

Structural, Morphological and Optical Properties of electrochemically Synthesized $\text{Al}_2\text{O}_3/\text{ZnO}$ Nanocomposite

Wasan Najm Abdullah

Department Of Chemistry, Faculty of Science,
Kufa University, Najaf City,
Iraq

Corresponding Author: wasann.hassan@uokufa.edu.iq

Abstract

In the present era, nanomaterial's have many uses due to their superior chemical and physical characteristics. The electrochemical deposition approach, which is quick, simple, and affordable, is one of the key techniques for creating Nano composites ($\text{Al}_2\text{O}_3/\text{ZnO}$). Advanced methods like x-ray Diffraction (XRD), field-emitted scanning electron microscopy (FESEM), microscopy using transmission electrons (TEM), ultraviolet-visible (UV-Vis), energetic dispersive x-ray (EDX), and atomic force microscopy, also called AFM, are used to analyze the Nano composite. According to the investigation, the Nano composite had an average crystal size of 8.4 nm, a band gap value of 3.23 (e.v), and a Nano rod form.

Keyword: Aluminum Foil; Composite; Electrochemical Cell; Solar Cell; Zinc Foil

1. Introduction

Introduction Metal oxide nanoparticles have demonstrated superior stability, low toxicology, high immovability, and selectivity compared to related organic compounds [1]. Moreover, the particle size of these particles is responsible for the changes in their basic physical and chemical characteristics. These microscopic particles show remarkable uses in catalysis, drug delivery, treatment of water, sensor gadgets, semiconductor materials, and solid oxide fills [2]. The main advantages of metal oxide nanoparticles are: -changes in surface properties that result in an extreme increase inside the band gap, which affects conductivity and chemical activity; -changes in electrochemical qualities due to the influence of quantum regulation; -structural changes that permit the modification of structural symmetry and cell parameters [3]. A material made up of multiple components is called a composite. One or more fillers, that include particles, sheets, having fibers with a high volume-to-surface ratio and diameters smaller than 100 nm, are usually supported by a matrix [4]. Nano composite is more electrically conductive, lighter, stronger, less brittle, resistant to scratches, recyclable, and flame-retardant than traditional composite materials. Adding nano fillers, such as carbon nanotubes, to the composite boosts its durability and resilience to wear, tear, overall breaking while preserving the material's surface transparency and high standards [5].

The properties of Nano composites at the Nano scale level are unique. Water purification, super capacitors, electro conductive scaffolds, anticorrosive and antiballistic optoelectronic devices, solar cells, hard coatings, biosensors, Nano devices, and renewable energy generation are just a few of the several industries in which they are extensively utilized. Silver/polyaniline (AgPani) Nano composites, which were produced by in-situ reduction of silver in aniline through photolysis at 265 nm, may be employed in additional bio sensing applications in addition to offering a viable technique for electro-catalytic hydrogen oxidation [6]. The use of Nano composites in state-of-the-art technological advancements, such as Nano biosensor-powered bio analysis and detection applications, is advantageous for the environment [7].

Structural, Morphological and Optical Properties.

2. Materials and Methods

2.1 Materials

97% aluminum foil, zinc foil, and polyvinyl alcohol (Fluka, Germany). Graphite, 99% ethanol, 98% acetone, 97% polyethylene glycol, 95% potassium chloride, and 97% potassium iodide (CDH, India). 99% iodine (Thomas Baker, India).

