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GREEN SYNTHESIS OF COPPER NANOPARTICLES FROM *TERMINALIA ARJUNA* (L.) BARK EXTRACT: CHARACTERIZATION AND POTENTIAL FOR MERCURY DEGRADATION

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ABSTRACT. The focus of this study is on synthesizing copper nanoparticles through a green approach, utilizing *Terminalia arjuna* bark extract. The ultra violet (UV) spectral analysis of copper nanoparticles synthesized through environmentally friendly methods revealed distinct absorption peaks at 287 nm, 575 nm, and 898 nm, indicative of significant light absorption. These peaks elucidate the nanoparticles' optical characteristics, shedding light on electronic transitions and surface plasmon resonance phenomena. Fourier transform infrared spectroscopy (FTIR) analysis displayed various peaks, suggesting vibrations associated with copper nanoparticles and functional groups in *T. arjuna* bark extract. The X-ray diffraction analysis (XRD) data exhibited characteristic peaks corresponding to metallic copper's crystallographic planes, confirming the formation of highly crystalline copper nanoparticles. Atomic force microscopy results depicted surface morphology and particle size distribution. Copper nanoparticles show promise in mercury degradation due to their high surface area and catalytic activity. They interact effectively with mercury ions through adsorption, reduction, and oxidation processes, leading to sequestration or transformation into less toxic forms. Functionalization enhances their affinity towards mercury, while synergies with other nanomaterials boost efficiency. Green synthesized copper nanoparticles offer an eco-friendly solution for effective mercury remediation, promising advancements in sustainable nanotechnological approaches for global environmental sustainability.

KEY WORDS: Mercury, Copper, *Terminalia arjuna*, Remediation

INTRODUCTION

Mercury, a heavy metal element with the symbol Hg and atomic number 80, holds a peculiar place in the periodic table due to its unique properties and profound environmental implications [1, 2]. Known for its silver-like appearance and fluidity at room temperature, mercury has found extensive use in various industrial, medical, and consumer applications throughout history [3]. However, its ubiquity in human activities has led to widespread concern regarding its detrimental effects on both environmental and human health [4].

Unlike many other metals, mercury exists in several forms, each with its distinct chemical properties and toxicity levels. Elemental mercury, often referred to as metallic mercury, is the pure form commonly used in thermometers, barometers, and dental amalgams [4]. In contrast, mercury compounds, such as methylmercury and mercuric chloride, are organic and inorganic compounds that arise from industrial processes and chemical reactions in the environment [5].

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The primary concern surrounding mercury stems from its persistence in the environment and its ability to bioaccumulate and biomagnify within the food chain. Once released into the air, water, or soil, mercury undergoes complex transformations, cycling between various environmental compartments and posing risks to ecosystems and human populations alike. In particular, methylmercury, a highly toxic organic form of mercury, accumulates in aquatic organisms, leading to contaminated seafood and potential health hazards for consumers [6]. The adverse effects of mercury pollution are well-documented, ranging from neurological disorders and developmental abnormalities in humans to reproductive impairments and population declines in wildlife. As such, addressing mercury pollution has emerged as a critical environmental and public health priority, prompting global efforts to regulate mercury emissions, promote cleaner technologies, and raise awareness about its risks [6, 7].

