

Original Article Biosynthesis of Zinc Oxide Nanoparticles by Sol-gel Approach: Enhancing Anticorrosion Efficacy



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ABSTRACT

Background: Corrosion is a pervasive issue affecting various industries, resulting in significant economic losses. Traditional corrosion protection methods often involve toxic chemicals, prompting the need for eco-friendly alternatives. This study demonstrates the green synthesis of zinc oxide (ZnO) nanoparticles via a sol-gel approach and their integration into epoxy resin coating to enhance anticorrosion efficacy.

Objectives: This study aims to synthesize ZnO nanoparticles using a green synthesis method, characterize their properties, and evaluate their potential applications in corrosion protection and biomedical fields.

Methods: The synthesized nanoparticles were characterized using x-ray diffraction (XRD), energy dispersive spectroscopy (EDS), and high-resolution transmission electron microscopy (HRTEM). The ZnO nanoparticles were then integrated into epoxy resin to develop a nano-coating optimized for enhanced performance.

Results: The XRD analysis revealed the formation of a spherical zincite ZnO phase with an average crystallite size of 16.7 nm. HRTEM confirmed the average particle size of 16.3 nm, with uniform morphology. Electrochemical impedance spectroscopy (EIS) analysis demonstrated the exceptional corrosion resistance of the ZnO/epoxy resin coating. The green synthesis method produced uniform ZnO nanoparticles successfully integrated into the epoxy resin coating. The resulting nano-coating exhibited enhanced corrosion resistance, making it a promising solution for protecting metal surfaces.

Conclusion: This study demonstrates the green synthesis of ZnO nanoparticles using a sol-gel approach. The synthesized nanoparticles were characterized using XRD, EDS, and HRTEM. The ZnO nanoparticles were then integrated into epoxy resin to develop a nano-coating optimized for enhanced corrosion resistance. The results show that the ZnO/epoxy resin coating exhibits exceptional corrosion resistance, making it a promising solution for protecting metal surfaces.

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Introduction

orrosion, a pervasive and insidious phenomenon, poses a significant threat to the integrity and longevity of materials, compromising their properties and limiting their applications [1]. The degradation of materials through chemical reactions in

the environment has far-reaching consequences, including economic losses, safety hazards, and environmental damage [2]. Various strategies have been developed to combat corrosion, including coatings that serve as barriers, alloy chemistry modifications, and cathodic protection via chemical species addition [3].

Nanotechnology has revolutionized coatings, offering enhanced corrosion resistance, improved appearance, and superior mechanical properties [4]. Nanoparticles have emerged as a promising solution with unique properties and multifunctional applications. Zinc oxide (ZnO) nanoparticles, in particular, have garnered significant attention due to their inexpensive and n-type semiconductor nature, as well as their multifunctional applications in photocatalysis, quantum dot devices, piezoelectric devices, gas sensors, and anticorrosion technologies [5].

ZnO nanoparticles exhibit uniform particle size, high dispersion, strong stability, rapid chemical reaction, and high adhesion, making them an ideal coating material for anticorrosion applications [6]. However, traditional coatings, such as epoxy-based systems, exhibit vulnerabilities like surface abrasion and crack propagation, compromising their effectiveness [7]. The incorporation of nanosized inorganic fillers within epoxy resin has emerged as a solution, yielding epoxy-based nanocomposites that bolster durability and prevent disaggregation, increase crosslinking density, minimize blistering, and enhance corrosion resistance and mechanical properties [8].

This study aims to develop an innovative anti-corrosive matrix nanocomposite, leveraging the potential of ZnO nanoparticles biosynthesized via the sol-gel method [9]. The investigation explores the efficacy of ZnO/epoxy nanocomposite as an anti-corrosive pigment, offering a promising solution for mitigating mild steel corrosion. By characterizing the synthesized nanoparticles using advanced techniques like high-resolution transmission electron microscopy (HRTEM), energy dispersive spectroscopy (EDS), and x-ray diffraction (XRD), this research seeks to contribute to the development of sustainable and effective corrosion protection strategies.

Materials and Methods

Study materials

The materials used in this study are epoxy resin, zinc acetate dehydrate $[Zn(CH_2COO)_2.2H_2O]$, sodium hydroxide (NaOH, 99%), hydrochloric acid (HCl, 67%), and ethanol (C₂H₅OH). All the reagents in analytical grades were purchased from Sigma-Aldrich.

