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CuO Nanoparticles: Synthesis and Characterization

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Abstract
In this paper, Copper oxide (CuO-NPs) was synthesized via the Chemical Co-Precipitation Method in the reaction temperature of 80°C, using copper acetate monohydrate [Cu (CH3CO2)2. H2O] & sodium hydroxide (NaOH) as a raw material and DI water (H2O) as solvent. The synthesized CuO nanoparticles were characterized by UV (Ultra violet) spectroscopy, Fourier transformed infrared (FTIR), Scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDX), and X-ray diffraction pattern (XRD) technique. The visible spectroscopy showed an absorption peak of CuONPs at 295nm. The composition of nanostructures and functional groups confirmed by analysis of FTIR spectra. The XRD patterns and EDX spectra showed that the prepared (CuO-NPs) were highly pure, crystalline and nano-sized with average size about 8.4nm. The SEM image suggested that nano-particles were spherical, and there was a tendency of agglomerations.

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1. Introduction

The field of nanotechnology is the science of producing and using nanoscale materials to create novel products and processes (Porter et al., 2008). Metal oxide nanomaterials exhibit unique thermal, magnetic, chemical, mechanical, electronic, catalytic, and optical properties compared to bulk concentrates (Khandagale & Shinde 2007; Xin et al., 2007). CuO nanoparticles have attracted great interest due to their potential applications in various fields, including electronic and optoelectronic devices such as microelectromechanical systems (Zhang et al., 2010), field effect transistors (Liao et al., 2009), electrochemical cells (Morales et al., 2005), and gas sensors (Cruccolin et al., 2004; Kattiet et al., 2003), magnetic storage media (Fan et al., 2004), solar energy conversion (Jess et al., 2009), field emitters (Hsieh et al., 2003) and quick-precipitation (Zhu et al., 2007; Rujun et al., 2010). Recently, it has also been emphasized that in addition to size, the shape of nanostructures is equally important in controlling various properties such as light absorption and catalytic activity of CuO nanostructures (Chen et al., 2007; Kimura et al., 2008). Up to now, there are some reports on the preparation of CuO nanomaterials such as nanowires (Kaur et al., 2006), fish bone-like nanostructures (Jia et al., 2009), nanorods (Yang et al., 2008), nanoplates (Zarate et al., 2007) and shuttle-like nanostructures (Zhang et al., 2006). Most of these nanostructures are randomly distributed. Chemical reaction methods are considered important among the various manufacturing methods due to the fact that they are safe and environmentally friendly, synthesis procedures are also carried out at relatively low temperatures (Li et al., 2005). In this study, Copper oxide nanoparticles (CuO-NPs) were synthesized by chemical solution-based co-precipitation, and their shape, size, and microstructure were investigated.

2. Materials & Methods

2.1. Synthesis of CuO NPs

Copper acetate monohydrate 0.05M [$Cu(CH_3CO_2)_2.H_2O$], & NaOH & DI water as a disseminated solvent were used to prepare CuO nanoparticles. To take the 1.0 gr of [$Cu(CH_3CO_2)_2$. H_2O] and dissolve in 100 ml DI water as solvent to get a certain molar concentration at room temperature. Then, obtained solution was magnetically stirred for 2 h, in the reaction temperature of 80°C**.** Afterwards; the 50 ml NaOH was added drop wise to the solution. Hence, nanoparticles of CuO were fabricated by chemical reaction as follow:

$$\mathbf{Cu\ (CH_3CO_2)_2 + 0.02\ NaOH = CuO + 2Na\ (CH_3CO_2)_2 + H_2O}$$

After the reaction was complete, the obtained lime black precipitate washed with DI water and filtration process, dried at oven 50 °C temperature for 24hr. Then, dried samples were calcined at 120 °C temperatures for 4hrs, to obtain CuO nanoparticles. Figure 1 shows the steps of (CuO) nanoparticle preparation by precipitation method.

Figure. 1: Synthesis of CuO NPs.

2.2. Characterization methods

The morphology, size & structure of Copper oxide nanoparticles were characterized by X-ray Diffraction Spectroscopy type (Shimadzu XRD-6000), Scanning Electron Microscopy SEM, (Model JSM-6700F), Energy dispersive spectrometer (EDS) that were used in the College of Chemical Sciences Kabardino-Balkarian State University (KBSU), Russia, and UV-visible spectroscopy that was used in the RFA Pharmaceutical industries, Hadhramout, at room-temperature. The FTIR spectrum of the prepared powdered sample was recorded in a wide range of wavenumbers 450- cm^{-1} to 4000 cm^{-1} by the FTIR spectrophotometer (Bruker, Berlin, Germany). Since the temperature difference between the four samples is slight and the results are close, the focus is on studying the characterization of the samples calcining at 120 ºC.