Mercury pollution presents significant environmental problems due to its toxicity, persistence, and ability to bioaccumulate and biomagnify within ecosystems [8, 9]. Here are some key environmental issues associated with mercury: Mercury can enter aquatic environments through various pathways, including industrial discharges, runoff from land surfaces, and atmospheric deposition. Once in water bodies, mercury can undergo transformations into methylmercury, a highly toxic organic form that accumulates in aquatic organisms, posing risks to both aquatic life and organisms higher in the food chain, including humans. Mercury bioaccumulates in organisms at each trophic level of the food chain, meaning that concentrations of mercury increase as organisms consume contaminated prey or plants [6, 10]. Biomagnification occurs when mercury concentrations become progressively higher in predators at the top of the food chain, leading to significant health risks for these organisms, including neurological damage, reproductive impairments, and population declines. Mercury can also contaminate soil and sediments through atmospheric deposition, agricultural runoff, and disposal of mercury-containing products. Contaminated soils can impact plant growth and biodiversity and serve as a source of exposure for terrestrial organisms [11-13]. Mercury is a global pollutant that can travel long distances through atmospheric currents. This global transport leads to mercury deposition in remote regions far from pollution sources, affecting ecosystems and indigenous communities that rely on local resources for food and livelihoods. Mercury pollution can disrupt ecosystem functioning by impairing the health and reproductive success of aquatic and terrestrial organisms [6]. In aquatic ecosystems, mercury toxicity can lead to population declines and altered community dynamics, while in terrestrial ecosystems, it can affect soil microbial activity, nutrient cycling, and plant productivity. Exposure to mercury through contaminated food, water, or air can pose significant health risks to humans. Chronic exposure to methylmercury, primarily through consumption of contaminated fish and seafood, can lead to neurological disorders, developmental delays, cardiovascular problems, and other adverse health effects, particularly in vulnerable populations such as pregnant women and children [14]. Mercury pollution presents regulatory challenges due to its complex chemistry, widespread distribution, and transboundary transport. Efforts to regulate mercury emissions and mitigate its environmental impacts require international cooperation and coordination to address both point and non-point sources of pollution effectively [15]. Addressing the environmental problems associated with mercury pollution requires interdisciplinary approaches that integrate scientific research, policy development, public awareness, and technological innovations. Strategies for mitigating mercury pollution include reducing mercury emissions from industrial processes, promoting cleaner technologies, improving waste management practices, and implementing measures to protect vulnerable ecosystems and human populations from exposure to mercury contamination [16].

In this context, understanding the sources, pathways, and impacts of mercury pollution is essential for devising effective strategies to mitigate its adverse effects and safeguarding both environmental integrity and human well-being. Through concerted international cooperation, regulatory measures, and technological innovations, it is imperative to address the challenges posed by mercury pollution and pave the way towards a sustainable and mercury-free future [6, 17].

In recent years, the field of nanotechnology has witnessed significant advancements in the synthesis of nanoparticles with environmentally friendly methods, leading to the emergence of green synthesis approaches [18]. Among these, green synthesized copper nanoparticles (CuNPs) have garnered considerable attention due to their unique properties and potential applications, particularly in environmental remediation, including heavy metal degradation [19].

Green synthesis methods involve the utilization of natural sources, such as plant extracts, microorganisms, or other biocompatible materials, as reducing and stabilizing agents for nanoparticle synthesis [20-22]. In the case of copper nanoparticles, green synthesis offers several advantages over conventional chemical methods, including reduced environmental impact, cost-effectiveness, and the absence of hazardous chemicals [23]. Plant extracts rich in bioactive compounds, such as polyphenols, flavonoids, and terpenoids, serve as excellent reducing agents for the synthesis of CuNPs. These compounds facilitate the reduction of copper ions (Cu^{2+}) to zero-valent copper (Cu^0), leading to the formation of nanoparticles with controlled size, morphology, and stability [24]. Furthermore, the phytochemicals present in plant extracts impart additional functionalities to CuNPs, enhancing their performance in various applications, including heavy metal degradation.

Heavy metal contamination poses a significant threat to environmental ecosystems and human health due to their persistence and toxic effects. Traditional methods for heavy metal remediation, such as chemical precipitation and ion exchange, often suffer from drawbacks such as high cost, limited efficiency, and generation of secondary pollutants [25]. In this context, green synthesized CuNPs offer a sustainable and effective alternative for heavy metal degradation and removal from contaminated environments. The unique properties of CuNPs, including high surface area, catalytic activity, and tunable surface chemistry, enable them to effectively interact with heavy metal ions and facilitate their conversion into less toxic forms or immobilization onto nanoparticle surfaces [26]. Through processes such as adsorption, reduction, and oxidation, CuNPs can selectively capture heavy metal ions from aqueous solutions, leading to their sequestration or transformation into insoluble precipitates [27]. Moreover, the incorporation of functional groups or surface modifications on CuNPs further enhances their affinity towards specific heavy metal contaminants, enabling tailored approaches for targeted remediation. Additionally, synergistic effects between CuNPs and other nanomaterials, such as carbon-based nanomaterials or metal oxides, can result in enhanced performance and efficiency in heavy metal degradation applications [28]. Green synthesized CuNPs have a high surface area, enabling them to adsorb heavy metal ions through physical or chemical interactions, which are enhanced by functional groups from plant extracts. The reducing agents in the plant extracts facilitate the synthesis of CuNPs and reduce heavy metal ions to less toxic or insoluble forms, aiding in their removal. Biomolecules from plant extracts can form stable complexes with heavy metal ions, effectively binding and removing them from contaminated environments.

