Preparation of ZnO nanoparticles by Electrolysis and evaluation of their antibacterial activity

Shaymaa N. Ismail 1, Ahmed N. Abd 2*, Osama Abdul Azeez Dakhil 2

Applied Physics Branch -Department of Applied Science/ Technology Universit / Iraq
Department of Physics, College of Sciences, Mustansiriyah University, Iraq2.

E-mail: ahmednajiaabd@uomustansiriyah.edu.iq

Abstract
This study is to prepare and characterize ZnO NPs by electrolysis method. The detailed characterization of ZnO nanoparticles was performed using ultraviolet spectroscopy, (FTIR), X-ray diffraction (XRD) analysis, electronic ice analysis and SEM scanning analysis demonstrating that the analysis of prepared ZnO nanoparticles is flower. The results of the antibacterial activity showed inhibitory activity against Pseudomonas aeruginosa, Staphylococcus aureus, E. coli, Pseudomonas aeruginosa and Bacillus subtilis.

Keywords: ZnO nanoparticles, Electrolysis, SEM, XRD

1. Introduction
Nanomaterial are very important in applications because the measurement properties vary from largest to nanometer size (10^-9m). Surface-to-volume ratio plays an important role in nanoparticles, which become more reactive [1,2]. “The unique physical and chemical properties of zinc oxide molecules such as high chemical stability, high electrochemical coupling coefficient and a wide range of radiation absorption and high noise on multifunctional materials [3,4]”. Several of preparation approaches have been studied for preparation zinc oxide nanoparticles as chemical precipitation, organometallic synthesis, microwave method, spray pyrolysis and thermal evaporation synthesis [5,6]. “The formation of nanoparticles includes nucleation and growth steps, the morphology and size of nanoparticles depend on different parameters such as: concentration and nature of precursor, solvent and type pH of the mixture, type of stabilizer and their concentrations, time and temperature, and the annealing temperature of the precipitated particles [7]”. Ananoparticles of metal oxides have been studied as antibacterial, it depose on surface of bacteria or accumulate in cytoplasm (periplasmic space) cause disrupt of cell function and membrane or disorganization of membranes [8,9]. Because zinc oxide nanoparticles ZnO NPs have antibacterial activity due to disorganization of bacterial mem-branes, which increases the permeability of the membrane leading to the accumulation of it in the membrane and cytoplasm, others refer to a mechanism of zinc oxide nanoparticles that prevent increased adhesion and internal.
Tight junction permeability and cytokine modulation [10]. In this paper, ZnO NPs were prepared by electrolysis method and deposited on glass as thin films, then measured by UV-VIS spectroscopic, XRD, SEM and EDX, and FTIR. These nanoparticles test antibacterial activity.

2-Material and methods

Preparation of ZnO nanoparticles

Electrolysis method was done to prepare bare ZnO nanoparticles, the prepared solutions were dissolved in distilled water, 0.10 M from zinc nitrate and 0.50 M urea (NH2CONH2) were mixed in a 1:1 solution and stirred to obtain a transparent and homogeneous solution, as shown in figure 1.

![Electrolytic Cells and Electrolysis](image)

3-Characterization Biological effects of ZnO NPs

UV-Vis Spectrophotometer was used to calculate the absorption spectra of zinc oxide nanoparticles. The morphology of ZnO NPs was characterized by scanning electron microscopy (SEM), equipped with an energy dispersive X-ray spectrometer (EDX) to analyze the elemental composition of the sample. An infrared shift Fourier analysis (Shimadzu / 84005) was used to investigate the functional groups of the prepared extracts. The crystal structure of the particles was determined by X-ray diffraction (XRD). The antibacterial efficacy of ZnO NPs was tested against Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa, and Bacillus subtilis with good diffusion agar methods. A loop filled with each test isolate was used to prepare the bacterial suspension in sterile saline. The density of the bacterial suspension was adjusted to 0.5 McFarland standards, then 100 ml of the bacterial suspension was inoculated with Muller-Hinton agar plates and spread as a L-glass rod, then 4 wells were made on the surface on the inoculation plate by cork borer an additional compound compound was added (0.01, 0.02, 0.04 and 0.06 mg / mL) per well and at 37 °C for 24 hours, the plates were incubated. Finally, the zone of inhibition was measured after 24 h of incubation.

