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Synthesis and characterization of copper nanoparticles using *Allium cepa* (L.) outer peel at ambient temperature

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ABSTRACT

Nanotechnology is increasingly finding applications in various fields like material engineering, medicine, and environmental science. Due to their antimicrobial, catalytic, and electrical properties, copper nanoparticles (CuNPs) have been used in biomedical treatments, cleaning up the environment, and making electronic devices. However, toxic chemicals and high energy consumption are the challenges of conventional synthesis methods of nanoparticles. This study explores the eco-friendly synthesis of copper nanoparticles (CuNPs) using the outer peel extract of *Allium cepa L.* at ambient temperature (25 ± 2°C). The phytochemicals in the extract were used as reducing agents in the process. The formation of CuNPs was confirmed by a color change to dark brown when they encountered Cu^{2+} ions. Using UV–Visible spectroscopy to characterize the sample showed broad absorbance band around 380-600 nm, which means nanoparticles are forming. Using Fourier Transform Infrared (FTIR) analysis, functional groups like hydroxyl (O-H), Cu-H, and C-C were found to be reducing Cu ions. X-Ray Diffractometry (XRD) confirmed the crystalline structure of the CuNPs, while Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) showed pseudo-spherical, rod-like, and polygonal particles with diameters ranging from 8.31 to 179.75 nm. Energy Dispersive X-Ray Spectroscopy (EDS) analysis indicated the presence of copper, oxygen, and sulfur. The results indicate a sustainable alternative for advanced nanotechnology synthesis and applications by demonstrating an efficient, cost-effective, and environmentally friendly approach to synthesizing CuNPs under ambient conditions.

Keywords: Copper nanoparticles, Green synthesis, *Allium cepa* (L.), Ambient temperature, Phytochemicals, Sustainable nanotechnology

Introduction

Copper is a type-p semiconductor material with a narrow band-gap energy of 1.2 eV. It is a nanostructure. This type of p-semiconductor possesses magnificent electromagnetic, optical properties, biological and catalytic properties.[1] Copper nanostructures have unique properties that make them useful in many ways. Due to these properties, they have been used in catalytic processes, biosensors, solar cells, and lithium batteries.[2] The agriculture and health sectors have also applied copper in combination with other elements as antimicrobial agents and photocatalysts for wastewater treatment, respectively.[3]
However, reports have indicated that traditional methods like chemical and physical processes produce nanomaterials.[4] These procedures have been associated with some challenges and setbacks, such as environmental and human biotoxicity, which is the case with chemicals. Physical nanoparticle synthesis is associated with the consumption of a tremendous deal of energy.[5] Furthermore, physically synthesized nanoparticles lack the right nano-composition, morphology, crystallinity, and size distribution, which affects their use and application.[6]

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A research on the economical and fast synthesis of silver nanoparticles from olive-leaf extract for biomedical applications. They reported that conventional techniques (physical, thermal, hydrothermal, and chemical) synthesis are expensive and risky to operate and utilize substances that are harmful to human health, which makes green synthesis a better alernative.[7] Green synthesis involves the use of biological resources and materials for the safe formation of nanoparticles. Reports suggest that this method for making nanoparticles is safe for the environment because it uses materials that are known to be safe for the environment and can be recycled, which act as both reducing and capping agents.[8] Using biomolecules like vitamins, enzymes, and algae, along with biological materials like biodegradable polymers and waste plant parts, has been used in green chemistry, to synthesize nanoparticles. These biomolecules help make nanoparticles more stable and have a controlled size distribution, thus making them safe for use in the environmental, agriculture, and health sectors.[9] Plant materials (leaves, roots, and fruits) have been employed in the safe biosynthesis and applications of nanoparticles.[10] It has also been employed in extensive studies on the pharmacological evaluation of a new anti-cancer triterpene (Nummularic acid) from *Ipomoea batatas*.[11] Green chemistry minimizes hazardous waste while ensuring nanoparticle stability and biocompatibility. Using biological materials is safe for use in biosynthesis applications. Advances in nanotechnology have brought about the application of green synthesis of metal components of plant extracts and components for medical research, microelectronics, and biological research.[7,12] Nanotechnology using green synthesis has been reported to be highly resistant to heat, conserving large amounts of energy, which makes it stable, cheap to adopt, and easy to synthesize.[13] Nanotechnology has also led to important discoveries and uses in materials science, especially in

