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Microwave-Sample-Preparation-System-Assisted Biogenic Synthesis of Copper Oxide Nanoplates Using *Saussurea costus* Root Aqueous Extract and Its Environmental Catalytic Activity

Amel Taha [1,2]

[1] Department of Chemistry, College of Science, King Faisal University, P.O. Box 400, Hufof 31982, Al-Hassa, Saudi Arabia; ataha@kfu.edu.sa
[2] Department of Chemistry, Faculty of Science and Technology, Al-Neelain University, Khartoum 11121, Sudan

Abstract: Using safe and environmentally benign materials is considered one of the green chemistry approaches to avoid waste production. This research reported the biogenic synthesis of CuO nanoplates using *Saussurea costus* root extract assisted by a microwave sample preparation system. The phytochemical contents in the *Saussurea costus* root aqueous extract work as the reducing and capping agents for the nanoparticles. The biosynthesized CuO nanoplates were analyzed using UV–Vis spectroscopy, FT-IR, XRD, HR-TEM, DLS, FESEM, and EDS techniques. According to the HR-TEM and FE-SEM results, the CuO nanoparticles exhibited a plate-like shape with a mean size of 29 nm. Furthermore, the XRD results showed a typical agreement with the pattern of the monoclinic phase of copper oxide. The catalytic efficiency of the CuO nanoplates in the reduction of 4-nitrophenol to 4-aminophenol using $NaBH_4$ was examined in terms of environmental catalytic activity. The reaction time took less than 10 min. Thus, CuO nanoplates synthesized via *Saussurea costus* root aqueous extract show high catalytic-activity potential for the environmental catalytic application of the removal of nitro pollutants.

Keywords: Cu nanoplates; microwave system; biogenic synthesis; *Saussurea costus*; catalytic activity

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1. Introduction

Nitrophenols are considered important intermediate compounds for numerous pharmaceuticals, petrochemicals, pesticides, insecticides, preservatives, dyes, and leather industries [1]. However, the derivatives of nitro compounds such as 4-nitrophenol (4-NP) have been considered dangerous pollutants and their degradation to nondangerous products is difficult since they have high stability and low solubility in water. Therefore, it is necessary to find alternative methods for removing these pollutants before they can be released into the environment, and working on developing materials with high ability in 4-NP reduction is highly required [2]. There is a need to find effective and eco-friendly metal nanocatalysts for the environmental dropping of these organic contaminants [3–5]. At the nanoscale, CuO exhibits a unique structure and properties among mono-metal oxide. Furthermore, it can be used potentially in gas sensors, catalytically in dye removal, in the formulation of pesticides, and as antibacterial agents in the inhabitation of pathogenic bacteria [6–8]. There are numerous approaches for the preparation of nanomaterials with size control, different morphology, and a highly crystalline nature; the literature has reported various physical and chemical methods that have been used for the synthesis of nanomaterials including electrochemical synthesis [9], the sol–gel method [10], and sonochemical synthesis [11]. Nevertheless, many of these synthetic strategies involve the use of hazardous chemicals and harsh reaction conditions which may lead to environmental concerns. Thus, it is important to find alternative green processes for metal nanoparticle synthesis through plant extracts [12,13], bacteria [14,15], and fungi [16]. Among these, plant extracts may offer a better alternative over microorganisms for metal nanoparticle synthesis since they

have a low cost, are abundantly available, can be handled safely, and their phytochemical content works both as a reducing and capping agent [17]. Additionally, the high cost of preparation of cell culture will be reduced. The assistance of microwave energy for the biogenic synthesis of metal nanoparticles by plant extracts is considered a viable and new route for the facile biogenic synthesis of metal nanoparticles. It shows many advantages such as fast reaction time, low energy consumption, and high yield [18]. The microwave digestion system is uniform as a heat medium of reaction so it provides good conditions for homogeneous nucleation and growth of metal nanoparticles. Siby Joseph et al. used microwave radiation for the synthesis of Ag and Au nanoparticles via *aserverlanata* leaf extract [18]. Theophil Anand and co-workers prepared copper oxide nanoparticles using a microwave-assisted method with *Moringa oleifera* leaf extract [19]. Mamatha Susan et al. reported the formation of hexagonal gold nanoparticles using *Myristica fragrans* extract assisted by microwave irradiation [20].

