synthesis of CuO Nano structure and their Charecterizations. *International journal of Scientific and engineering research*, **3(9):** 1- 4.

تحضير ووصف جسيمات اوكسيد الزنك النانوية المحظرة بالطريقة (الحرارية– المائية)

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الملخص

تم في هذا البحث تحضير جسيمات اوكسيد الزنك النانوية من خلال الطريقة (الحرارية المائية), باستخدام نترات الزنك المائية السداسية وهيدروكسيد الصوديوم كمواد اولية. تم دراسة طوبوغرافية السطح ; والتركيب البلوري لجسمات اوكسيد الزنك النانوية. حيود الاشعة السينية اظهرت بأن التركيب البلوري لجسيمات اوكسيد الزنك مثالي, اذ يمتلك التركيب البلوري المسمى (Wurtzite). التحليل البصري باستخدام جهاز (UV); اوضح لنا بأن جسيمات اوكسيد الزنك النانوية تنحرف باتجاه المنطقة الفوق بنفسجية ذات فجوة طاقة بصرية (4.9 eV), وان هذا ربما يعود الى تأثير الحصر الكمي للجسيمات النانوية. نتائج FT_IR اوضحت لنا وجود مجموعات OH,COO, H2O بالإضافة الى ان انماط اهتزاز جسيمات Zn-O النانوية تم تأكيدها. كما ان التحليل باستخدام مجهر القوة الذرية (AFM) اكد لنا بان القطر للجسيمات هو بحدود النانومتر اذ يتراوح من - 70 (74) نانومتر.