2.2 Methods

2.2.1 Preparation of Al₂O₃/ZnO Nano composite.

An inert electrode made of graphite and a working electrode constructed of aluminum and zinc foil were used to make the Al₂O₃/ZnO Nano composite using the electrochemical technique. A power supply is also utilized. Acetone and ethanol were used to wash the cathode and anode, followed by deionized water [8]. A 200 mL solution containing 5 mL of 10 g/100 mL electrolyte (KCl) and 10 mL of 10 g/100 mL stabilizer (polyvinyl alcohol (PVA) and ionized water, respectively) is poured into the electrochemical cell. Zinc foil (0.5 cm x 4 cm) and aluminum foil (0.5 cm x 4 cm) make up the working electrode, which is placed face-to-face with a graphite electrode (2 cm x 5 cm) in the cell electrolyte [9]. At a temperature of less than 30°C, the electrolysis process was carried out in an undivided electrolytic cell for sixty minutes while stirring. The voltage ranges from 9 to 15 volts. After centrifuging the resulting white precipitate of Al₂O₃/ZnO and repeatedly washing it with ethanol and deionized water, it was transferred into a drying vessel, dried for 60 minutes at 60°C, and then calcined for 60 minutes at 700°C [10].



Figure 1: The Al₂O₃/ZnO Nano composite is prepared using an electrochemical method.

3. Results and Conversations

3.1 Al₂O₃/ZnO Nanocomposite X-Ray Diffraction Analysis.

The Al₂O₃/ZnO Nano composite's XRD patterns were created at room temperature using an electrochemical process. The electrochemically produced Al₂O₃/ZnO NPs' XRD spectrum. The XRD patterns have revealed interference across the peaks that exist in the same location or close to both Al₂O₃ and ZnO NPs, and some maxima appear isolated where interference does not occur. Additionally, it has been found that the intensities of these peaks vary, as illustrated in figure 2. Several diffraction peaks were observed at 2θ (32.023°, 36.856°, 48.352°, 57.320°, 63.253°, and 69.643°) in relation to miller indices (100), (101), (102), (110), (103), and (112) of ZnO NPs, and 35.972°, 46.860°, and 74.784°) in relation to miller indices (311), (400), (440), and (620) of Al₂O₃NPs. A shift in the majority of peaks toward different diffraction angles, a decrease in the XRD intensity of some peaks, an increase in other peaks, and the removal of some peaks for Al₂O₃ NPs result in the formation of Al₂O₃/ZnO Nano composite, a new nanomaterial [11].

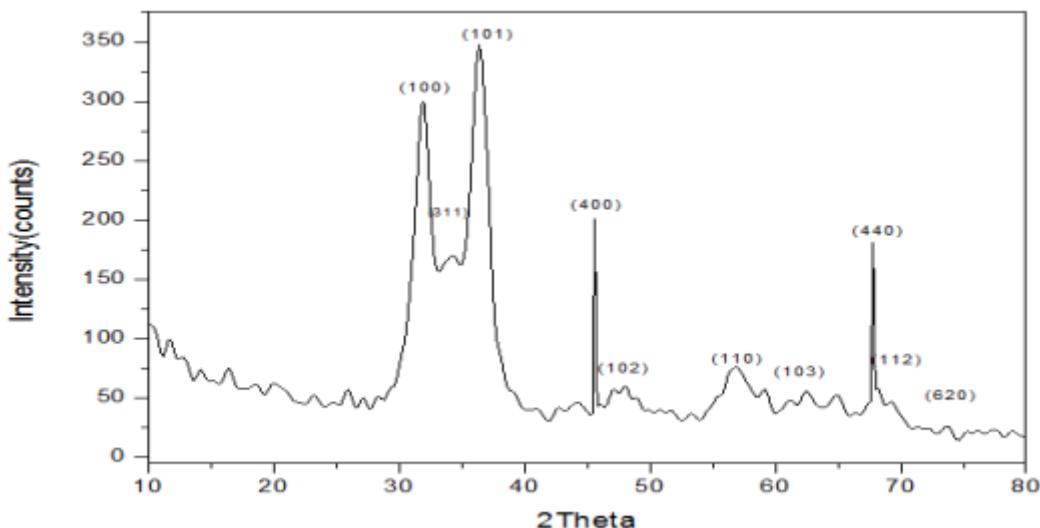


Figure 2: The $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite's X-ray diffraction pattern.

3.2 $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite Analysis Using Transmission Electron Microscopy (TEM).

Figure 3 displays TEM micrographs of $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite that were produced electrochemically at room temperature. The electrochemically produced $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite crystalline forms have a nanorods shape, according to the TEM data [12].