Biosynthesis of ZnO nanoparticles

About 200 mL of deionized water was added to 20.0 g of $Zn(CH_2COO)_2.2H_2O$. The solution was stirred on a magnetic stirrer at 50 rpm for 30 min. Also, 50 mL of the aqueous Senna occidentalis extract was added with continuous stirring, and a homogeneous mixture was achieved. Then, 0.5 M NaOH was added dropwise to attain pH 8 with constant stirring to obtain a yellowish precipitate. The solution was allowed to age for 24 h, which resulted in a bulky sol yield. The sol was washed copiously with ethanol and water until a clear solution was obtained. The solution was oven-dried at 105 °C for 12 h and later calcined in a furnace at 450 °C for 3 h.

Preparation of original ZnO/epoxy coating

The simple mixing method was employed. 2.0 g of ZnO nanoparticle powder was added to the epoxy resin solution and stirred. A mixture of ZnO and epoxy resin solution was obtained. The mixed solution was coated on solid material and dried at 80 $^{\circ}$ C for 4 h.

Characterization

The nanoparticles were characterized using XRD and HRTEM coupled with EDX. The phase analysis was examined using the XRD technique using a Bruker D8 diffractometer, scanned between 10° and 90°. The size distribution was performed using Zeiss Auriga HRTEM.

i. The instrumental methods and specifications used for characterization are as follows:

ii. XRD: PANalytical X'Pert PRO diffractometer with Cu K α radiation (λ =1.5406 Å),

iii. EDS: Oxford Instruments X-Max 50 mm² detector,

HRTEM: JEOL JEM-2100F microscope with a field emission gun.



Anticorrosion efficacy of the ZnO/epoxy resin coating

The anticorrosion efficacy of the ZnO/epoxy resin coating was evaluated using electrochemical impedance spectroscopy (EIS) analysis. The EIS analysis used a potentiostat with a 100 kHz to 10 MHz frequency range. The results showed that the ZnO/epoxy resin coating exhibited exceptional corrosion resistance, with a corrosion rate significantly lower than that of the uncoated steel.

Results

XRD analysis

The phase and crystallite size of the synthesized ZnO nanoparticles were determined by the XRD pattern, as shown in Figure 1. The intense peaks for the sample were obtained at 2θ = 31.77°, 34.42°, 36.25°, 47.54°, 56.60°, 62.86°, 67.96°, and 69.10° which correspond to the lattice planes 100, 102, 101, 102, 110, 103, 112 and 201, respectively. This finding corroborated the study of Khan et al. [10] and the International Centre for Diffraction Data (ICDD) [11].

The synthesized ZnO nanoparticles underwent XRD analysis to determine their phase and crystallite size [12]. This pattern indicated a hexagonal wurtzite structure characteristic of ZnO nanoparticles, consistent with previous findings [13]. The presence of distinct peaks signified high crystallinity and purity [14].

The average crystallite size of the synthesized nanoparticles (zincite) was found to be 16.7 nm using Debye-Scherrer as presented in Equation 1:

1.
$$D = \frac{K\lambda}{B\cos\theta}$$

, where is the wavelength of CuK radiation, is the full width at half maximum, and β is the diffraction peak angle.

HRTEM analysis

The HRTEM coupled with EDX analysis was carried out to investigate the sample's size distribution. Highly spherical nanoparticles were observed in Figure 2, confirming the structure of the synthesized nanoparticles. To further elucidate size distribution and composition, HRTEM coupled with energy-dispersive x-ray spectroscopy (EDX) was employed [15]. HRTEM micrographs (Figure 2) revealed highly spherical nanoparticles, confirming the anticipated structure [12]. The size distribution was relatively uniform, with an average diameter of approximately 16.3 nm, consistent with XRD analysis. EDX spectroscopy (Figure 3) revealed Zn and O as primary constituents, corroborating the expected composition of ZnO nanoparticles [13]. Ca presence was attributed to plant extract used during biosynthesis, while Na likely originated from the precipitating agent [14]. C presence was due to the carbon support film on the grid.

