

SYNTHESIS AND CHARACTERIZATION OF COPPER OXIDE NANOPARTICLES USING AQUEOUS EXTRACT OF IRANIAN VIOLACEAE FLOWER

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Abstract. In this work, we have synthesized copper oxide nanoparticles using Iranian violaceae flower extract and explored its biological activity. Green synthesis has emerged as a reliable, sustainable and ecofriendly protocol for synthesizing a wide range of nanomaterials and hybrid materials. In this paper, we report the synthesis of Copper oxide nanoparticles by a simple biological route using the extract of Iranian violaceae flower and CuSO₄ · 5 H₂O was used to synthesis the copper oxide Nanoparticles. The synthesized copper oxide nanoparticles were characterized using UV-visible spectroscopy, FTIR spectroscopy, FESEM, EDAX, and XRD techniques. UV –Visible analysis shows a characteristic peak around 266 nm for copper oxide nanoparticles and which is characteristic copper oxide nanoparticles. FTIR spectroscopy was used to characterize various capping and reducing agents present in the plant extract responsible for nanoparticle formation. The surface morphology was characterized using FESEM. The EDAX and XRD pattern suggested that prepared copper oxide nanoparticles were highly pure. The average particle size was calculated as 78.5 nm and α-copper oxide for all diffraction peaks (JCPDS card No. 41-1449) using the XRD technique. Our finding also support the synthesis of CuO NPs from Iranian violaceae flower sources due to relative abundance of plants for the production of reducing and stabilizing agents required for CuO NPs synthesis, potential efficiency of plant biomolecules in enhancing the toxicity effect of CuO NPs against microbes, prevention of environmental pollution due of nontoxic chemicals and degradation effectiveness of CuO NPs synthesized from plant sources. Furthermore, this study provides useful information on the rapid synthesis of CuO NPs with desired properties from plant extracts. Copper oxide NPs can have a good candidate for different applications.

Key words: Copper Oxide, Extract, Nanoparticles, Violaceae.

Introduction. Formulation of the problem

Today, nanotechnology has attracted a great attention in different fields. The nanoparticles have nanoscale dimensions in a range of 1–100 nm with unique properties due to small size and high surface area [1]. Metal oxide nanoparticles have received a research attention in many areas with unique and wide-ranging physicochemical properties [2]. copper oxide nanoparticle is a good candidate of metal oxide for various applications in preparation of nanostructures, photocatalyst, solid oxide fuel cell, gas sensor, catalyst for oxidation of hydrocarbons, catalytic performance for reduction, water purification, photovoltaic, biomedical, and antibacterial effect [3]. Various polymorphs of copper oxide oxide have included α-CuO Nps , β- CuO Nps , γ-CuO Nps, δ- CuO Nps, ε- CuO Nps, and ω- CuO Nps based on temperature. The

stable polymorph is monoclinic α- CuO Nps 3 in low temperature and cubic δ- CuO Nps in high temperature [4,5]. The increase of temperature caused the decrease of tetragonal β- CuO Nps structure and the show monoclinic α- CuO Nps in XRD patterns [6].

Analysis of recent research and publications

Copper oxide nanostructures can be fabricated by several methods such as solution, solution combustion, solvothermal, hydrothermal, laser ablation, microwave, so-gel, flame spray pyrolysis, thermal decomposition, electrodeposition, thermal oxidation, chemical vapor deposition, and green synthesis [3]. The green synthesis is a challenge for preparation of monodispersed nanoparticles with specific sizes and shapes [7]. Biosynthesis methods have more advantages than other classical synthesis procedures due to the easy availability, rich biodiversity, and eco-