3. Results and discussion

3.1. X-Ray Diffraction (XRD) Analysis

The X-ray diffraction of CuO NPs shows, in Figure. 2, noticeable peaks at 2θ values of 33º, 36º, 39.5º, 49º, 54.2º, 59º, 62.5º and 67º, 68.8º corresponding to **(1 1 0), (0 0 2), (1 1 1), ($\mathbf{\bar{2}}$ 0 2), (0 2 0), (2 0 2), ($\mathbf{\bar{1}}$ 1 3), (3 1 1) and (113)** plane orientations, respectively (Dhineshbabu *et al.*, 2016). The formation of narrow peaks with the Bragg's angle suggests the crystalline nature of CuO nanoparticles. Using the Debye–Scherrer equation (Eq. 1), the average crystal

size of CuO NPs is calculated, it can be clearly seen that all the peaks in the XRD patterns are consistent agreement with the previously reported (Mote *et al.*, 2012).

$$D = K\lambda/\beta \cos\theta \quad (1)$$

In this formula, D is the average crystallite size, K shows the crystal shape factor (approximately 0.9), λ denotes the wavelength of the X-ray source (1.54A°), is the peak width at half maximum height (FWHM), and θ is the diffraction angle (Mote *et al.*, 2012).

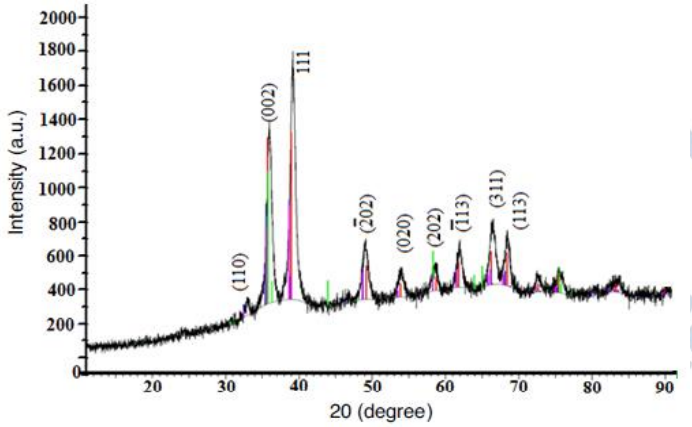

Figure. 2. XRD pattern of CuO NPs after calcinations.

3.2. SEM Analysis

Figure.3. represents the SEM image of CuO-NPs with magnification (Kx500). From the SEM image of CuO-NPs, it was observed that the particles are well-dispersed spherical, accompanying almost well-defined and uniform crystalline structure. There was also a higher tendency of agglomerations with average size between20µm to200µm which is consistent with previous study (Zhao *et al.*, 2022).

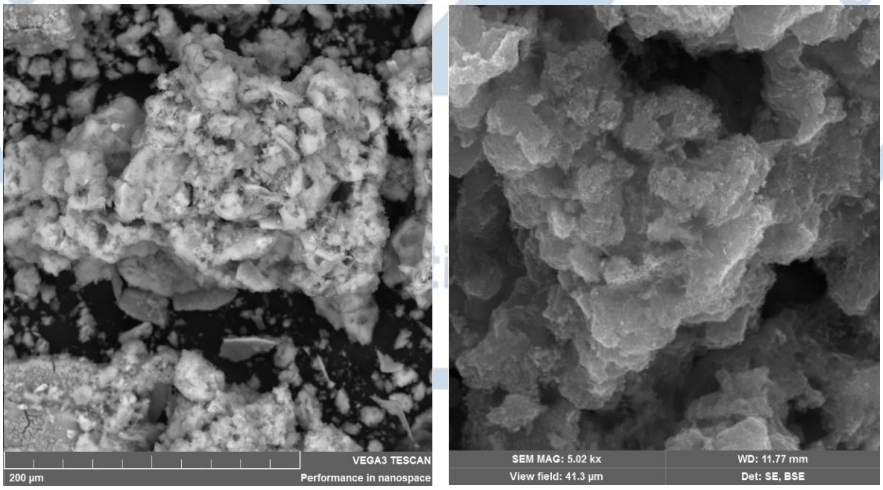

Figure. 3. SEM image of CuO-NPs.