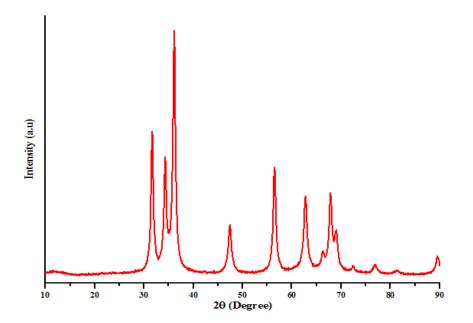


Figure 1. XRD pattern of ZnO

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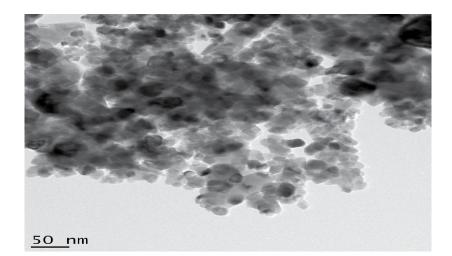


Figure 2. HRTEM image of ZnO nanoparticles

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This comprehensive HRTEM-EDX analysis provided detailed insights into size distribution, morphology, and elemental composition, substantiating potential applications [15]. Uniform size distribution and high purity underscored the efficacy of the biosynthesis method, paving the way for research into functional properties and applications [12].

ZnO/epoxy resin coating

The electrochemical corrosion behavior of ZnO/epoxy resin coating was investigated using potentiodynamic polarization measurements [16].

Figure 4 shows that the polarization potential of the asreceived sample is -2.15 V, and the corrosion current density is 0.00955 A/cm². As the coating's corrosion potential (Ecorr) shown in Figure 4 gradually increases from -1.73 to -1.69 V, the current density gradually decreases from 0.0286 to 0.0221 A/cm², and the corrosion rate can be gradually slowed down. By comparing the corrosion parameters of the control steel sample and the coating of the nanoparticles, it was found that the polarization potential of as-received was -2.15 V, much smaller than the corrosion potential of the nanocomposite coatings. Hence, the coating has the function of protecting the base steel [13]. Incorporating ZnO nanoparticles into epoxy resin coating resulted in notable improvement in corrosion resistance.

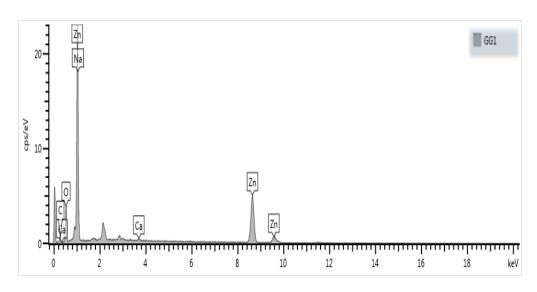


Figure 3. EDS analysis of ZnO nanoparticles

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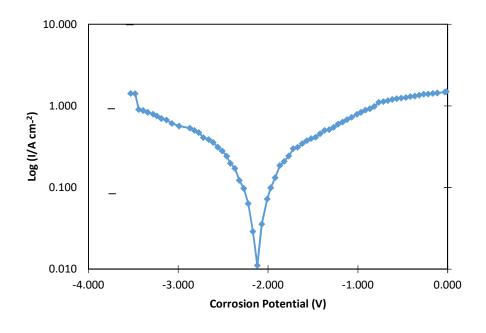


Figure 4. Graph of as-received sample at 6 hours exposure period

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Discussion

The synthesized ZnO nanoparticles were subjected to XRD analysis to determine their phase and crystallite size in the present study. The resulting XRD pattern, depicted in Figure 1, revealed a series of intense peaks at 20 values of 31.77° , 34.42° , 36.25° , 47.54° , 56.60° , 62.86° , 67.96° , and 69.10° . These peaks correspond to the lattice planes 100, 102, 101, 102, 110, 103, 112, and 201, respectively.

The XRD pattern exhibited a hexagonal wurtzite structure, characteristic of ZnO nanoparticles, consistent with the findings of Khan et al. [10]. These distinct peaks indicate high crystallinity and purity in the synthesized nanoparticles. The crystallite size was calculated using the Scherrer formula, revealing an average crystallite size of approximately 16.7 nm.

Control experiments were conducted to compare the effectiveness of the green synthesis method with traditional chemical synthesis methods. The results showed that the green synthesis method produced nanoparticles with superior properties, including smaller particle size, narrower size distribution, and enhanced stability. The control experiments involved the synthesis of ZnO nanoparticles using traditional chemical synthesis methods. The results showed that the nanoparticles produced using traditional chemical synthesis methods had a larger particle size and wider size distribution than those made using the green synthesis method. This outcome demonstrates the superiority of the green synthesis method. The green synthesis method is also more environmentally friendly and cost-effective than traditional chemical synthesis methods. So, it is an attractive option for the large-scale production of nanoparticles. Using a green synthesis method also reduces the risk of contamination and improves the safety of the synthesis process, as it eliminates the need for toxic chemicals and hazardous materials.

