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Green Synthesis of High Impact Zinc Oxide Nanoparticles

Ahmed Samy[1], Amal E. El-Sherbiny[2] and Abd El-Rahman A. Menazea[3,4]*

[1]*Department of Animal Production, National Research Centre, 12622, Dokki, Egypt.*
[2]*Department of Animal Production, Faculty of Agriculture, Cairo University, Egypt.*
[3]*Spectroscopy Department, National Research Centre, 33 El-Bohouth St., 12622, Dokki, Giza, Egypt.*
[4]*Laser Technology Unit, National Research Centre, 33 El-Bohouth St., 12622, Dokki, Giza, Egypt.*

THIS study was conducted as an attempt to reach the best method to synthesis zinc oxide nanoparticles (ZnONPs) using chemical methods. ZnO nanoparticles was prepared using Sol-Gel method from zinc chloride ($ZnCl_2$) and sodium hydroxide (NaOH) as chemical reactors and polyethylene glycol with low molecular weight as surfactant. The effect of the different reaction's temperature on the produced ZnO nanoparticles was studied. Four samples of zinc oxide nanoparticles were prepared on different reaction's temperatures; 0 °C, 50 °C, 90 °C, and 0-RT°C (the reaction beginning at 0 °C and after that increase the temperature to reach the RT). The obtained products were examined using different techniques; X-ray Diffraction (XRD), High Resolution Scanning Electron Microscope (HRSEM), Fourier Transform Infrared (FT-IR), Ultraviolet–Visible Spectroscopy (UV-Vis), and Energy Dispersive X-ray Analysis (EDAX). The results showed that XRD, FT-IR, and EDAX confirmed the presence of ZnO nanoparticles with high purity. The UV-Vis. absorption spectrum showed an absorption band at 376nm due to ZnO nanoparticles. HRSEM showed the superiority of ZnO nanoparticles that synthesized at 50°C and the inferiority of that synthesized at 0-RT °C.

Keywords: Zinc Oxide; Nanoparticles, Temperature, Sol-Gel, HRSEM.

Introduction

Nanotechnology is dealing with materials whose structures showed significantly unique and improved chemical, physical, and biological properties as a result of their particle size in the nano scale [1-2]. Nanotechnology has a huge impact in commercial application of nano minerals in the fields of information technology, engineering, medicine, food, pigments, pharmaceutical, biological, and electronics applications and etc. [3-6]. Therefore, the researchers have focused on studying the physical, chemical, electrical, and optical properties of nanoparticles [7-11]. It is also been used as a novel tool in the fields of biotechnology, biology (molecular and cellular), physiology, mineral nutrition, pharmacology, and reproduction, etc. in both human and animal models [12]. Minerals play a vital role in the nutrition of animal production system. Minerals perform digestive and reproductive process and growth of animals [2]. Zinc is one of the most abundant trace elements in the animal body and it should be added to the animal diets because it is not stored in the body [12]. Also, zinc is essential to the structure and function of myriad proteins which are classified as regulatory, structural and enzymatic. In the central nervous system, zinc has an additional role as a neurosecretory product or cofactor [13, 14]. Nano minerals have the range of particle size between 1-100 nm [15]. As a result of this very small size (high surface area), there have high absorption, reactivity and efficiency than conventional zinc. So, these can be used at very lower doses compared to conventional zinc

*Corresponding author e-mail: aanter7@gmail.com, Phone: +2 01064260397, Fax: +2 0233370931
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to enhance the animal performance, immunity, reproduction, act as antimicrobial agent and reducing environmental contamination [2, 6, 16-27]. Numerous techniques have been evolved for the synthesis of ZnO in nano form such as vapour-liquid-solid (VLS) [28], pulsed laser deposition [29], electro chemical deposition[30], metal vapour transport [31, 32], chemical vapour deposition [33], metal organic chemical vapour deposition [34]; the hydrothermal growth approach [35], Mechano-Chemical Method [36], chemical precipitation method [37],and sol-gel method [38, 39]. This work aimed to reach the best reaction's temperature for synthesis ofhigh impact zinc oxide nanoparticles.Four samples of zinc oxide nanoparticles were synthesized on different reaction's temperatures; 0 °C, 50 °C, 90 °C, and 0-RT °C via green sol-gel method.