Saussurea costus belongs to the family Asteraceae which includes near to 1000 generations that are distributed widely in different regions in the world [21]. Nevertheless, different species are grown in India (Pandey et al., 2007), Pakistan, and the Himalayas [22]. *Saussurea costus* is also well-known in Arab countries and has been used in Islamic medicine. It is known as "Al-Kost Al-Hindi". *Saussurea costus* is an effective medicinal plant used in folk medicine for the treatment of different diseases, e.g., asthma, inflammatory diseases, fever, microbial infections, and stomach problems [23]. There are many bioactive phytoconstituents in the *Saussurea costus* root extract such as polyphenols, sesquiterpene lactones, alkaloids, triterpenes, lignans, and tannins as the main bioactive constituents [24]. These active constituents in *Saussurea costus* root are reported to exhibit efficient anti-inflammatory, anticancer, antioxidative, antiulcer, and hypoglycemic activity properties. *Saussurea costus* plant extract is used for the synthesis of the oxides of the silver [25], magnesium [22], selenium [23], and zinc [26], but there is no literature available on the synthesis of CuO nanoparticles. In light of the above, with consideration of the importance of the chemical and biological constituents of *Saussurea costus* root extract, herein, we report the biogenic synthesis of CuO nanoparticles assisted by a microwave sample preparation system using *Saussurea costus* root extract as a new and fast technique for metal nanoparticle synthesis, and its environmental catalytic activity in the reduction of P-nitrophenol was examined.

2. Results

2.1. Characterization

The initial formation of copper oxide nanoparticles was visually detected through the changing in color and rapidly confirmed using UV-Vis spectroscopy as shown in Figure 1. When the *Saussurea costus* root extract was added to the solution of copper chloride, an immediate change in color was observed in a short period from a blue color to dark brown, demonstrating CuO nanoparticle formation. The UV-Vis spectrum of copper oxide nanoparticles shows a typical absorption peak at 286 nm [27].

The FT-IR spectra of the CuO nanoplate records were carried out in the range of 400–4000 cm^{-1}. The FTIR spectrum of *Saussurea costus* root extract in Figure 2a shows a broad peak at 3155–3406 cm^{-1} which may correspond to aromatic and aliphatic OH groups present in the alkaloid in the plant extract [27,28]. The peak at 2936 cm^{-1} represents the strong stretching vibrations of C-H of amines. The sharp peak at around 1586–1669 cm^{-1} is assigned to the existence of carbonyl groups or bending of the aromatic. The band at 1004–1036 cm^{-1} corresponds to the stretching of C-O. The FTIR spectrum of the CuO nanoplates in Figure 2b shows the repeating of all function groups of *Saussurea costus* root extract and also the presence of a sharp peak between 400 and 600 cm^{-1} assigned to the stretching frequency of metal–oxygen binding [29–33].

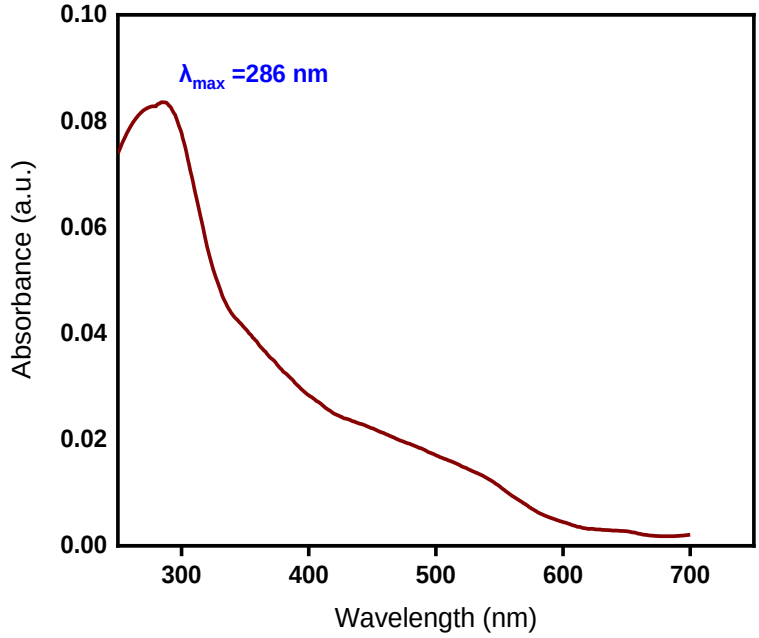


Figure 1. UV-Vis Spectrum of CuO nanoplates.

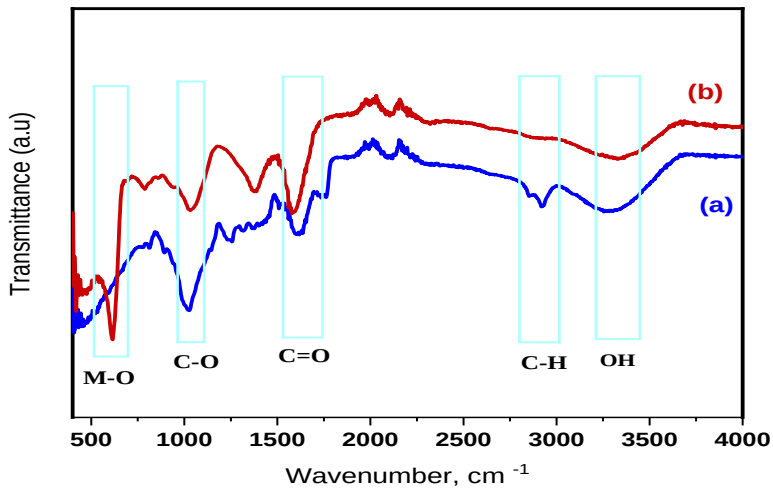


Figure 2. FTIR spectrum of (**a**) *Saussurea costus* root extract and (**b**) CuO nanoplates.

