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ORIGINAL ARTICLE

Synthesis of Copper(I) Oxide Nanoparticles using *Dillenia retusa* Fruit Extract as a Sustainable Capping Agent

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Abstract

Objectives: Natural and biocompatible capping agents are increasingly sought as sustainable alternatives to synthetic surfactants in nanoparticle synthesis. This study evaluated whether *Dillenia retusa* (Godapara), an endemic Sri Lankan plant, fruit extract which was not previously investigated for copper-based nanoparticle synthesis can act as an effective natural capping agent for the green synthesis of copper(I) oxide (Cu_2O) nanoparticles. **Method:** Cu_2O nanoparticles were synthesized via chemical reduction of $CuSO_4 \cdot 5H_2O$ with ascorbic acid under alkaline conditions, both with and without *D. retusa* fruit extract as a capping agent. Synthesis of each condition was independently repeated in triplicate to confirm reproducibility. The uncapped nanoparticles served as the benchmark for comparison. Products were characterized by FTIR, SEM, EDXS, and XRD, and crystallite size/lattice strain were quantified using the Williamson–Hall method. **Findings:** FTIR confirmed Cu–O bonding in both samples and revealed extract-derived phenolic and aliphatic bands unique to the capped sample. SEM showed that capping reduced the average particle size from 64 nm (38–86 nm range, uncapped) to 47 nm (28–68 nm range, capped), with the capped particles well-dispersed and free of agglomeration. XRD confirmed phase-pure cuprite, Cu_2O, in both samples, and Williamson–Hall analysis showed the crystallite size decreased from 51.1 nm (uncapped) to 29.2 nm (capped), a 43% reduction, accompanied by a small increase in microstrain (0.405% to 0.447%), consistent with phytochemical adsorption restricting crystal growth. **Novelty:** Although *D. retusa* extracts have recently been employed in the green synthesis of silver nanoparticles, their use in the synthesis of copper-based nanoparticles has not been reported to date. To the best of our knowledge, this study represents the first report of *D. retusa* fruit extract mediated synthesis of Cu_2O nanoparticles and the first demonstration of its role as a capping agent in the preparation of a copper oxide nanomaterial. This work therefore expands the reported applications of *D. retusa* for the synthesis of nanoparticles further with the quantitative data on particle and crystallite sizes.

Keywords: Cu_2O Nanoparticles; *Dillenia retusa*; Green Synthesis; Natural Capping Agents; SEM Characterization

1 Introduction

Nanotechnology has rapidly evolved into a significant interdisciplinary field due to its broad applications across scientific and industrial domains. Nanomaterials with at least one dimension in the 1–100 nm range exhibit unique physicochemical properties arising from their high surface-to-volume ratio and quantum effects, and metal nanoparticles particularly of copper, silver, zinc, platinum, and gold occupy a central position in nanotechnology owing to their catalytic, optical, antimicrobial, and electronic properties, with broad spectrum of applications spanning from drug delivery and imaging to optoelectronic and environmental remediation technologies [1].

A recent review of copper oxide nanoparticle synthesis highlights the role of capping agents in the formation of nanoparticles. Newly formed nanoparticles possess high surface energy and therefore have higher tendency to agglomerate. Capping agents adsorb onto the nanoparticle surface through electrostatic interactions, coordination bonding, or hydrogen bonding, thereby reducing the surface energy and preventing the possible agglomeration [2]. While synthetic surfactants remain widely used as capping agents [3], natural alternatives derived from plant extracts offer low toxicity, biodegradability, and cost-effectiveness [4]. Plant materials such as aloe vera [5] and *Dillenia indica* bark extract [6] have been employed as capping agents for metal nanoparticle synthesis.

Dillenia retusa (Godapara) (Figure 1), an endemic Sri Lankan medicinal plant, is traditionally used for ailments including fever, dandruff, cough, oral diseases, and some neurological disorders, and its fruits and leaves are a documented source of phytochemicals such as flavonoids and tannins with reported antimicrobial activity [7], [8].

Phytochemical extracts of leaf, fruit peel, flower, bark, stem, and seed of *D. retusa* have very recently been used to green-synthesize silver nanoparticles for catalytic and cytotoxicity applications [9]. However, to the best of our knowledge, the potential of *D. retusa* phytochemicals as capping agent for copper-based nanoparticles, especially in the synthesis of copper(I) oxide (Cu_2O) nanoparticles remains unexplored. Addressing this gap, the present study investigates the use of *D. retusa* in the green synthesis of Cu_2O nanoparticles.