friendly processes [8,9]. Green synthesis is very easy and cost-effective method for production of nanoparticles using the extract. The plant extract can act as reducing and capping agent for the reduction of metal ions and the formation of nanoparticles because of presence of the various biomolecules such as flavonoids, enzymes, proteins, phenolic acid, alkaloids, and terpenoids [10,11]. Silver nanoparticles were synthesized by *Mentha pulegium* (pennyroyal) leaf extract for antibacterial application [12,13]. The ultraviolet light can be caused the increase of risk for skin cancer and ocular damage. The UV radiation included three regions UV-A (320–400 nm), UV-B (280–320 nm), and UV-C (180–280 nm). The earth's atmosphere traps all UV-C and more than 99% of UV-B radiation. The UV-A blocking is the most important consideration for hazard prevention of exposure to direct sunlight [14]. Recently, CuO oxide was reported as the UV-absorber [5]. UV blocking ability is different in nanomaterial compared with bulk material because of small size and large surface area to volume ratio [15,16]. Antibacterial activity is another good application of CuO oxide nanoparticles against some pathogenic Gram-negative bacteria [17,18]

The purpose of this research was to synthesis and characterization of copper oxide nanoparticles using aqueous extract of Iranian *Violaceae* flower to achieve the following **objectives**:

- Green nanoparticle synthesis has been achieved using environmentally acceptable plant extract and ecofriendly reducing and capping agents. Plants are currently used for nanoparticle synthesis;
- Plants for synthesis of nanoparticles is rapid, low cost, eco-friendly, and a single-step method for biosynthesis process;
- Develop a novel approach for the green synthesis of copper oxide nanoparticles using Iranian *violaceae* flower extract aqueous which has been Less than 100 nm;
- Demonstrating of monoclinic α -copper oxide in XRD patterns that this type of structure is more stable because of small size and large surface area to volume ratio.

Research materials and methods

All the chemical reagents used in this experiment were of analytical grade purchased from Sigma Aldrich. The *Violaceae* plant was collected from Quchan herbarium, then it was washed with distilled water to remove the dust particles present on the plant, dried and powdered in a laboratory grinder (Mulinex, Iran).

Preparation of the Extract

The powder of *Violaceae* flower was weighed 5g and dissolved in 100ml of distilled water and allowed to boil at 50°C for 30 min, then it was cooled down to room temperature. The extract is filtered by whatmann paper No1. The filtrate was stored at 4 °C for experiments [19].

Synthesis of Plant CuO Nanoparticles

10 ml of extract was added to 100 ml of 1 mM aqueous CuSO 4.5H₂O solution. Reaction mixture stirred magnetically at room temperature for 12 hours. Afterwards, the mixture was treated with 1M sodium hydroxide drop by drop. Within a particular time, the color of solution was changed, which indicates the formation of copper oxide nanoparticles. Then the solution was centrifuged for 15 min at 12,000 rpm and dispersed in double distilled water to remove any unwanted biological materials. Then a brown powder was obtained after drying at 60°C in vacuum oven overnight [19].

Characterization of copper oxide Nanoparticles

UV-Visible spectroscopy

Ultraviolet visible spectroscopy analysis was done by using UV-visible spectrophotometer (Shimadzu 2100, Japan). UV spectral measurement was done in a range of wavelength between 250–480 nm [20].

Particle size analyzing

The size distribution and average particle size of copper nano particles (NPs) were obtained using particle size analyzer. The sizes of NPs were measured using dynamic light scattering (Vasco, U.K.) [20].

Fourier transform infrared spectroscopy

To determine the functional group present in the copper oxide nanoparticles, FTIR analysis was performed. Fourier transform infrared spectroscopy (FTIR) of extract were recorded on Perkin Elmer 1750 FTIR Spectrophotometer in a diffuse reflection mode, operating at a resolution of 4 cm⁻¹. The spectrum was recorded in the range between 4000 and 400 cm⁻¹ [20].

Scanning electron microscopy

Scanning electron microscopic (SEM) analysis was done using Hitachi S-4500 scanning electron microscopy. The copper oxide nanoparticle powder was dissolved in a suitable solvent and a thin film is prepared on the carbon coated copper grid. Excess solvent was removed by blotting paper then allowed the sample to stand for 5 min. The SEM images were recorded at 40, 000 X magnification operating at 20 kV [20].

XRD analysis

X-Ray diffraction (XRD) spectra of the copper oxide nanoparticle samples was done using Bruker AXSD8 (D8 FOCUS 2200 V Bruker AXS, Bruker Optik GmbH, Ettlingen, Germany), using Cu Ka radiation ($\lambda = 1.5418 \text{ \AA}$) with 2θ ranging from 20° to 80° [20].