XRD analysis was conducted to attain information about the crystalline nature of the CuO nanoplates. The X-ray diffraction spectrum of the synthesized CuO is shown in Figure 3. It shows characteristic diffraction peaks at 2θ of 32.52, 35.44, 38.68, 48.76, 53.56, 58.18, 61.48, 66.12, 67.84, 72.02, and 74.96, which were assigned to (110), (11-1), (111), (20-2), (202), (113), (022), (220), (31-2), and (004) planes, respectively. These values matched with (JCPDS card no. 96-901-5925) of the CuO monoclinic phase [34].

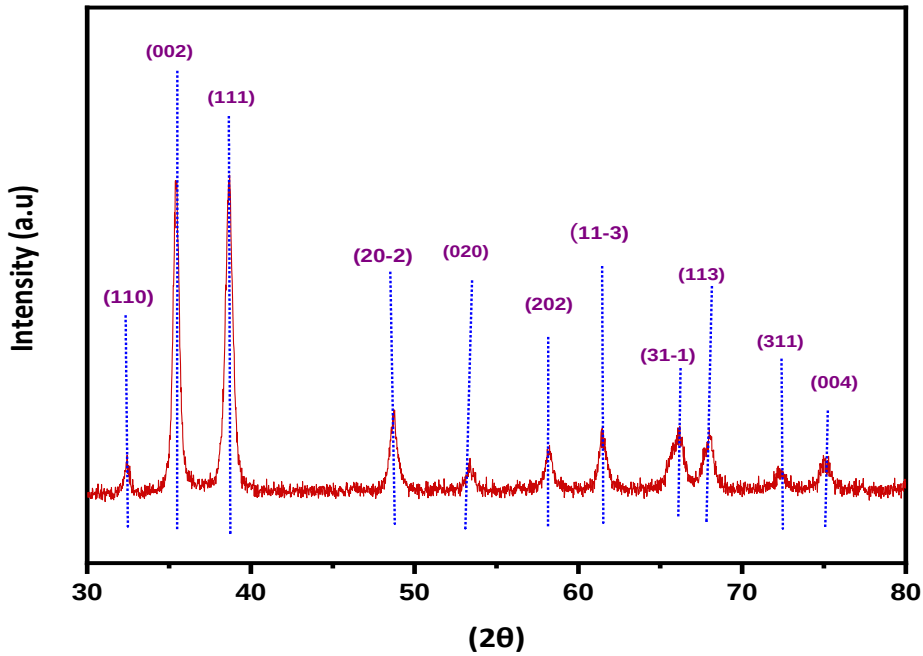
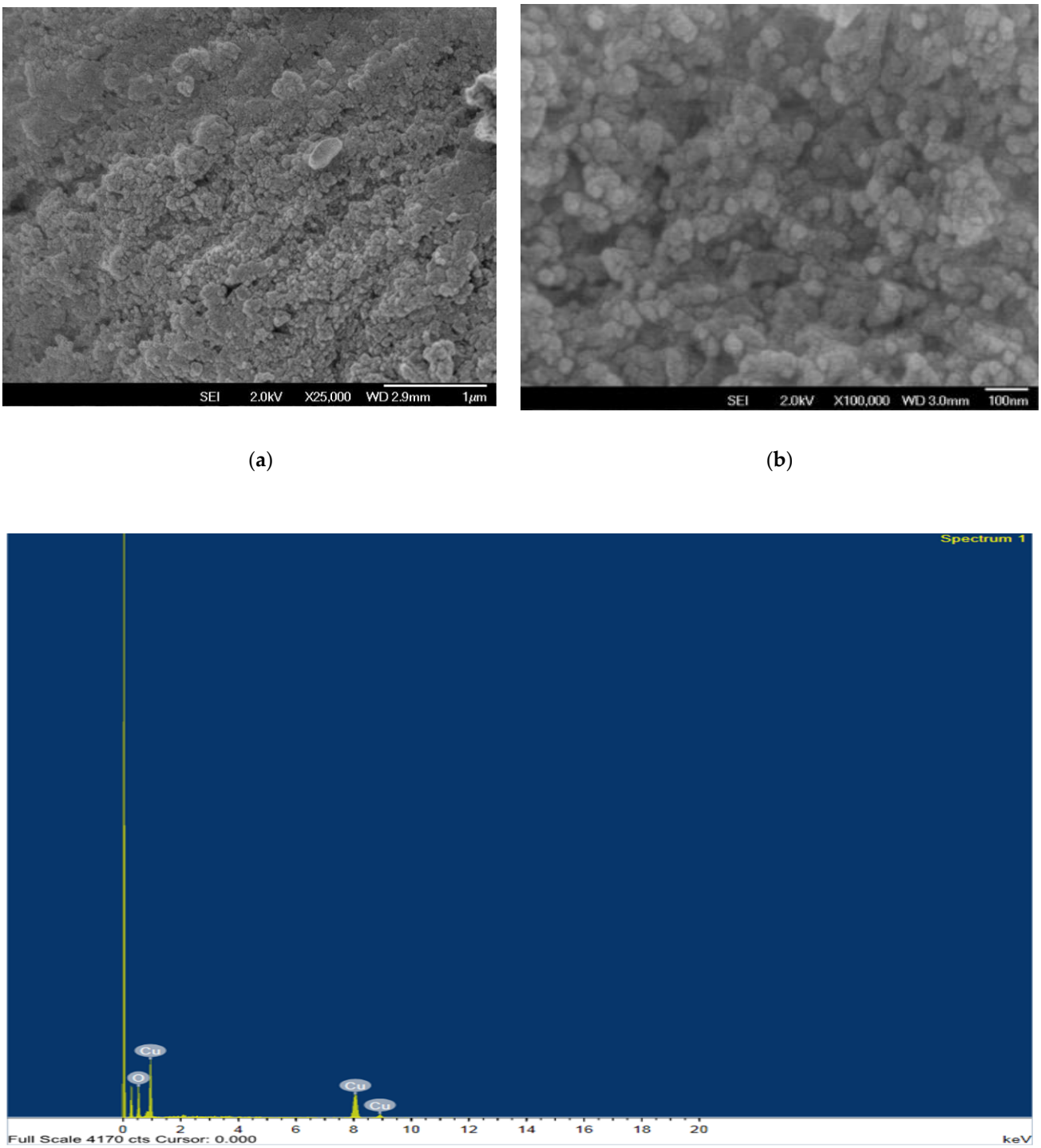


