



P-ISSN: 2349-8528

E-ISSN: 2321-4902

www.chemijournal.com

IJCS 2022; 10(2): 21-25

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Received: 13-01-2022

Accepted: 15-02-2022

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Green synthesis of copper oxide nanoparticles using *Justicia Carnea*

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DOI: <https://doi.org/10.22271/chemi.2022.v10.i2a.12176>

Abstract

This study manifests the simple, environmentally-friendly and inexpensive synthesis of small sized copper oxide nanoparticles (CuO NPs) using the aqueous extracts of *Justicia carnea*. The aqueous extracts of *Justicia carnea* serve as effective reducing and capping agents for the green-synthesized NPs. The reduction of Cu^{II} was visually observed by the change of colour from light green to brown with formation of precipitate. The reaction was followed by UV-Visible spectrophotometry and the characterization of the copper oxide nanoparticles by Fourier Transform Infrared spectrophotometry. The UV-Visible spectra of aqueous solutions containing the synthesized copper nanoparticles showed a maximum absorption peak at 240 nm corresponding to surface plasmon resonance (SPR) of copper oxide nanoparticles. The FTIR spectrum of CuO NPs shows peaks corresponding to bands associated to Cu-O stretching and bands which represent characteristic peaks believed to be due to the interaction of CuO with biomolecules of extract.

Keywords: *Justicia carnea*, copper oxide nanoparticle, spectroscopy, nanoparticle, green synthesis

Introduction

The submicron size particles with size between one and one hundred nanometre that exhibit unique physical and chemical properties are well known as nanoparticles (NPs). Among the severally researched metal nanoparticles synthesized from plant extracts are those of coinage metals such as copper, silver and gold because of their diverse properties and applications (Singh *et al.* 2018, Nzekekwa and Abosede, 2019; Hamilton-Amachree and Abosede 2019) [21, 3]. The preparation of copper nanoparticles at ambient conditions in aqueous medium often leads to oxidation and result to formation of copper oxide nanoparticles. Copper oxide (CuO) has a vast array of applications of applications in various fields, from energy conversion and storage through environmental science, electronics and sensor (Ren *et al.*, 2009) [19]. CuO nanoparticles have gained increasing prominence because copper is the simplest and cheapest member of the coinage metals, and it bears numerous useful physical properties such as electron correlation effects, optical properties and myriads of uncommon crystal morphologies (Sankar *et al.*, 2014) [20]. The diverse properties of CuO nanoparticles have resulted in their applications in industrial fields, e.g. as p-type semiconductors and transistors in the design and production of batteries (Kwak and Chongyup, 2005) [16], solar cells (Gawande *et al.*, 2016) [9], gas sensors (Khatoun *et al.*, 2018) [9], and field emitters (Singh *et al.*, 2015) [17]. Currently, CuO nanoparticles have been demonstrated to function as catalysts (Gawande *et al.*, 2016) [9], antioxidants, water purifiers, antimicrobials, drug delivery, and contrast agents (Ren *et al.*, 2009; Sankar *et al.*, 2014; Akintelu *et al.*, 2020) [19, 20, 2].

Two major approaches commonly employed for nanoparticles synthesis are the chemical and physical methods. Chemical synthetic method employs chemical reduction, electrochemical techniques and photochemical reduction (Guzman *et al.*, 2009; Sumitha *et al.*, 2016) [24]. The most commonly used method known as the classical chemical method involves the use of reducing agents such as sodium borohydride and hydrazine (Khatoun *et al.*, 2018) [14]. Chemical methods often result to successful production of pure and precise nanoparticles, they are in many cases not economical, inefficient and have toxic by-product which are harmful to the environment. Hence, alternative and sustainable methods of synthesis are usually explored. Green synthesis method such as plant-mediated synthesis of nanoparticles has become a conventional, ecofriendly substitute to the physical and chemical methods. It is a less-complicated, affordable and more expeditious method that involves the use of benign environmentally-friendly materials than other methods (Mohan *et al.*, 2015; Kavitha *et al.*, 2013) [17, 13].

