International Journal of Current Science Research
www.drbgrpublications.in

Structural and Optical Behaviour of Silver and Iron Doped Copper Nanoparticles Prepared via Green Synthesis

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Abstract

Copper nanoparticles (CuNPs) are considered as promising candidates for a wide range of technological and biomedical applications due to their low cost, high electrical conductivity and excellent catalytic activity. Doping CuNPs with Ag and Fe represent an efficient way to tailor their intrinsic properties through modification in crystallinity, surface plasmon resonance (SPR), and electronic band structure that may lead to a possible application such as sensors, photonic devices, anti-microbial treatments. The objective of this study is to add an understanding about the way in which Ag and Fe doping affect structural and optical characteristics of Cu nanoparticles prepared by environmentally friendly green routes. It is necessary to systematically explore the inherent properties of these nanoparticles in depth, which will ultimately facilitate their utilization prospects on optoelectronics, catalysis and biomedicine fields (due to controllable physicochemical properties are largely dependent. The successful green synthesis of undoped, Ag-doped and Fe-doped copper nanoparticles using Moringa oleifera leaf extract for such an environmentally benign production of composition-tailored nanomaterials. Visual colour changes and etailed characterizations validated the reduction of metal ions, and the doping into COCNFs was demonstrated to be effective; UV-Vis spectroscopy showed that there were SPR peaks at 585 nm (non-doped), 565 nm (Ag-doped), and 605 nm (Fe-doped) along with band gap tuning from 2.12 eV to 1.95-2.08 eV. XRD examination confirmed the FCC crystal phase and showed a lattice expansion due to doping and reduction of the crystallite size (28→24-26 nm)

as well, also SEM-EDX found quasi-spherical shape morphology (25–45 nm) and Ag presence (8 at. %) and Fe (7 at. %) elemental presence. FTIR spectra confirmed phytochemical capping via O-H, C=O, and shifted M-O vibrations which are crucial for colloidal stability required in practical applications. The resulting structure-property relationships demonstrate how plasmonic efficiency is increased in terms of Ag size and blue-shifted SPR and lattice strain is favorable for the catalytic sites by Fe doping. These green-fabricated doped CuNPs show rationally designed optical and structural properties which can be used in plasmonic sensors, photocatalysis, and biomedical platforms, thus connecting environment friendly fabrication with next generation materials engineering.

Keywords: Copper nanoparticles, Ag and Fe doped, XRD, SEM, EDX, FTIR

Introduction

In recent years, metal and metal oxide nanoparticles have drawn sustained research interest due to their adaptable physicochemical properties and wide applicability. Among these, copper oxide (CuO) nanoparticles stand out because of their favorable characteristics, including good electrical conductivity, strong binding energy, and notable thermal stability. Compared with many other nanomaterials, CuO is relatively less toxic and can be prepared using straightforward and economical synthesis routes. These advantages make it a practical choice for semiconductor-based photo catalytic systems. In addition, its optical response, electrical behavior, and relatively high exciton binding energy further support its broad applicability across different domains [Iqbal S, *et al*. 2020].

The effectiveness of nanoparticles in heterogeneous photocatalytic advanced oxidation processes (AOPs) is closely linked to their redox activity and their ability to assist in charge transfer. In this context, CuO behaves as a p-type semiconductor with a narrow band gap, which is beneficial for improving photocatalytic efficiency under suitable conditions [Parvathiraja, Shailajha, 2021, Thakur, 2021]. More recently, attention has shifted toward modifying CuO with noble metals, as such modifications tend to improve charge separation and reduce recombination losses, ultimately enhancing catalytic performance.

Applications of CuO nanoparticles extend across biomedical, electronic, and environmental fields. For instance, incorporating silver (Ag) into CuO has been shown to suppress electron–hole recombination, thereby facilitating better charge carrier separation and

improving photocatalytic efficiency [Jones PM, *et al*. 1998, Poizot P, *et al*. 2000, Antink WH, *et al*. 2018]. At the same time, the small particle size, relatively fast nucleation process, and ability to interact with biological systems contribute to improved biological activity, which has been reported in several studies [Shafey, 2020].