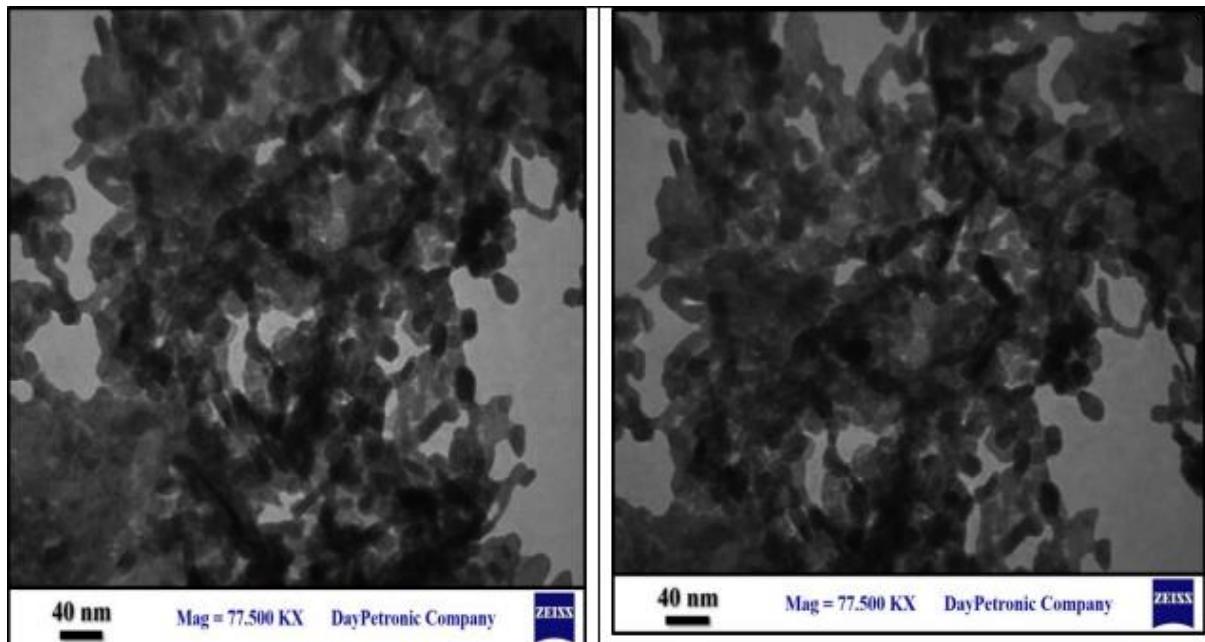


Figure 3: TEM images of $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite.

3.3 The $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite Field Emissions Scanning Electron Microscope (FESEM).

It is utilized by FESEM to ascertain the shape and structure of the produced films. The FESEM in secondary electrons mode at 100,000x magnification and 10 kV operating voltage may produce high-resolution pictures of a sample surface. The arrangement of the particles was seen in the FESEM picture. As seen in figure 4, the external layer of the $\text{Al}_2\text{O}_3/\text{ZnO}$ composite had a full and consistent coating of compacted nano sheets that were coupled to one another to produce a three-dimensional porous structure. In order to create the $\text{Al}_2\text{O}_3/\text{ZnO}$ composite, Al_2O_3 NPs were essential since they not only provided structural support for the ZnO NPs but also stopped them from aggregating [13].

