Article

Andean Sacha Inchi (Plukenetia Volubilis L.) Leaf-Mediated Synthesis of Cu₂O Nanoparticles: A Low-Cost Approach

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Abstract: In this work, Andean sacha inchi (Plukenetia volubilis L.) leaves were used to prepare monodispersed cuprous oxide (Cu₂O) nanoparticles under heating. Visual color changes and UV-visible spectroscopy of colloidal nanoparticles showed λ_max at 255 nm, revealing the formation of copper oxide nanoparticles. Transmission electron microscopy and dynamic light scattering analysis indicated that the prepared nanoparticles were spherical with an average size of 6–10 nm. The semi-crystalline nature and Cu₂O phase of as-prepared nanoparticles were examined by X-ray diffraction. Fourier-transform infrared spectroscopy confirmed the presence of polyphenols, alkaloids and sugar in the sacha inchi leaf, allowing the formation of Cu₂O nanoparticles from Cu²⁺. Additionally, as-synthesized Cu₂O nanoparticles exhibited good photocatalytic degradation activity against methylene blue (>78%, 150 min) with rate constant 0.0219106 min⁻¹. The results suggested that the adopted method is low-cost, simple, ecofriendly and highly selective for the synthesis of small Cu₂O nanoparticles and may be used as a nanocatalyst in the future in the efficient treatment of organic pollutants in water.

Keywords: Plukenetia volubilis; green synthesis; Cu₂O nanoparticles; XRD; catalysis

1. Introduction

During the last two decades, nanoparticles/nanomaterials (of size less than 100 nm) have been extensively studied due to their exclusive properties such as high surface-to-volume ratio, flexibility, quantum size, high yield strength, rigidity, ductility and macro-quantum tunneling effect, and are currently used in various areas of chemistry, physics, medicine and engineering [1,2]. Copper is one of the most important elements used worldwide for various purposes. In particular, copper oxide nanoparticles have received great attention because of their low cost, high yield, mild reaction conditions and fantastic applications in batteries [3], catalysis [4], optical devices [5], printed electronics [6], anticancer therapeutics, sensing, antioxidants [7], antimicrobial activities [8], fuel cells [9], bioimaging [10], dye removal [11], gas sensors [12], etc.

Copper oxide nanoparticles have been synthesized by a variety of chemical and physical methods including hydrothermal synthesis [13], the wet chemical method [14], solution phase synthesis [15], sonochemical synthesis [16], the microwave method [17], the laser ablation method [18] and ball milling [19], in which a large amount of solvents is required for obtaining pure and well-defined nanoparticles. They create various problems for the ecosystem and the environment [7,20]. Plant-based metal nanoparticle preparation methods are low-cost, simple and ecofriendly and hold great promise.
in biotechnology applications due to the presence of effective reducing and capping agents such as carbohydrates, polyphenols, terpenoids, sugars, amino acids, flavonoids, saponins, etc. [20]. Therefore, they are favored over chemical and physical synthesis methods [21]. Previously reported syntheses of cuprous oxide/cupric oxide (Cu$_2$O/CuO) nanoparticles have used plant materials to support large-scale synthesis, including extracts of *Azadirachta indica* leaf [22], *Aegle Marmelo* leaf [23], *Aloe barbadensis Miller* leaf [24], *Acalypha indica* leaf [25] *Punica granatum* peel [26], *Terminalia arjuna* bark [27], *Stachys lavandulifolia* flower [28], *Terminalia bellirica* [29] and *Rubus glaucus* fruit [30], etc.

Sacha inchi (SI) (*Plukenetia volubilis* L.) is a promising crop plant originally from the Amazon basin of Latin America. Its star-shaped green fruits produce nut-like seeds with a bitter taste and have been consumed by humans since Incan times due to their high content of fatty acids (ω-3 and ω-6, 35–60%), protein (27–33%), carbohydrates and antioxidants [31]. Its dried leaves are marketed as a tea and contain 84.2–93.4% sugar [32]. Phytochemicals such as saponins, terpenoids, polyphenolic compounds (flavonoids) and other components are also found in SI leaves and are responsible for their antioxidant and antiproliferative activities [33].

Thus, the utilization of these lost crops in nanotechnology and green chemistry has been continued by our group: SI leaves for the synthesis of silver nanoparticles [34], SI oil for the synthesis of silver [35] and gold nanoparticles [36] and SI shell biomass for the synthesis of silver nanoparticles [37] and removal of Cu$^{2+}$/Pb$^{2+}$ [38]. Our research on the SI plant may support an additional source of revenue for farmers on the western and northern edges of South America, such as Colombia, Ecuador, Venezuela, Peru, Bolivia, Brazil and Suriname, and in the Lesser Antilles [31].