Cu_2O nanoparticles are widely applied in perovskite solar cells, photovoltaic and optoelectronic devices, and sensing technologies. They have also demonstrated a significant antimicrobial activity against bacteria, fungi, algae, and viruses [10]. In this study, Cu_2O nanoparticles were synthesized through a green route using *D. retusa* fruit extract as a natural capping agent and the resulting nanoparticles were compared with those synthesized under identical conditions in the absence of the plant extract.



Fig 1. *Dillenia retusa* (Godapara) fruits and leaves

2 Methodology

2.1 Materials

All chemicals were of analytical grade and used without further purification. Copper(II) sulphate pentahydrate (98.5%, Sigma-Aldrich, USA), sodium hydroxide (>98%, Emparta, Germany), L-ascorbic acid (99%, Merck, Germany), ethanol (98%, Sigma-Aldrich, USA), and starch (Sigma-Aldrich, USA) were used as received.

2.2 Preparation of *D. retusa* extract

D. retusa fruits were collected from the western part of Sri Lanka. The plant was identified as *D. retusa* (Godapara) by comparing its morphological features such as leaf shape, flower structure, and fruits with the published description and photographic record of the species [11] and in consultation with a plant biologist.

The extraction method was adapted from a previously reported procedure [8]. Fresh *D. retusa* fruits were air-dried in sunlight for 48 hours before being ground into a coarse powder. The powder (100 g) was soaked in ethanol (99%, 500 mL) overnight. The mixture was stirred at 1400 rpm for 1 hour at room temperature (25 $\pm$ 2 °C) using a magnetic stirrer and subsequently filtered using vacuum filtration. The filtrate was concentrated under reduced pressure at 40 °C using a rotary evaporator, and the concentrated extract was stored at −20 °C until further use.

2.3 Synthesis of copper oxide nanoparticles for the control test

The uncapped nanoparticles served as the control for those synthesized in the presence of *D. retusa* extract. The synthesis was adapted from a chemical-reduction procedure reported for green Cu_2O nanoparticle synthesis [12]. $CuSO_4 \cdot 5H_2O$ solution (0.1 M, 60 mL) was mixed with ascorbic acid (0.2 M, 50 mL) under continuous stirring (1500 rpm). Subsequently, NaOH (1 M, 30 mL) was added dropwise, and the mixture was heated at 80 °C for 2 hours. A colour change from blue to ochre to dark brown was observed. The mixture was allowed to settle overnight, centrifuged at 12000 rpm for 20 min, and the collected solid was freeze-dried for 5–6 hours. The resulting dark brown nanoparticles were stored under nitrogen. This synthesis procedure was performed three times to confirm reproducibility.

2.4 Synthesis of *D. retusa* fruit extract-capped copper oxide nanoparticles

$CuSO_4 \cdot 5H_2O$ solution (0.1 M, 60 mL) was added dropwise to the *D. retusa* fruit extract solution (1.2% w/v, 120 mL). The amount of plant extract used was restricted to the mentioned values due to challenges encountered during the purification of the synthesized nanoparticles. Furthermore, this concentration is aligned with those reported in the literature with regards to the usage of phenolic-rich fruit extracts in comparable green-synthesis studies. The mixture was stirred vigorously at 1500 rpm for 30 min. Subsequently, ascorbic acid (0.2 M, 50 mL) and NaOH (1 M, 30 mL) were added following the same procedure used for the uncapped nanoparticles. The mixture was heated at 80 °C for 2 hours and left to settle overnight, then centrifuged at 12000 rpm for 20 min, and the collected solid was freeze-dried. The resulting dark brown nanoparticles were stored under nitrogen. Similar to the control synthesis, this synthesis procedure was performed three times.