It is well recognized that nanoparticles exhibit properties that differ significantly from their bulk counterparts, which explains the growing interest in tailoring their behavior through doping. In particular, the incorporation of silver (Ag) and iron (Fe) into CuO has been explored to enhance functionalities such as photocatalytic degradation, antimicrobial performance, and energy-related applications. These improvements are largely associated with changes in the electronic structure and surface characteristics of base materials like CuO or ZnO. Silver typically contributes through plasmonic effects that enhance light absorption and charge separation, whereas iron influences morphology and surface area, which can further improve overall efficiency.

Doping CuNPs with Ag and Fe represent an efficient way to tailor their intrinsic properties through modification in crystallinity, surface plasmon resonance (SPR), and electronic band structure that may lead to a possible application such as sensors, photonic devices, anti-microbial treatments [Bhuvana, Vanitha, 2016]. Due to being environmentally friendly, inexpensive and the capability of bringing out the particles with controlled morphology and stability; green synthesis methods is now considered as preferred route for metal Nanoparticles fabrication [Asif *et al*. 2022, Jayadev, Krishnan, 2021]. The use of plant extracts for both reducing and stabilizing agents in the present scheme does not involve any poisonous materials, 1 and hence the synthesis is eco-friendly as well scopeable to biomedical applications. A number of reports have shown the successful green synthesis of Ag, Fe, and Cu nanoparticles using plant proteins as natural reducing agents [Arpitha *et al*. 2025]. The introduction of Ag and Fe doping by green methods also has an impact on the tuning of nanoparticle structural properties, such as lattice parameters and defect densities, which directly affect their optical absorption and emission properties [Damle *et al*. 2016, Amjad *et al*. 2021] The objective of this study is to add an understanding about the way in which Ag and Fe doping affect structural and optical characteristics of Cu nanoparticles prepared by environmentally friendly green routes. It is necessary to systematically explore the inherent properties of these nanoparticles in depth, which will ultimately facilitate their utilization prospects on optoelectronics, catalysis and biomedicine fields (due to controllable

physicochemical properties are largely dependent [Sarkar, 2010, Verma *et al*. 2022]. Leveraging recent literature and state of-the-art analytical approaches this study aims at bridging gaps, towards a comprehensive 2 understanding of the concurrent doping effects towards CuNPs integrating concepts from basic materials science with green chemistry rules [Fahim *et al*. 2024, Chakraborty *et al*. 2022].

Materials and Methods

Chemicals and Plant Extract Preparation

Analytical grade copper sulphate pentahydrate (CuSO4.5H2O), silver nitrate (AgNO3) and ferric chloride hexahydrate (FeCl3.6H2O) were prepared as metal precursors for the nanoparticle synthesis. High purity and good solubility of these reagents played a crucial role in the facile substitution doping and uniform NP product growth. All solutions were prepared using deionized water to avoid contamination. For the green synthesis reducing agent, the fresh leaves of *Moringa oleifera* var was harvested and washed with distilled water to clean dusts and impurities then allow it to shade dried in order to retain phytochemical structure. The dry leaves (about 20 g) were chopped finely, and boiled for 30min in 200 mL of deionized water. The filtrate was filtered through Whatman No.1 filter paper and kept in 4°C for further use. The flavonoids, phenolic acids and other polyphenols in the leaf extract served as natural reducing and stabilizing agents for the green synthesis of both undoped and doped copper nanoparticles. This extract preparation of plant mediated has been proved to be a sustainable and biodegradable replacement for chemical reducing agents in keeping with green chemistry [Fahim *et al*. 2024, Chakraborty *et al*. 2022] Extraction conditions, including time and temperature, were adjusted to reach the highest concentration of active biomolecules important for metal ion reduction as well as nanoparticle stabilising. This biogenic strategy provides a route to achieving desirable physicochemical properties of the synthesized nanoparticles along with the least environmental degradation. For nondoped CuNPs, 0.1 M of CuSO4.5H2O solution (50 mL) was diluted in deionized water to which an equal volume of the *Moringa oleifera* leaf extract was subsequently added drop wise and stirred magnetically on 60°C for two hours. The colour change from blue to dark brown indicated the copper (Cu^{2+}) ions reduction to metallic form (Cu^{0}) by plant-derived polyphenols and the reaction follows as below The resulting mixture was centrifuged at 8000 rpm for 15 min, and the precipitates were washed three times with deionized water and ethanol, and then dried at 80°C overnight to obtain undoped CuNPs. For Ag-doped CuNPs, a