Experimental

Materials

Zinc chloride ($ZnCl_2$) and sodium hydroxide (NaOH) were used as a reacted material. Polyethylene glycol, with low molecular weight, was used as surfactant and deionized water was used as solvent material. All chemicals used were analytical grade purchased from El-Nasr Chemical Company.

Methods

Four samples of zinc oxide nanoparticles were prepared using Sol-gel method as a chemical method from the same source of Zinc chloride ($ZnCl_2$) and sodium hydroxide (NaOH) as reacted materials. The 0.6 moles of NaOH was dissolved in deionized water after that heated the solution with different temperatures 0, 50, and 90 °Cas shown in Fig. 1-A (First Stage) under constant stirring after reach to the desired temperature, adding 5% Polyethylene glycol, with low molecular weight, was used as surfactant then adding a solution of 0.3 M $ZnCl_2$ drop by drop after the complete of addition. All samples are maintained at the desired temperature; 0, RT (the solution at 0°C from the first stage was increased the temperature to reach the room temperature), 50 °C,and 90°C as shown in Fig. 1-B (Second Stage). After that each solution remained agitated for three hours, maintaining the desired temperature. The suspension solution is converted from transparent to white color due to the reaction. After that make centrifuge at 5000 rpm for 15 min and washed three times by deionized water and two times by ethanol. Each sample was dried in a vacuum oven at 70°C for several hours. After that all samples were calcinated at 300 °C.

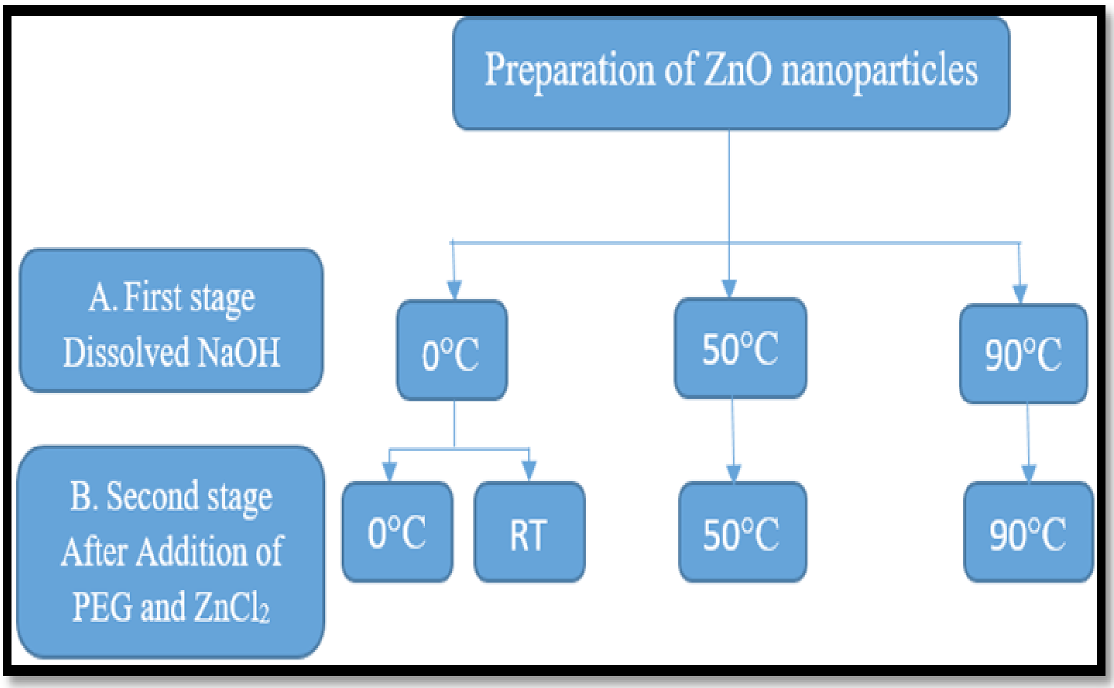

Fig. 1.The preparation of ZnO nanoparticles at different temperature the first stage A. 0°C, 50°C and 90°C and the second stage B. 0°C, RT, 50°C, and 90°C.