2.5 Characterization of synthesized nanoparticles

Fourier-transform infrared (FTIR) spectroscopy was performed using a Thermo Scientific Nicolet S10 FT-IR spectrometer equipped with an attenuated total reflectance (ATR) accessory, recording spectra over 4000–500 cm^{-1} at 4 cm^{-1} resolution. Scanning electron microscopy (SEM) was performed using a Carl Zeiss EVO 18 instrument at 10 kV; particle size was measured by analyzing approximately 50 particles per sample using ImageJ software. SEM combined with energy-dispersive X-ray spectroscopy (EDXS) was used to investigate elemental composition. The chemical nature and crystallite size of the nanoparticles were assessed using a CuKα radiation source (wavelength 1.54178 Å, 40 mA) on a Rigaku Ultima IV X-ray diffractometer operating in the 5–80° range.

3 Results and discussion

3.1 Synthesis of copper oxide nanoparticles

Copper oxide nanoparticles were synthesized using a modified chemical reduction method using *D. retusa* fruit extract as an environmentally friendly capping agent. *D. retusa* is recognized as a medicinal plant species endemic to Sri Lanka. Nanoparticle synthesis used $CuSO_4 \cdot 5H_2O$ as the copper precursor, ascorbic acid ($C_6H_8O_6$) as the reducing agent, and NaOH to adjust the reaction pH. The overall reduction of Cu^{2+} to Cu^{+} by ascorbic acid under alkaline conditions can be represented as:

$$2Cu^{2+} + C_6H_8O_6 + 4OH^{-} \rightarrow Cu_2O + C_6H_6O_6 + 2H_2O \quad (1)$$

The initial reaction mixture was light brown, gradually changing to dark brown during synthesis; nanoparticles prepared with and without the *D. retusa* fruit extract as a capping agent exhibited a similar dark brown appearance. This colour development and settling behaviour was consistent across all independent batches synthesized for each case.

3.2 Characterization of synthesized copper oxide nanoparticles

3.2.1. FTIR spectroscopy

Figure 2 presents the FTIR spectra of (a) uncapped and (b) *D. retusa* fruit extract-capped copper oxide nanoparticles as an overlay. Both spectra exhibit characteristic Cu–O stretching vibrations in the 500–650 cm^{-1} region, confirming the formation of the Cu_2O phase in both samples. However, the capped sample shows five distinct Cu–O bands (620, 610, 591, 572, and 560 cm^{-1}) compared to three in the uncapped (611, 572, and 557 cm^{-1}), suggesting increased lattice distortion induced by the capping layer[13].

The C–H stretching bands at 2920 and 2850 cm^{-1} are present exclusively in the *D. retusa* fruit extract-capped sample and entirely absent in the control (in uncapped copper oxide), providing unambiguous evidence that aliphatic phytochemicals from the *D. retusa* fruit extract are specifically adsorbed on the copper oxide nanoparticle surface. The mid-infrared fingerprint region (700–1700 cm^{-1}) of the two samples is markedly different: the control shows bands at 1632, 1383, 1317, and 1104 cm^{-1} characteristic of residual ascorbate species, while the capped sample shows bands at 1573, 1366, and 1081 cm^{-1} arising from phytochemical functional groups. This indicates that *D. retusa* phytochemicals effectively displaced surface-adsorbed ascorbate residues during capping, establishing a stable organic phytochemical layer on the nanoparticle surface[14].

