مجلة العلوم البحثة والتطبيقية
Journal of Pure & Applied Sciences
www.Suj.sebhau.edu.ly ISSN 2521-9200
Received 15/02/2019 Revised 15/04/2019 Published online 01/05/2019

الخواص التركيبية لمساحيق اكسيد الزنك المحضرة بطريقة المحلول الجيلاتيني

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الملخص حضرت مساحيق اكسيد الزنك بطريقة المحلول الجيلاتيني. العينات حرقت عند درجتي الحرارة C600 و C700 °. الخواص التركيبية للعينات قيست باستخدام مقياس حيود بالأشعة السينية (XRD) ، رامان والمجهر الإلكتروني الماسح (SEM). كشفت أنماط XRD أن العينة المحضرة أظهرت بنية غير متبلورة. أظهرت العينات المكلسة عند C600 ° و C700 ° بنية سداسية (wurtzite). زادت شدة قمم XRD للعينات مع زيادة درجة حرارة التكلس بسبب تحسين تبلورها. كانت قيم حجم البلورية للعينات عند C600° و C700° تساوي 34 و 22 نانومتر على التوالي. وأظهرت العينة ZnO في 700 درجة مئوية قضبان في شكل سداسي. ، يشير طيف رامان إلى أن الذروة الحادة ذات الكثافة العالية التي تقع عند 437 سم $^{-1}$ - كانت ذات علاقة بالارتفاع E_2 بسبب التناسق الجيد للعينة. لذلك، سيتم تنفيذ المزيد من القياسات لمساحيق ZnO المطعمة باليود لتعزيز خصائصها البصرية لتكون مناسبة كمحفزضوئي.

الكلمات المفتاحية: ZnO، المحلول الحيلاتيني الخصائص التركيبية ، مساحيق النانو.

Structural Properties of Zinc Oxide Powders prepared by Sol-Gel method

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Abstract The zinc oxide (ZnO) nanopowders were prepared by the sol-gel method. The calcination process to calcine the samples was executed at different temperature values of 600° C and 700° C . The structures and morphology of samples were characterized using the X-ray diffractometer (XRD), Raman and the scanning electron microscope (SEM). An amorphous structure of as-prepared sample was confirmed by XRD patterns. The calcined samples at 600° C and 700° C exhibited the hexagonal (wurtzite) structure. The intensities of samples were increased with increasing of calcination temperature due to crystallinity improvement. The crystalline size values of the samples at 600° C and 700° C were 34 and 22 nm, respectively. At 700° C, the ZnO sample showed rods -like with the hexagonal shape. Also, Raman spectra indicate a sharp peak with high intensity located at 437 cm^{-1} related to E_2 high mode due to well crystalline of the sample. Further measurements will execute for iodine doped ZnO powders to enhance their optical properties to be proper for the photocatalytic applications.

Keywords: ZnO, sol-gel ,Structural Properties , nanopowders.

1.Introduction

The Zinc Oxide (ZnO) becomes a wide known and it has an importance role in many different aspects for researches such as science, technology, medicine and renewable energy [1] [3]. The ZnO belongs to II- VI semiconductors family and its iconicity locates between the covalent and ionic semiconductor. However, the crystal structure of ZnO commonly occurs in the wurtzite structure with a hexagonal unit cell and it has two lattice parameters *a* and *c* [2]. This is ascribed to both its bond symmetry and pond polarity [4]. Many methods have been employed to fabricate ZnO powder such as spray pyrolysis [5], hydrothermal synthesis [6] , coprecipitation, micro emulsion [7] and sol-gel method [3].The sol–gel method is favourable among researchers for

considerable advantages such as, simple preparation method, low equipment and low preparation temperature [2] [8]. Therefore, this work reports the structural properties of ZnO powders prepared via sol-gel.

2. Experimental details

The Zinc acetate dihydrate ($Zn(O_2CCH_3)_2$ $(H_2O)_2$: 99% purity) was dissolved in ethanol and stirred at 65°C for 1h to get a transparent solution. The solution was maintained at pH7 by adjusting the amount of sodium hydroxide in the solution. Then, the solution was stirred again at (65 ±1) °C for 1 h to obtain a homogenous gel. The gel was dried at room temperature for 24h. The ZnO powders were calcined in air at 600° C and 700° C for 2h.

The sample's structures were characterized by an X-ray diffractometer (XRD و Bruker D8 Advance diffractometer using monochromatic Cu-Kα radiation (λ= 1.5406 Å)) in the 2θ range of 20o - 80 o and Raman spectroscopy (Renishaw inVia Reflex Spectrometer System) with an excitation wavelength of 514 nm.

. The field emission scanning electron microscope (SEM, Zeiss FESEM Ultra PLUS) was used to study the morphology of the samples

3. Results and discussion

3.1. Structural characterization

Fig.1. shows the XRD patterns of pure ZnO powders calcinated at 600° C, 700° C and as prepared. The resulted peaks were indexed and compared according to the JCPDS card No. 00-005-0664 for pure ZnO. It can be seen that, as prepared sample showed an amorphous structure. The samples have a hexagonal wurtzite structure, no other phases were observed in the patterns. The increase of the calcination temperature up to 700° C results in sharper peaks with the increased intensity and higher crystallization without changes in the obtained phases due to the enhance of crystal structure of the sample [3].The average crystalline sizes of samples were calculated using Debye- Sherrer`s equation

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

where D is the crystallite size, λ= 0.15406 nm is wavelength of CuKα beam, β is FWHM of the XRD peak at the diffraction angle θ and k= 0.9 is the shape factor of average crystallite size [9,10]. An average crystallite size (D) of the samples at 600° C and 700° C was 34 nm and 22nm, respectively. The decreasing in crystallite size could due to the defect in the samples. The lattice parameter of ZnO was calculated according to Bragg`s law, where the lattice constants a and c of wurtzite structure can be computed using the following equation:

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2} \qquad (2)$$

where $d_{hkl}d_{hkl}$ is the interplaner spacing and hkl are the Miller indices, a and c are the lattice parameters [11].