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recycling metal particles for food, engineering, pharmaceuticals, and other fields. The biosynthesis of metal nanoparticles, such as CuNP particles, benefits the environment and the economy.[9] CuNPs have been made from different kinds of fruit peels and waste; papaya peel,[1] banana peel,[14] *Bauhinia tomentosa* leaves,[15] *Lantana camara* flower,[16] *Capparis spinosa* (caper) leaves,[17] as well as *Aloe-vera* leaf,[18] and other related plant parts and leaves. However, only a handful of studies have explored the potential of using the outer peels of *Allium cepa* to produce CuNPs.

Onions (*Allium cepa*) a biennial plant of the family *Amaryllidaceae* that is grown and consumed for its herbaceous edible fleshy bulb and cherished for their rich flavor of unique cooking and culinary application, which makes them of great value as flavor enhancers in Africa to meals and dishes, stews, soups. They can also be cooked with vegetables in some vegetarian dishes.[19] During the processing and utilization of onions, large amounts of waste are produced that can be considered a valued source of underutilized agricultural waste that can be processed through valorization processes into valuable products for human and animal uses.[20] *Allium cepa* peel extracts have been reported to slow down and halt the growth of Gram-positive and negative bacteria. Bacteria such as *Bacillus cereus, Staphylococcus aureus, Pseudomonas aeruginosa, Salmonella typhimurium, and Klebsiella pneumoniae*.[21] Onions are rich sources of phytochemicals such as flavonoids, phenolics, anthocyanin, and organosulfur compounds, besides microbial inhibitory properties.[22] Also, various species of onions have been reported to be effective in the treatment of diseases such as vascular diseases, cough and asthma because of their antioxidant, anti-inflammatory, antimicrobial, and cytotoxic.[23] Additionally, onion peels constitute a significant portion of agricultural waste, particularly in countries struggling with large-scale agro-waste management issues.[24] Many developing nations, including India, Nigeria, and China, generate substantial onion waste due to high production rates, leading to environmental disposal challenges.[25,26] The use of onion peel waste provides a possible solution with agricultural waste and offers an eco-friendly way to make nanoparticles.[27-29]

The research methodology adopted in this study is highly relevant to developing a green synthesis approach for CuNPs and using the outer peel extract of *Allium cepa* as a reducing and stabilizing agent.[30,31] The synthesis of copper nanoparticles (CuNPs) involves their characterization which gives information on their shape, size distribution, morphology, purity (crystalline structure) and elemental analysis.[32] These properties have effects on their stability and area of application, such as medicine, biosensing, electronics, and catalysis. Therefore, this study employed multiple analytical techniques to ensure the synthesized CuNPs possess the desired properties. Different types of techniques such as X-ray diffractometry (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy dispersive X-ray spectroscopy (EDS) are some of these.[33] Each method plays a critical role in confirming the composition, morphology, and structural integrity of the nanoparticles: The surface plasmon resonance (SPR) peak can be found using UV-Vis spectroscopy. This shows how CuNPs are made and how stable their optical properties are. This is important for their use in biosensing and catalysis.[34] The X-ray diffractometry (XRD) technique finds out about the crystallinity and phase composition of a material.[35] It indicates the diffraction peaks that line up with the CuNPs crystal planes. This shows how structured, reactive, and long-lasting CuNPs would be for use in electronics and catalysis. SEM and TEM provide detailed imaging of nanoparticle morphology and size distribution. Controlled shape and uniformity influence CuNP dispersion, antimicrobial efficiency, and drug delivery potential.[36] Energy Dispersive X-ray Spectroscopy (EDS) verifies the elemental composition and confirms the presence of copper. It also confirms the presence of other elements or bioactive compounds from the plant extract.[37] The results of the characterization would show that the green synthesis method could be used in biomedical, environmental, and industrial settings. The comprehensive use of these techniques ensures a rigorous and scientifically validated approach to nanoparticle synthesis.