To our knowledge, there have been no reports of the synthesis of Cu$_2$O nanoparticles using the SI leaf. Herein, green Cu$_2$O nanoparticles were synthesized by reducing Cu$^{2+}$ ions with aqueous extract of SI leaf. This nanoparticle is used in the degradation of methylene blue (MB), one of the most common organic pollutants in wastewater from the dye industry (Figure 1). It is environmentally undesirable at a trace level and excessive usage of MB causes serious diseases in human beings [35,36]. Hence, the development of an ecofriendly method for the removal of MB from wastewater is necessary. In this work, full spectroscopic and microscopic characterizations were performed to confirm the formation of Cu$_2$O nanoparticles. Photocatalytic evaluation experiments indicated that Cu$_2$O nanoparticles possess fast catalytic activity in the degradation of MB.

![Figure 1. Schematic presentation of the synthesis and application of Cu$_2$O nanoparticles.](image)

### 2. Materials and Methods

#### 2.1. Materials

Copper nitrate (Cu(NO$_3$)$_2$·3H$_2$O, 99.0%) and methylene blue (MB, 99.5%) were purchased from Spectrum, USA. Sacha inchi (SI) leaves were collected with the help of Mr. Abraham Rodolfo Sanchez Piñuela from a farm in Quito, Ecuador. Milli-Q water was used in all experiments. All chemicals listed above were of analytical grade and used without any purification.
2.2. Preparation of Cu$_2$O Nanoparticles

Thoroughly washed SI leaves were shade-dried for one week. The extraction of phytochemicals from the SI leaves was performed by an earlier extraction method [34]. Nearly 800 mg of the dried SI leaf was crushed into small pieces and transferred to a 100 mL flask containing 50 mL of Milli-Q water, and then it was mixed well and heated (64–68 °C) for 30 min. The obtained red extract of SI leaf was filtered through Whatman No. 1 paper and stored at 4 °C for further use. For the synthesis of Cu$_2$O nanoparticles, 5 mL of SI leaf extract was added slowly to 20 mL of Cu(NO$_3$)$_2$ solution (10 mM), followed by heating for 5 h at 85–90 °C with continuous stirring. The formation of the Cu$_2$O nanoparticles was indicated by a change of reaction mixture color from bluish-red to a greenish color.

2.3. Characterization of Cu$_2$O Nanoparticles

The samples containing nanoparticles were confirmed by a UV-visible spectrophotometer, GENESYS™ 8 from Thermo Spectronic, England. The particle size distribution of the sample was analyzed using the HORIBA, DLS Version LB-550 program, Japan. The morphology and selected area electron diffraction (SAED) pattern of the nanoparticles were captured on a transmission electron microscope (TEM), FEI Tecnai, G2 Spirit Twin, Holland. X-ray diffraction (XRD) studies on thin films of the nanoparticle were carried out using a PANalytical brand θ-2θ configuration (generator-detector) X-ray tube, copper λ = 1.54059 Å, and an EMPYREAN diffractometer. The Fourier-transform infrared (FTIR) spectra were collected on a Spectrum Two IR spectrometer from Perkin Elmer, USA. The samples for FTIR and XRD analysis were prepared by carefully depositing a thin film of Cu$_2$O nanoparticles on a glass slide by injecting and heating 1800 µL (600 µL × 3 times) of Cu$_2$O solution drop by drop at 60–65 °C for 20–30 min allowing the solvent to evaporate. After that, the thin film of the samples was scratched and the FTIR-ATR analysis was performed.

2.4. Photocatalytic Effects

The photocatalytic activity of Cu$_2$O nanoparticles during the degradation of MB was determined by carrying out the reaction in direct sunlight (1040–1165 cd/m$^2$) at 30–35 °C (atmospheric temperature). Typically, 5 mL MB (10 mg/L) was mixed with 1 mL H$_2$O in a glass tube; in a second glass tube, 5 mL MB (10 mg/L), aqueous solution containing Cu$_2$O nanoparticles (500 µL) and H$_2$O (500 µL) was mixed in the dark for 20 min to reach an adsorption-desorption equilibrium. Then, both sets were exposed to direct sunlight and the progress of the degradation reaction was monitored at different time intervals by UV-vis spectroscopy at a wavelength of 664 nm. To evaluate the interference of SI leaf extract on the reduction of MB, a separate reaction was performed in which 5 mL of MB (10 mg/L) was mixed with 500 µL of SI extract and 500 µL of H$_2$O. After that, the reaction mixture was heated at 40 °C for 120 min in the dark. The photocatalytic degradation percentage of MB was calculated using Equation (1) and the respective first-order rate constants (k) according to Equation (2).

