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\textsubscript{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.
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 of high 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 °C as 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.](image-url)
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\(^{\text{Pert}}\) Pro target Cu-K\(^{\alpha}\) with secondary monochromator Holland radiation (\(\lambda= 0.1540 \text{ nm}\), the tube operated at 45kV, scans were collected over a 20 range of 5 - 60\(^{\circ}\)), 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.
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 Microscopes 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].

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.

Acknowledgment
This study was financially supported by National Research Centre of Egypt through Internal Research Project No. 11030113.

References


47. Imran K; Structural and optical properties of Zr doped ZnO nanoparticles, Optmater, 35, 1189-1193 (2013).


تجهيز أمVien محيمى للذئاب النانومترية عالية التأثير

أحمد سامي، أمال الشربينى، المرزوق العنتر، مصري 1

قسم الإدارة الحيواني، المركز اليدوي للبحوث، مصر

قسم الإدارة الحيواني، كلية الزراعة، جامعة القاهرة، مصر

قسم الطيف، المركز اليدوي للبحوث، مصر

وحدة تكنولوجيا الليزر، المركز اليدوي للبحوث، مصر

أجريت هذه الدراسة كمحاولة للوصول إلى أفضل طريقة لتحضير جزيئات أكسيد الزئبق النانومترية (ZnO NPs) باستخدام ZnCl2 و Sil-Gel و H2O2 في مصادرية كيميائية و باستخدام الطرق الكيميائية بواسطة البولي ايثيلين جليكول ذو الوزن الجزيئي المنخفض. تم دراسة تأثير درجات الحرارة المختلفة على التفاعل على أنتاج الزئبق الأكسيد النانومترية. حيث تم نواحي عينات من الزئبق النانومترية تحت درجات حرارة مختلفة وهي صفر ، 50 و 90 درجة حرارة حرارة الغرفة (التي بدأ الفرق في درجة حرارة حرارة عينة بدرجة حرارة حرارة عينة ثم يتم رفع درجة الحرارة للوصول إلى درجة حرارة حرارة الغرفة). تم فحص واختبار العينة الناتجة باستخدام التحليل الطيفي-Fourier (FT-IR)، التحليل الامتصاصي-x-ray (XRD) من حيود الأشعة السينية، التحليل الأشعة السينية المشتتة (EDAX) التي يحدها النوع الباكر للأشعة السينية المضخة. كما أظهر طيف الامتصاصي EDAX و FT-IR و XRD نتائج تهيئة أكسيد الزئبق النانومترية Breaeja عادلة الأشعة السينية. كما أظهرت نتائج الأشعة السينية السامحة على الدقة أن أفضل حجم للعينات كان عند درجة حرارة هي 50 درجة حرارة و/or حجم محكم كان عند درجة حرارة حرارة الغرفة (التي يحدها نوع طيف الامتصاصي FT-IR) ثم ارتفعت للوصول إلى درجة حرارة حرارة الغرفة. 