Measurement Techniques

Fourier Transform Infrared (FT-IR) measurements were taken using JASCO, FT/IR-6100 in the spectral range of 4000-400 cm^{-1}, ultraviolet-Visible absorption spectra (UV-Vis) were measured in the wave length region of 200-800 nm using JASCO V- 630 spectrophotometer, X-ray Diffraction (XRD) was performed using PANalyticalX'Pert Pro target Cu-Kα with secondary monochromator Holland radiation (λ= 0.1540 nm, the tube operated at 45kV, scans were collected over a 2θ range of 5 - 60°), High Resolution Scanning Electron Microscope (HRSEM) was performed using SEM Model Quanta 250 FEG and energy dispersive analysis by X-ray (EDAX).

Results and Discussion

Fourier Transform Infrared Analysis (FT-IR):

FT-IR spectroscopy was performed to identify the functional groups of the synthesized ZnO nanoparticles. The IR absorbance spectra of the samples were obtained in the range from 4000 - 400 cm^{-1}. Fig.2 showed the FT-IR spectrum of all prepared samples of ZnO NPs. Fig. a, b, c, and d represent FT-IR spectrum of ZnO at the different reaction's temperature; 0 °C, 50 °C, 90 °C, and 0-RT °C, respectively. The peaks at 3440 $cm^{-1}$and 1600 cm^{-1} are characteristics band attributed to hydroxyl group (O-H) stretching and bending vibration, respectively[40].The absorption band at 438 cm^{-1} is attributed to the Zn–O stretching mode of the ZnO lattice[41]. All the observed peaks confirm the characteristic beaks of ZnO nanoparticles according the previous literatures and the FT-IR results have shown to be high purity of synthesized ZnO NPs. These results of FT-IR showed no differences among all treatments and confirmed the chemical compositions of the obtained Zinc oxide [42, 43].

Ultraviolet-Visible Spectroscopy (UV-Vis)

UV-visible absorption spectroscopy is a powerful technique to examine the optical properties of nano-sized particles, also performed to further confirm the formation of zinc oxide nanoparticles. The absorption spectra of ZnO NPs prepared at different temperatures were shown in Fig.3. An absorption peaks was observed at 376 nm, 376 nm, 376 nm and 374 nm for 0°C, 50 °C, 90 °C, and Mix 0-RT°C, respectively, which is a characteristic band for the pure ZnO [44, 45]. From this UV spectra, SPR peak for ZnO nanoparticles is obtained at 376 nm indicating combined vibration of electron of nanoparticle with the light wave [46]. No other peak was observed which confirmed the high purity of ZnO and these results were confirmed with the FT-IR, XRD, and HRSEM results [47].