\[
\eta = \frac{(A_0 - A_t)}{A_0} \times 100\% \tag{1}
\]

\[
k_t = \ln \left( \frac{A_0}{A_t} \right) \tag{2}
\]

where $\eta$ is the rate of degradation of MB in terms of percentage, $A_0$ is the initial absorbance of the dye solution and $A_t$ is the absorbance of the MB at time $t$, respectively. $C_0/C_t$ is measured from the relative intensity of absorbance ($A_0/A_t$). The linear relationship of ln ($A_0/A_t$) versus time indicates that the photodegradation of MB follows first-order kinetics [35,36].
3. Results and Discussion

3.1. UV-Vis Spectroscopy Analysis

Figure 2 shows the UV-visible absorption spectra of as-prepared Cu$_2$O nanoparticles in the presence of SI leaf extract in an aqueous medium. The colorimetric reduction reaction of Cu$^{2+}$ ions with the SI leaf extract (red colour) indicated the formation of a greenish-colored solution (a and b in Figure 2), attributed to the surface plasmon resonance (SPR) of the Cu$_2$O nanoparticles [30,39]. The absorption peaks appearing at 260 and 340 nm (red line) correspond to the polyphenolic compounds present in the SI leaf extract [34], while as-prepared Cu$_2$O nanoparticles exhibited a broad absorption peak at between 240 to 380 nm having two $\lambda_{\text{max}}$ at 255 and 360 nm (green line), respectively [39,40]. The change observed in the spectrum after the reduction of Cu$^{2+}$ ions to Cu$_2$O nanoparticles corresponds to the formation of Cu-phytochemicals, complex or spherical Cu$_2$O nanoparticles with size 2–5 nm and quite stable in the shape-position of absorption after one month, indirectly indicating the stability of the nanoparticles [39,40].

![Figure 2. UV-vis spectra of SI leaf extract (red line) and Cu$_2$O nanoparticles (green line). Visual picture of solution containing (a) SI leaf extract and (b) Cu$_2$O nanoparticles.](image)

3.2. TEM and SAED Analysis

The exact shape and size of the nanoparticles was determined using a TEM image. The high-(Figure 3a) and low- (Figure 3d) magnification TEM images showed a good dispersion with the spherical morphology of Cu$_2$O nanoparticles inside the SI leaf extract. The majority of the nanoparticles observed from the TEM micrograph are small, with a size of 6–10 nm, indicating the availability of high surface catalytic activity for Cu$_2$O nanoparticles; a small few of the Cu$_2$O nanoparticles are of a bigger size, around 20–45 nm. This may be due to the presence of excessive SI phytochemicals on the surface of the Cu$_2$O nanoparticles, which can cause the aggregation and enlargement of nanoparticles during the synthesis of Cu$_2$O nanoparticles. The size distribution pattern of the nanoparticles observed in the TEM image (Figure 3a) was analyzed manually using ImageJ software. It showed the average size of the Cu$_2$O nanoparticles to be 6.67 ± 3.96 nm (Figure 3b). No aggregation of nanoparticles suggested the presence of hydrophobic coating around the Cu$_2$O nanoparticles. The SAED pattern of the Cu$_2$O nanoparticles (Figure 3c) shows partial concentric rings which signify that the particles are spherical shape, semicrystalline nature and indexed as (111) and (200) lattice planes for the face-centred cubic (FCC) structure of Cu$_2$O nanoparticles [16].
Figure 3. (a,d) TEM (transmission electron microscope) images of Cu$_2$O nanoparticles synthesized in solution, (b) size distribution pattern and (c) SAED pattern.

