

SYNTHESIS AND CHARACTERIZATION OF CuO/NiO/ZnO NANOCOMPOSITES

Abstract

Over the past ten years nano science and technology have become the cutting edge of science and technology. The morphology, composition, size, crystallinity, shape of a nanoparticle plays a significant role in determining its intrinsic properties. To achieve a uniform metal response, samples were synthesized using the chemical sol-gel method. Scanning Electron Microscopy, Fourier Transform Infrared Spectroscopy was used to characterize the composite samples. The presence of characteristic peaks of the nanocomposites revealed the successful preparation of CuO/NiO/ZnO nanocomposites.

Keywords: Metal Oxides, Nanocomposites, CuO, NiO, ZnO, Sol-gel Synthesis

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I. INTRODUCTION

Nanotechnology has a long history and has led to the production of materials at the nanoscale (dimensions $< 100\text{nm}$). The field involves interdisciplinary contributions from biology, physics, chemistry, and material sciences. One key application is the development of therapeutic nanosized materials for biomedical and pharmaceutical applications. Metal oxide nanoparticles are a focal point in nanomaterials research due to their diverse applications in areas like electronics, optics, energy storage, and catalysis. The synthesis of these nanoparticles has seen significant advancements, enabling the tailoring of their properties for specific applications. The synthesis of nonmaterial's can be categorized into two approaches: top-down and bottom-up. Wet chemical synthesis methods have been used to achieve reproducible control over the size and shape of metal oxide nanoparticles. The factors influencing size and shape include precursor conversion, surface stabilizing agents, and reaction mechanisms. Metal oxide nanoparticles like silver oxide, copper oxide, zinc oxide, and nickel oxide have shown antimicrobial properties. This effect is attributed to their small size and high surface-to-volume ratio, which enable close interaction with bacterial membranes. The release of metal ions is also considered a contributing factor to their bactericidal effectiveness. The goal is to create nanoparticles with antimicrobial activity against bacterial strains at sub-micromolar concentrations. The specific metal oxides of interest are NiO, CuO, and ZnO [1].

CuO is a P-type semiconductor with a band gap ranging from 1.21 to 1.51 eV. It is considered a favorable oxide due to its versatile properties, covering various aspects of chemistry and physics [2]. CuO semiconductors exhibit strong optical absorption, are non-toxic, and can be manufactured at an affordable cost. Advances in nanoscience and nanotechnology have opened avenues for investigating the antimicrobial properties of innovative nanomaterials containing metals like Cu, Ni and Zn [3]. Nanomaterials with metal content have demonstrated extended and enhanced bioactivities compared to bulk metals. This enhanced bioactivity is attributed to factors like surface stabilizers, the size-dependent properties of nanometals, and their high surface-to-volume ratio. Capping chemicals play a significant role in modifying nanoparticle behavior, including ionic release and anti-biofilm properties. These modifications are relevant for achieving efficient and eco-friendly antimicrobial materials.

NiO has semitransparent and stable characteristics, a high direct band gap, and p-type semiconducting activity with weak absorption bands. Nickel oxide (NiO) is a semiconductor material with intriguing physical and chemical properties that make it an attractive candidate for antimicrobial research. NiO nanoparticles exhibit several features that contribute to their antibacterial activity: At the nanoscale, materials can exhibit size-dependent properties that differ from their bulk counterparts. NiO nanoparticles' enhanced surface area-to-volume ratio can lead to increased interactions with bacterial cells, potentially enhancing their antibacterial efficacy [4]. NiO nanoparticles' surface chemistry can be tailored, allowing for the attachment of functional groups that enhance their interaction with bacterial membranes. This modification can lead to the disruption of bacterial cell membranes and subsequent cell death. NiO nanoparticles have been reported to generate reactive oxygen species upon exposure to light or in the presence of certain chemical environments. ROS can cause oxidative stress within bacterial cells, leading to cellular damage and death. NiO nanoparticles can release metal ions, such as nickel ions, which have been shown to exhibit toxic effects on bacterial

cells. The controlled release of these ions can contribute to the antibacterial activity of NiO nanoparticles. NiO nanoparticles can potentially be used in combination with existing antibiotics, creating synergistic effects that enhance the overall antibacterial efficacy. This approach could mitigate the development of antibiotic resistance.