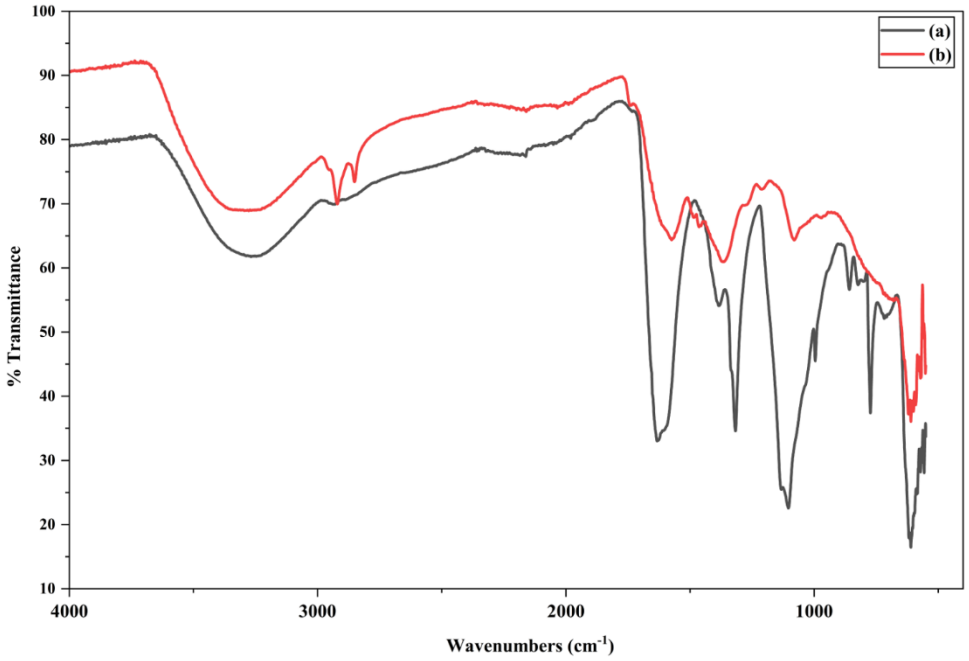
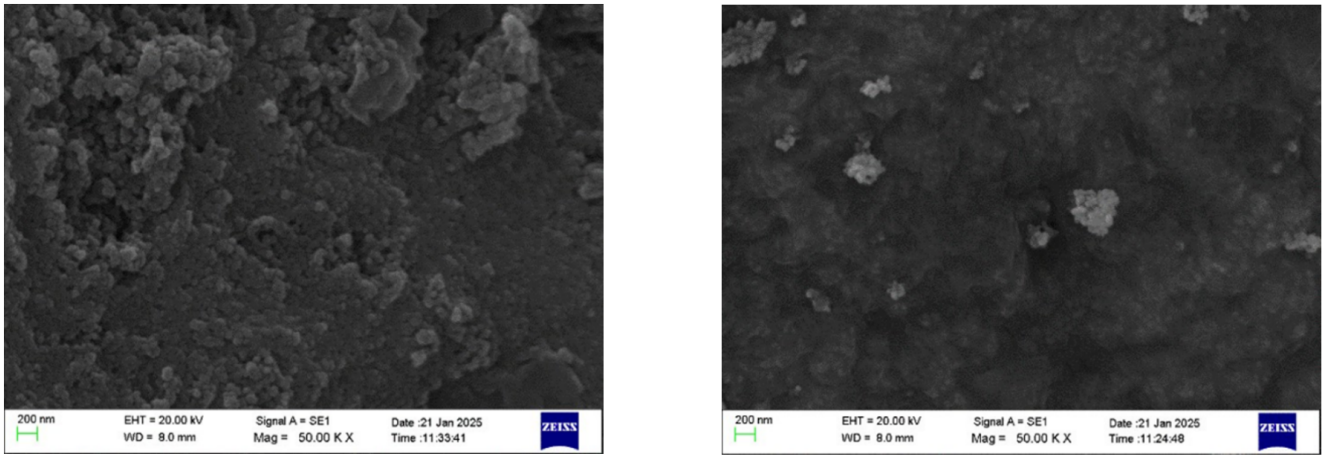


Fig 2. FTIR spectra of (a) uncapped and (b) *D. retusa* fruit extract-capped copper oxide nanoparticles

3.2.2. SEM analysis

Morphological analysis was performed to evaluate the size, shape, and size distribution of the synthesized copper oxide nanoparticles and to verify their nanoscale dimensions. Figure 3 shows the SEM images of the copper oxide nanoparticles synthesized without a capping agent (Figure 3a) and with *D. retusa* fruit extract as capping agent (Figure 3b). The average particle size of the nanoparticles synthesized without the capping agent (Figure 3a) was 64 nm, with a size distribution of 38–86 nm; the particles were spherical with some agglomeration. *D. retusa* fruit extract-capped copper oxide nanoparticles (Figure 3b) exhibited an average particle size of 47 nm, with a size distribution of 28–68 nm and showed no signs of agglomeration highlighting the role of the extract as an effective stabilizing agent.



(a) (b)

Fig 3. SEM images of the synthesized copper oxide nanoparticles (a) without capping agent and (b) with capping agent (*D. retusa* fruit extract)

3.2.3. EDXS analysis

EDXS analysis of the *D. retusa* fruit extract-capped nanoparticles (Table 1) confirmed the presence of Copper, Oxygen, and Carbon. The detected oxygen likely arises both from Cu–O bonds and from oxygen-containing compounds in the extract, while the carbon content is attributed to organic constituents of the *D. retusa* extract.