Therefore, the aim of this study is the green synthesis and characterization of Copper Nanoparticles using *Allium cepa* (L.) outer peel extract at an ambient temperature. A key novelty in this study is the synthesis of CuNPs at room temperature (25 ± 2°C), reducing energy consumption during synthesis compared to traditional thermal and hydrothermal methods that need high temperatures. This is an economical and energy-efficient method of CuNPs synthesis, which reduces the carbon footprint of CuNP synthesis, and aligns with global sustainability goals.

Materials and Methods

Materials

Sodium hydroxide 99% purity (BDH Poole, England), Ethanol Analar 96% purity (VWR Chemicals, England), Copper sulfate 99% purity (Fisher-Scientific, England), Saline solution (Biomed Diagnostics USA), *Allium cepa* Outer Peel obtained from Mushin Market Lagos, Nigeria (Latitude: 6.5244° N, Longitude: 3.3558° E). All chemicals used were of analytical grade.

Preparation of aqueous extract of outer peel of Allium cepa (AEOPAC)

The preparation of the aqueous extract from the outer peel of *Allium cepa* (AEOPAC) was conducted according to the method employed by Islam et al.,[28] with slight modifications. It was initiated by thoroughly cleaning the peels to remove dirt and impurities using running water. The cleaned peels were air-dried under ambient conditions (26 °C, 80 % relative humidity) for seven days. Once dried, the peels were ground into a fine powder to enhance the surface area. A portion (100 g) of this powder was mixed with water in a 500 mL glass beaker and heated at 60 °C for 30 minutes until a consistent pink hue appeared. The resulting peel broth was filtered through fabric and centrifuged at 2500 rpm for 5 minutes. The filtrate, termed AEOPAC, was separated and stored at 4 °C for later use.

Synthesis of copper nanoparticles (CuNPs) at ambient temperature.

The synthesizing copper nanoparticles (CuNPs) at ambient temperature was conducted according to the method employed by Amatya and Joshi [38] with slight modification. 100 mL of AEOPAC was combined with an equal volume of copper sulfate (1 mM) solution under vigorous stirring at room temperature (25 ± 2 °C) for 3 hours. Sodium hydroxide (0.01 M) was added to adjust the pH to 11, ensuring an effective reaction. The resulting precipitate was centrifuged, washed three times with ethanol and water, and then dried at 50°C for 12 hours. The obtained CuNPs were stored in labeled amber containers for further characterization.

Characterization of Copper nanoparticles (CuNPs)

The characterization of CuNPs was carried out was conducted according to the method employed by Ismail, [39] using multiple techniques to assess their structural, morphological, and compositional properties. UV–Visible spectroscopy (Jenway UV-Vis 7205 Cole-Parmer Ltd., United Kingdom) confirmed the formation of CuNPs by measuring the absorbance of the nanoparticle suspension in distilled water over the 400–800 nm wavelength range. Scanning Electron Microscopy (SEM), coupled with Energy Dispersive X-Ray (EDS) analysis, was employed to analyze the morphology and elemental composition of the nanoparticles. Using a JEOL JSM-7100 FLV field emission SEM, Japan samples were prepared on aluminum stubs (25 mm diameter) coated with a 25 nm carbon layer. Imaging was performed at 20 kV accelerating voltage with a backscattered electron detector, capturing snapshots at various magnifications. Further analysis through Transmission Electron Microscopy (TEM) with EDS provided high-resolution imaging and size distribution of the nanoparticles. Samples were dispersed, sonicated, and a drop placed on a formvar/carbon-supported nickel grid. Excess liquid was removed, and the samples were dried before imaging at 200 kV using a Jeol JED-2300T-SDD, Japan. Nanoparticle sizes were measured with ImageJ software, National institutes of Health USA, Version 1.54m, 2024, and size distribution graphs were plotted using OriginPro version 2025 software, OriginLab Corporation, USA. Fourier Transform Infrared (FTIR) spectroscopy identified functional groups associated with the nanoparticles. The analysis was conducted using an Agilent Technologies Cary 630 FTIR spectrophotometer, USA over the frequency range of 4000–400 cm^{-1}, with powdered samples placed directly on the spectrophotometer aperture. Crystal structure and size were determined using X-Ray Diffractometry (XRD) with a Rigaku Smartlab Powder Diffractometer, Japan (Model: Bragg-Brentano mode). Cu Kα radiation (λ = 1.5406 Å) and a nickel monochromator were utilized, with scans conducted from 5 ° to 80 ° at 2 °/min and a resolution of 0.02 °. The average crystallite size was calculated using Scherer's equation: $D = \frac{k\lambda}{\beta \mathrm{Cos}\theta}$