Results of the research and their discussion

The properties of CuO nanoparticles

After 12 hours of reaction, the color of reaction mixture changes to dark color, indicating the synthesis of CuO nanoparticles due to the reduction of Cu⁺ ions and due to the excitation of Surface plasmon vibration in metal nanoparticles.

Optical characterization

UV-Visible absorption spectrum is the preliminary characterization to know the optical

property of synthesized nanoparticles. The reduction of copper nanoparticles was monitored by UV-spectrophotometer range of absorbance from 250–480 nm. The spectrum showed the absorbance peak at 266 nm corresponding to the characteristic band of copper oxide nanoparticles (Figure 1).

The absorption spectra at 266 nm suggest the formation of copper oxide nanoparticles. Gans modification of Mie theory predicts that the shift in the surface plasmon resonance occurs when the particles deviates from spherical geometry. The single sharp SPR band in the absorption spectra predicts the spherical nature of copper oxide nanoparticles and was confirmed by UV-Visible spectroscopy. These results are agreement with Guin et al. [20].

FTIR Analysis

FTIR spectra of biosynthesized copper oxide nanoparticles were recorded to identify of metal

nanoparticles. The FTIR spectrum of extract and synthesized CuO nanoparticles is shown in Figure 2. For copper oxide nanoparticles, peak values at 3450, 2932, 2890, 1619, 1404, 1097 cm^{-1} were observed. Peak at 1619 cm^{-1} corresponds to C=O stretching of amides and O-H stretching of phenolic compound respectively. The peak obtained in copper nanoparticle sample are 3450 cm^{-1} due to O-H Stretching of hydrogen bonded alcohols and phenols. Fig. 3 shows the FTIR results of extract. The spectra show frequency bands 3580 cm^{-1} , 2928 cm^{-1} , 1470 cm^{-1} and 1079 cm^{-1} which is also present in the IR spectra of copper oxide nanoparticles which confirm the presence of Phyto-constituents in the nanoparticles surface that may act as the capping agent.

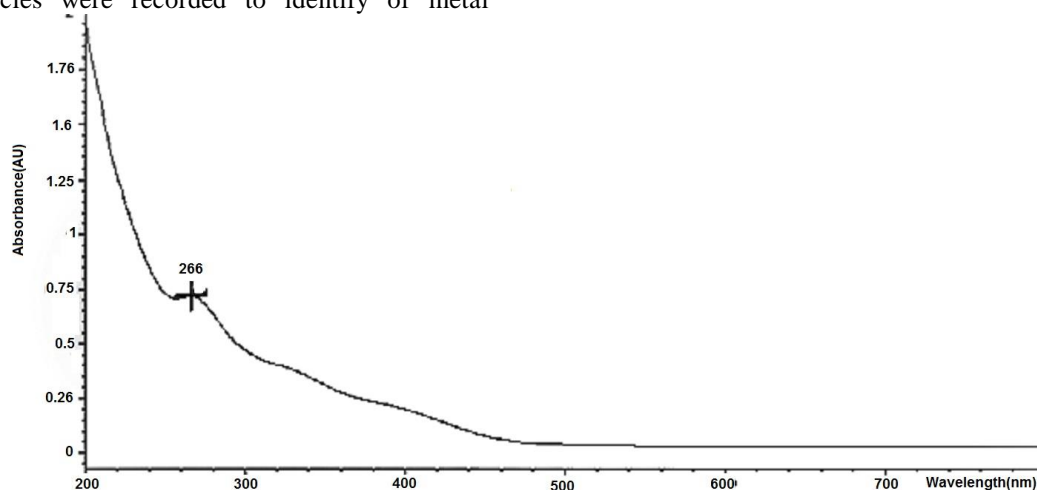


Figure 1. UV-visible spectra of Copper oxide nanoparticles synthesized using plant extract

