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Original paper

Synthesis of Montmorillonite-Copper Oxide Hybrid Nanocomposite

Narges Sadr¹*, Masoud Fardin², Pardis Masaeli³, Elahe Aliheydari⁴, Erfaneh Dalghi⁵

¹ Department of Biotechnology, Faculty of Basic Sciences, Urmia Branch, Islamic Azad University, Urmia, Iran

² Department of Microbiology, Ardabil Branch, Islamic Azad University, Ardabil, Iran.

³ Department of Biology, Faculty of Basic Sciences, Isfahan University, Isfahan, Iran

⁴ Department of Biology, Faculty of Basic Sciences, Shahr-Rey Branch, Payame Noor University, Tehran, Iran

⁵ Department of Biology, Faculty of Life Science and Biotechnology, Shahid Beheshti University, Tehran, Iran

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Abstract

Background and aim: The synthesis of nanoparticles, especially nanocomposite is of particular importance due to their application in the fields of medicine and industry. The most crucial reason for the synthesis of nanoparticles, particularly nanocomposites, to be paramount in the fields of medicine and industry lies in their unique properties at the nanoscale. Nanoparticles often exhibit enhanced reactivity, increased surface area, and distinct physicochemical properties compared to their bulk counterparts. These characteristics open avenues for novel applications, such as targeted drug delivery in medicine and improved catalysis in industry. Building upon this foundation, the current investigation focuses on the synthesis of a copper oxide hybrid nanocomposite.

Materials and methods: During this experimental-laboratory research, the chemical precipitation method of the composite structure was used for the synthesis of copper oxide-clay hybrid nanocomposite and it was confirmed by using FTIR and XRD. In addition, the morphology of nanoparticles was determined by SEM/TEM measurements.

Results: The results of the present study show that the processed nanocomposites were pure and had dimensions of 250-200 nm.

Conclusion: The findings of this study demonstrate that the copper oxide and clay nanocomposite, as prepared, possesses the characteristics of an advantageous nanoparticle, including desirable dimensions. This nanoparticle holds potential applications in both medical and industrial contexts. **Keywords:** *Synthesis, Hybrid nanocomposite, Copper-clay oxide*

*Corresponding author: Narges Sadr, Department of Biotechnology, Faculty of Basic Sciences, Urmia Branch, Islamic Azad University, Urmia, Iran. E-mail address: narges.sadr@yahoo.com

Introduction

Composites, encompassing polymer-based, ceramic-based, and metal-based variants, incorporating nanoparticles as the dispersed phase are referred to as nanocomposites [1]. These materials, utilizing reinforcements such as clay and metals like copper, exhibit enhanced strength and reduced weight in the case of polymer nanocomposites [2]. Copper, recognized for its biocidal properties, has been historically instrumental in various applications, including water purification, algaecide, fungicide, and as an antibacterial agent [3].

The effectiveness of copper surfaces in reducing bacterial viability, surpassing materials like stainless steel [4], extends to inhibiting bacterial pathogens in food processing stages [5] and suppressing microbial growth in water distribution systems [6], [3]. Polymer clay nanocomposites (PCN), featuring classes like montmorillonite, vermiculite, and bentonite as reinforcements, offer unique properties such as thermal stability, flame retardancy, and anti-corrosion features [7]. PCNs find significance in environmental applications, including anti-pollution initiatives and water system enhancements, particularly in soil and air purification [8], [9].

Amid the global challenges posed by bacterial contamination and the rising prevalence of antibiotic-resistant strains, the synthesis of copper oxide-clay hybrid nanocomposites gains importance [10], [11]. Unlike traditional studies focusing solely on standalone nanoparticles, this research explores the synergistic potential of nanocomposites, specifically combining copper oxide and clay in a hybrid form. The resulting nanocomposites exhibit multifaceted advantages, including enhanced antibacterial efficacy, stability, and versatile applications across various fields. Beyond their antimicrobial properties, copper oxide-clay nanocomposites demonstrate versatility in environmental applications. The synthesis of these nanocomposites holds promise for addressing both bacterial infections and environmental challenges, contributing to global initiatives combating pollution and improving environmental quality. This dual functionality underscores the significance of the current research in addressing complex societal issues.

As bacterial infections continue to rise, and conventional antibiotic treatments face challenges, the synthesis of copper oxide-clay hybrid nanocomposites emerges as a cutting-edge approach. The outcomes of this research not only expand our understanding of nanocomposites' potential but also offer practical solutions to the complex interplay between bacterial contamination, antibiotic resistance, and environmental concerns. In the broader context of medical and environmental sciences, the results have the potential to pave the way for novel applications and interventions, ultimately benefiting public health and ecological well-being.