where D represents the crystallite size, k is the Scherer constant (0.89), λ(lambda) is the X-ray wavelength (1.5406 Å), θ(theta) is Bragg's angle, and β(beta) is the full width at half maximum (FWHM) in radians.

Statistical analysis

All measurements of X-ray diffractometry (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy dispersive X-ray spectroscopy (EDS) were performed in triplicate measurements. Means and standard deviation of CuNPs particle sizes (n=60) calculated from TEM Image using the ImageJ software, National institutes of Health USA, Version 1.54m, 2024, was obtained using IBM SPSS Statistics Version 28. Statistical significance was established for experiments with p-values< 0.05.

Results and Discussion

The *Allium cepa* extract absorbs increased light at shorter wavelengths (200–350 nm), meaning organic compounds like flavonoids and phenolics are present. The extract does not exhibit any absorbance peak in the visible region, indicating the absence of metal nanoparticles. The copper sulfate solution shows a broad absorbance band around 700–800 nm, characteristic of copper ions in the solution. The absence of a sharp peak in the UV-Vis range suggests that the copper is still in the form of ions and has not been broken down into nanoparticles yet. The Surface Plasmon Resonance (SPR) of the copper nanoparticles reveals a broad absorbance band around 380-600 nm. The obtained result agrees with the previously reported literature by Ginting et al.,[40] where a progressive SPR band appeared at the wavelength of 480-620 nm from copper nanoparticles synthesized using *Blumea balsamifera* Leaf extract. Similar results were obtained within the observed range of the absorption peak of CuNPs synthesized using *Cissus arnotiana* plant extracts at 350–380 nm in a study by Rajeshkumar et al.,[41] It also agrees with a study by John et al.,[42] which obtained UV-visible spectra of the CuNPs with maximum absorbance in the 380–385 nm range from copper nanoparticles synthesized using bacterial strains. This is a crucial indicator that CuNPs are synthesized because they differ from the onion extract and copper sulfate spectra. As expected, the blank, distilled water exhibits no significant absorbance across the measured wavelength range, serving as a control. The peak for the onion extract is mainly in the UV range, but the CuNPs show an SPR peak in the visible range (~558 nm), which proves that the CuNP spectrum is different from the plant extracts. This indicates the successful reduction of copper ions in the extract. The copper sulfate spectrum does not have the typical SPR peak in CuNPs. This shows that the copper ions have changed into metallic nanoparticles. The observed SPR peak at 380–600 nm in the CuNPs spectrum, absent in both the onion extract and $CuSO_4$ spectra, strongly supports that the spectra are indeed those of the synthesized copper nanoparticles and not a mere overlap of the starting materials (see Supplementary Figure S1 for spectra).

The FTIR spectrum shown in Figure 1 revealed absorption bands indicating various functional groups associated with CuNPs synthesized at room temperature. Bands observed included 3220.42–3242.78 cm^{-1} (OH stretching and NH primary amine), 2855.14–2922.23 cm^{-1} (CH3 stretching), 1625.12 cm^{-1} (CH2 and carbonyl stretching), 2102.22 cm^{-1} (Cu-H stretching), and 797.65 cm^{-1} (Cu-O stretching). These vibrations confirmed the presence of carbon, nitrogen, hydrogen, copper, and oxygen as primary constituents. The amine and carboxylate groups in the *Allium cepa* extract contributed to binding proteins on the Cu surface, aiding in stabilizing the biosynthesized nanoparticles. This result is in accordance with the previous report by Rafique et al.,[43] as well as Kenneth and Nwodo[44] in which CuNPs was synthesized using *Allium cepa* (onion) peels indicating functional groups such as hydroxyl and amine groups.