Figure 3. XRD pattern of bio-synthesized CuO nanoplates.

The surface morphology of the synthesized sample was investigated using scanning electron microscopy. Figure 4a,b show the scanning electron microscopy (FESEM) images of the biogenic CuO nanoplates prepared using *Saussurea costus* root extract assisted by the microwave at different magnifications. We found that the CuO sample had a plate-like structure and was uniform and well-defined. Aklilu Guale Bekru et al. reported the formation of a copper oxide nanocluster by Cordia africana Lam extract [35,36]. *M. cochinchinensis* and *Abutilon indicum* leaf extracts were used for the synthesis of spherical CuO nanoparticles [37,38]. In these studies, it was noticed that the different plant extract led to the formation of different geometry in the CuO nanoparticles. Figure 4c reveals the (EDS) characterization of the CuO. The graph confirmed the presence of only Cu and O elements. So, the active components of the *Saussurea costus* extract were involved in the formation of CuO nanoplates and no presence of any other peak confirms the high purity of the synthesized sample [19].

The high-resolution transition electron microscopy (HRTEM) images in Figure 5a show a well-dispersed and highly collected form of plate morphology for the synthesized CuO. The size of the synthesized sample ranged from 20 to 60 nm with a mean size of 29 nm (ImageJ software was used to measure the particle size; Figure 5b. The SAED images of the CuO nanoplates show the reflected concentric rings Figure 5c which prove the formation of crystalline materials. Dynamic light scattering (DLS) characterization was carried out to find the metal size and surface surrounding the nanoparticles. Figure 5d shows the biogenically synthesized copper oxide nanoparticles' average particle size distribution was 400 nm. It shows that the addition of *Saussurea costus* root extract plays a significant role in the agglomeration reduction and also controls the growth during the microwave process.



(**a**) (**b**)

(**c**)

Figure 4. (**a**,**b**) FESEM images of bio- synthesized CuO nanoplates at different magnification and (**c**) Energy-Dispersive Spectroscopy (EDS) for Cu Nanoplates.