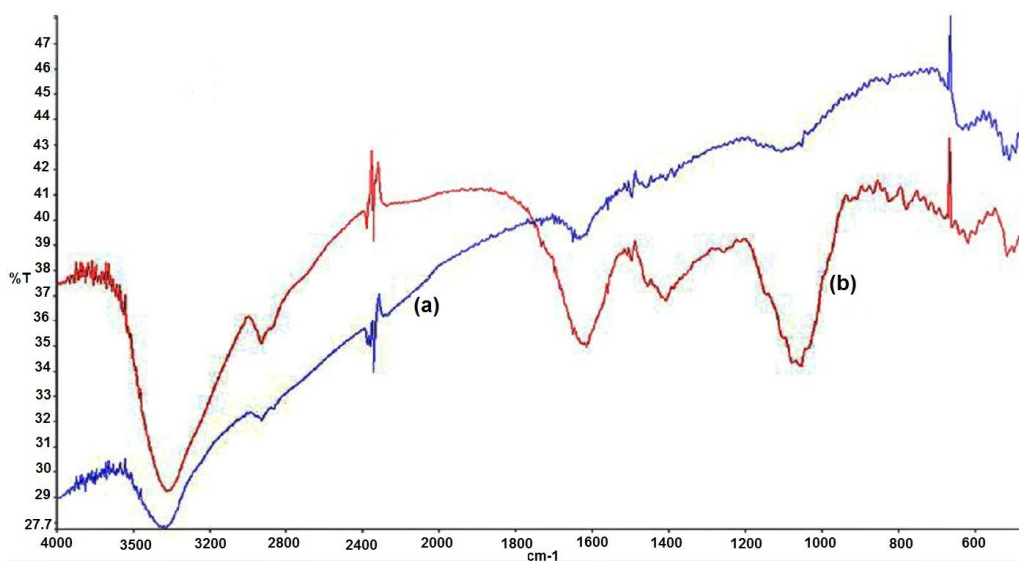


Figure 2. Fourier transform infrared spectroscopy (FTIR) spectra of (a) Iranian Violaceae flower extract (b) FTIR spectra of biosynthesized copper oxide nanoparticles

In order to understand the presence of different functional groups responsible for the synthesis of mono dispersed copper oxide nanoparticles, FTIR measurement was carried out. Swarnkar et al [21] described the spectra of copper oxide nanoparticles using desmodium gangeticum extract showed main frequency band 617 cm^{-1} corresponds to Copper oxide in Cu_2O phase. Also, the spectra show frequency bands 3746 cm^{-1} , 2928 cm^{-1} , 1400 cm^{-1} and 1079 cm^{-1} which is also present in the IR spectra of copper oxide nanoparticles which confirm the presence of DG phyto-constituents in the nanoparticles surface that may act as the capping agent [21]. Ethiraj and Kang [22] represents the FTIR spectrum recorded for the CuO nanowires in the range of 400 to 4,000 cm^{-1} . The three characteristic bands observed at 432.3 cm^{-1} , 497 cm^{-1} , and 603.3 cm^{-1} can be assigned to the Au mode, Bu mode, and the other Bu mode of CuO [23]. The high-frequency mode at 603.3 cm^{-1} may be attributed to the Cu-O stretching along the direction, while the peak at 497 cm^{-1} can be assigned to the Cu-O stretching vibration along the direction [24]. Moreover, no other IR active mode was observed in the range of 605 to 660 cm^{-1} , which totally rules out the existence of another phase, i.e., Cu_2O [25].

Particle size analyzing

The range of nanoparticles size measured by particle size analysis between 24–99 nm. The average particle sizes of the nanoparticles are 77 nm.

SEM Analysis

The morphology of the prepared nanoparticles was examined using scanning electron microscopy. Figure 3 shows the scanning electron microscopy of copper oxide nanoparticles synthesized by the plant extract of Iranian Violaceae flower is obtained from the proposed by bio-reduction method. The shape of the copper oxide nanoparticles was confirmed in the range of 1 μm , 2 μm and 200 nm.