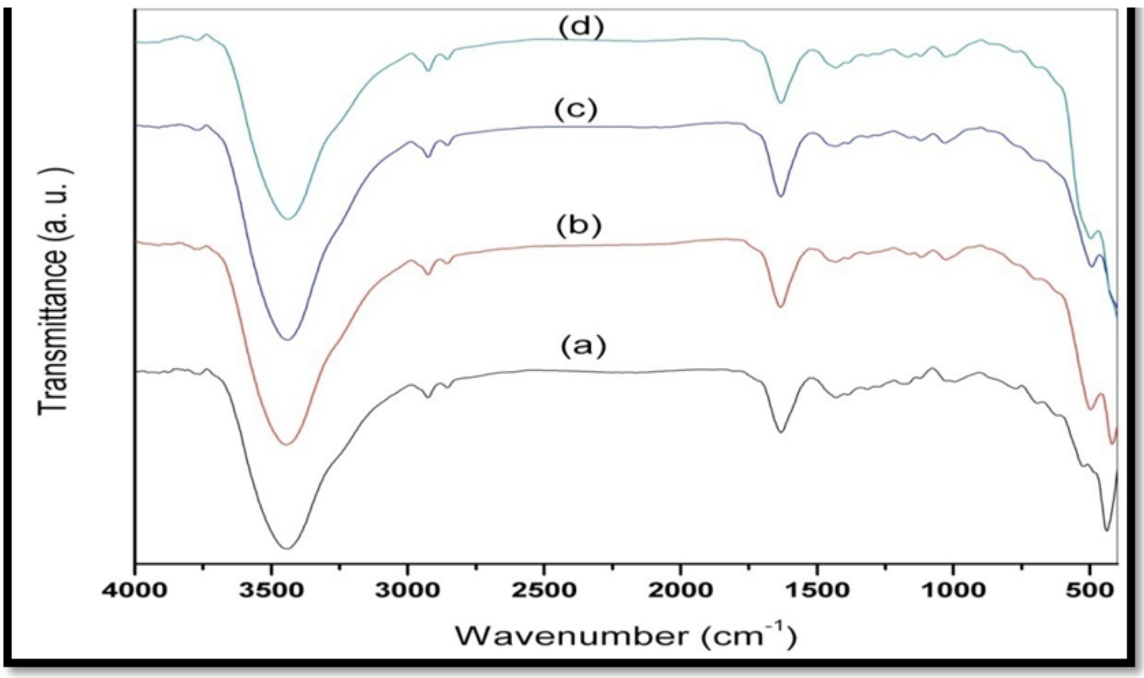

Fig. 2. FT-IR of ZnO nanoparticles prepared at different temperature (a) 0°C, (b) 50 °C, (c) 90 °C, and (d) 0-RT°C.