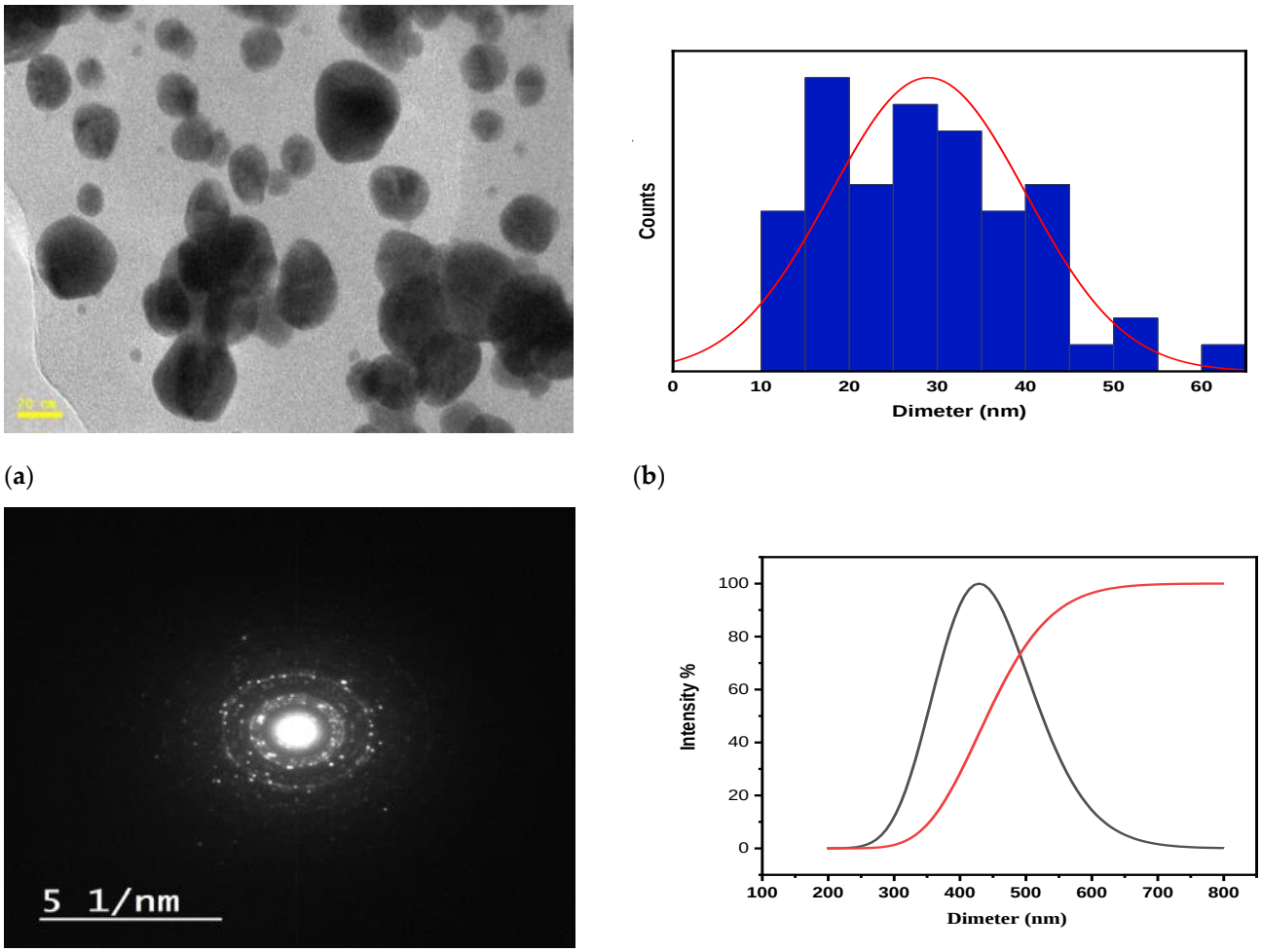


Figure 5. (**a**) HRTEM images of Cu nanoplates at different magnifications, (**b**) particle size distribution calculated from TEM, (**c**) SEAD pattern of nanoparticles, and (**d**) DLS particle size distribution.

2.2. Mechanism of CuO Nanoplate Synthesis

Plant extracts have a variety of phytochemical constituents; some of these constituents are responsible for nanoparticle synthesis. Important function groups have been reported as the main functional groups responsible for the reduction and stabilization of nanoparticles and these functional groups are existing in plant metabolites, such as flavonoids, alkaloids, carbohydrates, polyphenols, and proteins [35]. The CuO nanoplate synthesis mechanism can be summarized as: The polyphenols present in *Saussurea costus* bind with Cu^{2+} ions and form metal complexes, reducing it into Cu. In the second step, when the mixture is transferred to the microwave sample preparation system, it undergoes direct decomposition to form CuO through the heating process; then, the formed CuO seed particles undergo aggregation, followed by further growth of nucleation. A graphical representation of the mechanism of CuO nanoplate synthesis is presented in Figure 6. Buazar et al. and Zahrah Alhalili suggested similar mechanisms for nanoparticle formation [39,40].

