Preparation of CdO Nanoparticles Using Two Different Salts and Investigation of their Antibacterial Activity

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Abstract: This study reports synthesizing two types of CdO NPs using CdSO4 and CdCl2 salts as precursor materials and Petroselinum extracts as a reducing agent. The nanoparticles were characterized by XRD, TEM, FE-SEM, EDX, and DLS techniques. The results showed that the properties of the prepared CdO nanoparticles varied depending on the type of salt used as a precursor. The XRD confirmed that the two prepared CdO nanoparticles have particle diameters of 58.35 CdSO4 and for CdCl2 is 44.02 nm. FE-SEM micrographs confirmed that the obtained particles have different shapes and sizes, and the average particle diameters were 77.16 and 109.57 nm for CdO nanoparticles from CdSO4 and CdCl2, respectively. The CdO nanoparticles that obtained from CdSO4 have a narrow particle size distribution was 265.6, in contrast, in the case of CdCl2, the distribution of the particles was 356.3 nm with a broad peak. Anti-bacterial evaluation of the prepared CdO exhibits revealed a significant inhibition against four types of bacteria Staphylococcus aureus, Streptococcus pneumonia, Klebsiella pneumonia, and Escherichia coli.

1 INTRODUCTION

Nanotechnology is a branch that is applied in many fields, such as biology, physics, chemistry, and engineering, due to their special physicochemical properties [1, 2]. The metallic nanoparticles have applications in many areas, specifically cosmetics, coating, electronics, packaging, and biotechnology, and enormous potential for their use in humans [3, 4]. The antibacterial agents were used extensively, which generates pressure to escalate rates of antibiotic resistance [5]. However, new resistant bacterial strains to current antibiotics have become a serious issue for public health, which has awakened the need to find new bactericidal agents [6]. Oxide nanoparticles with heavy metals such as Cd can eliminate cancer cells at a low concentration and are non-toxic to mammalian cells [7], [8] . Nanoparticles are being studied more deeply, and several therapeutic modulations of nanoformulations have been created as a new brand perspective for cancer treatment [9].

However, the synthesis of new compounds like nanoparticles with anti-microbial properties is essential, with potential applications which can help fight antibiotic resistance towards pathogenic bacteria [10]. Nanoparticles can be used in small amount of cadmium oxide as medicines with tiny sizes [11], [12], [13], [14], [15]. Scine, the synthesis of NPs has been a matter of concern because chemical methods are toxic. On the contrary, biological methods are considered eco-friendly. Whoever, many reports have confirmed that plant extracts can be used as reducing and capping agents in forming NPs since they contain various phytocompounds: phenols, alkaloids, and terpenoids. So, the aim of this study is to use two types of salts the CdSO4 and CdCl2 as precursor materials to synthesize the cadmium oxide nanoparticles (CdO NPs) with using leaf extract petroselinum crispum as a reducing agent and investigate their effect on the selected pathogenic bacteria (Staphylococcus aureus, Streptococcus pneumonia, Klebsiella pneumonia and Escherichia coli).

2 MATERIALS AND METHODS

Petroselinum crispum leaves were collected from the local gardens at Diyala governorate in Iraq. All chemicals involved in the experiments are reagent grade. Cadmium sulfate (CdSO4), Cadmium chloride (CdCl2), and Sodium hydroxide were purchased from Sigma Aldrich and BDH. Deionized water was used for the experiment. The anti-bacterial test includes Staphylococcus aureus, Staphylococcus epidermidis, Escherichia coli, and Klebsiella pneumonia, obtained from Baqubah General Hospital, Diyala, Iraq.

2.1 Preparation of the Plant Extract

The green reducing and stabilizing agent was prepared from the petroselinum crispum leaves which were collected from the Diyala governorate gardens in Iraq. The leaves were washed using deionized water to remove impurities, and then cut into small pieces. 25 g of cut leaves were put in 100 mL of deionized water and set the temperature at 80 °C. After some time, the water turned greenish, indicating leaf extract formation in the water. The filtered extract was transferred into the burette to be used as a reducing agent for cadmium oxide nanoparticle synthesis, Figure 1.

Figure 1: Synthesis of leaves extract petroselinum crispum.

2.2 Synthesis of Cadmium Oxide Nanoparticles

Cadmium sulfate (CdSO4) and cadmium chloride (CdCl2) salt solutions were prepared using deionized water. Leaves extract petroselinum crispum was added dropwise, and sodium hydroxide solution was introduced slowly to the solution under a stirrer until the pH=12. After 2 hours, the obtained precipitates were filtered using a Buckner funnel, washed with distilled water several times, and dried in an oven for 2 hours. Then, calcination was carried out in the furnace at 600 ºC for 6 hours to remove any contamination [16]. As shown in Figure 2.