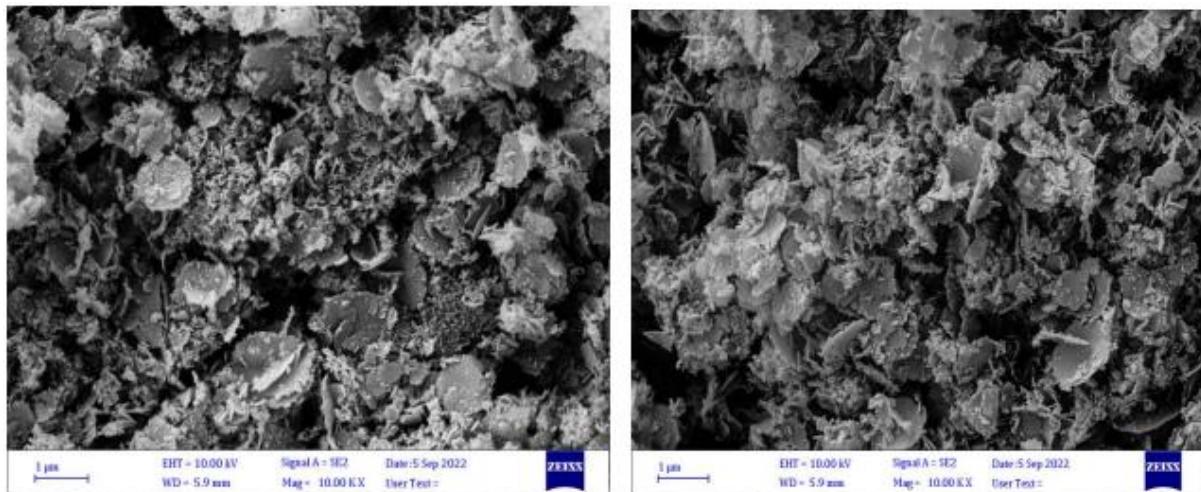


Figure 4: FESEM images of $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite.

3.4 $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite Energy Dispersive X-Ray Spectroscopy (EDX).

Energy dispersive X-ray spectroscopy (EDX) was used to assess the purity and stoichiometry of the electrochemically produced $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite. The results, which are displayed in Figure 5, demonstrate that the samples were extremely clean because only particular signals for oxygen, aluminum, and zinc were present. As can be observed in the EDX chart for ZnO NPs, the appearance of gold in the chart is a result of gold covering the sample during testing. $\text{Zn}=58.1\%$, $\text{Al}=22.8\%$, and $\text{O}=19.1\%$ are the weight proportions of $\text{Al}_2\text{O}_3/\text{ZnO}$. These ratios show that the ZnO : Al_2O_3 ratio is 2:1, in conjunction with the presence of free metal deposits of zinc and aluminum that function as doping [14].

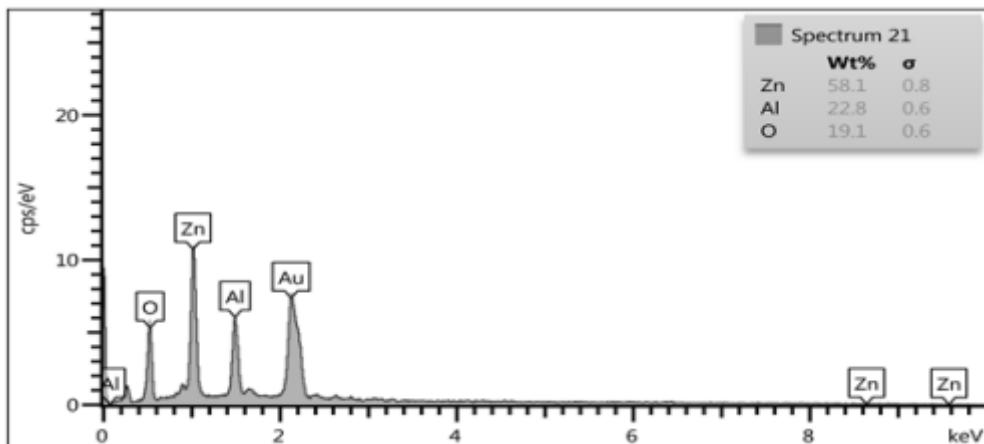


Figure 5: $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite's EDX spectrum.