Material and Methods

- Instruments

To explore the vibrational characteristics of chemical bonds, the Fourier-Transform Infrared Spectroscopy (FT-IR) method was employed. This method detects variations in bond length or angle within molecules. The measurements were conducted using a Brucker Tehsor 27 spectrometer, manufactured in Germany, at the research laboratory of Mohaghegh University of Ardabil [12]. The X-ray diffraction (XRD) pattern of the samples was analyzed by bombarding finely powdered samples with X-ray beams, with a wavelength ranging from 0.1 to 100 angstroms. The diffraction patterns were identified and recorded at the Razi Metallurgy Research Center laboratory, comparing them with standard diffraction patterns to determine the crystal type of each compound [13]. The morphology of nanoparticles was investigated using Field Emission Scanning Electron Microscopy (FESEM) with the MIRA3TESCAN-XMU model at the Razi Metallurgy Research Center [14]. Additionally, Transmission Electron Microscope (TEM) micrographs were

prepared and registered at the laboratory service center of Sharif University of Technology using the Zeiss EM900 transmission electron microscope [15].

In the synthesis of the copper oxide-clay hybrid nanocomposite, the chemical precipitation method was employed. The experiments underwent multiple optimizations within a temperature range of 60-90 degrees and at pH levels ranging from 9 to 11, using various amounts of raw materials.

- Synthesis of Copper Oxide-Clay Hybrid Nanocomposite

For the synthesis, 143.5 cc of double-distilled water was poured into a container and placed on a stirrer heater. Subsequently, 2 cc of 2% glucose solution, 0.5 cc of 1% soda solution, 1 gram of cotton goods, and 1 gram of clay were added, with the temperature and pH controlled to maintain the desired conditions, typically set at pH 9. Then, 4 cc of 2% copper sulfate was introduced. The synthesis duration for this nanocomposite was 1 hour. Following the synthesis, the deposits were collected and dried in an oven at 90°C for 30 minutes. Post-synthesis, the structure, morphology, and size of nanoparticles were analyzed and investigated using spectroscopy (FT-IR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) [16].

Results

- Interpretation of FTIR Spectra of Silver and Clay Nanocomposite

The FTIR spectrum analysis of the nanoclay revealed distinctive peaks corresponding to various vibrational modes and chemical groups. Notably, the peak observed at 1050 is attributed to Si-o-Si vibrations, signifying a fundamental characteristic of nanoclay. Additionally, the peak at 465 corresponds to the bending vibrations of the Si-o-Si group, while the one at 526 is linked to Si-o-Al vibrations. The presence of Mo (oxygen bound to the metal) manifests in the peak at 626, and the peak at 726 is indicative of Al-MgOH nanoclay vibrations.

Further scrutiny reveals a peak in the 918 region, signaling Al-AlOH vibrations. Moreover, the peaks observed at 3632-1637-800 correspond, respectively, to the bending, out-of-plane, and stretching OH groups of the hydroxyl groups on the nanoclay surface (Figure 1). These distinctive peaks offer insights into the structural composition and functional groups present in the nanoclay. The presence of copper oxide was unequivocally confirmed through complementary analyses conducted in this research, including X-ray Diffraction (XRD) and Field Emission Scanning Electron Microscopy (FESEM). The synthesis of the copper oxide-clay hybrid nanocomposite was validated through a comprehensive examination of its structural and chemical characteristics, providing a foundation for further understanding and potential applications in various fields.



Figure 1. Spectra of synthetic nanoparticles

- Interpretation of the XRD Pattern

The X-ray diffraction pattern analysis unequivocally identified the distinctive pattern (Figure 2) as the characteristic X-ray diffraction pattern associated with the copper oxide and clay nanocomposite. This recognition further validates the successful synthesis of the nanocomposite, providing crucial insights into its crystalline structure.



Figure 2. The spectra derived from the synthetic nanoparticles reveal distinct features. Peaks marked with the symbol "&" are associated with copper oxide, and the corresponding three-digit codes are indicated on each respective peak.

Furthermore, the diffraction peaks at angles of 37° , 5° , and 42° (as illustrated in Figure 3) were found to align with the crystal planes of copper oxide. Utilizing Scherer's relation, the distances between these crystal planes and the size of the crystals associated with this structure can be derived from the respective diffraction angles.