The focus of this study is on synthesizing copper nanoparticles through a green approach, utilizing *T. arjuna* bark extract and utilizing the mercury degradation.

EXPERIMENTAL

Plant sample collection and preparation

Collected *T. arjuna* tree bark (Figure 1) from coutrallam hills, Tamil Nadu, India at the geographical ranges of 8° 55' 35.652" and N 77° 16' 2.0172" E. The collected bark was washed thoroughly with distilled water to remove any impurities. Dried the bark in shade to preserve its phytochemical constituents. Ground the dried bark into a fine powder using a mortar and pestle. Prepared an aqueous extract by adding the 5 g powdered bark to 100 mL distilled water and boiled the mixture for a specific duration (30 min). Filtered the extract using filter paper to remove any solid particles, obtaining a clear solution of *T. arjuna* bark extract. This extract contains phytochemicals like flavonoids, phenolics, and tannins, which act as reducing and stabilizing agents in the synthesis of CuNPs.

Figure 1: *Terminalia arjuna* tree.

Synthesis of copper nanoparticles

Prepare a copper salt solution (0.1 M) by dissolving a copper precursor such as copper sulfate ($CuSO_4$) in distilled water. The concentration of the copper salt solution can vary based on the desired nanoparticle size and concentration. Mixed the 90 mL of copper salt solution with the 10 mL of *T. arjuna* bark extract in a 90:10 ratio. The bark extract acts as a reducing agent, converting the copper ions into copper nanoparticles. Stirred the reaction mixture at an optimal temperature 60 °C for a specific duration (2 hours) to facilitate the reduction process. Monitored the color change of the reaction mixture from the initial color of the copper salt solution to reddish-brown, indicating the formation of copper nanoparticles. Once the reaction is complete, allowed the mixture to cool to room temperature and then centrifuged it to separate the synthesized CuNPs from the reaction mixture [29].

Characterization of copper nanoparticles

Characterize the synthesized CuNPs using various analytical techniques such as UV-Vis spectroscopy, which can confirm the formation of nanoparticles based on their characteristic absorption peak in the visible range. Use techniques like scanning electron microscopy (SEM) and AFM to determine the size, morphology, and distribution of the synthesized nanoparticles.

Employ techniques like X-ray diffraction (XRD) to analyze the crystalline structure of the CuNPs and confirm their identity.

Environmental remediation study

The solar catalytic degradation of mercury using copper nanoparticles synthesized from *T. arjuna* tree bark. The catalyst was prepared by mixing 10 mL of the green CuNPs with 100 mL of aqueous 0.1 M mercury(II) chloride solution. Solar light was utilized as the energy source for the degradation process, following the method outlined by Mariselvam *et al.* [30]. To assess the degradation of mercury, UV/visible spectrophotometry was employed, with kinetic measurements conducted at ambient temperature under solar radiation. The concentration of mercury was monitored by measuring the optical density (OD) at various time points within the wavelength range of 200–1000 nm [31].

RESULTS AND DISCUSSION

In the UV spectral analysis conducted for copper nanoparticles synthesized via green methods, notable absorption peaks were identified at wavelengths of 287 nm, 575 nm, and 898 nm (Figure 2).