3.5 Analysis of the $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite by Atomic Force Microscopy (AFM).

Atomic force microscope (AFM) topographical imaging is a useful method for gathering detailed information on the morphology, topography, and texture of different surfaces. The faint structures are represented by opaque colors in AFM pictures, whereas the soaring structures are represented by bright colors due to the various orientations of the particle grains. Surface skewness, Root Mean Square, and surface roughness Average (Sa) were among the many important features that were included in the comprehensive data that was obtained from AFM measurements. On the other hand, AFM measurements offer superior information on the average nanoparticle diameter, size dispersion, and uniformity of nanoparticle surfaces [15]. Figure 6 illustrates how $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite can be used to create images and configurations in two and three dimensions (D and 3D).

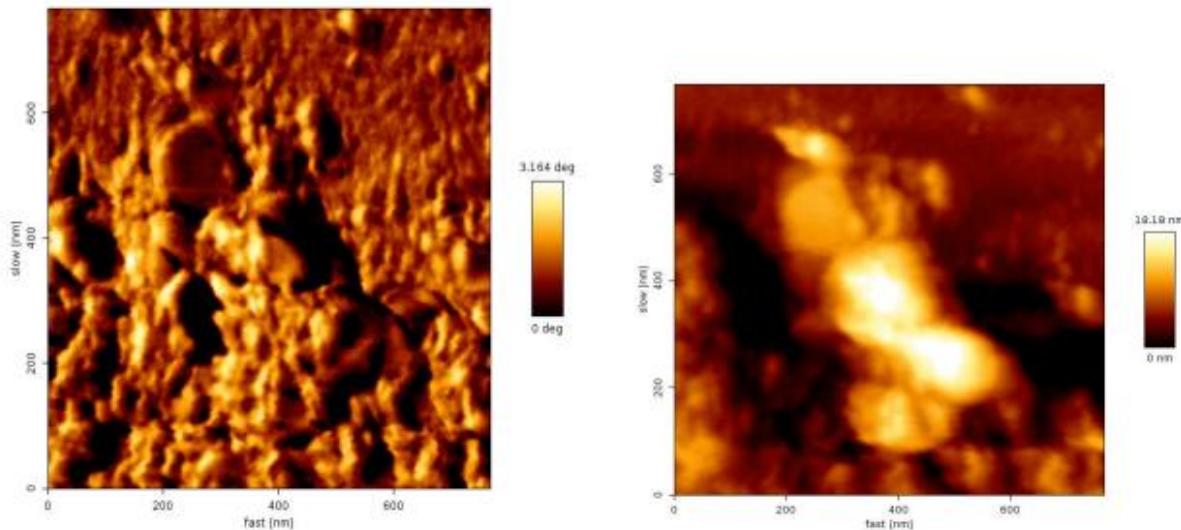


Figure 6: $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite AFM 2D and 3D pictures

3.6 $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite Fourier transformation infrared spectroscopy analysis (FT-IR).

FTIR spectroscopy was employed to get additional insight into the chemical relationship between Al_2O_3 NPs and ZnO NPs. The $\text{Al}_2\text{O}_3/\text{ZnO}$ FTIR spectrum are shown in Figure 7. The presence with Al-O and Zn-O bonds was shown by the typical absorption peaks of the $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite that were mainly visible in the FTIR spectrum. The contraction vibration from Zn-O was linked to the peak at 464 cm^{-1} , and the Al-O expansion mode was suggested by the stronger absorption at $500\text{--}750\text{ cm}^{-1}$ [16].