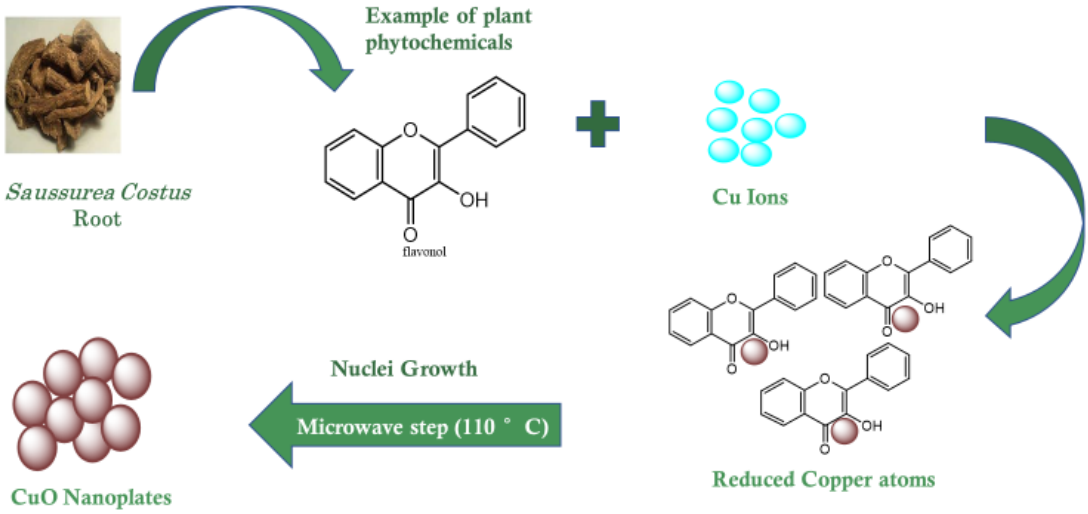


Figure 6. Possible mechanism of formation of CuO nanoplates using *Saussurea costus* root extract.

2.3. Catalytic Reaction

The performance of CuO nanoplate samples, prepared using *Saussurea costus* root extract assisted by a microwave sample preparation system, as a catalyst in 4-NP reduction in the presence of NaBH4 was investigated. Figure 7a demonstrates that the 4-nitrophenol was reduced into 4-aminophenol. UV-Vis spectroscopy was used to monitor the reduction process. The 4-NP displayed a peak at around 318 nm in the absence of $NaBH_4$. However, after the addition of $NaBH_4$, a change in the 4-NP solution was noticed—to the darker yellow of the p-nitrophenolate ion—which was shown by a sharp absorption peak around 402 nm. After the addition of the CuO nanoplate catalyst, a gradual decrease in the peak at 402 nm was observed while another peak appeared at 300 nm, representative of the p-nitrophenolate ion conversion into p-aminophenol, as shown in Figure 7b. The complete conversion of 4-nitrophenol into 4-aminophenol was measured in the presence of the CuO catalyst based on the disappearance of the peak at around 402 nm [33,34]. The time for the whole conversion of 4-nitrophenol into aminophenol in the presence of the CuO catalyst was 10 min.

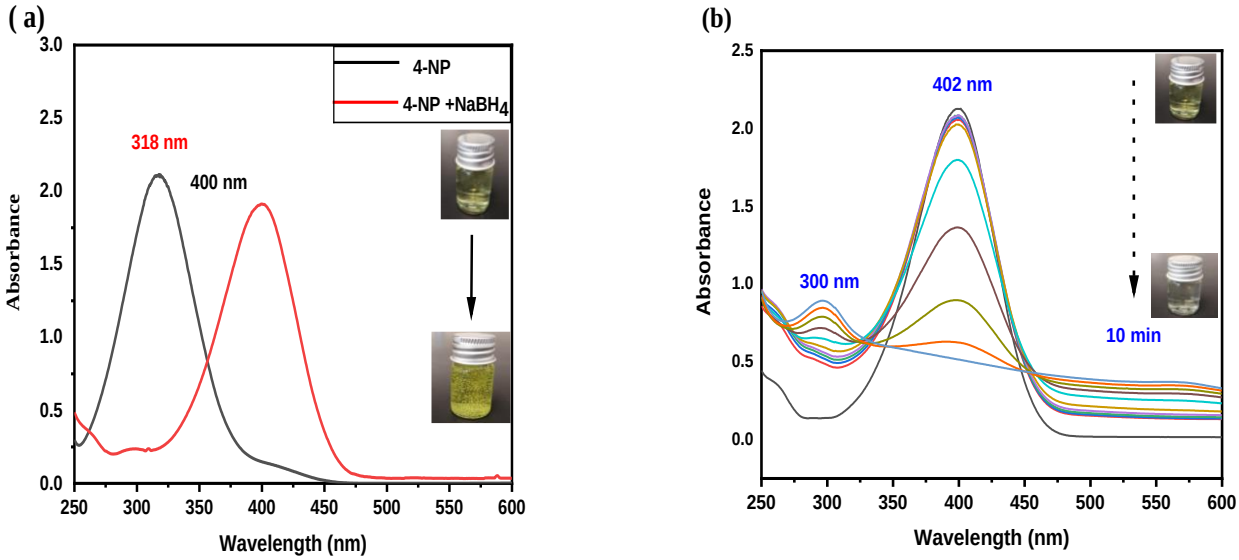


Figure 7. (**a**) Absorption spectra of 4-NP before and after the addition of aqueous $NaBH_4$ solution, (**b**) reduction of 4-NP with $NaBH_4$ in the presence of a CuO nanoplate catalyst.

Table 1 shows a comparison of the catalytic performance of the present nanocatalyst with previously reported CuO-based nanocatalysts, prepared via different methods, in 4-NP reduction. The results indicated that the CuO NPs synthesized using *Saussurea costus* root extract assisted by the microwave sample preparation system exhibited a faster reduction time than that reported for a Cu nanocluster, CuO nanoleaves, CuO@C, and spherical CuO nanoparticles [31,34,39–41].