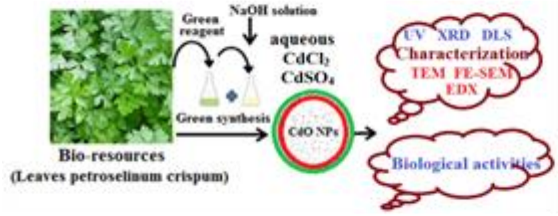


Figure 2: Synthesis of CdO-NPs using CdSO4 and CdCl2 salts as a sources of cadmium metal by petroselinum crispum leaves extract [17].

2.3 Characterization

The characterization of CdO NPs was done using several techniques, including Fourier transform infrared (FTIR) spectroscopy in the range of 400–4000 cm-1 for functional groups. Field Emission Scanning Electron Microscopes (FE-SEM) and Transmission Electron Microscopes (TEM) were utilized to examine the CdO nanoparticles morphology. Energy-dispersive X-ray spectroscopy (EDX) was used to study the FE-SEM micrographs and for elemental mapping. X-ray diffraction (XRD) was utilized to calculate the average crystalline size, and DLS technique was used to examine the surface area, pore size, and distribution of CdO NPs, respectively.

3 RESULTS AND DISCUSSION

3.1 XRD Analysis

Figure 3 exhibits the XRD patterns for the prepared CdO, from which the intensity and quality of XRD peaks vary depending on the salt that used and the variation of the precursor; the size and morphology of NPs also vary. The observed diffraction patterns showed strong reflections from the (111), (200), (220), (311), and (222) planes, which correspond to the face-centered-cubic (FCC) consistent with Monteponite structures of CdO NPs (from CdSO4 salt) and CdO NPs (from CdCl2 salt), with the purity of CdO NPs (from CdCl2 salt) which very well as compare to CdO NPs (from CdSO4 salt). The prepared cubic phase of the polycrystalline Monteponite CdO (JCPDS card number 050640) is agreed with previous literatures [18]. The crystallite size (D) at the highest reflection plane (111) for the prepared CdO nanostructures is calculated following Debye–Scherrer’s equation:

$$D = \frac{\kappa * \lambda}{\beta \cos\theta} . \quad (1)$$

Where λ is x-ray wavelength, K corresponds to the shape constant (0.9), θ is Bragg angle, and β the full width height maximum (FWHM) of the diffracted peak.

The average size for the CdO nanoparticles was 55.36 nm and 45.69 nm for CdO NPs (from CdSO4 salt) and CdO NPs (from CdCl2 salt), respectively. The lattice parameters are listed in Table 1. Lattice parameter (a) was calculated using the relation:

$$a = d_{hkl}\sqrt{h2 + k2 + l2}\,, \tag{2}$$

d_{hkl} is the interplanar spacing, and h, k, and l are the miller indices.

Table 1. The indexing XRD of two types CdO nanoparticles.

Nanomaterials	Plane	d_{111}(A°)	a (A°)	D, nm
CdO from $CdSO_4$ salt	(111)	2.6937	4.6656	58.35
CdO NPs from $CdCl_2$ salt	(111)	2.6903	4.6598	44.02

3.2 FE-SEM Micrographs

The surface morphology for the synthesized nanoparticles was observed using a scanning electron microscope. Figure 4 (a and b) shows the surface morphology of the CdO (from $CdSO_4$ salt) and CdO (from $CdCl_2$ salt) nanoparticles respectively, in which the nanoparticles are found to be cylindrical in shape with the average size of nanoparticles at 77.16 nm, see Figure 5a. Figure 5b exhibits CdO NPs (from $CdCl_2$ salt) as spherical shape nanoparticles with the average size of nanoparticles at 109.57 nm. The results confirm the effect of the anion associated with cadmium metal is very clear on the size and shape of the CdO nanoparticles since the different anions gave agglomeration of the nanoparticles and could be related to van der Waals forces among.