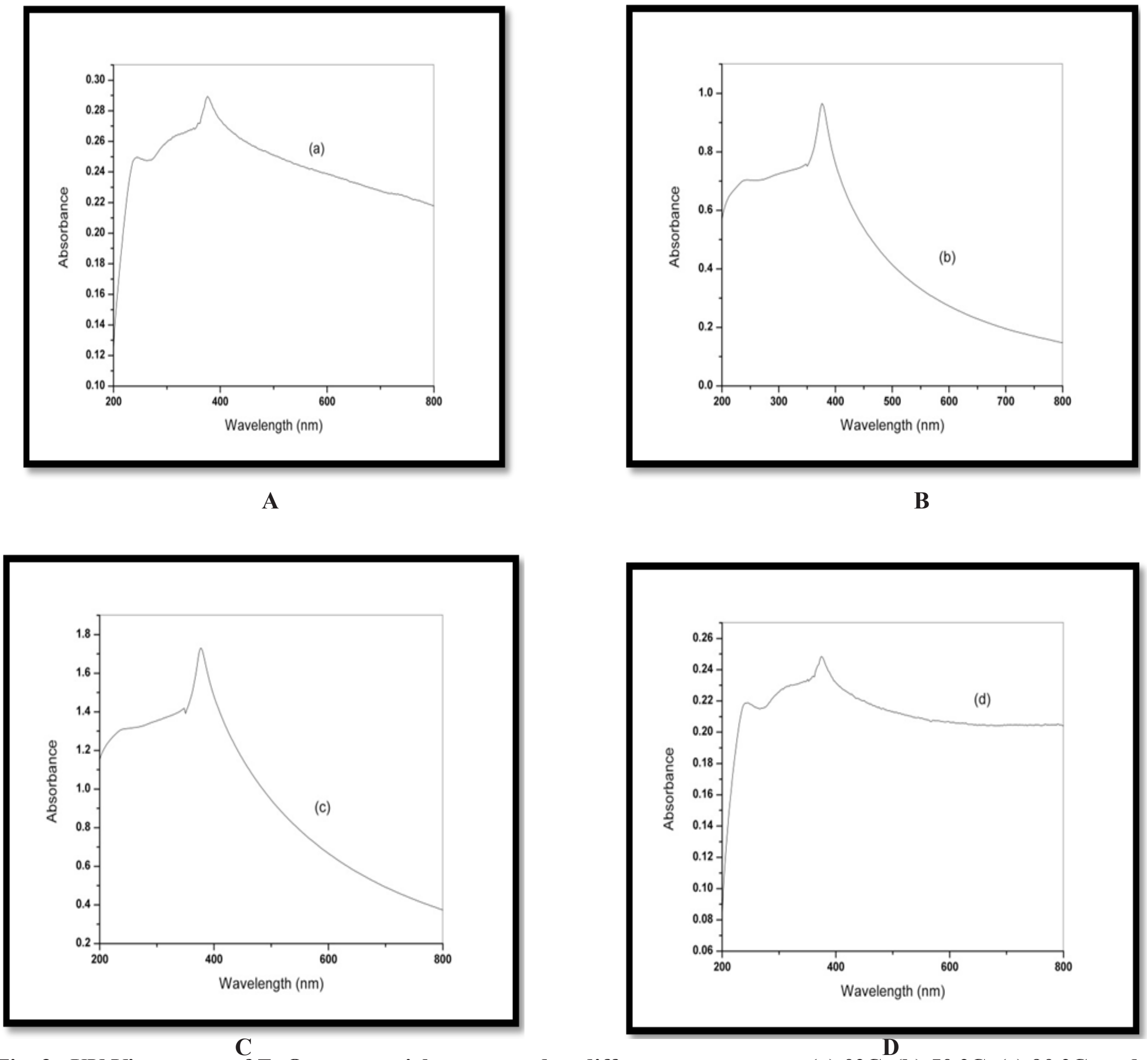

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Fig. 3. UV-Vis spectra of ZnO nanoparticles prepared at different temperature (a) 0°C, (b) 50 °C, (c) 90 °C, and (d) (0-RT) °C.

X-ray Diffraction (XRD)

X-Ray Diffraction is a perfect technique to examine the structure of materials. The crystallinity and phase of the synthesized ZnO nanoparticles at 0°C, 50 °C, 90 °C, and 0-RT°C, respectively are shown in Fig.4. It shows diffraction peaks at 2θ values of 31.8°, 33.4°, 36.2°, 47.5°, and 56.5°. These peaks correspond to (100), (002), (101), (102), and (110) planes, respectively, which are characteristic of the hexagonal structure of ZnO nanoparticles [4]. All the diffraction peaks matched well with ZnO according to the standard JCPDS file for ZnO (No. 89-1397). No other peaks related to impurities were seen indicating that the high purity ZnO nanoparticles were obtained. At 0-RT, the diffraction peak sharpness improved and the full-width at half-maximum (FWHM) values were decreased. These results confirmed that there is an improvement in the crystallinity of the ZnONPs and thus the size of ZnONPs increases at 0-RT. This is due to the change of growth rate between the different crystallographic planes[42, 43].

High-Resolution Scanning Electron Microscope (HRSEM)

High Resolution Scanning Electron Microscopeis powerful technique used for providing information for the topographical, morphological and composition. Fig.5 shows HRSEM image for ZnO nanoparticles at 0 °C, 50 °C, 90°C, and 0-RT°C, respectively. It is clear from the HRSEM micrographs that the particles have rod shape[48].HRSEM images show that the best size of ZnO nanoparticles found at 50 °C and the worst one is at 0-RT°C[49, 50].