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Green synthesis mitigates environmental problems. The plant extracts in the green synthesis often play vital role(s) in the size and surface morphology of the nanoparticle synthesized by green methods. The plant extracts possess active compounds that often serve as both reducing and capping agent. Furthermore, producing nanomaterials from plant extracts frequently enhances biocompatibility which contributes to biofunctionality of the nanomaterials (Yuvakkumar and Hong 2014) [29]. Another advantage of the green synthetic route over chemical method is higher yield of nanoparticles than other methods (Buazar *et al.*, 2019) [6]. Fabrication of copper/copper oxide NPs with good physico-chemical properties have also been achieved using fungal and bacterial agents such as *Botrytis cinerea* (Kovacec *et al.*, 2017), *Escherichia coli* (Singh *et al.*, 2010) and *Serratia sp.* (Hasan *et al.*, 2008) [10] as reductants. Likewise, leaf extracts from plants such as *Stachys Lavandulifolia* (Veisi *et al.*, 2021) [26], *Psidium guajava* (Singh *et al.*, 2019) [22], *Abutilon indicum* (Ijaz *et al.*, 2017) [11], *Malva sylvestris* (Awwad *et al.*, 2015) [5], *Aloe vera* (Kumar *et al.*, 2015), *Carica papaya* (Sankar *et al.*, 2014) [20], *Camellia sinensis* (Sutradhar *et al.*, 2014) [25] and *Ixoro coccinea* (Vishveshvar *et al.*, 2018) [27] have been used for the preparation of CuO NPs.

Though some studies have been carried out on the synthesis of CuO NPs using different plant extracts and broth, nevertheless, no detailed research has been carried out on the synthesis and characterization of CuO NPs using *Justicia carnea* (locally known as five minutes blood leaf, figure 1). These precedents informed our interest in the preparation of CuO NNPs using *Justicia carnea* leaves aqueous extracts.

The rationale for the choice of *Justicia carnea* is because of its wide usage among the local populace for different therapeutic effects especially for the treatment of anemia. Different species of *Justicia carnea* are being employed for different therapeutic effects e.g. in the treatment of diseases of the digestive tracts and respiratory organs as well as diabetes, headache, HIV and nervous disorders (Corrêa and Alcântara, 2012) [7].

The major objective of the present study is to synthesize copper oxide nanoparticles by an environmentally friendly method using aqueous leaf extract of *Justicia Carnea* (five minutes blood leaf) and to characterize the nanoparticles generated using UV-Vis spectroscopy and FTIR. Isolated extracts of *Justicia* leaves have been shown to possess essential elements four times higher than that obtained in blood plasma (Wood *et al.*, 2020) and phytochemicals such as steroids, phenolic and flavonoid compounds (Anigboro *et al.*, 2021) [4].



Fig 1: Picture of Fresh *Justicia Carnea* (five minutes blood leaf)

Materials and Methods

Materials and Instrumentals

Copper sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) (99.5%), distilled water were sourced from commercial suppliers and

used as received. *Justicia Carnea* leaves (well known as five minutes blood leaf) were bought from Otuoke community market in Ogbia Local Government Area of Bayelsa State, Nigeria. UV-Vis spectra were taken on JASCO V-730 Spectrophotometer between wavelength of 200 and 600 nm at bandwidth of 1 nm and scan speed of 1000 nm/min. FTIR spectra were taken on Shimadzu FTIR-8400S Spectrophotometer from wavenumber range of 400 – 4000 cm^{-1} .

Methodology

Aqueous extracts from the ambient-dried leaves of *Justicia Carnea* was prepared by weighing 15 g of the grounded dried leaves into 500 mL beaker with the addition of 150 mL of distilled water and then stirred for about 10 minutes. The mixture was incubated for 48 hours at 25 °C. The aqueous extract was collected by filtration using Whatmann filter paper.

Copper sulphate pentahydrate (molecular weight of 249.68 g/mol) was used to prepare a concentration of 0.1 M copper sulphate solution. 6.240 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ was weighed using analytical weighing balance and then dissolved in distilled water in 250 mL volumetric flask.

For reduction of Cu^{2+} ions, 5 mL of aqueous extract of *Justicia Carnea* was added into a clean beaker and then the respective volume of 0.1 M of aqueous $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ solution was added into the extract. On addition of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ to the extract, precipitate was noticed after about 40 minutes. The copper oxide nanoparticle preparation was carried out at different mixing ratios of the extracts to 0.1M $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (1:0, 1:1.04, 1:1, 1:2, 1:3) at 10% extract concentration.