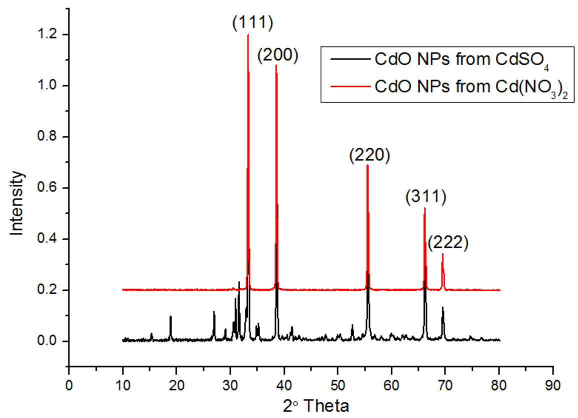
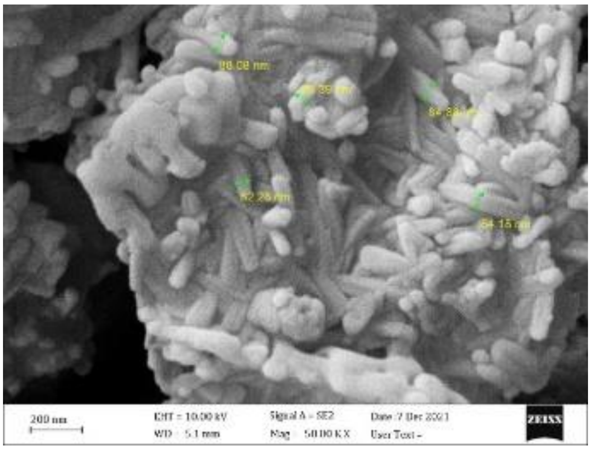


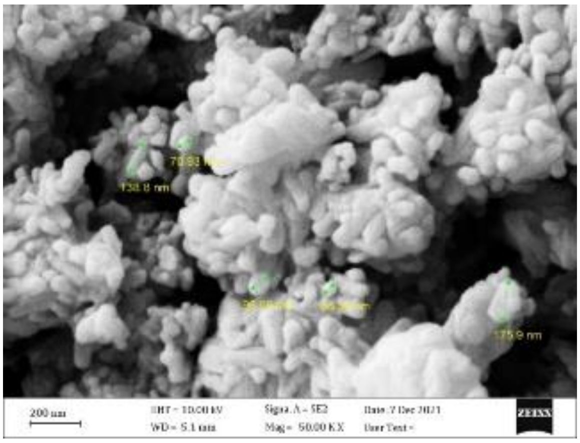
Figure 3: XRD patterns of CdO NPs from CdCl2 salt and CdO NPs from CdSO4 salt.

3.3 EDX Analysis

The Energy-dispersive X-ray (EDX) spectra show the components in the prepared samples, in which the fabricated samples are CdO nanoparticles. The EDS result in Figure 5 (a and b) shows the presence of Cd and O (Cd: 90% and O: 10%) for both samples.



(a)



(b)

Figure 4: FE-SEM images of CdO nanoparticles, a) CdO NPs from CdSO4 salt, and b) CdO NPs from CdCl2 salt.

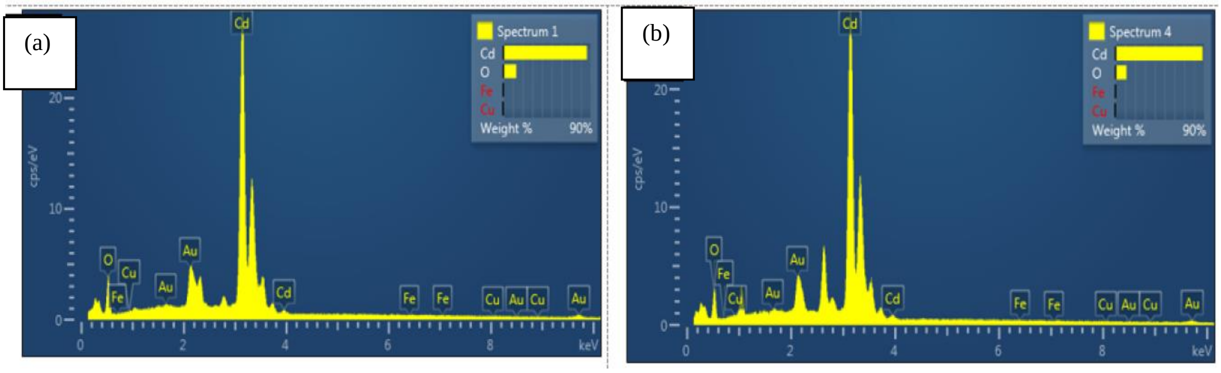


Figure 5: EDS images of CdO nanoparticles, a) CdO NPs from CdSO4 salt, and b) CdO NPs from CdCl2 salt.

3.4 TEM Image

The images of TEM for CdO nanoparticles are illustrated in Figures 6 (a and b). The formation of CdO nanoparticles was prepared by the Eco-Friendly method. The diameter was confirmed using TEM micrographs (28.83 nm to 37.2) nm for CdO NPs from CdSO4 salt and (27 nm to 36 nm) for CdO NPs from CdCl2 salt.