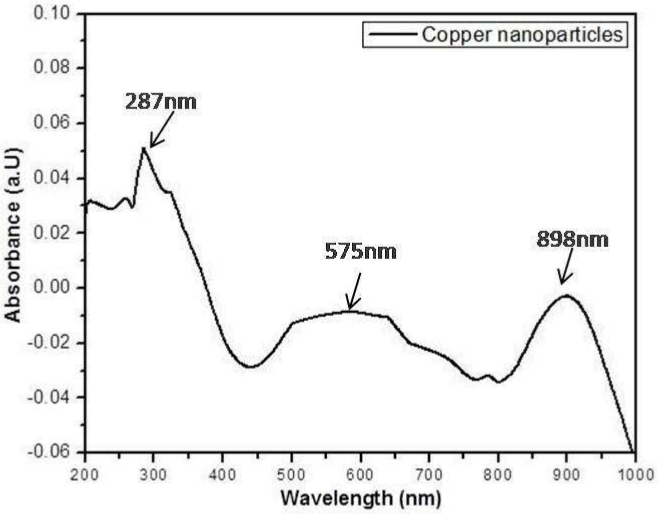

Figure 2. UV spectral analysis of green synthesized copper nanoparticles.

These distinctive bands represent the wavelengths at which the copper nanoparticles exhibit significant absorption of light. Such absorption patterns are indicative of the nanoparticles' inherent optical characteristics, providing insights into their electronic transitions and surface plasmon resonance phenomena. The utilization of environmentally friendly approaches in the synthesis process underscores the sustainable nature of the produced copper nanoparticles, aligning with the principles of green chemistry and nanotechnology [26].

The FTIR (Fourier-transform infrared spectroscopy) result shows various peaks at different wavenumbers (Figure 3).

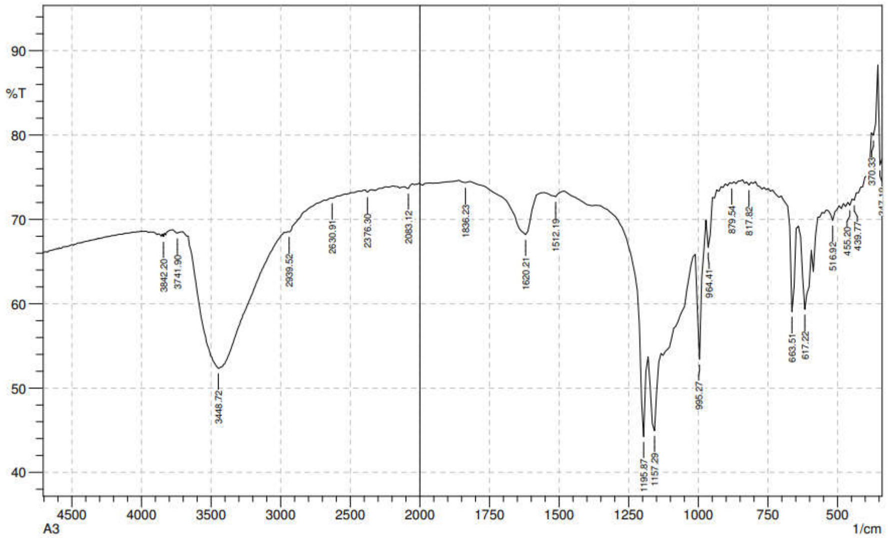

Figure 3. FT-IR spectra of green synthesized copper nanoparticles.