1 mL of the extracts were collected periodically to monitor the complete ion bioreduction of $\text{Cu}(2+)$ ion in aqueous solution, and subsequent scan in UV-Visible (Vis) spectra, between wavelength of 200 and 600 nm in a spectrophotometer (JASCO V-730 Spectrophotometer), at a bandwidth of 1 nm and scan speed of 1000 nm/min. Distilled water was used as the blank.

Results, Discussion

The formation of CuO NPs was monitored by the visual observation of change in colour of the aliquot as the reaction progresses with time. Initially, the colour of the precursor solution of *Justicia Carnea* extract was light green (figure 2), the colour of the reaction solution gradually changed into light brown, with increase in time. Finally, the precipitation of brown solids was observed on the inner wall of the vessel after 40 minutes of reaction signifying the formation of copper nanoparticles. The change in colouration is attributed to the bio-reduction of Cu^{2+} ions to CuO nanoparticles (Sukumar *et al.* 2020) [23].



Fig 2: Shows copper nanoparticles synthesized at different ratios. 1:0; 1:04; 1:1; 1:2 and 1:3

The FTIR spectra of CuO NPs were presented in figure 3 with series 1, 2 and 3 representing the spectrum of CuO nanoparticles obtained from 1:1, 1:2 and 1:3 mixing ratio of extract to 0.1 M Cu²⁺ respectively. The FTIR spectra of CuO NPs (figure 3) showed peaks at 3421, 3402, 3303, 3250, 2926, 2931, 2362, 2366, 1633, 1627, 1192, 1261, 744, 657, 1404 and 1560 cm⁻¹. From the indicated peaks, the broad band around 3402 cm⁻¹ in the spectrum of Cu NPs represent the presence of phenolic compounds present in the aqueous leaf extract of *Justicia carnea* which have adsorbed on the surface of the CuO NPs.

Strong peak around 3427 cm⁻¹ corresponds to the -OH stretching of phenolic compounds present in the *Justicia carnea* aqueous extract, and the band at 1635 cm⁻¹ corresponds to the C=O stretching of ketones or acids. The sharp peak around 1405 cm⁻¹ shows the presence of -COO carboxylic acid stretching and the absorption peak at 1103 cm⁻¹ corresponds to the C-NH₂ primary aliphatic amines present in the *Justicia carnea* extract.

The intense peaks shown 2926 cm⁻¹ and 2931 cm⁻¹ correspond to asymmetric C-H stretching.

The biomolecules present in *Justicia carnea* extracts serves as reducing, stabilizing, and capping agents in the CuO NP synthesis.

The medium peak at 1192 and 1261 cm⁻¹ corresponds to C-O stretching of phenolic compounds. The last peak at 1404 and 1560 cm⁻¹ corresponds to C-C (Aryl). In addition to this, the FTIR spectrum of CuO NPs shows peaks at 831, 744, 607 and 455 cm⁻¹ which represents a characteristic peak believed to be due to the interaction of CuO NPs with biomolecules of extract. The bands centered at 744 and 657 cm⁻¹ confirm the formation of CuO NPs.

The UV-visible absorbance spectrum recorded for CuO NPs exhibited λ_{max} of 240 nm as shown in (figure 4). This absorption band is basically due to surface plasma resonance of CuO NPs (Sukuma *et al.* 2020) [23]. Another weak and broad peak at 305 was observed. Peaks in the range of 300–600 nm are usually attributed to the SPR band of some Cu colloid formations of the nonoxidized Cu Nps. Similar results were reported after the analysis of CuO NPs synthesized using *Caesalpinia bonducella* seed extract (Akhina *et al.* 2021, Sukuma *et al.* 2020) [1, 23].