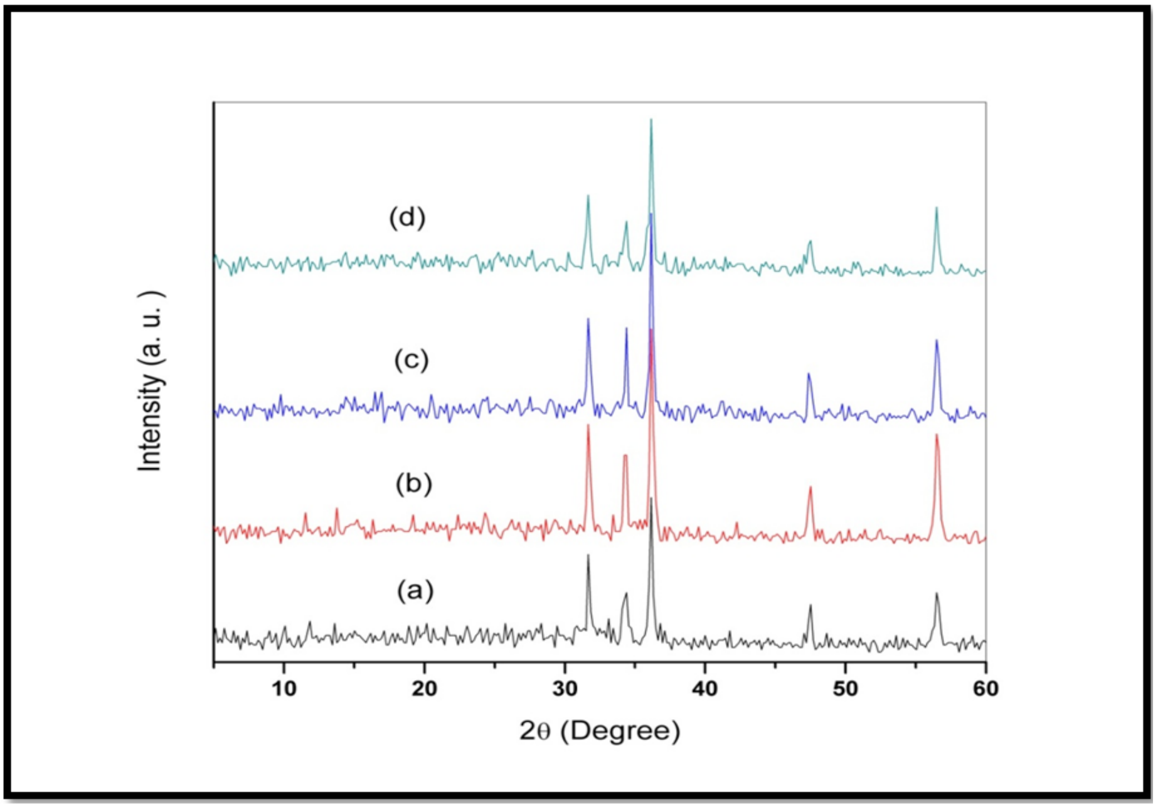


Fig. 4. XRD of ZnO NPs at different calcinations temperature (a) 0°C, (b) 50 °C, (c) 90 °C, and (d) 0-RT°C.

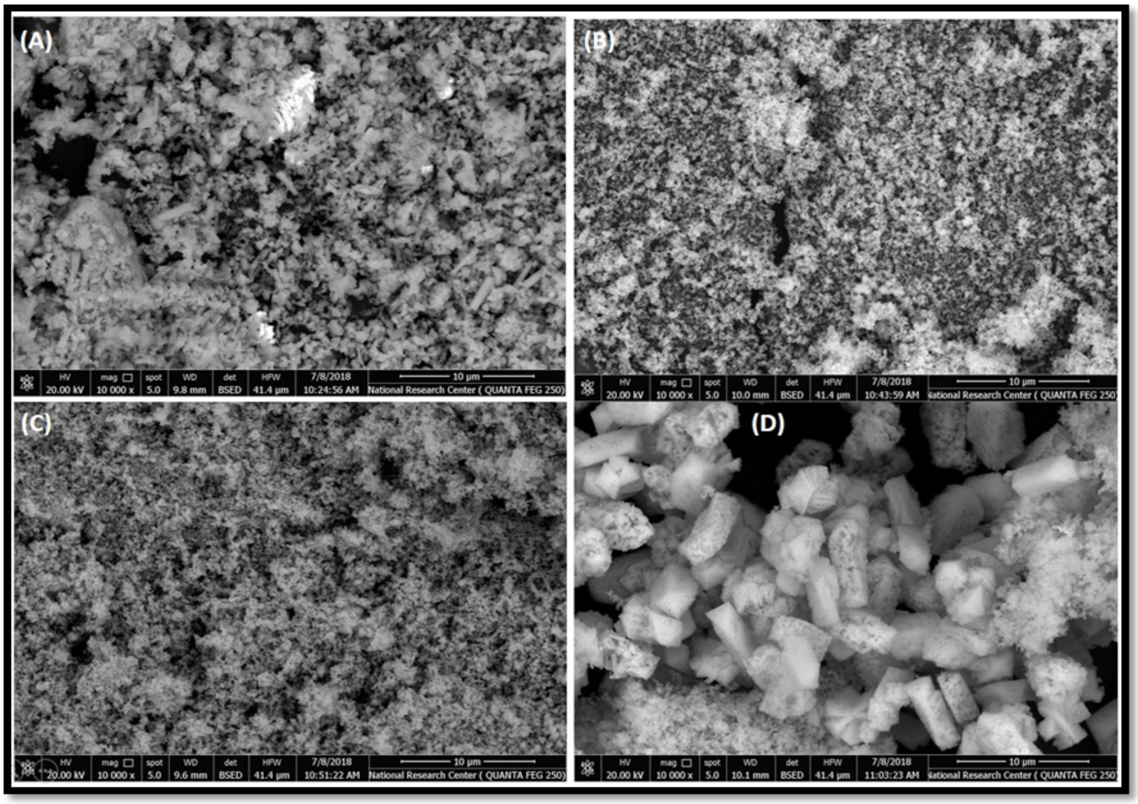
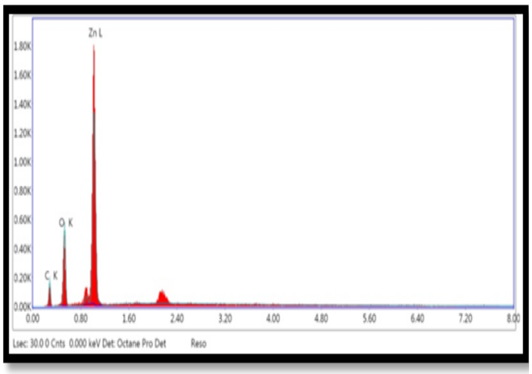