3.3. EDX Analysis

The EDX analysis confirmed the chemical composition of the CuO NPs. Results indicate that the elements Cu and O. The K-series of Cu content were 80.3 at % while O content 19.7 at %. Spherically nano size was successfully produced due to the suitable selection of calcination temperature (Saleh *et al.,* 2023). The elemental analysis demonstrates that a synthesized CuO nanoparticles consists of Cu and O with a molecular ratio of 1:1. Table **1** shows the weight and atomic percentage of the constituents of the EDX-characteristic CuO NPs as shown in Figure. 4.

Table 1. EDAX analysis of element CuO

Element	Atomic Number	Normal Weight (%)	Atomic Weight (%)
Cu	29	80.3	51.12
O	8	19.7	48.88

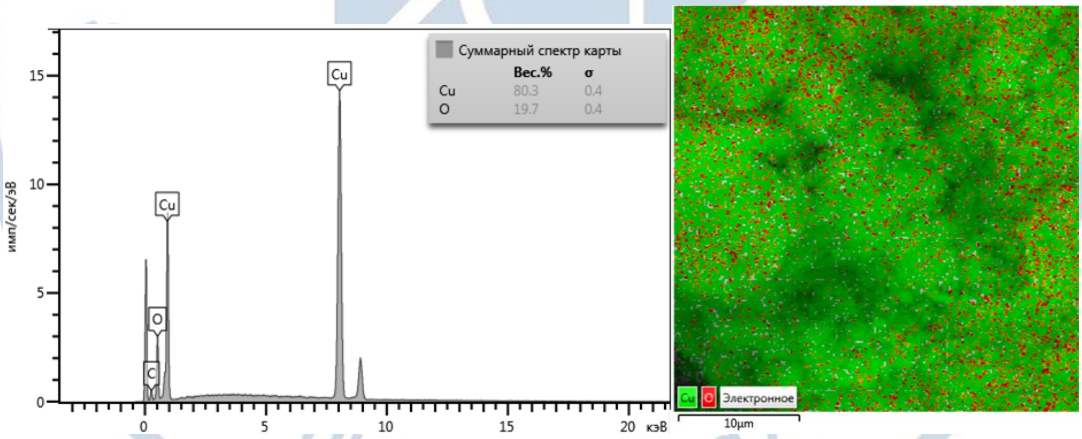

Figure. 4. EDX Analysis of CuO NPs.

3.4. UV-Vis Spectral Analysis

The synthesized CuO nanoparticles via the chemical precipitation method were characterized through UV–Vis spectra, that in the RFA Pharmaceutical industries, Hadhramout, at room-temperature. The absorption spectrum for all tested samples was acquired in the range from 200nm to 800 nm. In the present study, the synthesized CuO nanoparticles showed an absorption peak at 295 nm as shown in Figure. 5. This absorption peak is within the range of literature values (Sharma & Kumar 2020).

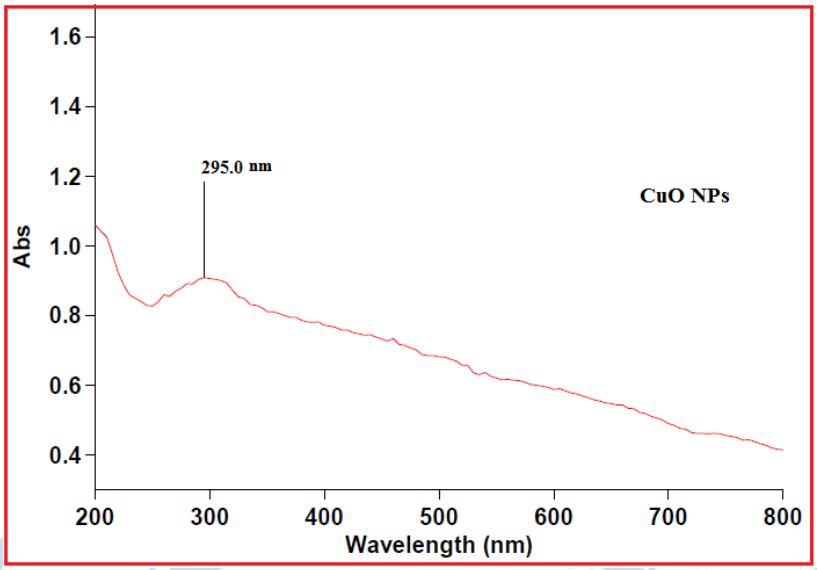

Figure 5: UV-visible analysis of CuO NPs.