Table 1. Comparison of the catalytic activity of CuO NPs with the previously reported CuO-based catalysts for the reduction of 4-NP.

Catalyst	Preparation Method	Time (min)	Reference
CuO nanocluster	Microwave-assisted leaf extract and coprecipitation method	12	[31]
CuO nanoleaves	NaOH using the microwave-heating method	15	[39]
CuO@C	Solvothermal method	18	[40]
Spherical CuO nanoparticles	Coprecipitation	11	[41]
Spherical CuO nanoparticles	Hydrothermal + leaf extract	14	[27]
Cu nanoplates	Microwave + leaf extract	10	*Current work*

The reusability tests for the CuO nanoplate catalyst were carried out through its repeated use (three times) in the 4-NP reduction. The percent reduction values for the reaction were calculated using Equation (1) below:

$$Reduction\ \% = \frac{C0 - C}{C0} = \frac{A0 - A}{A0} \quad (1)$$

and data were plotted as shown in Figure 8.

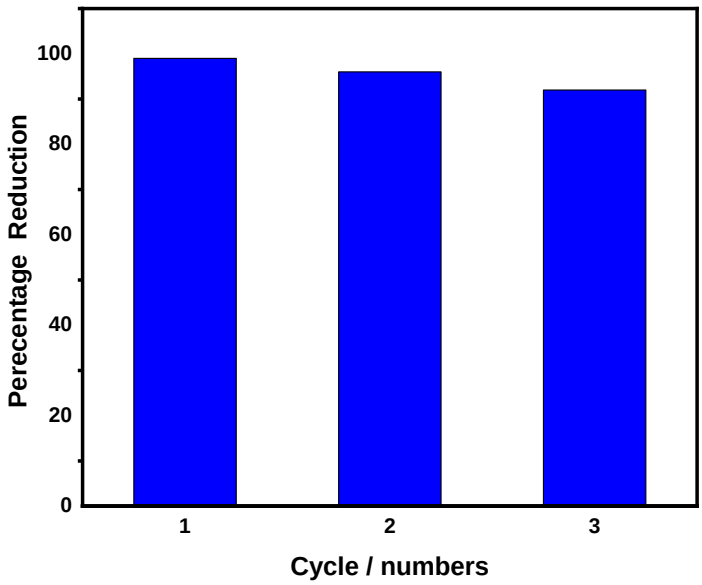


Figure 8. Efficiency of CuO nanoplate's multiple usage in 4-NP reduction.

2.4. Mechanism of the Reduction of 4-NP

The mechanism of the reduction of 4-NP using CuO nanoplates includes electron transfer from the $NaBH_4$ as a donor to the nanoplate catalyst's surface [20]. Due to the presence of the phytochemical components surrounding the nanosurface, all the reactant becomes much closer by means of electrostatic forces and enables electron relay to the

acceptor 4-nitrophenolate ions. Next, by gaining electrons, nitrophenolate ions are reduced as shown in Figure 7b.

3. Experimental Section

3.1. Materials

Copper chloride hexahydrate ($CuCl_2{\cdot}6H_2O$) was bought from Sigma-Aldrich (St. Louis, MO, USA). All chemicals used in this work were of analytical grade. All solutions were prepared with deionized water.

3.2. Saussurea costus Root Extraction

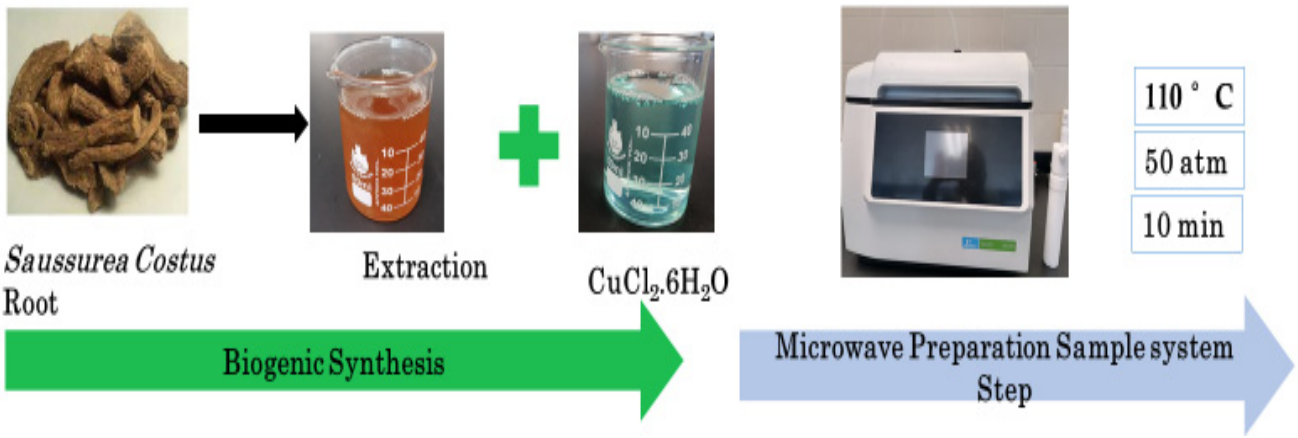
Saussurea costus roots were obtained from the local market in Hofuf, Saudi Arabia. The roots were washed three times using tap and distilled water, then dried at room temperature and ground to a fine powder. For extract preparation, 10 g of *Saussurea costus* root was added to 100 mL distilled water and boiled for 15 min. The extract was left to settle down at room temperature and filtered with Whatman No. 1 filter paper.