Peaks around 347.19, 370.33, and 439.77 cm^{-1} may correspond to vibrations associated with the copper nanoparticles or functional groups present in the *T. arjuna* bark extract. Peaks at 455.2 and 516.92 cm^{-1} could indicate stretching vibrations of metal-oxygen bonds present in the copper nanoparticles. The peak observed at 617.22 cm^{-1} might be related to bending vibrations of C-H bonds or other organic groups present in the bark extract. Peaks around 663.51, 817.82, and 879.54 cm^{-1} may correspond to specific functional groups or molecular vibrations present in both the copper nanoparticles and the bark extract. Peaks at 964.41 and 995.27 cm^{-1} could be attributed to vibrations associated with aromatic ring structures present in organic compounds within the bark extract. The peak observed at 1157.29 cm^{-1} might correspond to stretching vibrations of C-O bonds present in polysaccharides or other organic compounds. Peaks around 1195.87 and 1512.19 cm^{-1} could be indicative of stretching vibrations of C-C and C=C bonds present in organic molecules. The peak observed at 1620.21 cm^{-1} might correspond to vibrations associated with carbonyl groups (C=O) present in organic compounds within the bark extract. Peaks around 1836.23 cm^{-1} may indicate vibrations associated with aldehyde or ketone functional groups present in the organic constituents of the bark extract. The peak observed at 2083.12 cm^{-1} could be attributed to vibrations associated with triple bonds present in certain organic compounds. Peaks at 2376.3 and 2630.91 cm^{-1} might correspond to vibrations associated with carbon dioxide (CO2) or other atmospheric gases present in the sample. The peak observed at 2939.52 cm^{-1} could indicate stretching vibrations of C-H bonds present in organic molecules. Peaks around 3448.72 and 3741.9 cm^{-1} may correspond to stretching vibrations of O-H bonds present in hydroxyl groups or water molecules. The peak observed at 3842.2 cm^{-1} might indicate vibrations associated with hydrogen bonding or intermolecular interactions present in the sample. Overall, the FTIR spectrum suggests the presence of various organic functional groups and vibrations associated with both the copper nanoparticles and the *T. arjuna* bark extract, providing valuable information about the chemical composition and structure of the synthesized material.

The XRD (X-ray diffraction) data shows (Figure 4) peaks at various 2θ angles along with their corresponding intensities. The XRD (X-ray diffraction) data for copper nanoparticles (CuNPs) typically exhibits characteristic peaks corresponding to the crystallographic planes of metallic copper. These peaks are attributed to the face-centered cubic (FCC) crystal structure of copper. The observed XRD peaks at 2θ angles of 15.2968°, 16.0010°, 17.0167°, 18.6117°, 22.0905°, 23.8305°, 24.9604°, 25.6132°, 26.7973°, 29.1068°, 31.0002°, 31.4779°, 32.3637°, 33.4417°, 36.9997°, 44.5558°, 47.6728°, 49.7260°, 51.2858°, and 56.3839° are indicative of the presence of copper nanoparticles. These peaks correspond to the (111), (200), (220), (311), (222), (400), (331), (420), (422), (511), (440), (531), (533), (600), (622), (800), (711), (731), (800), and (822) crystallographic planes of face-centered cubic (FCC) copper, respectively. The distinct positions and intensities of these peaks suggest the formation of highly crystalline copper nanoparticles in the sample, confirming the successful synthesis of CuNPs. The average crystallite size of copper nanoparticles was 18.143nm to 52.583 nm, calculated by the Scherrer Equation: $D=K\lambda/\beta\cos\theta$.

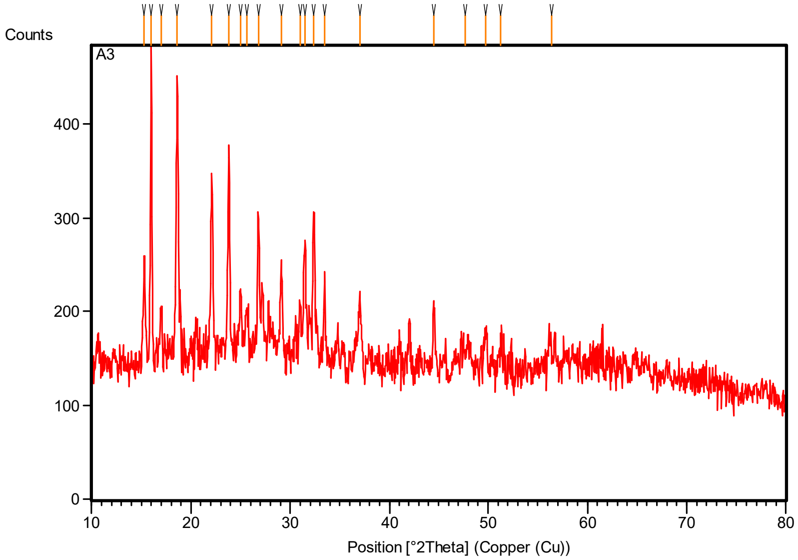

Figure 4. XRD data of copper nanoparticles.