In SEM analysis, the synthesized copper oxide nanoparticles of spherical shape show less aggregation of particles with diameters ranging from 24–99 nm with an average size of 77 nm. The morphology of the CuO samples without organic molecule thioglycerol (TG) shows the formation of CuO flowers consisting

of individual nanowires, whereas when the same synthesis is carried out in the presence of organic molecules TG, isolated CuO nanowires were obtained [24].

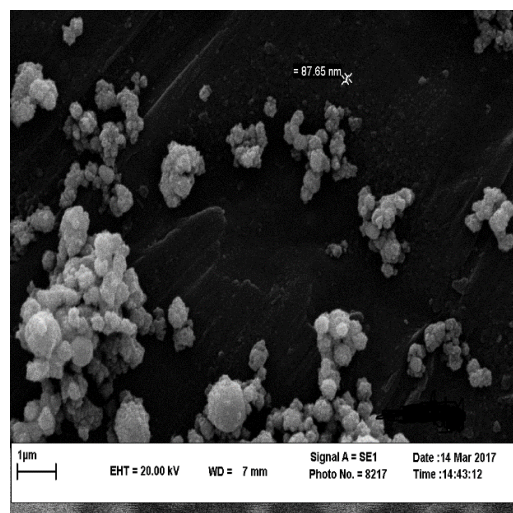


Figure 3. SEM images of the synthesized copper oxide nanoparticles

XRD Analysis

The dry powders of the copper oxide nanoparticles were used for XRD analysis. The diffracted intensities were recorded from 20° to 80° at 2theta angles. XRD values of 2θ showed peaks at 34.56°, 35.59°, 37.76°, 44.22°, 48.16°, 52.93°, 57.93°, and 73.44° which are comparable to JCPD Standards (Figure 4). The XRD pattern indicates that the copper Nanoparticles had a cubic structure. To determine the average particles size of the copper Nanoparticles, the Debye Sherrer's equation is used:

$$D = K\lambda / B \cos\theta$$

Where D is the particle size (nm), K is a constant equal to 0.94, λ is the wave length of X-ray radiation (1.5406 Å), β is the full-width at half maximum (FWHM) of the peak (in radians) and 2θ is the Bragg angle (degree). The average crystallite size was found to be in the range 78.5 nm.

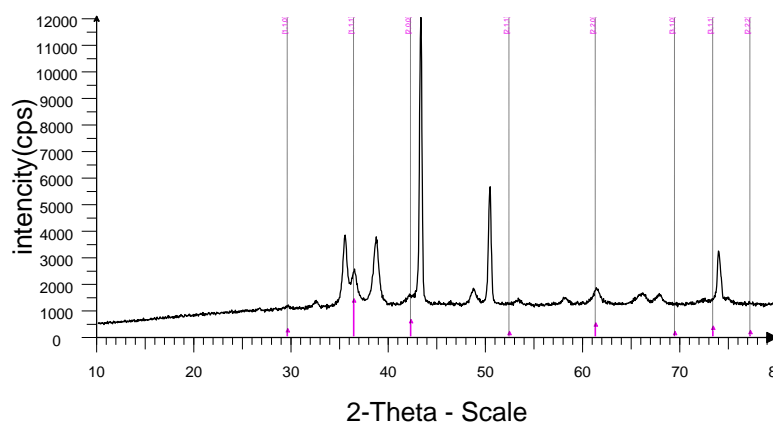


Figure 4. XRD pattern of synthesized copper oxide nanoparticles

The XRD spectrum of biosynthesized copper oxide nanoparticles indicates the cubic cuprous oxide (Cu_2O). The peaks observed in the spectrum with 2θ Bragg's angles of 34.56° , 35.59° , 37.76° , 44.22° , 48.16° , 52.93° , 57.93° , and 73.44° which are comparable to JCPD Standards. This confirms the presence of copper oxide nanoparticles with face centered cubic structure [26, 27]. These results agreed with the data reported for the Cu (2p) in CuO [28, 29, 30, 31].