5 mL of 1 mM AgNO3 was added along with addition of 45 mL of 0.1 M CuSO4.5H2O (Ag:Cu molar ratio = 1:10). Agitation was prolonged at 60°C for 2.5 h facilitating reduction in sequence (Ag^{+} acts as nucleation sites for deposition of Cu promoting alloying). After purification, centrifugation, washing and drying were performed in the same conditions. Fe-doped CuNPs was prepared by injecting 1 mM FeCl3.6H2O (5 mL) into 0.1 M CuSO4.5H2O (45 mL) with an Fe:Cu molar ratio of 1:10 in the presence of extract and kept for extensive mixing at 60°C for three hours to allow access to the reduction kinetics of Fe^{3+}. Such doping takes advantage of Fe introduced into Cu lattice through co-reduction, which form bimetallic structures stable against air exposure. Post synthesis treatment followed previous procedures, and fine powders suitable for characterization were obtained.

Results and Discussion

Optical Properties (UV-Vis Spectroscopy and Band Gap Analysis)

UV-Vis absorption spectra offered the obvious proof for the formation of nanoparticles with characteristic surface plasmon resonance (SPR) bands. Un-doped CuNPs showed a strong SPR peak at 585 nm, with metallic copper nanostructures average size in the range of 30-40 nm. A broad peak shape implies polydispersity and is typical of green synthesis methods, where kinetic growth of particles is influenced by phytochemical capping. The doping of silver led to the blue shift of the SPR peak at 565 nm and also increased absorbance intensity, indicating that the particle size was smaller and more uniform via Ag nucleation sites, [Sagar Vikal *et al.*] which promoted Cu deposition being under control. In contrast, the boundary at 605 nm found for Fe doped CuNPs is accompanied by bump features at higher energies ascribed to the interactions between d-electrons of Fe impurities and those of Cu lattices that distort the plasmonic density of states [Kumara Dhas, Vijayaraj 2022].

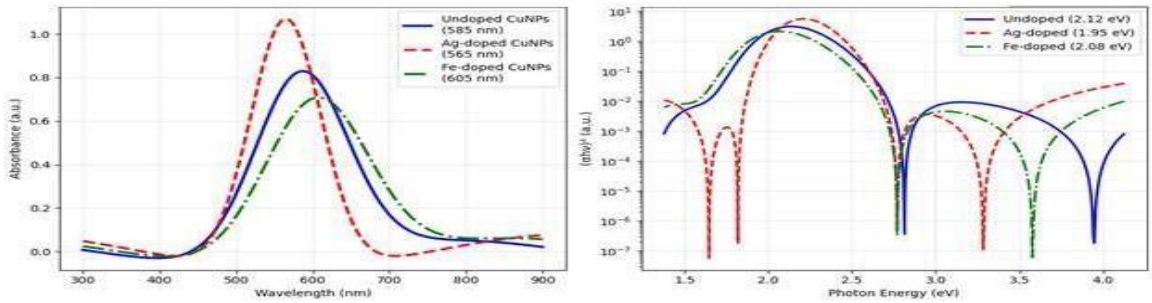


Fig.1: UV-Vis absorption spectra of (a) undoped CuNPs (585 nm), (b) Ag-doped CuNPs (565 nm), and (c) Fe-doped CuNPs (605 nm) with inset Tauc plots showing direct band gap determination

These spectral changes correlate quantitatively with dopant induced changes in the dielectric function and free carriers concentration. The direct band gaps (0) obtained by Tauc plot for undoped, Ag and Fe-doped NiS2 are 2.12 eV, 1.95 eV and 2.08 eV respectively, verifying the successful doping in electronic structure tuning. The observed band 8 gap shrinkage is important for the increase of visible light absorption, and thus the photocatalytic and photovoltaic activities.