3.5. FTIR Analysis of the CuO-NPs

The FT-IR spectra of the CuO NPs in Figure. 6 shows absorption peaks in the range 4000 to 450 cm^{-1} at room temp. Samples were prepared by mixing samples powder with KBr. The strong intensity peak at 3544. 73 cm^{-1} on the FT-IR spectrum is related to O-H bond. The absorption at 1421.27 cm^{-1} attributed to hydroxyl groups. The absorption bonds at 1343.57 cm^{-1} to 1047.93 cm^{-1} indicates the existence of carbonates and the bond at 2800 cm^{-1} correspond to C-H stretching mode (Dubal *et al.*, 2010; Phiwdang *et al.*, 2013). The absorption bonds at 465.12 and 506.35 cm^{-1} are associated to Cu-O vibration bond, but absorption bond at 677.05 cm^{-1} is assigned to Cu-O-H stretching bond. The above information confirmed the formation of pure CuO nanoparticles.

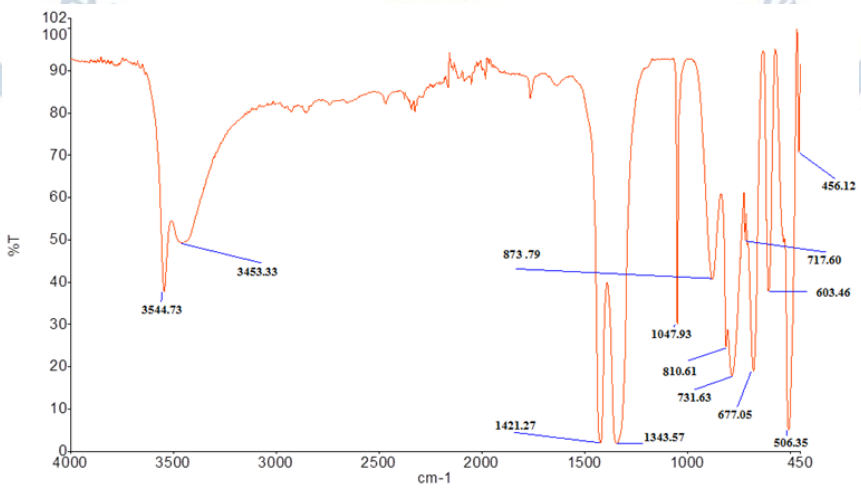

Figure. 6. FTIR spectrum of the CuO NPs.

4. Conclusions

Copper oxide nanoparticle have been successfully synthesized via the solution method in the reaction temperature of 80°C (using copper acetate & sodium hydroxide). The SEM analysis has shown that the synthesized NPs were spherical and homogeneous with average size of CuO NPs. The XRD results indicated that the sample has a high degree of crystallinity nature CuO NPs, and that their average size is found to be 8.4nm. The results based on XRD, SEM and EDX showed that the formation of CuO nanoparticles was confirmed and pure nanoparticles formed. The composition of nanostructures and functional groups confirmed by analysis of FTIR spectra. EDX analysis shows highest peak for copper and oxygen indicating that the CuO NPs synthesized is highly pure and contains no impurities.

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Conflicts of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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الجسيمات النانوية لأكسيد النحاسCuO: التوليف والتوصيف

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الملخص

في هذا البحث، تم تصنيع جزيئات أكسيد النحاس النانوية المحضرة بطريقة الترسيب الكيميائي المشترك في درجة حرارة تفاعل C800 درجة مئوية. باستخدام خلات النحاس [Cu (CH3COO)2. H2O] وهيدروكسيد الصوديوم (NaOH) كمادة خام وماء منزوع الايونات DI (H2O) كمذيب. تم تشخيص جزيئات أكسيد النحاس النانوية باستخدام التحليل الطيفي للأشعة فوق البنفسجية (UV) والأشعة تحت الحمراء المحولة فوريه (FTIR) والمجهر الإلكتروني الماسح (SEM) ومطياف الأشعة السينية المشتتة للطاقة (EDX) وتقنية نمط حيود الأشعة السينية (XRD). أظهر التحليل الطيفي المرئي ذروة امتصاص CuO NPs عند295 نانومتر. تم تأكيد تكوين الهياكل النانوية والمجموعات الوظيفية من خلال تحليل أطياف (FTIR). أظهرت أنماط ((XRD وأطياف (EDX) أن الجسيمات النانوية من أكسيد النحاس المحضرة كانت نقية للغاية ومتبلورة وحجمها نانوي بمتوسط حجم حوالي 8.4نانومتر. اظهرت صورة المجهر الإلكتروني الماسح أن الجسيمات النانوية كانت كروية وكان هناك ميل للتكتل.

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الكلمات المفتاحية

الجسيمات النانوية, طريقة الترسيب المشترك, UV-Vis, FTIR, XRD, SEM & EDX