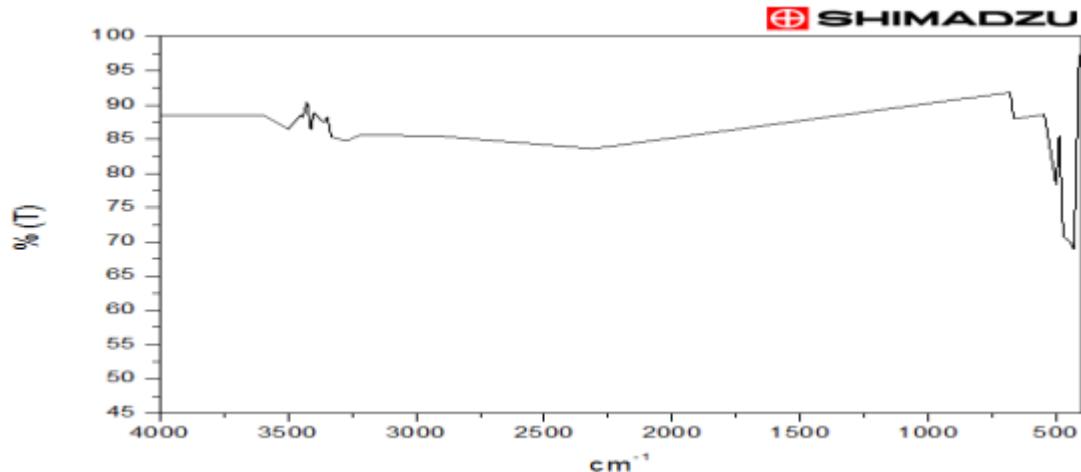


Figure 7: $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite FTIR spectrum; UV-Vis analysis of Al_2O_3 NPs.

3.7 UV-Visible Spectroscopy (UV-Vis Al_2O_3 and ZnO Nano Composite).

Solid state ultraviolet (UV)-visible spectroscopy was used to analyze $\text{Al}_2\text{O}_3/\text{ZnO}$ composites thin films made on glassy slides at temperatures below 30 C^0 . Figure 8 displays the generated UV-visible spectra, which seem to have a defined maximum wavelength [17].

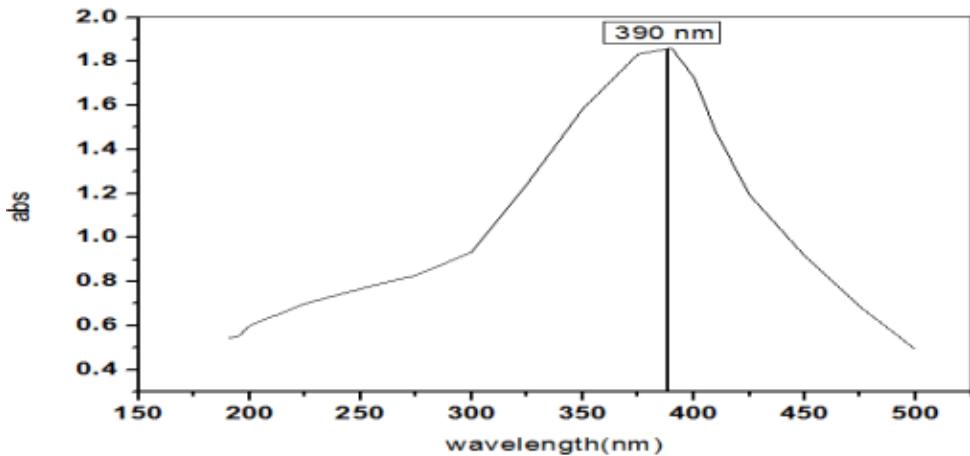


Figure 8: $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite spectrum.

Furthermore, optical absorption is an essential technique for determining the optical energies band gap of crystallized and amorphous materials, as seen in figure 9. The type and magnitude of the optical band gap can be determined using the fundamental absorption, which is equivalent to the acceleration of electrons from the band called the valence band to the band for conduction. All of the films made for this investigation were subjected to UV-Vis spectroscopy using a spectrophotometer that operated in the 150–500 nm range. By calculating radiation along the (hv) and (αhv) 2 lines on the Taucplot, the optical band gap of the fabricated films was also determined. The shifting characteristics of $\text{Al}_2\text{O}_3/\text{ZnO}$ films in the optical energy gap at 25 was studied using the UV area absorption peak [18].