Figure 3. XRD spectrum of copper oxide and clay nanocomposite

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- Investigation of FESEM Images of Copper Oxide and Clay Nanocomposite

The investigation into the morphology of nanoparticles, through field emission electron microscope images of the copper oxide and clay nanocomposite, revealed the polyhedral shape of copper oxide particles intricately positioned between the layers of nanoclay. The copper oxide nanoparticles exhibited a size range of 200-250 nm (Figure 4).



Figure 4. Field Emission Scanning Electron Microscope (FESEM) images of the "copper oxide and clay nanocomposite" at varying scales: 2 microns, 1 micron, 500 nm, and 200 nm.

- TEM Images

The findings from TEM provide a means to explore diverse aspects, including the microstructural characteristics of materials, plate formations, and crystal orientations. In this study, thin and transparent sections were meticulously prepared from the sample, as clearly depicted in the captured images. These images distinctly reveal the presence of nanoclay layers across all observations, with nano copper oxide consistently stabilized in a polyhedral crystal state between these layers (Figure 5).



Figure 5. TEM transmission electron microscope images of "silver, copper oxide and clay hybrid nanocomposite" at the scale of 100nm, 50 nm and 25 nm

Discussion

Numerous studies highlight the pivotal role of nanocomposite synthesis and their diverse properties, encompassing antibacterial, reinforcing, anti-fouling, and lightweight attributes, particularly within the health sector [3], [5], [17]. This research contributes to this field, shedding light on the widespread use of various synthesis methods for nanocomposites, broadly categorized into polymer-based, ceramic-based, and metal-based nanocomposites [18].

While acknowledging the antibacterial efficacy of nanocomposites, especially copper-clay oxide, this study delves into the broader spectrum of their applications in both the medical and industrial realms. The synthesis methods of specific nanocomposites, such as CuO-montmorillonite, are explored, emphasizing their characteristics, particle size, and potential applications [19].

In a significant advancement, new nanosorbents (Cr2O3@PA6, CrO@PA6, and CuO@PA6) as modified polyamide nanocomposites are successfully synthesized for efficiently removing U(VI) from aqueous solutions, showcasing their potential in industrial applications [20]. The study recognizes clay nanoparticles as stabilizing agents, demonstrating their effectiveness in preventing silver accumulation and positioning them for applications across various industries [21].

Exploring the characteristics of copper oxide nanoparticles (CuO), the study underscores their versatility on the nano scale, containing traces of pure Cu and Cu2O nanoparticles [22]. The unique features of clay polymers, such as anti-corrosion and high anti-pollution properties [8], [9], are emphasized, positioning nanocomposites as sought-after materials in engineering plastics and elastomers, with applications ranging from packaging to biomedical fields in the industrial sector [23].

As the synthesis of hybrid nanocomposites gains attention, this research accentuates their compatibility with environmental concerns, offering new opportunities in biotechnology, automotive, aerospace, and electronics industries [23]. The study delves into the definition of hybrid nanocomposites, where inorganic nanoparticles are dispersed in a macroscopic organic matrix, with a particular focus on systems like clay-polymer nanocomposites, positioning them as promising substitutes for traditional fiber-reinforced composites in various industrial applications [24].

Recognizing the need for more extensive studies to unravel the mechanisms enhancing the fundamental properties of polymers and understanding nanostructure creation, this study provides insights into the potential applications in the medical and industrial sectors. The research conducted within the scope of investigating the synthesis of lead oxide-hybrid nanocomposites underscores the importance of the nanoparticle processing method in these applications. Acknowledging limitations in the medical nanobiotechnology industry, the study advocates for further exploration in the synthesis of other nanocomposites, evaluation of their industrial and medical applications, and exploration of potential attributes, such as anticancer and antiviral properties. Additionally, the consideration of these materials in pharmaceutical synthesis is recommended for further exploration in the medical sector.

Conclusion

The findings from this research underscore the remarkable characteristics of the copper oxide and clay nanocomposite synthesized, revealing its inherent attributes as an optimal nanoparticle. The dimensions of this nanocomposite, ranging from 200 to 250 nm, align with the desirable criteria for various applications. Notably, the versatility of this nanoparticle extends beyond conventional bounds, making it highly suitable for utilization in medicine, health-related applications, and various industrial sectors. The established dimensions and inherent properties of this nanocomposite position it as a valuable and versatile entity with potential applications across diverse fields, promising advancements in medicine, healthcare, and industry alike.

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Conflict of interests

The authors declare no conflict of interest.

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