Table 1. EDXS analysis results of copper oxide nanoparticles synthesized using *D. retusa* fruit extract as the capping agent

Element	Weight (%)	Atomic (%)
C	21.30	42.48
O	24.85	37.21
Cu	53.86	20.31

3.2.4. XRD analysis

The crystalline structure and phase purity of the uncapped and capped nanoparticles were determined by XRD (Figure 4). Both patterns show a well-defined, sharp diffraction pattern with minimal background noise, indicating high crystallinity. Both exhibit five characteristic peaks indexed to the (110), (111), (200), (220), and (311) planes of the cuprite (Cu_2O) phase (space group *Pn*-3*m*, No. 224; ICDD 00-005-0667), confirming phase-pure Cu_2O with no CuO or metallic Cu peaks detected. The refined lattice parameter of the capped sample (a = 4.2629 Å) agrees closely with the standard value (a = 4.2696 Å; < 0.2% deviation). Systematic peak broadening in the capped sample indicates reduced crystallite size and/or increased lattice strain from phytochemical surface interactions.

Crystallite size (D) and lattice strain (ε) were evaluated using the Williamson–Hall (W-H) method [15], [16], which deconvolves crystallite-size and micro-strain contributions to peak broadening (Eq. 2). Tables 2 and 3 give the full width at half maximum (FWHM) values used for the uncapped and capped samples, respectively.

Table 2. FWHM values from XRD analysis (Williamson–Hall method) for uncapped Cu_2O nanoparticles

Peak	hkl	2θ (deg)	FWHM (deg, 2θ)
1	(110)	29.55	0.450
2	(111)	36.43	0.492
3	(200)	42.27	0.480
4	(220)	61.32	0.680
5	(311)	73.49	0.960

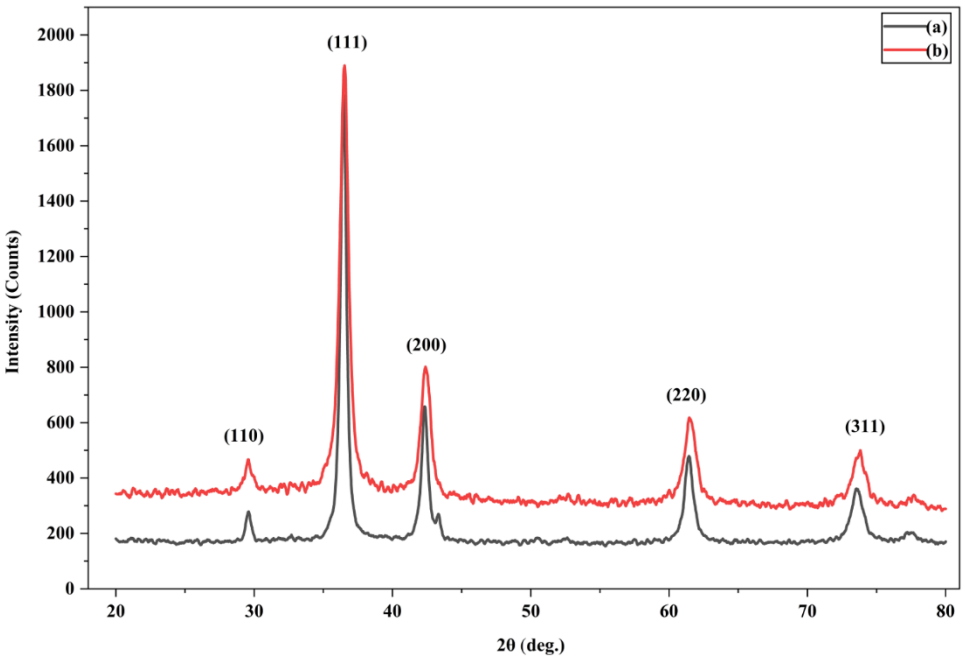


Fig 4. XRD patterns of (a) uncapped and (b) *D. retusa* fruit extract-capped copper oxide nanoparticles

Table 3. FWHM values from XRD analysis (Williamson–Hall method) for *D. retusa*-capped Cu_2O nanoparticles

Peak	hkl	2θ (deg)	FWHM (deg, 2θ)
1	(110)	29.52	0.440
2	(111)	36.50	0.706
3	(200)	42.37	0.800
4	(220)	61.47	0.940
5	(311)	73.65	1.070

$$\beta \cdot \cos\theta = K\lambda/D + 4\epsilon \cdot \sin\theta \quad (2)$$

where β is FWHM in radians; θ is the Bragg angle (radians); D is volume-weighted crystallite size; ε is micro-strain; K = 0.94 (Scherrer constant for spherical nanoparticles)[16] and λ = 1.5406 Å.

