Synthesis and Characterization of Copper Oxide Nanoparticles (CuO NPs) using Mangifera indica Leaf Extract

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ARTICLE DETAILS

Article history:
Received 20 April 2019
Accepted 28 May 2019
Available online 12 June 2019

Keywords:
Mangifera indica
CuO NPs
Biocatalyst

ABSTRACT

Metal oxide nanoparticles (NPs) receiving a significant consideration for their probable applications in nanodevices, optoelectronics, nanosensors, nanoelectronics, catalysis and information storage. The objective of the work was to be investigate the synthesis of CuO NPs and evaluation of its characterization of CuO NPs using leaf extract of Mangifera indica. Mangifera indica leaf extract was found suitable for the copper oxide nanoparticles synthesis through biological method ambient conditions. Spherical, polydispersity of CuO NPs of particle sizes ranging from 18 to 106 nm. The average size was 52.54 nm. The formation of color changes indicates that the synthesis of CuO NPs due to plasmon resonance with the active compounds present in the Mangifera indica leaf extract. CuO NPs further confirmed by FT-IR, UV–vis spectroscopy, XRD, EDX and SEM. The functional groups confirmed that the alcohol, phenol, aromatics and alkynes have a probable adherence capacity with copper oxide, which acting as a capping agent and responsible for the bioreduction process.

1. Introduction

Nanotechnology is one of the important areas which are used for utilization and creation of materials with structural geographies between those of bulk materials and atoms with at least one dimension in the nano range [1]. Metallic oxide nanoparticles (NPs) receiving a significant consideration for their probable applications in nano devices, optoelectronics, nano sensors, nano electronics, catalysis and information storage [2]. The copper oxide nanoparticles are of great importance among the various metal nanoparticles due to their low cost of preparation and excellent chemical and physical properties. Copper oxide nanoparticles have extensive solicitations as temperature transfer systems, super strong materials, antimicrobial materials, catalysts and sensors. Copper oxide nanoparticles are very reactive because of can effortlessly engage with other debris and their high floor-to-volume ratio [3] and increase their antimicrobial efficiency.

The synthesis nanoparticles used physical and chemical methods but recently biological method widely used. The drawbacks of chemical methods including the development of toxic by-products, high energy consumption and use of toxic solvents, which risks to the environment and human health. Therefore, the green method is environmentally friendly and cost-effective over physical and chemical methods of synthesis of nanoparticle [4]. The green method of nanoparticle synthesis involved in this are microorganism and plant extracts [5]. In current years, the usage of plant and plant products has gained more importance in the synthesis of nanoparticles. The CuO NPs synthesis from many plant and its products have been investigated by many researchers [6-8]. Hence in this study, we evaluated the synthesis and characterization of CuO NPs using leaf extract of Mangifera indica.

2. Experimental Methods

2.1 Preparation of Leaves Extract

Fresh leaves of healthy Mangifera indica were collected from Thanjavur. The dust particles of leaves were removed by washed with water and shade dried two weeks. The aqueous extract of Mangifera indica leaves was prepared by added 10 g of dried fine leaf powder with 400 mL of sterile distilled water was taken in 500 mL beaker. The aqueous solution color was changed in to brown-yellow from watery after boiled for 10 min. Then filtered with Whatman No. 1 filter paper and remove biomaterials by centrifuge at 1200 rpm for 5 min. The extract was stored in order to use for further experiments.

2.2 Copper Oxide Nanoparticle (CuO NPs) Synthesis

The copper acetate monohydrate (2.8 g) was dissolved in deionized water (500 mL) and stirred magnetically for 5 min at room temperature. Afterwards, added dropwise Mangifera indica leaves aqueous extract under stirring as soon as the leaves extract comes in contact with copper ions to change the colour from blue to green color. After 10 min, the formation of water-soluble monodispersed copper oxide nanoparticles was observed [9].

2.3 Characterization of Nanoparticles

2.3.1 UV and FTIR Spectroscopic Analysis

For UV and FTIR spectrophotometer analysis, the reduction of pure Cu+ ions were scanned in the wavelength ranging from 260-900 nm using Perkin Elmer Spectrophotometer and the characteristic peaks were detected. FTIR analysis was performed using Spectrophotometer system, which was used to detect the characteristic peaks in ranging from 400-4000 cm⁻¹ and their functional groups. The peak values of the UV and FTIR were recorded.

2.3.2 Electron Microscopy and EDX Analysis of Copper Oxide Nanoparticles

ZEISS-SEM machine were used to characterize mean particle size, morphology of CuO NPs. A thin layer of platinum was coated to make the samples conductive ZEISS-SEM machine was operated at a vacuum of the order of 10−5 torr. The accelerating voltage of the microscope was kept in the range 10 kV. Compositional analysis on the sample was carried out by the energy dispersive X-ray spectroscopy (EDS) attached with the SEM. The EDX analysis of Cu sample was done by the SEM machine.