XRD patterns showed in Figure 2 distinct peaks at 2θ = 43.28°, 50.4°, and 74.1°, corresponding to 111, 200, and 220 Bragg reflections, respectively.

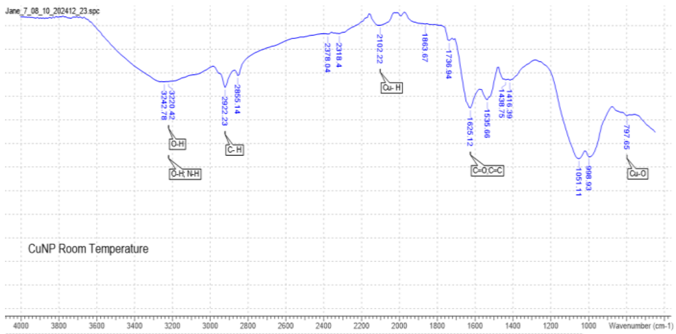

Figure 1: FTIR spectra of CuNP synthesized from outer peel of *Allium cepa (L.)* at room temperature indicating the functional groups observed.

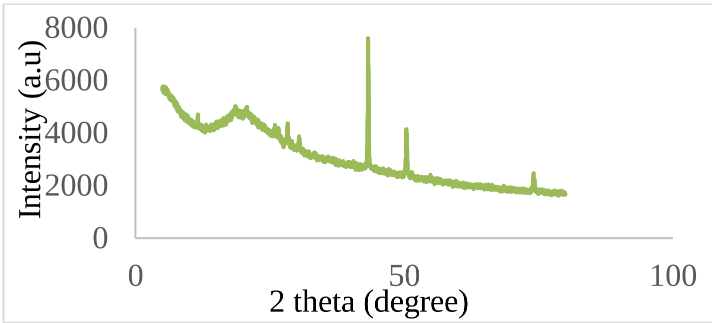

Figure 2: X-ray diffraction spectra of synthesized CuNPs at room temperature.

These are alignment with the reference data from the International Centre for Diffraction Data (JCPDS-45-0937), confirming the crystalline nature of CuNPs and the purity of the nanoparticles. This result also matched those of pure copper in a study by Mohamed, 2020 which revealed the presence of the characteristic peaks of copper at 2θ = 43.2745°, 50.4083° and 74.1706°. Research by Hajizadeh et al.,[45] in which CuNPs was synthesized using Iranian propolis extracts reported XRD peaks corresponding to face-centered cubic copper structures, aligning with our data. In contrast, a study reported by Ali et al.,[46] 2023 indicated XRD patterns with peaks major peaks observed at 36.2 and 38.4 degrees correspond to the crystal planes 11-1 and 111 of CuO, respectively suggesting oxidation of nanoparticles, which contrasts with findings in this study. Another study by Nzilu et al.,[47] which observed additional peaks indicating the presence of Cu_2O, differing from our XRD results. A study by Achamo et al.,[48] reported broader XRD peaks at (002), (111), (202), (220) and (311) planes with 2 theta values of 36.4, 38.7, 48.7, 61.5, and 73.6°, suggesting smaller particle sizes or amorphous structures, in contrast to the crystalline nanoparticles in this study.

Copper was one of the dominant elements represented by EDS in Figure 3 in the sample, with contributions from other elements such as carbon, oxygen and sulphur, possibly from phytoconstituents or biomolecules in the *Allium cepa* extract. This was similar to the elements reported by Ginting et al.,[40] from CuNPs synthesized using *Blumea balsamifera* leaf extracts.