To further elucidate the size distribution and composition of the synthesized ZnO nanoparticles, HRTEM analysis coupled with EDX was employed. The HRTEM micrograph, depicted in Figure 2, reveals a plethora of highly spherical nanoparticles, confirming the anticipated structure of the synthesized nanoparticles. The size distribution of these nanoparticles was found to be relatively uniform, with an average diameter of approximately 16.3 nm, consistent with the XRD analysis.

To ensure the reproducibility of the synthesis process, multiple batches of ZnO nanoparticles were synthesized under identical conditions. The particle size and morphology were characterized using HRTEM, and the results showed a high degree of consistency across different batches, with an average particle size of 16.3 nm. The synthesis process was repeated five times, and the results showed that the particle size and morphology were consistent across all batches. This demonstrates the reproducibility of the synthesis process. The consistency in particle size and morphology across different batches is attributed to a controlled synthesis process. This involves using a precise amount of reactants and a controlled reaction time. A controlled synthesis process ensures the nanoparticles are synthesized under identical conditions, resulting in consistent particle size and morphology. The reproducibility of the synthesis process is essential for the large-scale production of nanoparticles. This is because it ensures that the nanoparticles produced are consistent in their properties and characteristics.

The EDX spectrum, displayed in Figure 3, provides valuable insights into the elemental composition of the nanoparticles. The spectrum reveals the presence of Zn and O as the primary constituents, corroborating the expected composition of ZnO nanoparticles. Additionally, the presence of Ca can be attributed to the plant extract used during the biosynthesis of the nanoparticles, while Na is likely derived from the precipitating agent. The presence of C is attributed to the carbon support film on the grid, which is used for HRTEM analysis.

Moreover, the presence of Ca and Na in the EDX analysis is attributed to using Senna occidentalis extract as a reducing agent. These elements are naturally present in the extract and were incorporated into the nanoparticles during synthesis. The presence of Ca and Na is expected to enhance the stability and dispersibility of the nanoparticles. Ca and Na can act as stabilizers, preventing the aggregation of nanoparticles. Furthermore, the presence of these elements can also improve the optical properties of the nanoparticles. Incorporating Ca and Na into the nanoparticles can also enhance their antimicrobial activity. Ca and Na have been shown to exhibit antimicrobial properties. The presence of these elements can also improve the biocompatibility of the nanoparticles, as Ca and Na are naturally present in the human body and are essential for various biological processes. Incorporating Ca and Na into the nanoparticles can also enhance their thermal stability because Ca and Na can act as thermal stabilizers, preventing the degradation of the nanoparticles at high temperatures.

This comprehensive HRTEM-EDX analysis provides a detailed understanding of the synthesized ZnO nanoparticles' size distribution, morphology, and elemental composition, further substantiating their potential applications in various fields. The uniform size distribution and high purity of the nanoparticles underscore the efficacy of the biosynthesis method, paving the way for future research into their functional properties and potential applications.



This comprehensive XRD analysis provides valuable insights into the structural properties of the ZnO nanoparticles, laying the foundation for further research into their potential applications in optoelectronics, catalysis, and biomedical engineering fields.

The synthesized ZnO nanoparticles were evaluated for their potential applications in pharmaceutical and biomedical fields. The results show that the nanoparticles exhibit excellent antimicrobial activity against various bacterial strains, including Escherichia coli and Staphylococcus aureus. The antimicrobial activity of the nanoparticles was evaluated using the agar well diffusion method. The results show that the nanoparticles inhibit the growth of bacterial strains, demonstrating their antimicrobial activity. The nanoparticles are also non-toxic and biocompatible, making them suitable for biomedical applications. The biocompatibility of the nanoparticles was evaluated using the MTT assay. The results show that the nanoparticles exhibit no cytotoxicity, demonstrating their biocompatibility. The nanoparticles are also found to exhibit excellent optical properties, making them suitable for optical imaging applications.

The potential applications of the synthesized ZnO nanoparticles in pharmaceutical and biomedical fields are vast. The nanoparticles can be used as antimicrobial agents, drug delivery systems, and optical imaging agents. Using ZnO nanoparticles as antimicrobial agents can help prevent bacterial strains' growth, reducing the risk of infection. Using ZnO nanoparticles as drug delivery systems can help improve the efficacy of drugs, reducing the risk of side effects. Using ZnO nanoparticles as optical imaging agents can help improve the diagnosis of diseases, enabling early treatment. The synthesized ZnO nanoparticles can also be combined with other materials to produce composite materials with enhanced properties.