Fig.5. HRSEM of ZnO NPs at different temperature (a) 0 °C, (b) 50 °C, (c) 90 °C, and (d) (0-RT) °C.

Energy Dispersive Analysis by X-ray (EDAX)

This technique is known as energy dispersive analysis of X-ray (EDAX) and is used extensively to study the elemental composition of the sample. The results of EDAX were shown in Fig.6. At all Fig., the ratio of Zn/O are close to ZnO nanoparticles and the peak of carbon appeared in the Fig.6. A, B, and D but it is disappeared at C because in this case the 90°C is more than the melting point of the Polyethylene glycol. No other signals were detected in the detection limits of EDAX which confirm the purity of the zinc oxide nanoparticles [48].



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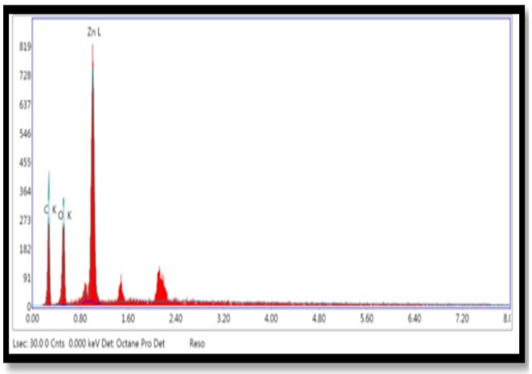

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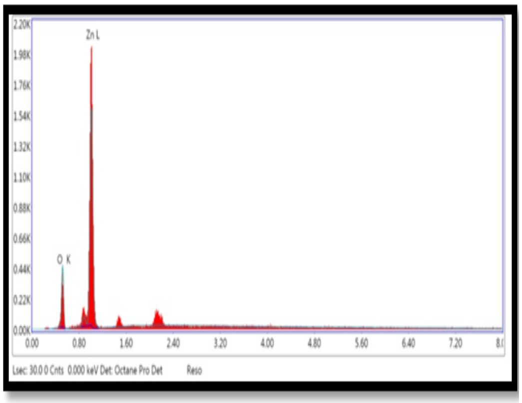