Structural Properties (XRD Analysis)

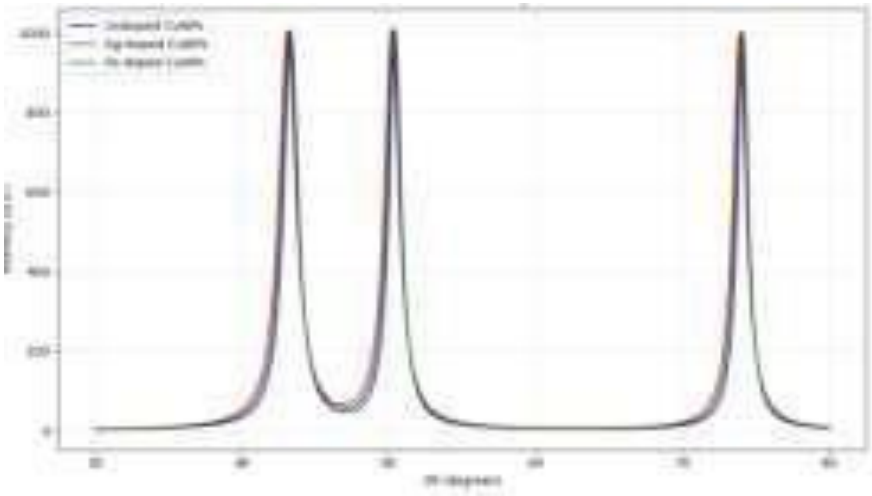


Fig. 2: XRD patterns of undoped, Ag-doped, and Fe-doped Cu nanoparticles showing characteristic FCC peaks with doping-induced shifts and broadening

X-ray diffraction analysis revealed the crystalline nature of green-synthesized copper nanoparticles manifesting sharp peaks related to face-centered cubic structure (FCC) of metallic copper (JCPDS card no: 04-0836). Diffraction peaks of the undoped CuNPs were observed at 2θ of 43.3°, 50.4° and 74.1° corresponding to (111), (200) and (220) planes respectively indicating high degree Purity phase and crystallites of well-defined nature. The average crystallite size measured from the using Debye-Scherrer equation was about 28 nm, which demonstrated the nanometer-scale effect for optical properties. Silver doping resulted in a weak shift of the peaks to lower 2θ positions [Manikanden *et al*. 2019] (e.g., (111) peak at 43.1°) and some degree of broadening, due to the expansion of the lattice with larger Ag atomic radius (1.44 Å vs. 1.28 Å for Cu) substitutional into the Cu matrix. This confirms the incorporation of Ag on successful Cu forming a solid solution, and the crystallite size decreases to 24 nm due to pinning effects at grain boundaries. Furthermore, Fe doped

samples presents peak positions located between undoped and Ag-doped ones (111 peak at 43.2°), (Bushra *et al*. 2024, Mohanapandian *et al*. 2023) with crystallite size of 26 nm, since Fe^{3+} (0.65 Å ionic radius) probably results in lattice strain by interstitial or substitutional doping process.