Zinc oxide nanoparticles, as a wide-bandgap semiconductor, possess distinctive physical, chemical, and biological characteristics that render them highly effective as antibacterial agents. Several factors contribute to their potent antibacterial activity: At the nanoscale, materials exhibit size-dependent properties that deviate from their bulk counterparts. ZnO nanoparticles' increased surface area-to-volume ratio facilitates greater contact with bacterial cells, enabling enhanced antibacterial interactions. ZnO nanoparticles display remarkable photo catalytic properties, generating reactive oxygen species (ROS) under UV irradiation. ROS, such as hydroxyl radicals, have potent antibacterial effects by causing oxidative damage to bacterial cell membranes and biomolecules. ZnO nanoparticles can adhere to bacterial surfaces and exert mechanical stress, leading to structural disruption and eventual cell death. This mode of action is particularly effective against biofilm-forming bacteria. ZnO nanoparticles release zinc ions, which can infiltrate bacterial cells and disrupt essential cellular processes. Zinc ions have been shown to interfere with bacterial metabolism and DNA replication, further enhancing their antibacterial potential. ZnO nanoparticles can synergistically enhance the antibacterial efficacy of existing antibiotics. This combination approach addresses the rising concern of antibiotic resistance and boosts the overall therapeutic outcome [5].

Nanocomposite materials, consisting of nanoscale constituents combined to create new functionalities, hold immense importance across a wide range of scientific, technological, and industrial applications. Their significance arises from their ability to combine the unique properties of different components at the nanoscale, resulting in enhanced performance, improved properties, and novel functionalities that traditional materials cannot achieve. Here are some key reasons highlighting the importance of nanocomposites: Nanocomposite materials allow the integration of disparate materials with complementary properties, resulting in improved mechanical, thermal, electrical, optical, and magnetic characteristics. This enhancement can lead to materials with unprecedented strength, conductivity, and other desirable properties. By manipulating the composition, size, and arrangement of nanocomponents, it's possible to create materials with custom-designed functionalities [6].

This tailoring enables the development of materials for specific applications, ranging from lightweight yet strong structural materials to advanced electronic devices. Nanocomposites often exhibit multiple functionalities simultaneously. For instance, a nanocomposite material might possess both electrical conductivity and high thermal stability, making it suitable for diverse applications in electronics and energy storage. Nanocomposite materials can outperform conventional materials in terms of efficiency, durability, and overall performance. This is particularly crucial in sectors such as aerospace, automotive, and energy, where materials must withstand harsh conditions while maintaining optimal performance. Nanocomposite materials can achieve the same level of performance as conventional materials using significantly fewer quantities. This reduction in material usage can lead to cost savings, resource conservation, and more sustainable practices. Nanocomposite materials can contribute to energy-efficient technologies by enabling the

development of lightweight materials with high strength and durability. This, in turn, leads to reduced energy consumption in transportation and other industries. The nanoscale components in these composites enable the creation of miniature devices and structures, driving advancements in fields like microelectronics, medical devices, and sensors. Nanocomposites are finding growing use in biomedicine, enabling targeted drug delivery, tissue engineering, and medical diagnostics. Their unique properties allow for precise control over interactions with biological systems.

Nanocomposites can be tailored for environmental applications such as water purification, air filtration, and pollution control. Their enhanced surface area and reactivity enable efficient removal of contaminants. The ability to combine different materials at the nanoscale encourages innovation and the exploration of uncharted territories in material science, leading to breakthroughs that can shape the future of technology. Many nanocomposite materials can be processed using scalable techniques like additive manufacturing and solution-based processes, enabling flexible and cost-effective production. The development of nanocomposites requires expertise from various disciplines, fostering collaborations between materials scientists, chemists, physicists, engineers, and more, which can lead to new discoveries and insights. In conclusion, nanocomposites play a pivotal role in advancing various fields by providing a platform to harness unique properties and functionalities. Their versatility and potential for innovation make them essential for addressing complex challenges and driving progress across diverse industries.

II. EXPERIMENTAL PROCEDURE

Nanotechnology has evolved into a cutting-edge technology with a broad spectrum of applications, notably within the pharmaceutical and various other industries. Over time, an array of nanoscale devices has emerged through various methodologies, marking the advent of nanotechnology. In recent times, there has been a growing emphasis on environmentally friendly 'green' techniques for producing nanomaterials. This shift is propelled by the pressing need to address the environmental challenges posed by conventional chemical and physical methods. The plant-mediated synthesis approach has emerged as a valuable strategy, offering both ease of manufacturing and engineering. Traditional methods for nanoparticle synthesis often suffer from drawbacks such as prolonged production periods, high costs, intricate processes, and the utilization of hazardous compounds. Consequently, the focus of much research has shifted towards the development of rapid and environmentally sustainable protocols for nanoparticle fabrication. In the realm of material science, there is a growing emphasis on eco-friendly techniques for generating nanomaterials. In this context, the utilization of various plant extracts for synthesizing nanoparticles has gained momentum within the framework of green chemistry. This approach is characterized by its simplicity, cost-effectiveness, and non-toxic nature [7].

Nanotechnology, in turn, has substantially improved human quality of life by addressing an array of challenges spanning industrial growth, climate change, food production, clothing, healthcare, and the treatment of severe ailments like cancer, respiratory infections, and Alzheimer's disease. The far-reaching impact of nanoscale technology on human well-being underscores its potential to revolutionize various facets of existence, forging a path towards sustainability and innovation.