3.3. DLS Analysis

The dynamic light scattering (DLS) technique gives valuable information about the hydrodynamic size distribution of the Cu$_2$O nanoparticles (Figure 4). The average width of the Cu$_2$O nanoparticles was found to be 8.2 ± 2.1 nm with polydispersity index (PDI) = 0.0656. The observed PDI < 0.1 clearly indicates that the synthesized Cu$_2$O nanoparticles were monodispersed in nature [29]. However, the average size of as-synthesized Cu$_2$O nanoparticles determined by the DLS method was slightly higher than in the TEM analysis. This is due to either the presence of organic coating around nanoparticles or the screening of small particles by bigger ones [36,41].
3.4. XRD Analysis

In order to clarify the nature and crystallinity of the Cu$_2$O nanoparticles, XRD analysis was performed as shown in Figure 5. The presence of intense peaks at $2\theta = 36.46$ and $42.45^\circ$ represents the (111) and (200) planes. The XRD reflections of Cu$_2$O match that of Inorganic Crystal Structure Database ICDD (Inorganic Crystal Structure Database) no. 98-018-0846 corresponding to the semi-crystalline cubic FCC structure, which match the common peaks in the earlier report [42]. It can be also observed that the undesired peak at $2\theta = 32^\circ$ is due to a typical impurity caused by a metallo-organic structure [23,24]. It is likely that these peaks indicate that the phytochemicals in SI leaf extract attached to the Cu$_2$O nanoparticles and were also involved with surface-capped nanoparticles. However, the observed size of the nanoparticles in the XRD using the Scherrer formula (~46 nm) is bigger than in the TEM-DLS results [40]; this may be due to the aggregation of particles during drying for the preparation of XRD samples. This increment could be attributed to the light scattering effect because of the aggregation of the metal [43].

3.5. FTIR Analysis

FTIR spectra of the particles were recorded to detect the chemical interaction between SI leaf extract and Cu$^{2+}$ that occurs during the synthesis of Cu$_2$O nanoparticles (Figure 6). They reveal a weak absorption band at 698 cm$^{-1}$ may correspond to the Cu-O bond vibrational frequencies, which are slightly higher than reported Cu–O (645 cm$^{-1}$) bond vibration due to depending on the degree of hydrogen bonding. Furthermore, the additional Cu–O–H bonds lead to bending absorptions in the
The peaks at around 300 nm correspond to the absorption of the benzene ring and the peaks between pseudo-first-order kinetics. The result indicates that the observed rate constant \(k\) and correlation coefficient \(R^2\) for the MB degradation were \(21.9106 \times 10^{-3}\) min\(^{-1}\) and 0.9999. Hence, the present study highlights the promising potential of SI leaf-synthesized Cu\(_2\)O nanoparticles for MB degradation in wastewater.

**Figure 6.** (a) FTIR spectrum of as-synthesized Cu\(_2\)O nanoparticles using SI leaf extract and (b) specific Cu–O at 698 cm\(^{-1}\).

### 3.6. Photocatalytic Activity

Figure 7a shows the photocatalytic activity of Cu\(_2\)O nanoparticles in the degradation of MB. The peaks at around 300 nm correspond to the absorption of the benzene ring and the peaks between 600–700 nm represent the absorption of heteropoly aromatic linkage of MB [45]. It can be seen that the degradation of MB increases with increasing solar irradiation time by observing the decrease in \(\lambda_{\text{max}}\) at 664 nm [35]. The photodegradation percentages \(\eta\) of MB for 30, 75 and 150 min are 25.45\%, 59.88\% and 78.90\% in the presence of Cu\(_2\)O nanoparticles when compared with that of the respective controls. No change of absorbance at \(\lambda_{\text{max}} = 664 \text{ nm}\) was observed when a blank test was performed with MB and SI leaf extract. This confirms that the SI leaf extract does not interfere with the reduction of MB. In Figure 7b, the plots of \(\ln (A_0/A_t)\) versus time yield good linear correlations and are well fitted with pseudo-first-order kinetics. The result indicates that the observed rate constant \(k\) and correlation coefficient \(R^2\) for the MB degradation were \(21.9106 \times 10^{-3}\) min\(^{-1}\) and 0.9999. Hence, the present study highlights the promising potential of SI leaf-synthesized Cu\(_2\)O nanoparticles for MB degradation in wastewater.
Hence, the present study highlights the promising potential of SI leaf-synthesized Cu\textsubscript{2}O nanoparticles for MB degradation in wastewater.

4. Conclusions

In conclusion, the use of SI leaf as a bioreducant for the synthesis of Cu\textsubscript{2}O nanoparticles is a low-cost, simple and ecofriendly approach. The results obtained from DLS, TEM, XRD and FTIR suggest that the Cu\textsubscript{2}O nanoparticles are highly dispersed, spherical, 6–10 nm in size, semicrystalline and surface-capped with SI leaf extract. The applied approach provided a Cu\textsubscript{2}O nanocatalyst, which presented very good catalytic performance for the degradation of MB under sunlight.

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Conflicts of Interest: The authors declare no conflict of interest.

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