The atomic force microscopy (AFM) and scanning electron microscope (SEM) image results of copper nanoparticles involve analyzing the surface morphology, size distribution, and particle size (Figure 5 and 6). According to the SEM and AFM results, the synthesized copper nanoparticles are spherical shape with smooth surface.

Green synthesized CuNPs hold promise for mercury degradation due to their unique properties and interactions with mercury species. Copper nanoparticles possess high surface area, catalytic activity, and tunable surface chemistry, which enable them to effectively interact with mercury ions and facilitate their conversion into less toxic forms or immobilization onto nanoparticle surfaces. Through processes such as adsorption, reduction, and oxidation, CuNPs can selectively capture mercury ions from aqueous solutions, leading to their sequestration or transformation into insoluble forms (Figure 7). Additionally, the incorporation of functional groups or surface modifications on CuNPs further enhances their affinity towards mercury contaminants, enabling tailored approaches for targeted remediation. Moreover, synergistic effects between CuNPs and other nanomaterials, such as carbon-based nanomaterials or metal oxides, can result in enhanced performance and efficiency in mercury degradation applications. By harnessing the capabilities of green synthesized CuNPs, researchers can develop innovative strategies for mitigating mercury pollution in various environmental matrices.

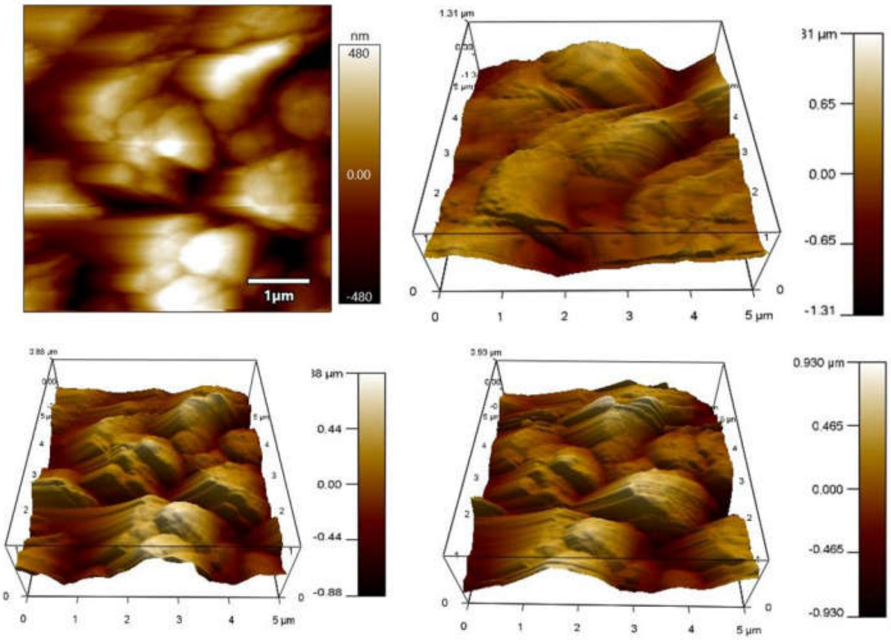

Figure 5. AFM images of green synthesized copper nanoparticles.