2.3.3 X-Ray Diffraction Method

The phase evolution of calcined powder as well as that of sintered samples was studied by X-ray diffraction technique (Philips Analytical, Netherlands, Model: Diffractometer system= PW3040/60 XPERT-PRO)
using monochromatic CuKa radiation of wave length 1.5418 Å. The generator voltage and current were set at 40 KV and 30 mA respectively. The scanning range 2θ/θ was selected. The scanning speed 10 min⁻¹ was employed for precise determination.

3. Results and Discussion

3.1 Copper Oxide Nanoparticle Synthesis

Phytosynthesis of copper oxide nanoparticles by the extract of Mangifera indica leaves were carried out in this work. During the visual observation, copper acetate and leaves extract stirred magnetically showed the green mixture after 10 min. The appearance of blue color of copper ions to green color is clear indication for the development of water-soluble monodispersed copper oxide nanoparticles (Fig. 1).

![Fig. 1 Colour changes before (Plant extract) and after (CuONPs) the process of reduction of Cu²⁺ to Cu nanoparticles and control (Copper acetate). (Plant = Mangifera indica extract only, copper acetate = copper acetate without Mangifera indica extract, CuONPs = CuONPs with Mangifera indica extract after 5 h of incubation (brown colour)](image)

3.2 UV-VIS Spectral Analysis

It is commonly accepted that size and shape-controlled nanoparticles in aqueous suspensions examine by UV–Vis spectroscopy. UV-Vis spectra recorded in Fig. 2. In the UV–Vis spectra of the reaction mixture of leaves extract of Mangifera indica with copper acetate solution, the peak was observed at 284 nm indicating the formation of CuO NPs through reduction of copper acetate monohydrate was completed at almost 10 min at room temperature. The peak was raised due to inter band transition of core electrons of CuO NPs in the reaction mixture and the broadening of peak indicated that the polydispersed particles. The absorbance wave length values agreement with earlier reported values [6-9].

![Fig. 2 UV-Vis Spectral analysis of CuONPs](image)

3.3 Fourier Transform Infra-Red Spectral Analysis of CuO NPs

FTIR spectrum of copper oxide nanoparticles was examined to identify the potential bioactive compounds responsible for capping and efficient stabilization of the copper oxide nanoparticles synthesized from leaves extract. The peaks observed (Fig. 3) for phytochemicals capped copper oxide nanoparticles formed by reduction by Mangifera indica leaves at 3439.91 cm⁻¹ (Alcohol, Phenol), 1638.27 cm⁻¹ (aromatic rings) and 668.76 cm⁻¹ (alkylenes) suggest the presence of flavonoids and phenols adsorbed on the surface of copper oxide nanoparticles. The outcomes of FTIR analysis established the presence of alcohol, phenol, aromatics and alkylenes compound. This study proved that the presence of secondary metabolites in the sample as proposed capping and stabilizing agent.

![Fig. 3 FTIR spectrum of copper oxide nanoparticles synthesized by reduction of Cu⁺ ions by Mangifera indica leaves extract](image)

3.4 Scanning and Transmission Electron Microscope (SEM) Analysis of CuO NPs

SEM and TEM analysis were carried out to understand the size of the CuO NPs and topology, which showed the synthesis of polydispersed spherical copper-NPs of various sizes with higher density. SEM analysis showed the ranges from 86.24 - 94.44 nm as well spherical and crystalline nature of the nanoparticles. Most of the nanoparticles gathered and only a little of them were dispersed, when observed under SEM (Fig. 4).

![Fig. 4 High resolution scanning electron microscopic (SEM) image of copper oxide nanoparticles (CuONPs), polydispersed (Cluster) CuO NPs ranged between 86.24 - 94.44 nm](image)

3.5 Energy-Dispersive X-Ray Spectroscopy (EDX) Analysis of CuO NPs

The data that is generated by EDX analysis consists of spectra with peaks corresponding to all the different elements that are present in the sample. EDS of CuO NPs revealed the presence of pure copper (Cu 98.74%) and was the major constituent element compared to carbon (1.26%) as represent in Table 1 and Fig. 5. The EDX reading demonstrated that the manditory phase of copper (Cu) was present in the CuO NPs.

![Fig. 5 EDX-spectroscopy view of the Mangifera indica showing synthesis of copper oxide nanoparticles and elemental copper signal in higher percentage](image)

<table>
<thead>
<tr>
<th>Elements</th>
<th>AN</th>
<th>Series</th>
<th>Weight %</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>29</td>
<td>K-series</td>
<td>98.74</td>
<td>93.70</td>
</tr>
<tr>
<td>C</td>
<td>06</td>
<td>K-series</td>
<td>1.26</td>
<td>6.30</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td></td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

3.6 The XRD Pattern of CuO NPs Synthesized from Leaves

XRD is generally used for shaping the crystal structure of a material and chemical composition. Therefore, identifying the incidence of copper oxide nanoparticles can be achieved by using XRD to observe the diffraction peaks of the CuO NPs. X-ray diffraction pattern of CuO NPs is shown in Fig. 6. The crystalline nature of Cu nanoparticles was proved from X-ray diffraction (XRD) analysis showing the XRD pattern of the dried nanoparticles obtained from colloidal samples. A number of Bragg reflections with 29 values of 31.41, 38.20, 46.07, 64.42, 67.49 and 77.20° indicate the (110), (111), (200), (220), (300) and (311) reflections of metallic copper clearly indicating the cubic crystalline face-centered cubic structure of copper which was compared with the standard powder diffraction card of JCPDS card 05-0661. Here, the constant is 28.81 (75.974-47.1564= 20.81). Hence XRD pattern thus clearly illustrated that the copper oxide nanoparticles formed in this present synthesis are
crystalline in nature. The line broadening of the peaks is primarily due to small particle size. Indexing has been done and data are in Table 2.