4-Results and Discussion Characterization of the synthesized ZnO NPs

The spectrum of UV-visible absorption ZnO NPs e is show in fig. (2). The λ max of the absorption value around 250 nm for zinc oxide nanoparticles, This indicates a blue shift in the spectrum. The appearance of a strong band in the spectral model is due to the excitation of local surface plasmas that cause light to be scattered strongly by an electric field with a wavelength in which resonance occurs. UV-Vis Spectra was found for peak zinc oxide nanoparticles at 370 nm and are the properties of the nanoparticles that make up the study conducted by Paul and Ban (2014) [11].
FTIR spectroscopy of ZnO nano-particles are shown in Figure 3. The main characteristic absorption bands appeared in the powder. The range at the wavenumber about (3509 cm⁻¹) is related to O-H expansion pattern of the hydroxyl group, while the range (1600 cm⁻¹) is related to CO. The range 680 cm⁻¹ is associated with zinc. In previous studies, the peak appeared at 500 cm⁻¹, which correlates with transverse optical expansion patterns of Zn-O [12].

Fig. 3: FTIR analysis of ZnO NPs

Fig. 4 illustrates the XRD pattern of the thin film ZnO prepared by electrolysis and deposition in the glass substrate. Many sharp and narrow peaks 2θ indexed to (26.56), (33.58), and (39.26) large peaks. This belongs to the asymmetric crystal structure according to the ASTM card [No. 00-021-1486]. The crystallite size G(S) was estimated using Sheerer equation found to be (85.46nm), (73.71) and (144.70 nm) [13]:

\[ G(S) = 0.9 \frac{\lambda}{FWHM} \cos \theta \]  

Where FWHM the full width at half maxima when \( \theta \) is the diffraction angle and \( \lambda \) (0.154056 nm) is the wavelength of the incident X-ray.
Fig. (4): XRD of ZnO thin film

It shows in figure (5) the SEM images of ZnO NPs obtained by electrolysis method, the image show flower like structure. The result of ZnO nanoparticles as a flower shape like is observed in flower-shaped zinc oxide microscopic structures (ZnO) that have been synthesized via the electrochemical method [14].

Fig. (5): SEM image of ZnO thin film

It shows in figure (6) the EDX spectrum of ZnO NPs prepared by electrolysis method. The strong peaks observed in the spectrum related to Z and O. It shows in Table (1) the constitution of element to prepare zinc oxide nanoparticles found that two major peaks have weight percentage of 57.61 of zinc and 42.39 of oxygen. ZnO NPs have atomic percentage of 24.96 of zinc and 75.04 of oxygen, which confirmed the formation of zinc oxide nanoparticles in this method. The result similar with researcher that obtained zinc oxide nanoparticles which synthesized by sol gel method, EDX characterize, it has good purity (content of zinc- 55.38%; oxygen - 44.62%) [15].
Antibacterial activity in ZnO NPs was tested by well diffusion agar methods in Figure 7. Furthermore, Table 2 illustrates an inhibition zone that indicates the antibacterial effect of zinc oxide nanoparticles against bacteria. Many of the former studies; ZnO NPs have antibacterial activity against many bacteria. It is suggested that zinc oxide molecules cause the formation of peroxide that may give an antibacterial effect. Some have suggested that it may affect the membrane of bacteria and inhibit their growth [16-18]. The efficacy of antibacterial increased with decreasing zinc oxide nanoparticles size. So the enhanced bioactivity of smaller nanoparticle probably is related to the higher surface area to volume ratio [19,20].
Fig. (7): image of inhibition zone of bacteria

5. Conclusion

The work demonstrates how the thin film was prepared by electrolysis. Simple, easy and fast method for installing nanostructures. The film behavior as shown in the figures proves that the film has good optical transmission with good crystallization. This study also concluded that ZnO nanoparticles have significant activity and potential effect in reducing the growth of pathogenic bacteria in practical applications.