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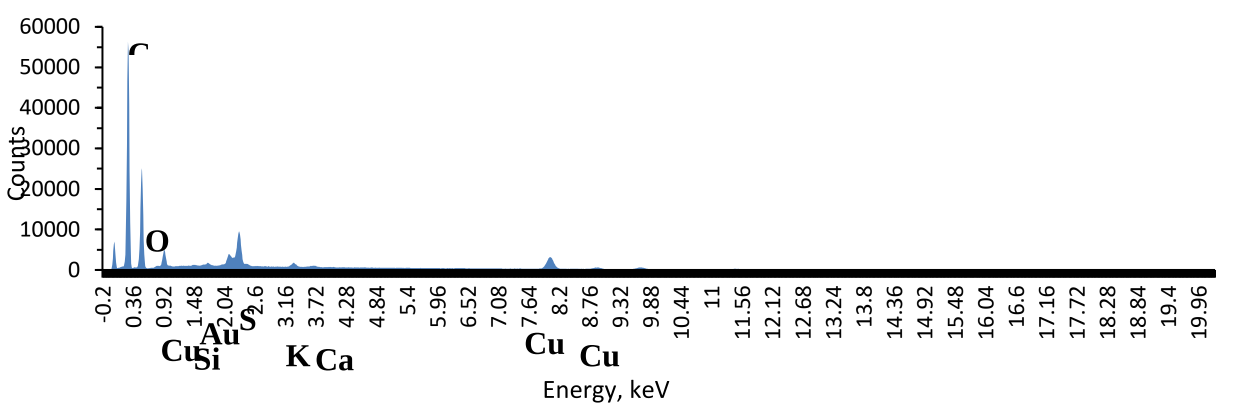

Figure 3: EDX Spectrum of synthesized CuNPs at room temperature.

SEM imaging represented in Figure 4 revealed the morphology of CuNPs synthesized at magnifications ×1000, x5000, ×10,000, and ×25,000, the particles exhibited uneven clustering and more significant agglomeration. The surface appeared rough with grainy features, consistent with the presence of aggregated nanospheres. Similarly, Phang et al.,[1] reported, that green synthesis (70–80 °C, pH not specified) is a cost-effective method for the synthesis of CuNPs from waste peel extract of fruits and showed that the utilization of waste for CuNPs was demonstrated. These authors synthesized CuNPs using the nontoxic and renewable aqueous extract of waste papaya peel, reporting spherical-like particle size from SEM and showing that biosynthesized CuNPs were densely agglomerated. These results compare favorably to findings obtained for onion peel extracts in this current study. However, this study is in accordance with a study by Amaliyah et al.,[49] which observed spherical and tended to form a random aggregate in CuNPs synthesized from *Piper retrofractum Vahl* extract. In contrast, another study by Renuga et al.,[50] indicated the presence of CuNPs with uniform shape but varying in sizes and no aggregation synthesized from *Brassica oleracea var. italic* extract.

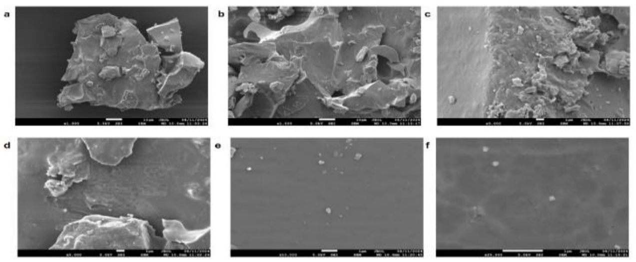

Figure 4: SEM Image of CuNP synthesized at room temperature a. x1,000 b. x1,000 c. x5,000 d. x5,000 e. x10,000 f. x25,000.