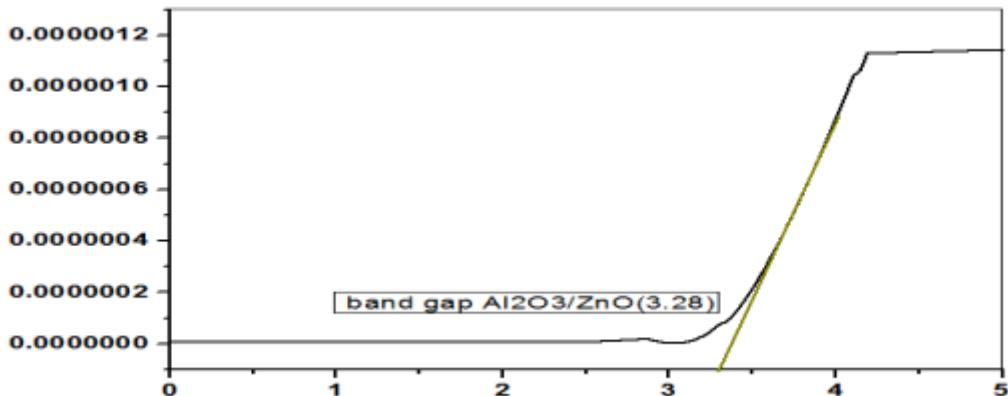


Figure 9: the optical band gap of $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composite.

4. Conclusion

A straightforward and inexpensive electrochemical deposition technique was used to successfully create the $\text{Al}_2\text{O}_3/\text{ZnO}$ nano hybrid. The creation of a new composite material was verified by XRD structural characterization, which showed peak shifting, intensity variations, and the emergence or disappearance of particular diffraction peaks. While FESEM pictures showed evenly distributed crumpled nano sheets producing a porous 3D structure, showing successful interaction between Al_2O_3 and ZnO nanoparticles, TEM investigation revealed that the nano composite had a nano-rod shape. Energy-dispersive X-ray spectroscopy (EDX) verified a $\text{ZnO}: \text{Al}_2\text{O}_3$ ratio of roughly 2:1 and demonstrated the exceptional purity of the produced nano composite. AFM investigation also revealed a homogeneous surface with appropriate roughness and well-distributed nanoscale features. The presence of $\text{Al}-\text{O}$ and $\text{Zn}-\text{O}$ functional groups was confirmed by FT-IR spectroscopy, indicating effective bonding within the composite. Tauc plot analysis revealed an optical band gap of 3.23 eV and UV-Vis measurements demonstrated a distinct optical absorption peak, indicating that the nano composite has promising optical properties appropriate for optoelectronic and photocatalytic

applications. Overall, the study shows that electrochemical synthesis is an effective way to create high-quality $\text{Al}_2\text{O}_3/\text{ZnO}$ Nano composites with desired structural, morphological, and optical characteristics that make them appropriate for use in advanced electronic devices, sensors, energy storage, and catalysis.