3.3. Biogenic Synthesis

A solution of copper (II) chloride hexahydrate ($CuCl_2{\cdot}6H_2O$ 5 mM) was prepared using deionized water in a 100 mL volumetric flask. For the biogenic synthesis of CuO nanoparticles, 20 mL of *Saussurea costus* root extract was added to the copper (II) chloride solution and it was vigorously stirred at 1000 rpm for 1 h at room temperature [40].

3.4. Microwave-Assisted Sample Preparation

A microwave sample preparation system was used to assist the biogenic synthesis of CuO nanoparticles. The prepared mixture was placed in the microwave vessels at a temperature of 110 °C and a pressure of 50 atm, and held for 10 min; then, it was cooled to room temperature. The obtained colloidal CuO nanoparticles were subjected to sonication for an hour in 100 mL distilled water. After that, the sample was left to settle down and the supernatant was disposed of. The sonication and settling down processes were repeated three times with water and one time with ethanol. The sonication was executed by using a Power sonic 405 ultrasonic bath to cleanse the samples [28]. The synthesized sample was oven-dried at 80 °C for 2 h. Scheme 1 shows the synthetic stages of the CuO nanoplates.



Scheme 1. The synthesis stages for CuO nanoplates, where they were prepared using biogenic synthesis assisted by the microwave method.

3.5. CuO Nanoplate Characterization

To verify the formation of CuO nanoplates, the UV-Vis spectrum was measured in the absorption wavelength ranging from 200 to 800 nm using a (UV 2450, Shimadzu, Kanagawa, Japan) spectrophotometer. FT-IR measurements for all samples were performed using an FT-IR spectrophotometer, model number (360) from Cary, South San Francisco, United States

of America. Field emission–scanning electron microscopy (FE-SEM) of the samples was conducted with a scanning electron microscope model (FEI, QUANTA FEG, 250) fitted with a high-angle, angular dark-field detector and an X-ray energy-dispersive spectroscopy system (EDS) was used to detect the morphology and purity of the particles. High-resolution transmission electron microscopy (HRTEM) images were collected using a model (JEOL JEM-2100, Tokyo, Japan). Samples were prepared through dispersion in ethanol and sonication for 30 min. On the carbon-coated copper grid (400 mesh), one drop of the sample suspension was placed and then dried at room temperature. The XRD analysis were performed on a DX-1000 X-ray diffractometer equipped with graphite-monochromatized Cu K α1 radiation (λ = 1.54056 Å) (DX-1000, Dandong Fangyuan Instrument Co. Ltd., Dandong City, Liaoning Province, China) to identify the crystalline phases. The nanoplates were prepared using the PerkinElmer microwave sample preparation system equipped with 16 low-volume perfluoroalkoxy (PFA)-lined vessels with safety rupture members (maximum operating pressure of 1380 KPa).

3.6. Catalytic Activity

The catalytic activity of the biogenically synthesized CuO nanoplates was examined by the reduction of p-nitrophenol in the presence of $NaBH_4$. The catalytic reduction was carried out as follows:

1. Mixing of 2.5 mL 4-NP (1×10^{-3} M) with a 0.5 mL $NaBH_4$ solution (1×10^{-3} M).
2. Additionally, a 0.5 mL solution of CuO nanoplates catalyst (400 ppm) was added to the prepared mixture in a quartz cell. The progress of the reaction was observed spectrophotometrically by following the absorbance decrease at λ_{max} of 400 nm [29,30]. The reusability of the catalyst was examined three times.

4. Conclusions

Copper oxide (CuO) nanoplates were successfully synthesized using *Saussurea costus* root extract assisted by microwave preparation sample system instruments as a new, green, eco-friendly, facile, and fast process for nanoparticle synthesis. The biogenically prepared CuO nanoplates had a monoclinic phase. Additionally, the EDS results confirmed that the synthesized material was composed of Cu and O elements. The average crystallite size of the nanoparticles was in the range of 10–60 nm. They showed high catalytic efficiency in the reduction of 4-NP with excellent reusability. The CuO nanoplates, synthesized using *Saussurea costus* root extract assisted by a microwave sample preparation system, displayed high capacity as a possible material for the reducing of other nitroamines and pollutant dyes in the environment.

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Data Availability Statement: Data only available upon request from the corresponding author.

Conflicts of Interest: The author declares that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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