TEM analysis shown in Figure 5 and Table 1 provided insights into particle shape and size. The images showed pseudo-spherical particles with visible secondary material capping. The particles were measured and showed diameters ranging from 8.31 to 179.75 nm, with an average size of 55.98nm. These observations suggest a porous microstructure with nanoparticles densely packed and exhibiting minimal gaps. These results obtained from this present study is similar to the report of Kenneth and Nwodo,[44] who researched on green synthesis of copper oxide nanoparticles using the outer layer of *Allium cepa* (27 °C, pH neutral to slight basic) and evaluation of its antimicrobial properties, reported TEM (28 - 45 nm), with a mean particle size of 32 nm. However, Baran et al.,[8] in a study on green synthesis of silver nanoparticles from *Allium cepa* peel extract (40 ºC, pH not specified reported TEM; 8.44 nm minimum and 19.93 nm maximum and a value of 19.47 nm and −13.1 mV for the crystal average size and zeta potential of nanoparticle respectively, the shape obtained were predominantly spherical. These authors concluded that nanoparticles that are synthesized biologically may have the potential for application in biomedical and pharmaceuticals, that metal nano particles can be synthesized from *Allium cepa* biological waste. In another report by Abdullah et al.,[51] green synthesis, characterization and applications of silver nanoparticle mediated by the aqueous extract of red onion peel, synthesized at 90 ºC (pH not specified), the structure of the AgNPs were synthesized and observed as spherical shape, with an average size of 12.5 nm as revealed by (TEM) analysis. The major setback is the physical conventional method is the inadequacy of the surface construction and formation of nanoparticles such as CuNPs. These flaws have been reported to have a substantial influence on physical and exterior interactions properties of the metallic nanoparticles.[52]

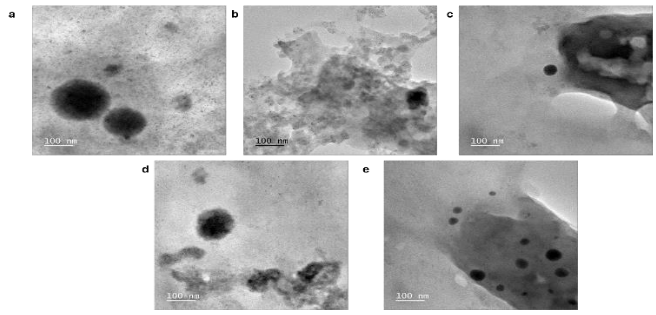

Figure 5: TEM image of CuNP synthesized at room temperature x100nm.

According to Abdelghany et al., [53] green synthesis of metallic nanoparticles is a novel approach and trusted methods, that are more effective for the generation of nanoparticles with merits of less chances of failure, low cost and ease of characterization, as well as utilization of waste. The use of green synthesis that has been reported to be save and less expensive with the possibility of using waste materials instead of expensive chemicals for the synthesis of nanoparticles. Nanoparticles synthesized by conventional methods are expensive and toxic to nature, risk of toxicity in the environment due to the different chemicals used particularly in physical and chemical methods, suggesting the use of alternate approaches such as green synthesis of metal nanoparticles Gour and Jain.[54]

Table 1: Average Particle size of CuNPs, calculated using IBM SPSS Statistics Version 28.

Nanoparticle	Particle Size (nm)**
CuNP Synthesized at	55.98± 41.09

**Values are Mean ± SD.*

According to Yin et al.,[55] the use of conventional (physical, chemical) methods usually involves the use of expensive chemicals for process completion. Also, these methods are associated with potential ecological damage and pose serious toxicity to plant and animal cells which could serve as source of carcinogens.[56,57]

Conclusion

This study revealed scientific evidence of the possibility of synthesis of copper nanoparticles using the outer peel of *Allium cepa*, which confirms onion peel valuable biological waste and green synthesis ecologically safe, low-cost and acceptable method. It also revealed that Cu nanoparticles can be synthesized safely at ambient temperatures making safe and cost effective in terms of energy consumption and utilization, showcasing its novelty in terms energy consumption and cost effectiveness. The nanoparticle from Allium. cepa displayed the potential to be employed as an antioxidant agent, confirming its application in the food, pharmaceutical and cosmetics industries. The future perspective of the of the synthesis of Cu nanoparticle using green synthesis at ambient temperature lies in its application for use in an environmentally safe and cost effect processes, this therefore calls for improvement catalytic efficiencies, toxicity evaluation and effective risk management processes for human safety and health. Further research should be carried out using waste from other *Allium* species and their environmental impact assessment should be ascertained.

Conflict of Interest

The authors declare no conflict of interest.

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Author Contribution

Jane Nnamani Akinniyi- Conceptualization and research design, research methodology, data interpretation and manuscript preparation. The author gave consent for publication.

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