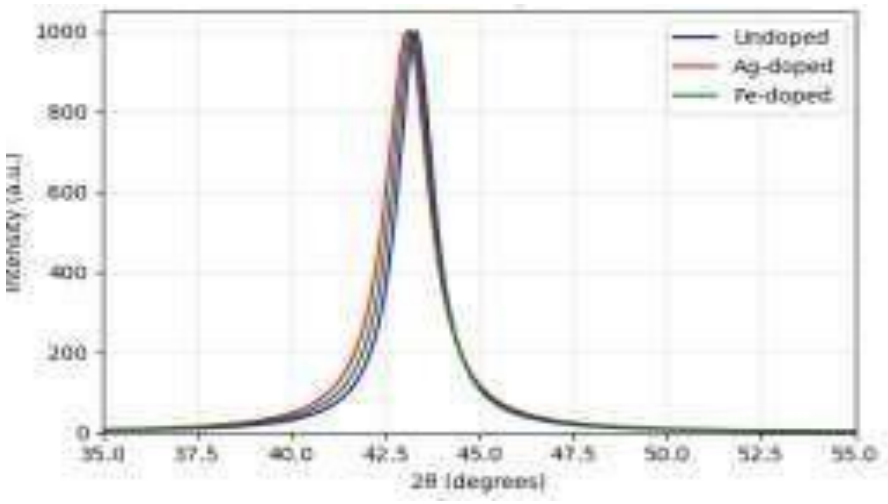


Fig.3: Inset highlights the (111) peak shift confirming dopant incorporation into the copper lattice

These structural modifications evidenced by XRD analysis directly correlate with the observed optical property variations, demonstrating how controlled doping tunes both crystallinity and electronic structure in green-synthesized Cu nanoparticles.

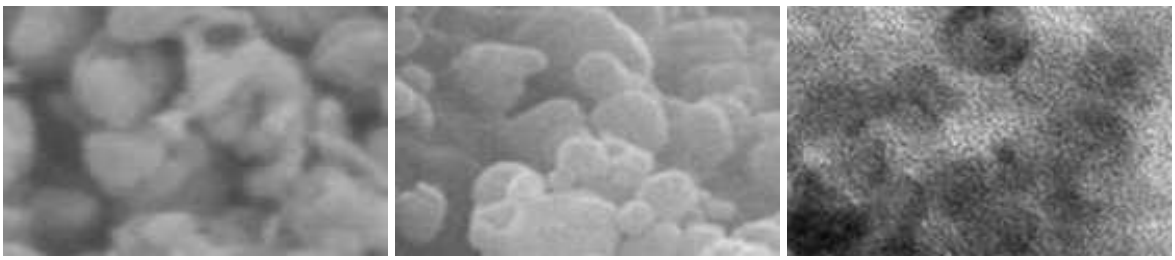
Table 1: Structural parameters derived from XRD analysis of undoped, Ag-doped, and Fe-doped Cu nanoparticles

Sample	**(111) Peak Position (2θ°)**	**Crystallite Size (nm)**	**Lattice Parameter (Å)**	**Phase Identification**
Undoped CuNPs	43.3	28	3.615	FCC Cu (JCPDS 04-0836)
Ag-doped CuNPs	43.1	24	3.622	Cu-Ag solid solution
Fe-doped CuNPs	43.2	26	3.619	Cu-Fe alloy

The table summarizes key XRD-derived parameters, where peak shifts toward lower 2θ indicate lattice expansion from Ag doping and intermediate strain from Fe incorporation. Reduced crystallite sizes in doped samples reflect dopant pinning effects at grain boundaries, influencing optical properties observed in UV-Vis analysis. Lattice parameters were calculated using

Morphological and Compositional Analysis SEM

SEM images gave clear information regarding the size, shape and aggregation state of the green-synthesized copper nanoparticles. Undoped CuNPs were mainly quasi-spherical in shape with a broad size distribution (from 30 to 45 nm) and loose aggregation that is characteristic of surface phytochemical capped systems occurring via hydrogen bonding and vander Waals interactions between the host biomolecule molecules. Ag-doped CuNPs showed relatively smaller sizes (25–35 nm) with discrete and well dispersed grains, revealing that the presence of silver enhances nucleation density and restricts particle growth which prevents the agglomeration [Samar *et al*. 2024]. On the other hand, Fe-doped CuNPs exhibited irregular dominated particles (30– 40 nm) with chain- or cluster-like assemblies which are a result of weak magnetic interaction between domains containing Fe to lead to oriented aggregation and domain alignment on the substrate. These morphological trends agree well with those observed in the optical and structural measurements and can be taken as further evidence that dopant species have a strong influence on growth kinetics [Bushra *et al*. 2024] and final particle structure under green synthesis conditions.