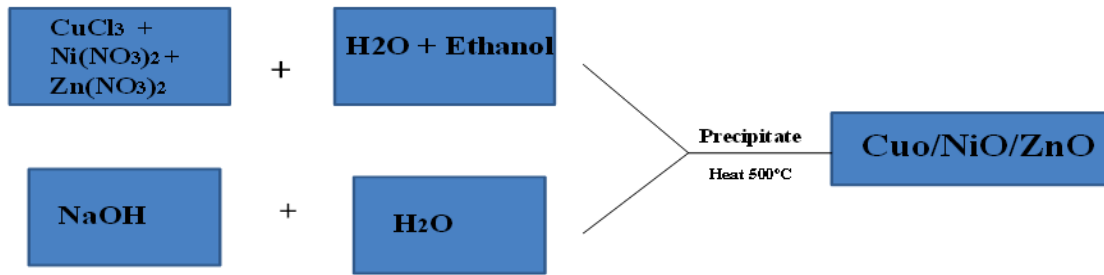


Figure 1: Synthesis Procedure of CuO/NiO/ZnO Nano Composites

Figure 1 depicts a summary of the synthesis process. Sigma-Aldrich analytical-grade chemicals were used straight out of the package to create the nanoparticles. The salts employed were copper (II) chloride dihydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) as the Cu salt, zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) as the Zn salt, and nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) as the Ni salt. Weighing 0.426 g of the Cu salt, 0.727 g of the Ni salt, and 0.744 g of the Zn salt, they were combined and dissolved in a solvent made up of water and ethanol at a volume ratio of 4:1 to create a 0.01 M precursor solution with a Cu:Ni:Zn mole ratio of 1:1:1. At 50 °C, the solution was magnetically swirled for 30 minutes. Separately, 2 g of NaOH pellets were dissolved in 5 minutes of double-distilled water by stirring, yielding a 50-ml aqueous solution of NaOH. The mixed precursor solution was then added to the transparent NaOH solution while being constantly stirred. The mixture's colour shifted from turquoise to a deep shade of green, and precipitate was seen to form right away. After five minutes, the temperature was increased and kept at 100 °C for another 30 minutes while the mixture was continuously stirred. The liquid was thereafter given time to cool to room temperature. The precipitate was obtained through filtering, rinsed three times with distilled water, and dried for two hours on a hot plate at 100 °C. A fine black powder was produced by annealing the powder for two hours at 500 degrees Celsius.

III. RESULTS AND DISCUSSION

The SEM image of CuO/NiO/ZnO nanocomposites is shown in Figure 2. The Image shows nanoparticles of spherical shapes. The size of the nanoparticles also varies. EDX mapping confirms the composition of CuO/NiO/ZnO nanocomposites and shown in Figure 2.

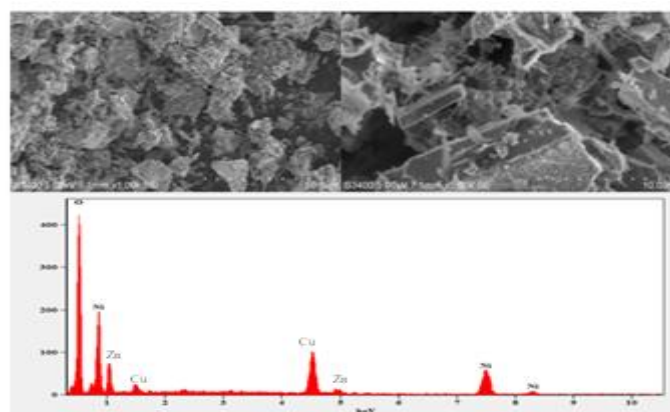


Figure 2: SEM and EDX Images of CuO/NiO/ZnO Nanocomposites

FTIR absorption peaks for vibrations of metal-oxygen bonds often occur below 1000 cm^{-1} . CuO contains six infrared vibrational bands, which are located at 147, 161, 321, 478, 530, and 590 cm^{-1} [8]. In accordance with the morphology of the nanoparticles, the Zn-O bond vibrational bands are located at approximately 395, 425, 470, and 515 cm^{-1} [9,10].