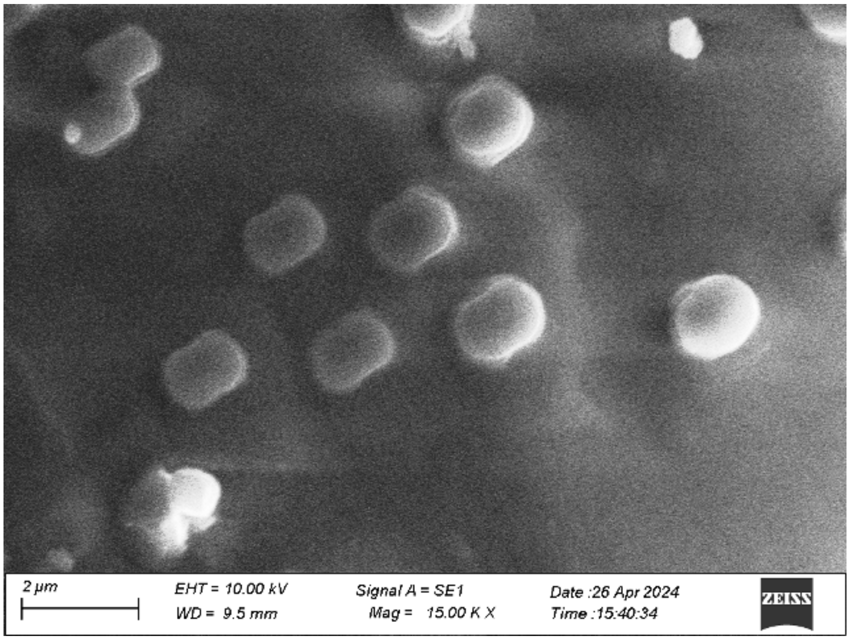

Figure 6. SEM image of green synthesized copper nanoparticles.

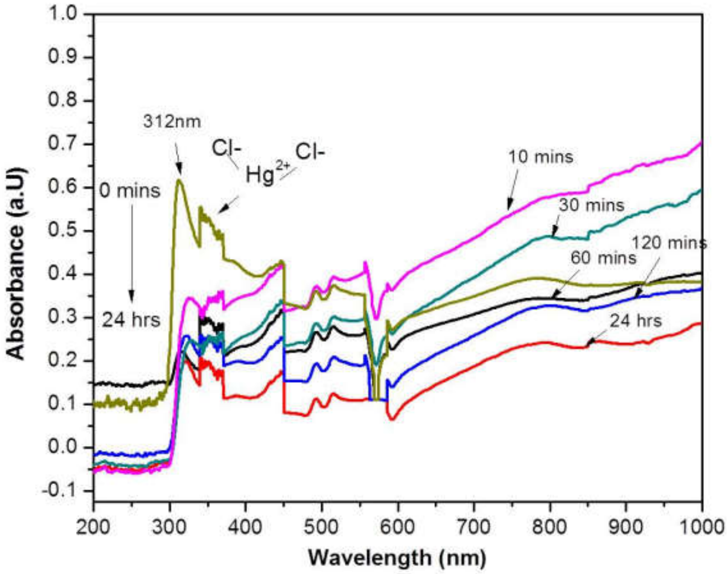

Figure 7. Mercury degradation by green synthesized copper nanoparticles.

Green synthesized copper nanoparticles offer a promising avenue for eco-friendly and effective mercury remediation. By leveraging the unique properties of CuNPs and employing green synthesis approaches, researchers can develop sustainable nanotechnological solutions for addressing the challenges posed by mercury contamination. Continued research efforts in this field hold the potential to advance environmentally friendly approaches for mercury degradation and promote global environmental sustainability.

CONCLUSION

This study highlights the successful green synthesis of copper nanoparticles utilizing *T. arjuna* bark extract, offering a sustainable and environmentally friendly approach. Through UV spectral analysis, distinct absorption peaks were identified, providing insights into the nanoparticles' optical characteristics and validating the efficacy of green synthesis methods. FTIR and XRD analyses further confirmed the formation of highly crystalline copper nanoparticles with characteristic peaks corresponding to metallic copper's crystallographic planes. AFM results provided valuable information regarding surface morphology and particle size distribution. The potential of green synthesized copper nanoparticles in mercury remediation was also demonstrated. These nanoparticles exhibit promising properties for interacting with mercury ions through various processes such as adsorption, reduction, and oxidation, leading to their sequestration or transformation into less toxic forms. Moreover, the incorporation of functional groups or surface modifications enhances their affinity towards mercury contaminants, offering tailored approaches for targeted remediation. Overall, this research underscores the significance of green nanotechnology in addressing environmental challenges, particularly in mercury pollution mitigation. By harnessing the capabilities of green synthesized copper nanoparticles, researchers can pave the way for sustainable and effective solutions for global environmental sustainability. Continued research efforts in this field hold great potential for advancing eco-friendly approaches for mercury degradation and promoting a cleaner and healthier environment for future generations.

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