The *D. retusa*-capped Cu_2O nanoparticles exhibited a crystallite size of D = 29.2 nm, significantly smaller than the uncapped nanoparticles (D = 51.1 nm) — a 43 % reduction — consistent with phytochemicals in the *D. retusa* fruit extract acting as growth-inhibiting capping agents that sterically restrict grain propagation[15]. The marginally higher microstrain of the capped sample ($\varepsilon = 4.465 \times 10^{-3}$, 0.447%) relative to the blank ($\varepsilon = 4.053 \times 10^{-3}$, 0.405%) is consistent with localized lattice distortions from phytochemical surface adsorption, as reported for other green-synthesized metal oxide nanoparticles[16]. The low absolute microstrain in both samples indicates the crystallographic integrity of the Cu_2O phase is well preserved[17].

3.3 Significance of the study

Within the *Dillenia* genus, previous studies have reported metallic copper nanoparticles synthesized using *D. indica* bark extract for antioxidant and anticancer evaluation[6]. In another study, monoclinic CuO nanoparticles (15 nm) synthesized using *D. indica* and *Mikania micrantha* leaf extracts used for photocatalytic and antibacterial applications[18], and selenium nanoparticles synthesized using *D. indica* leaf extract applied for larvicidal and antimicrobial activity[19]. More recently, it was attempted to use aqueous extracts of different *D. retusa* plant parts for the synthesize of silver nanoparticles (65 nm)[9]. However, placing

the finding of this research in the context of recent literature highlights the novelty of using *D. retusa* fruit extract for the first time as a capping agent for Cu_2O nanoparticle synthesis.

Beyond the Dillenia genus, green-synthesized Cu_2O nanoparticles have been reported using *Myrciaria dubia* juice for antibacterial functionalization of textiles, yielding smaller particles (10 ± 2.2 nm) than the corresponding chemical synthesis (75 ± 2.8 nm)[20]. Cu_2O nanoparticles synthesized using *Muntingia calabura* fruit extract have also been reported for catalytic applications[12], while CuO nanoparticles prepared from *Ziziphus jujuba* fruit extract have been investigated for anticancer and antioxidant activities[14]. These findings support the potential of Cu_2O nanoparticles for a wide range of applications across various fields.

In this context, the present study introduces *D. retusa* as a new plant source for Cu_2O synthesis and demonstrates a reduction in particle size from 64 nm to 47 nm and with 43% reduction in crystallite size due to fruit extract capping. The XRD results further confirm phase-pure Cu_2O formation, extending the application of *D. retusa* beyond previously reported silver nanoparticle synthesis.

4 Conclusion

This study demonstrated the green synthesis of Cu_2O nanoparticles using *D. retusa* fruit extract as a natural capping agent and presumably the first report about this. Upon the analysis of the resultant product, FTIR analysis confirmed Cu–O bonding and the presence of extract-derived phenolic and aliphatic functional groups in the capped sample. XRD confirmed the formation of crystalline, phase-pure Cu_2O in both capped and uncapped samples. The use of *D. retusa* extract reduced the average particle size from 64 nm to 47 nm and the crystallite size from 51.1 nm to 29.2 nm (43% reduction), while also minimizing the agglomeration observed in the uncapped sample. These findings highlight the potential of *D. retusa* fruit extract as a natural capping agent for Cu_2O synthesis and extend its reported application beyond silver. The study also adds another distinct nanoparticle system to the *Dillenia* genus, alongside previously reported *D. indica*-mediated copper and CuO nanoparticles. Future work should focus on evaluating the antimicrobial and catalytic activities of the capped nanoparticles, given the reported bioactivity of both *D. retusa* phytochemicals and Cu_2O. Further studies could also explore the synthesis of other useful nanoparticles using *D. retusa* fruit extract as a natural capping agent.

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