References

[1] Pandiyarasan, V., Shamugapriya, P., Thanuja, M.Y., Anusuya, T. and Vairavaraja, P., 2014. Synthesis and Characterization of ZnO Nanoparticles-A Green Chemistry Approach. Asian J. of Adv. Basic Sci, 3(1), pp.94-101–408.
[2] Getie, S., Belay, A., Chandra Reddy, A.R. and Belay, Z., 2017. Synthesis and Characterizations of Zinc Oxide Nanoparticles for Antibacterial Applications. J Nanomedic Nanotechnol S., 8, p.2.
[3] Segets D, Gradi J, Taylor RK, Vassilev V, Peukert W (2009) Analysis of optical absorbance spectra for the determination of ZnO nanoparticle size distribution, solubility, and surface energy. ACS Nano 3: 1703-1710.
[4] Lou X (1991) Development of ZnO series ceramic semiconductor gas sensors. J Sens Trans Technol 3: 1-5.
[5] G. Murugadhoss (2012), “Synthesis and Charaterization of Transition metals Doped Zno Nanorods”, Elsevier, J.Mater. Sci.Tecnol, 28(7), 587-593.
[6] S.R.Brintha, M.Ajihta (2015). Synthesis and characterization of ZnO nanoparticles via aqueous solution, sol-gel and hydrothermal methods. IOSR Journal of Applied Chemistry (IOSR-JAC). 8, 11, 66-72.
[7] Van Ngo G.V, Margaillan A, Sylvie Villain, Leroux C, Bressy C (2013). Synthesis of ZnO nanoparticles with tunable size and surface hydroxylation. J Nanopart Res. 15: 1332.
[8] Brayner R, Ferrari-Iliou R, Brivois N, Djediat S, Benedetti MF, et al. (2006) Toxicological impact studies based on escherichia coli Bacteria in ultrafine ZnO nanoparticles colloidal medium. Nano Lett 6: 866-870.
[9] Zhang L, Jiang Y, Ding Y, Povey M, York D (2007) Investigation into the antibacterial behaviour of suspensions of ZnO nanoparticles (ZnO nano-fluids). J Nanopart Res 9: 479-489.
[10] Shaymaa N. Ismail, Ahmed N. Abd , Osama Abdul Azeez Dakhil,(2020) "Comparison between preparation of ZnO nanoparticles by Electrolysis and standard ZnO Nanoparticles", Academic J.for Engineering and Science, NO.1, February.
[11] Paul S and Ban D. K. (2014). Synthesis, Characterization and the Application of ZnO Nanoparticles in Biotechnology. Int'l Journal of Advances in Chemical Engg., & Biological Sciences (IJACEBS). 1(1).
[12] Salahuddin N. A, El-Kemary M and Ibrahim E. M (2015). Synthesis and Characterization of ZnO Nanoparticles via Precipitation Method: Effect of Annealing Temperature on Particle Size. Nanoscience and Nanotechnology. 5(4): 82-88.
[13] S. Ponarulselvam, C. Panneerselvam, K. Murugan, N. Aarthi, K. Kalimuthu, and S. Thangamani, "Synthesis of silver nanoparticles using leaves of Catharanthus roseus Linn. G. Don and their antiplasmodial activities," Asian Pacific Journal of Tropical Biomedicine, vol.2, no. 7, pp. 574-580, Jul 2012.
[14] Venkatesha, T.G., Nayaka, Y.A., Viswanatha, R., Vidyasagar, C.C. and Chethana, B.K., 2012. Electrochemical synthesis and photocatalytic behavior of flower shaped ZnO microstructures. Powder technology, 225, pp.232-238.
[15] Hasmidawani J.N, Azlina H.N, Norita H, Bonnia N.N, Ratim S. and Ali E.S. (2016). Synthesis of ZnO Nanostructures Using Sol-Gel Method. Procedia Chemistry. 211- 216.
[16] J. Sawai, Quantitative evaluation of antibacterial activities of metallic oxide powders (ZnO, MgO and CaO) by conductimetric assay, J. Microbiol. Methods 54 (2003) 177–182.
[17] Shaymaa N. Ismail, Ahmed N. Abd, Osama Abdul Azeez Dakhil " Comparison of preparation of ZnO nanoparticles by chemical and aqueous hydrolysis and evaluation of their antibacterial activity", plant Archives Vol. 20 Special Issue, 2020,pp.3372-3376.
[18] Y. Liu, L. He, A. Mustapha, H. Li, Z.Q. Hu, M. Lin, Antibacterial activities of zinc oxide nanoparticles against Escherichia coli O157:H7, J. Appl. Microbiol. 107 (2009) 1193–1201.
[19] Emami-Karvani Z and Chehrazi P (2011). Antibacterial activity of ZnO nanoparticle on gram-positive and gram-negative bacteria. African Journal of Microbiology Research. 5(12). 1368-1373.
[20] Nagarajan P, Rajagopalan V (2008). Enhanced bioactivity of ZnO nanoparticles an antimicrobial study. J. Sci. Technol. Adv. Mater., 9(3): 035004.