![Graph showing X-ray diffraction peaks](image)

**Fig. 6 XRD patterns of copper nanoparticles synthesized using leaves**

### Table 2 Simple peak indexing

<table>
<thead>
<tr>
<th>Peak (2θ)</th>
<th>1000 + Sin2θ</th>
<th>1000 + Sin2θ/28.80</th>
<th>Reflection</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>18.87</td>
<td>47.15640956</td>
<td>1</td>
<td>100</td>
<td>11 + 0 + 0 = 1</td>
</tr>
<tr>
<td>24.02</td>
<td>75.96625967</td>
<td>2</td>
<td>110</td>
<td>11 + 1 + 0 = 2</td>
</tr>
<tr>
<td>29.20</td>
<td>111.4824695</td>
<td>3</td>
<td>111</td>
<td>11 + 1 + 1 = 3</td>
</tr>
<tr>
<td>31.71</td>
<td>130.9690266</td>
<td>4</td>
<td>200</td>
<td>2θ + 2θ + 0 = 4</td>
</tr>
<tr>
<td>37.83</td>
<td>184.357903</td>
<td>6</td>
<td>211</td>
<td>11 + 1 + 1 = 6</td>
</tr>
<tr>
<td>43.4</td>
<td>239.8452168</td>
<td>8</td>
<td>220</td>
<td>2θ + 2θ + 0 = 8</td>
</tr>
<tr>
<td>47.52</td>
<td>284.88888832</td>
<td>10</td>
<td>310</td>
<td>3θ + 1 + 0 = 10</td>
</tr>
<tr>
<td>50.46</td>
<td>318.760155</td>
<td>11</td>
<td>311</td>
<td>3θ + 1 + 1 = 11</td>
</tr>
<tr>
<td>56.4</td>
<td>13.99108832</td>
<td>14</td>
<td>320</td>
<td>3θ + 2 + 1 = 14</td>
</tr>
</tbody>
</table>

**Table 3 The copper oxide nanoparticle grain size**

<table>
<thead>
<tr>
<th>Intense peak (2θ) (deg)</th>
<th>Miller indices (hkl)</th>
<th>% of the intense peak (β) (deg)</th>
<th>FWHM of intense peak (β) radians</th>
<th>Size of the particle (D) (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18.87</td>
<td>100</td>
<td>9.435</td>
<td>0.164672</td>
<td>49.5371</td>
</tr>
<tr>
<td>24.02</td>
<td>110</td>
<td>12.01</td>
<td>0.209615</td>
<td>45.37157</td>
</tr>
<tr>
<td>29.20</td>
<td>111</td>
<td>14.6</td>
<td>0.254819</td>
<td>78.80114</td>
</tr>
<tr>
<td>31.71</td>
<td>200</td>
<td>15.855</td>
<td>0.276723</td>
<td>29.20921</td>
</tr>
<tr>
<td>37.83</td>
<td>211</td>
<td>18.915</td>
<td>0.33013</td>
<td>24.38523</td>
</tr>
<tr>
<td>43.4</td>
<td>220</td>
<td>21.7</td>
<td>0.378737</td>
<td>22.27469</td>
</tr>
<tr>
<td>47.52</td>
<td>310</td>
<td>23.76</td>
<td>0.414691</td>
<td>98.00582</td>
</tr>
<tr>
<td>50.46</td>
<td>311</td>
<td>25.23</td>
<td>0.440348</td>
<td>18.3367</td>
</tr>
<tr>
<td>56.4</td>
<td>320</td>
<td>28.20</td>
<td>0.492184</td>
<td>106.9842</td>
</tr>
<tr>
<td><strong>Average nanoparticle size</strong></td>
<td></td>
<td></td>
<td></td>
<td><strong>52.540</strong></td>
</tr>
</tbody>
</table>

### 3.7 Particle Size Calculation

From this study, the peak at degrees, Debye–Scherrer formula used for average particle size has been estimated [10-13], \( D = \frac{0.9 \lambda}{\beta \cos \theta} \), where \( \lambda \) is wave length of X-Ray (0.1541 nm), \( \beta \) is FWHM [full width at half maximum], \( \theta \) is the diffraction angle and \( 'D' \) is particle diameter size. According to Debye–Scherrer equation, the average crystalline size was found to be 52.54 nm and represent in Table 3.

### 4. Conclusion

In the present study concluded that the CuO nanoparticles synthesized by eco-friendly, and convenient green method from copper acetate monohydrate using aqueous extract of Mangifera indica leaf. Spherical and polydispersity of CuO NPs of particle sizes ranging from 18 to 106 nm with an average size of 52.54 nm were obtained.

### References