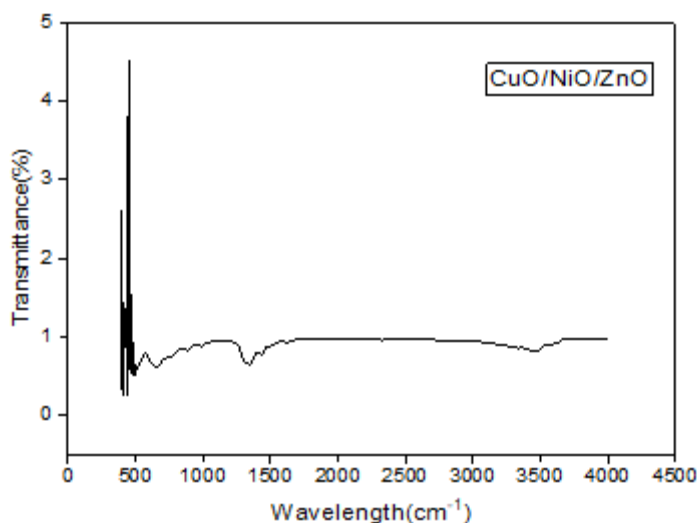


Figure 3: FTIR Studies of CuO/NiO/ZnO Nanocomposites

On the other hand, NiO exhibits IR absorption peaks at 454 cm^{-1} and 571 cm^{-1} that are caused by Ni-O vibration [11]. Figure 8 displays the FTIR spectrum of the ZnO-NiO-ZnO nanocomposite. It is evident that the CuO, NiO, and ZnO vibration peaks in the region below 600 cm^{-1} are overlapping. It is challenging to distinguish the distinct peaks for each oxide phase in the nanocomposite because their absorption peaks are near to one another.

IV. CONCLUSION

A new composition of CuO/NiO/ZnO was synthesized by sol-gel method and annealed at 500^o C. EDX data showed that the distribution of Cu, Ni and Zn was not uniform across the selected area of the sample, which suggests that the oxides were not uniformly intermixed in the nanocomposite. These results introduce a new multiphase mixed oxide nanocomposite that may have a variety of possible application in sensor, photonic and optoelectronic and Antibacterial Properties.

REFERENCES

- [1] Kim, S., Jeong, J.-E., Hong, J., Lee, K., Lee, M.J., Woo, H.Y. and Hwang, I. (2020) Improved Interfacial Crystallization by Synergic Effects of Precursor Solution Stoichiometry and Conjugated Polyelectrolyte Interlayer for High Open-Circuit Voltage of Perovskite Photovoltaic Diodes. *ACS Applied Materials & Interfaces*, 12, 12328- 12336. <https://doi.org/10.1021/acsami.9b22283>
- [2] Akhimoto K, Ishizuka S, Yanagita M, Nawa Y, Paul GK, Sakurai (2008) *T. Sol. Energy* 0:715.
- [3] C. C. Trapalis, M. KokkOris, G. Perdikakis, G. Kordas, Study of antibacterial composite Cu/SiO₂ thin coatings, *J. Sol-Gel Sci Techn.* 26 (2003) 1213–1218, <https://doi.org/10.1023/A:1020720504942>.
- [4] G. Faundez, M. Troncoso, P. Navarrete, G. Figueroa, Antimicrobial activity of copper surfaces against suspensions of salmonella enteric and campylobacter, *BMC Microbial.*(2004) 19–25, <https://doi.org/10.1186/1471-2180-4-19>.

- [5] Stoyanova, A., et al., *Synthesis and antibacterial activity of TiO₂/ZnO nanocomposites prepared via nonhydrolytic route*. J. Chem. Technol. Metall, 2013. 48(2): p. 154-161.
- [6] Franke ME, Koplin TJ Simon U. Metal and metal oxide nanoparticles in chemiresistors: does the nanoscale matter? Small, 2006; 2(1):36-50.
- [7] Morones JR, Elechiguerra JL, Camacho A, Holt K, Kouri JB and Ramirez JT. The bactericidal effect of silver nanoparticles. Nanotech 2005;16:2346–53.
- [8] K. Borgohain, J. Singh, M. Rama Rao, T. Shripathi, S. Mahamuni, Quantum size effects in CuO nanoparticles, Phys. Rev. B 61 (2000) 11093–11096, [http:// dx.doi.org/10.1103/PhysRevB.61.11093](http://dx.doi.org/10.1103/PhysRevB.61.11093).
- [9] T. Ivanova, A. Harizanova, T. Koutzarova, B. Vertruyen, Study of ZnO sol-gel films: effect of annealing, Mater. Lett. 64 (2010) 1147–1149, [http://dx.doi.org/ 10.1016/j.matlet.2010.02.033](http://dx.doi.org/10.1016/j.matlet.2010.02.033).
- [10] Z. Yang, Z. Ye, Z. Xu, B. Zhao, Effect of the morphology on the optical properties of ZnO nanostructures, Phys. E Low-Dimensional Syst. Nanostruct. 42 (2009) 116–119, <http://dx.doi.org/10.1016/j.physe.2009.09.010>.
- [11] K. Anandan, V. Rajendran, Morphological and size effects of NiO nanoparticles via solvothermal process and their optical properties, Mater. Sci. Semicond. Process 14 (2011) 43–47, <http://dx.doi.org/10.1016/j.mssp.2011.01.001>.