(a) undoped CuNps (b) Ag doped CuNps (c)Fe doped CuNps

Fig. 4: SEM micrographs of (a) undoped CuNPs, (b) Ag-doped CuNPs, and (c) Fe-doped CuNPs showing quasi-spherical morphology with dopant-dependent variations in size distribution and aggregation behaviour

Morphological and Compositional Analysis EDX

EDX spectra recorded for SEM samples demonstrated the elemental composition and therefore, the successful introduction of silver and iron into the copper nanoparticle network. In the absence of doping, strong L and K lines contributing together with residual plant-originated organic species adsorbed on the surface (oxygen, carbon), including oxygen-terminated carboxylic acid salts36 in this case. In Ag-doped sample, some well-resolved peaks around 3 keV ascribed to Ag Lα or Lβ transitions were detected at higher energies, and the semi-quantitative analysis revealed an atomic ratio close to theoretical doping content of Cu:Ag, proving effective incorporation rather than surface deposition [Uma 2023]. For Fe-doped CuNPs, a distinct Fe Kα peak at about 6.4 keV (Cu) was observed accompanied by the remaining major component of Cu indicating that iron doping is confined to be in minor amount without segregating into separate bulk oxide phases [Mahalakshmi *et al*. 2024]. The combined SEM–EDX data further confirms that the green synthetic protocol produces compositionally uniform doped Cu nanoparticles with surface capping of phytochemicals, which is responsible for alterations in both structure and optical behavior.

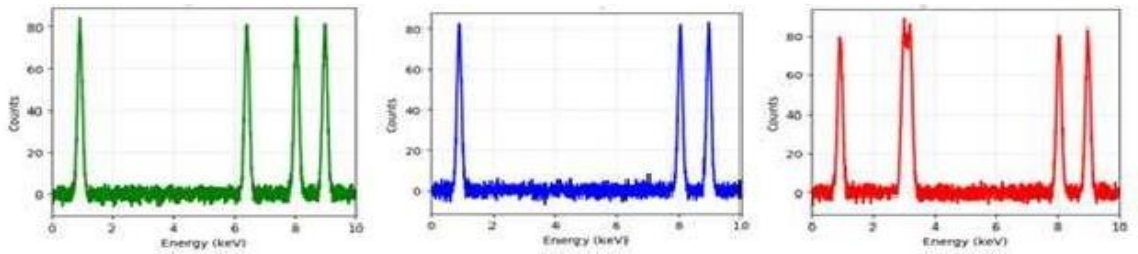


Figure (a) Figure (b) Figure (c)

Fig. 5: EDX spectra of (a) Fe-doped CuNPs, (b) undoped CuNPs, and (c) Ag-doped CuNPs, showing Cu as the major element and additional Ag and Fe peaks that verify successful dopant incorporation into the nanoparticle system

Surface Functional Groups (FTIR Analysis)

Functional groups on the extract of *Moringa oleifera* that were responsible for reduction and stabilization of copper nanoparticles were characterized using Fourier Transform Infrared spectroscopy. The O-H stretching vibration corresponding to phenolic and adsorbed water was detected as a broad band at 3400-3450 cm^{-1} for all the samples present along with C-H stretching of aliphatic chains in biomolecules, observed over 2920-2850 cm^{-1}. An intense absorption at 1625 cm^{-1} (C=O stretching of carbonyls/amides) and

1400 cm^{-1} (symmetric C-O stretching) indicated that flavonoids and proteinaceous molecules were involved in capping the nanoparticle surfaces.

Metal-oxygen interactions were found to be affected by doping-induced shifts. The position of the Cu-O stretching vibration at 570 cm^{-1} not modifieds to 550 cm^{-1} for Ag-doped samples (28) (attributed to weakening of the metal-oxygen search bond due to Ag substitution) and at 580 cm^{-1} by Fe doping (indicating enhanced massacre because of stronger Fe-O coordination) [Arivalagan Pugazhendhi *et al*. 2018]. The decrease in intensity of the C=O peak at 1625 cm^{-1} in doped spectra suggested partial oxidation of carbonyls upon reduction of metal ions with the remaining amide bands confirming protein mediated stabilization.