5. Conflict of interest. The authors declare that they have no conflict of interest in this article.

6. References

- [1] Anggraeni, S. D., & Kurniawan, F. (2020). Synthesis of Aluminium Nanoparticles Using Electrochemical Method. In *Journal of Physics: Conference Series* (Vol. 1445, No. 1, p. 012013). IOP Publishing.
- [2] Alghriany, A. A., Omar, H. E. D. M., Mahmoud, A. M., & Atia, M. M. (2022). Assessment of the Toxicity of Aluminum Oxide and Its Nanoparticles in the Bone Marrow and Liver of Male Mice: Ameliorative Efficacy of Curcumin Nanoparticles. *ACS omega*, 7(16), 13841-13852.
- [3] Chłędowska, J., Wyrwa, J., Rękas, M., & Brylewski, T. (2022). Effects of aluminum oxide addition on electrical and mechanical properties of 3 mol% yttria-stabilized tetragonal zirconia electrolyte for ITSOFCs. *Materials*, 15(6), 2101.
- [4] Shaislamov, U., Kim, H., Yang, J. M., & Yang, B. L. (2020). CuO/ZnO/TiO₂ photocathodes for a selfsustaining photocell: Efficient solar energy conversion without external bias and under visible light. *International Journal of Hydrogen Energy*, 45(11), 6148-6158.
- [5] Qi, W., Tian, Y., Lu, D., & Chen, B. (2022). Research Progress of Applying Infrared Spectroscopy Technology for Detection of Toxic and Harmful Substances in Food. *Foods*, 11(7), 930.
- [6] Praseptiangga, D., Zahara, H. L., Widjanarko, P. I., Joni, I. M., & Panatarani, C. (2020, May). Preparation and FTIR spectroscopic studies of SiO_2 -ZnO nanoparticles suspension for the development of M. K. A. A. AL-BYATI and A. M. J. Al-DUHAIDAHAWI, 2023 Azerbaijan Medical Journal 8034 carrageenan-based bio-nanocomposite film. In *AIP Conference Proceedings* (Vol. 2219, No. 1, p. 100005). AIP Publishing LLC
- [7] Janković, S., Mitić, M., Arsić, B., & Stankov-Jovanović, V. (2021). The kinetic and thermodynamic studies of solid-liquid extraction of apigenin-glycosides from parsley (*Petroselinum crispum*). *Separation Science and Technology*, 56(13), 2253-2265.
- [8] Al-Douri, Y. (2022). *Renewable Energy: Analysis, Resources, Applications, Management, and Policy*.
- [9] Micó-Vicent, B., Perales Romero, E., Jordán-Núñez, J., & Viqueira, V. (2021). Halloysite and Laponite Hybrid Pigments Synthesis with Copper Chlorophyll. *Applied Sciences*, 11(12),
- [10] Eid, M. M. (2022). Characterization of Nanoparticles by FTIR and FTIR-Microscopy. In *Handbook of Consumer Nanoproducts* (pp. 1-30). Singapore: Springer Singapore
- [11] Roslan, N., Ya'acob, M. E., Radzi, M. A. M., Hashimoto, Y., Jamaludin, D., & Chen, G. (2018). Dye Sensitized Solar Cell (DSSC) greenhouse shading: New insights for solar radiation manipulation. *Renewable and Sustainable Energy Reviews*, 92, 171-186.
- [12] Bouhamed, H., & Baklouti, S. (2014). Synthesis and characterization of $\text{Al}_2\text{O}_3/\text{ZnO}$ nanocomposite by pressure less sintering. *Powder technology*, 264, 278-290.
- [13] Di, S., Gong, L., & Zhou, B. (2020). Precipitated synthesis of Al_2O_3 -ZnO nanorod for highperformance symmetrical super capacitors. *Materials Chemistry and Physics*, 253, 123289.
- [14] kashel, N., Biu, J., Cheng, K., Nee, W., Ho, W., & Tang, H. (2019). Preparation of $\text{Al}_2\text{O}_3@ \text{ZnO}$ coreshell micro rod with excellent adsorption affinity for Congo red molecule. *Applied Surface Science*, 473, 251-260.
- [15] zenate, G., soraein, Z., & Abdelghany, M. (2021). Properties of inorganic Al_2O_3 -ZnO composites. *Applied Physics A*, 127(2), 1-6.

Structural, Morphological and Optical Properties.

- [16] Saffar, A., Abbastabar Ahangar, H., & Barati, S. (2021). The use of high concentration of zinc acetate solution for synthesis of Al₂O₃-ZnO nanocomposite and its application for dye removal. *Journal of Nano Chemistry and Electrochemistry*, 1(1), 37-48.
- [17] Mahdavi, R., & Talesh, S. S. A. (2021). Selective degradation of mixed dye pollutant in aqueous phase in the presence of ZnO/ Al₂O₃ as sonophotocatalysis based on central composite design. *Korean Journal of Chemical Engineering*, 38(6), 1260-1271.
- [18] Niu, H., Zhou, D., Yang, X., Li, X., Wang, Q., & Qu, F. (2015). Towards three-dimensional hierarchical ZnO nanofiber@ Ni (OH)₂ nanoflake core–shell hetero structures for high-performance asymmetric super capacitors. *Journal of Materials Chemistry A*, 3(36), 18413-1842