The lattice parameters values of both samples are a = 3.2 Å and c= 5.2 Å, which are the same as reported in the literature [2].

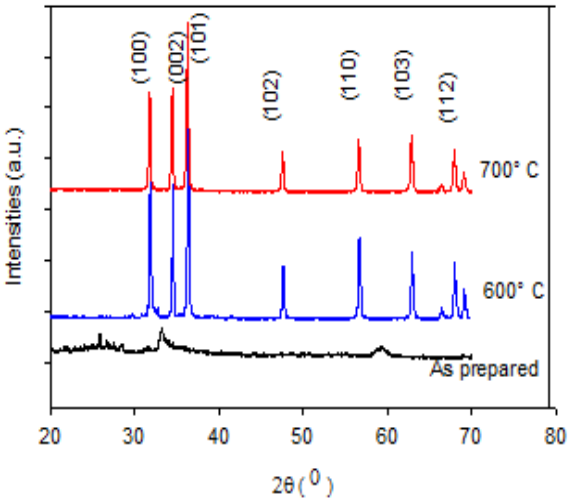

Fig1: The XRD patterns of ZnO powders calcined at 600° C and 700° C.

3.2. SEM measurements

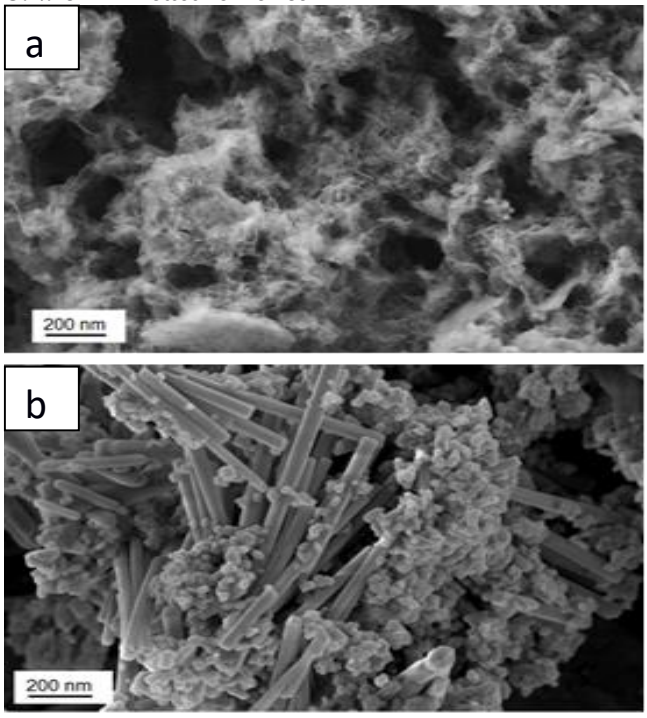

Fig.2: The SEM images of ZnO samples calcined at (a) 600° C and (b) 700° C

Fig.2. shows the SEM images of ZnO calcined at 600° C and 700° C. Regardless as prepared sample, it observed that the sample calcined at 600° C contains pores and voids. This could be due to the large gas amount escape during the composition [12] . Further increasing of the temperature up 700° C, the sample shows rods with average diameters about 69 nm. However, additional measurements will be performed to obtain the dimensions of rods.

3.3. Raman analysis

The Raman spectra of samples under excitation of 514 nm laser line are illustrated in Fig. 3. The spectra show the excitations mode of ZnO wurtzite (hexagonal) structure and Raman active modes can be divided as $A_1+E_1+ 2E_2$ [13]. A_1 and E_1 are polar modes, which will split into transverse optical (TO) and longitudinal optical (LO) phonons. Also, E_2 modes (E_2 low and E_2 high) are nonpolar [12]. Raman spectrum at 600° C reveals few peaks with the low intensities appear at 437 cm^{-1} and 1065 cm^{-1}. At 700° C, three peaks are indicated. The observed peaks are located at 396 cm $^{-1}A_1$ (TO) mode), 437 cm^{-1} (E_2 (high) mode) and 586 cm^{-1} A_1 (LO) mode. The E_2 is sharp and has a high intensity due to well crystallinity of the sample [11].

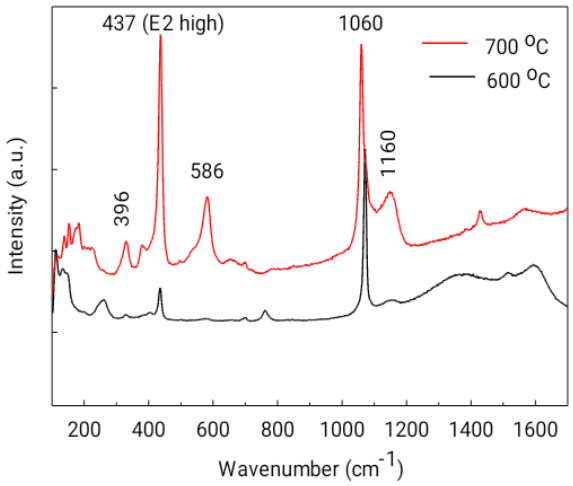

Fig. 3: Raman spectra of ZnO

Acknowledgments

Agreat thank goes to our team from Sebha University.

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