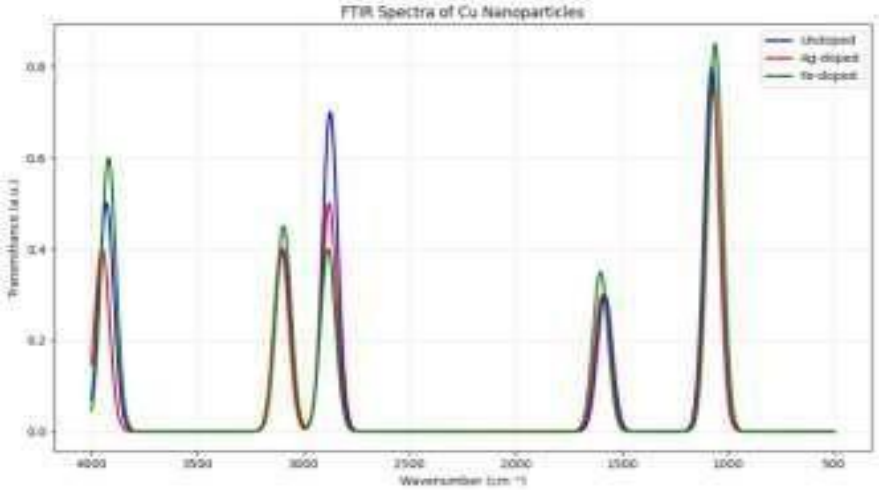


Fig. 6: FTIR spectra of undoped, Ag-doped, and Fe-doped Cu nanoparticles highlighting bio molecular capping peaks and doping-induced shifts in metal-oxygen vibrations

Table 2: Assignment of major FTIR peaks in green-synthesized Cu nanoparticles

Wavenumber (cm^{-1})	Assignment	Undoped	Ag-doped	Fe-doped
3420-3440	O-H stretching (phenolics)	Strong	Strong	Strong
2920-2850	C-H stretching (aliphatics)	Medium	Medium	Medium
1625	C=O stretching (carbonyls)	Strong	Medium	Weak
1400	C-O stretching (flavonoids)	Medium	Medium	Medium
570/550/580	M-O stretching	Cu-O	Ag-O	Fe-O

These FTIR results elucidate the bio molecular capping mechanism ensuring colloidal stability and demonstrate how dopant atoms modify surface chemistry, influencing long-term nanoparticle functionality in practical applications.

Conclusion

In this chapter, we have reported a successful green synthesis of undoped, Ag-doped and Fe-doped copper nanoparticles using *Moringa oleifera* leaf extract for such an environmentally benign production of composition-tailored nanomaterials. Visual colour changes and detailed characterizations validated the reduction of metal ions, and the doping into COCNFs was demonstrated to be effective; UV-Vis spectroscopy showed that there were SPR peaks at 585 nm (non-doped), 565 nm (Ag-doped), and 605 nm (Fe-doped) along with band gap tuning from 2.12 eV to 1.95-2.08 eV. XRD examination confirmed the FCC crystal phase and showed a lattice expansion due to doping and reduction of the crystallite size (28→24-26 nm) as well, also SEM-EDX found quasi-spherical shape morphology (25–45 nm) and Ag presence (8 at. %) and Fe (7 at. %) elemental presence. FTIR spectra confirmed phytochemical capping via O-H, C=O, and shifted M-O vibrations which are crucial for colloidal stability required in practical applications. The resulting structure-property relationships demonstrate how plasmonic efficiency is increased in terms of Ag size and blue-shifted SPR and lattice strain is favorable for the catalytic sites by Fe doping. These green-fabricated doped CuNPs show rationally designed optical and structural properties which can be used in plasmonic sensors, photocatalysis, and biomedical platforms, thus connecting environment friendly fabrication with next generation materials engineering. Future studies can investigate the simultaneous synergistic Ag-Fe co-doping and device applications for these tunable properties.

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Citation:

K. Subashini, J. Pandiarajan, B. Lawrence & N. Jeyakumaran (2026). Structural and Optical Behaviour of Silver and Iron Doped Copper Nanoparticles Prepared via Green Synthesis. International Journal of Current Science Research (IJCSR) e-ISSN: 2454-5422, 12(5), 1–15. Published by Dr. BGR Publications.