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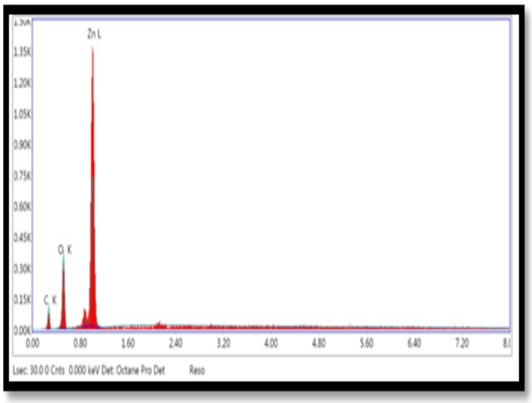

D

Fig.6. EDAX of ZnO NPs at different temperature (a) 0 °C, (b) 50 °C, (c) 90 °C, and (d) (0-RT) °C.

Conclusion

This study is aimed to reach the best method to produce high purity zinc oxide nanoparticles using a new idea of chemical methods. Four samples of nano zinc oxide using different reaction's temperature at 0 °C, 50 °C, 90 °C, and 0-RT°C and examined using, FT-IR, UV-Vis, XRD, HRSEM, and EDAX. The results showed that FT-IR, XRD and EDAX confirmed that the presence of ZnO nanoparticles. The UV-Vis. absorption spectrum shows an absorption band at 376 nm due to ZnO nanoparticles. HRSEM reveal that the best ZnO nanoparticles synthesized at 50°C but the worst one at 0-RT °C.

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تحضير أمن لجسيمات أكسيد الزنك النانومترية عالية التأثير

أحمد سامى[1]، أمال الشربينى[2]، عبدالرحمن عنتر منازع[3،4]
[1] قسم الإنتاج الحيواني ، المركز القومي للبحوث ، مصر
[2] قسم الإنتاج الحيواني ، كلية الزراعة ، جامعة القاهرة، مصر
[3] قسم الطيف ، المركز القومي للبحوث ، مصر
[4] وحدة تكنولوجيا الليزر ، المركز القومي للبحوث، مصر

أجريت هذه الدراسة كمحاولة للوصول إلى أفضل طريقة لتحضير جزيئات أكسيد الزنك النانومتري (ZnONPs) باستخدام الطرق الكيميائية. تم تحضير جزيئات النانو ZnO باستخدام طريقة Sol-Gel من كلوريد الزنك (ZnCl2) وهيدروكسيد الصوديوم (NaOH) كمتفاعلات كيميائية وتم استخدام البولي ايثيلين جليكول ذو الوزن الجزيئي المنخفض . تم دراسة تأثير درجات الحرارة المختلفة للتفاعل علي انتاج الزينك اكسيد النانومتري. حيث تم انتاج اربع عينات من الزنك اوكسيد تحت درجات حرارة مختلفة وهي صفر ، 50 ، 90 و من صفر لدرجة حرارة الغرفة (حيث يبدء التفاعل في هذة المعامله علي درجة حرارة صفر درجة مئوية ثم يتم رفع درجه الحرارة للوصول الي درجة حرارة الغرفة). تم فحص واختبار العينات الناتجة باستخدام كلا من حيود الأشعة السينية (XRD) ، الميكروسكوب الإلكترونى الماسح عالى الدقة (HRSEM) ، الأشعة تحت الحمراء (FT-IR) ، التحليل الطيفي للأشعة فوق البنفسجية المرئية (UV-Vis) ، تحليل الطاقة بالأشعة السينية المشتتة (EDAX). اتضح من نتائج XRD و FT-IR و EDAX تكوين جزيئات اكسيد الزنك النانومتري بدرجة عالية النقاوة. كما أظهر طيف الامتصاص عند 376 نانو متر الراجع لجزيئات اكسيد الزنك النانومتري المتكونة. كما أظهرت نتائج الميكروسكوب الالكتروني الماسح عالي الدقة أن افضل حجم للجزيئات كان عند درجة حرارة هي 50 درجة مئوية وان اسوء حجم منتج كان عند درجة حرارة 0-حرارة الغرفة (التي بدءت بصفر درجة مئوية ثم ارتفعت للوصول لدرجة حرارة الغرفة).