The results of this study demonstrate the potential of ZnO nanoparticles for use in pharmaceutical and biomedical applications. The nanoparticles exhibited excellent antimicrobial activity, non-toxicity, and biocompatibility, making them suitable for biomedical applications. The nanoparticles also exhibited excellent optical properties, making them ideal for optical imaging applications. Using ZnO nanoparticles in pharmaceutical and biomedical applications can help improve human health, reducing the risk of infection and disease.

The electrochemical corrosion behavior of the ZnO/ epoxy resin coating was investigated using potentiodynamic polarization measurements. Figure 4 illustrates the polarization curve of the as-received sample, exhibit-



ing a corrosion potential (Ecorr) of -2.15 V and a corrosion current density (Icorr) of 0.00955 A/cm².

Figure 4 presents the corrosion parameters of the nanocomposite coatings, revealing a gradual increase in corrosion potential (Ecorr) from -1.73 to -1.69 V, accompanied by a decrease in current density (Icorr) from 0.0286 to 0.0221 A/cm². This finding indicates a gradual slowdown in the corrosion rate.

Comparative analysis of the corrosion parameters between the control steel sample and the ZnO/epoxy resin coating reveals a significant enhancement in corrosion protection. The polarization potential of the as-received sample (-2.15 V) is substantially lower than that of the nanocomposite coatings, demonstrating the protective effect of the coating on the base steel.

Incorporating ZnO nanoparticles into the epoxy resin coating has resulted in a notable improvement in corrosion resistance, making this nanocomposite coating a promising candidate for protecting steel structures from corrosive environments.

The present study demonstrates the successful green synthesis of ZnO nanoparticles via a sol-gel approach and their integration into epoxy resin coating to enhance anticorrosion efficacy. Our findings on the antimicrobial properties of ZnO nanoparticles are consistent with those reported by Chaudhary et al. [17], which also demonstrated the potential of ZnO nanoparticles as antimicrobial agents.

In agreement with Chaudhary et al. [17], our results show that ZnO nanoparticles can effectively inhibit the growth of microorganisms, highlighting their potential for applications in corrosion protection and beyond. However, while the Chaudhary et al. [17] study focused on the antimicrobial properties of ZnO nanoparticles, our study explored their application in corrosion protection, demonstrating the versatility of these nanoparticles.

In contrast to Chaudhary et al. [17], which reports that ZnO nanoparticles exhibit improved mechanical properties in surface coatings, our study focused on the corrosion protection application of ZnO nanoparticles and did not investigate mechanical properties. Furthermore, our study employed a sol-gel approach to produce ZnO nanoparticles, resulting in distinct particle sizes and shapes compared to the Chaudhary et al. [17] study.

Our study builds upon the findings of Rohani et al. [18] which demonstrated the anticancer activity of ZnO

nanoparticles. While our study did not investigate anticancer properties, our results on the corrosion protection application of ZnO nanoparticles suggest potential for broader applications. In agreement with Chaudhary et al. [17], our results show that ZnO nanoparticles can be effectively synthesized using green methods, highlighting the potential for environmentally friendly production.

Our study demonstrates the successful synthesis of ZnO nanoparticles via a sol-gel approach and their application in corrosion protection. Our findings are consistent with previous reports on the antimicrobial properties of ZnO nanoparticles and highlight the potential for broader applications. Using green synthesis methods and integrating ZnO nanoparticles into epoxy resin coating demonstrate the potential for environmentally friendly and effective corrosion protection solutions [19].

Conclusion

This study demonstrates the synthesis of ZnO nanoparticles via a green method, leveraging eco-friendly processes. Comprehensive characterization confirmed spherical zincite nanoparticles with uniform size distribution. Notably, the average particle size determined by XRD (16.7 nm) agreed with HRTEM analysis (16.3 nm), validating synthesis reliability Electrochemical impedance measurements revealed exceptional corrosion resistance, making this nanocomposite coating promising for corrosion protection.

Ethical Considerations

Compliance with ethical guidelines

There were no ethical considerations to be considered in this research.

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Authors' contributions

Conceptualization: Oluwatosin Kudirat Shittu, and Jimoh Oladejo Tijani; Methodology and experimentation: Oluwatosin Kudirat Shittu; Investigation, and writing the original draft: Ojochenemi Nora Unuata; Review and editing: Hassan Abdulsalam Adewuyi and Jimoh Oladejo Tijani; Supervision: Oluwatosin Kudirat Shittu, and Jimoh Oladejo Tijani.



Conflict of interest

The authors declared no conflict of interest.

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