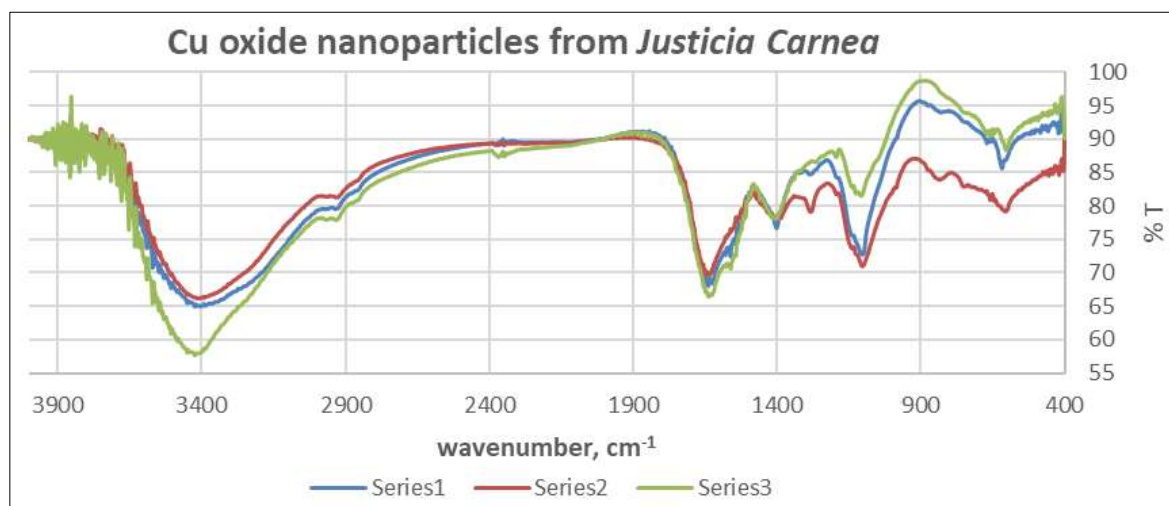


Fig 3: FTIR spectra of copper oxide nanoparticles synthesized from *Justicia carnea* leaf extract. Series 1 (1:1), Series 2 (1:2), Series 3 (1:3).

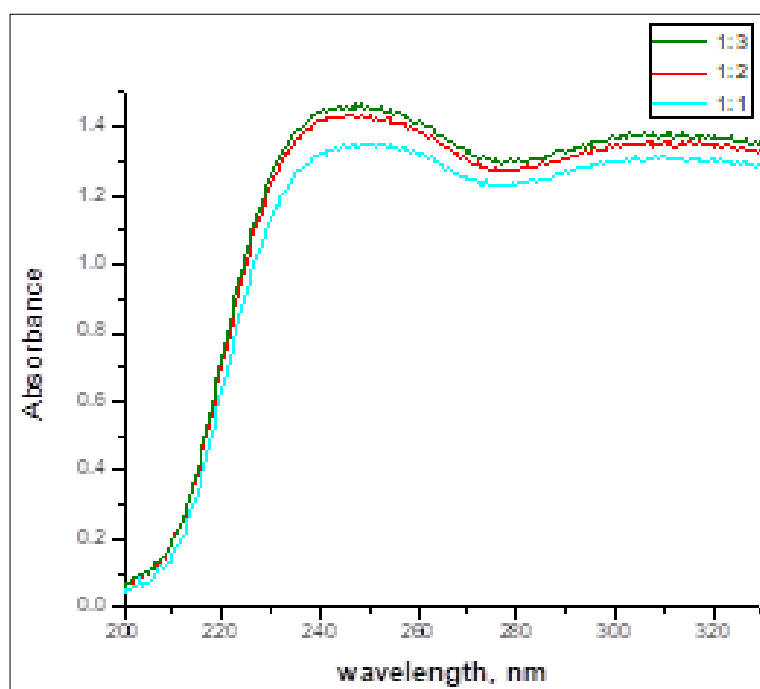


Fig 4: UV spectra of Copper Oxide nanoparticles synthesized from *Justicia Carnea* leaf extract.

Conclusion

Copper nanoparticles were successfully synthesized using the extract of *Justicia Carnea* (Five Minutes Blood Leaves). The biological synthesis of copper oxide nanoparticles using *Justicia Carnea* aqueous extracts provides environmentally friendly, simple and efficient route for the synthesis of benign CuO nanoparticles. The absorption spectra of the prepared CuO NPs showed maximum absorption peak at 240 nm after 40 minutes of stirring. This indicates the formation of CuO NPs. FTIR analysis illustrated the presence of functional groups of *Justicia Carnea* phytochemicals such as phenol, carbonyl and amide groups at the surface of the CuO NPs which are responsible for reducing and stabilizing the nanoparticles.

Acknowledgement

Olufunso O. Abosede appreciates International Foundation for Science (IFS), Stockholm, Sweden and the Organization for the Prohibition of Chemical Weapons (OPCW) for the donation of JASCO UV-Visible V730 spectrophotometer through IFS individual grant IFS 5780-1.

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