TEM Analysis

Figure 5 shows the TEM image of CuO Nps. The TEM study is carried out to understand the

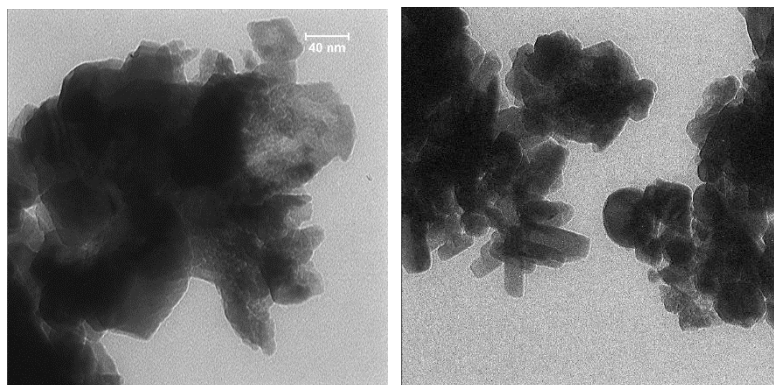


Figure 5. TEM image of CuO nanoparticles

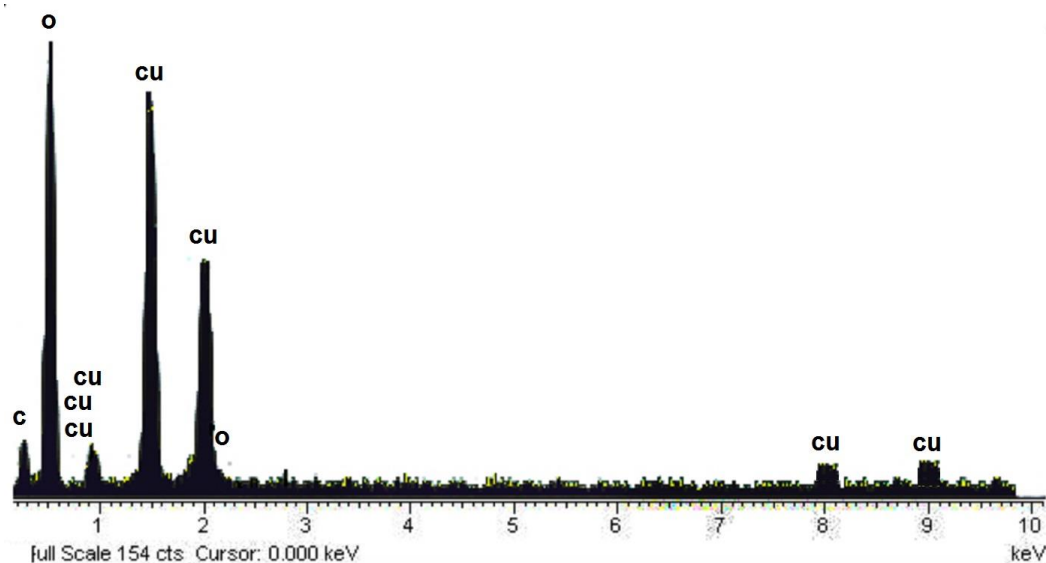


Figure 6. Energy dispersive X-ray (EDX) analysis of CuO-NPs

Conclusion

The copper oxide nanoparticles were prepared by green method using Iranian violaceae flower aqueous extract as a reducing agent. The XRD spectrum confirmed the monoclinic α -Copper oxide crystalline structure. The DLS, SEM and TEM results showed the

crystalline characteristics of the nanoparticles. The particles are observed to be cubic in shape.

X-ray (EDX) Analysis

Figure 6 showed the EDX analysis was carried out CuONPs at 10 keV. Results revealed the presence of copper (Cu) and oxygen (O) elements in CuO-NPs. Results of X-ray (EDX) analysis revealed the presence of copper (Cu) and oxygen (O) elements in copper oxide nanoparticles. Ethiraj and Kang [22] indicates the presence of copper [Cu], oxygen [O], and silicon [Si] in CuO.

size in nanometer scale with narrow distribution and the particle size was estimated 77 nm. In the present study, we successfully observed UV blocking and antibacterial activity applications of bismuth oxide NPs. These properties can be resulted in many advantages in the future with less harm and toxicity to the human health and more safety.

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