3.5 Dynamic Light Scattering (DLS)

The distribution function analysis of the suspension system was done using the Rayleigh-Debye model. The particle size distribution curves of CdO (from CdSO4 salt) and (from CdCl2 salt) nanoparticles are shown in Figure 7 (a and b). In Figure 7a, the dynamic light scattering (DLS) data for CdO NPs (from CdSO4 salt) gave one narrow peak with a particles distribution 265.6 nm [19]. In this case, CdO NPs (from CdCl2 salt) has one broad peak with a higher particles distribution 356.3 nm, Figure 7b. Increasing the average particle size is due to the anion-type effect, in which all particles have different sizes. The mean particle size increase can also be attributed to the agglomeration of the smaller particles.

Table 2: Anti-bacterial activity towards Staphylococcus Aureus, Staphylococcus Pneumonia, Escherichia Coli, and Klebsiella Pneumonia (50 and 100 mg/mL), Demeter (mm).

Sample	*S. Aureus*		*S.Pneumonia*		*E. coli*		*K.Pneumonia*	
Concentration (mg/mL)	50	100	50	100	50	100	50	100
CdONPs from $CdSO_4$ salt	18	23	22	24	21	23	20	22
CdONPs from $CdCl_2$ salt	20	23	21	24	18	20	19	21
Amoxicillin	20	21	R	R	R	10	R	11
DMSO	0	0	0	0	0	0	0	0

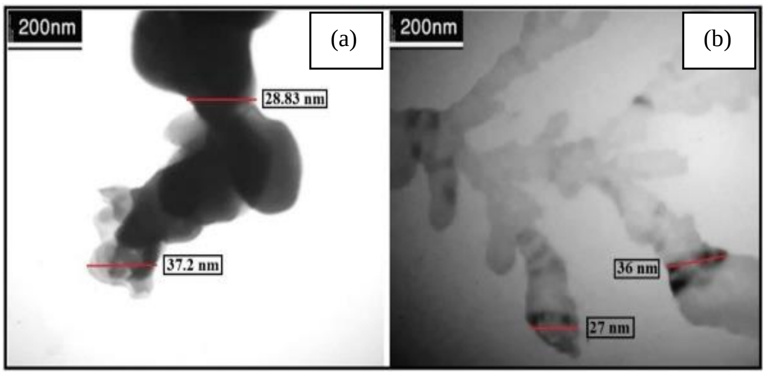


Figure 6: TEM images of CdO nanoparticles, a) CdO NPs from CdSO4, and b) CdO NPs from CdCl2.

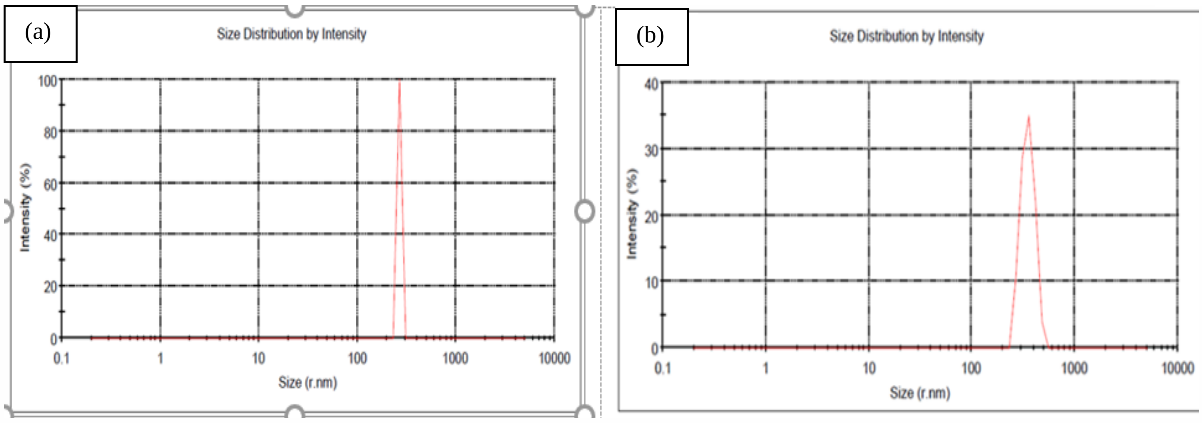


Figure 7: DLS analysis of CdO nanoparticles, a) CdO NPs from CdSO4 salt, b) CdO NPs from CdCl2 salt.

3.6 Antibacterial Activity

The anti-bacterial activities for the synthesized nanoparticles CdO NPs (from CdSO4 and CdCl2) have tested using disk diffusion method and the inhibition zone was measured in mm [20]. Amoxicillin (50 and 100 mg/mL) was used as a reference drug for the test. The synthesized compounds were evaluated for their anti-bacterial activity against the Staphylococcus aureus and Streptococcus pneumonia, Klebsiella Pneumonia, and Escherichia coli for their concentrations (50 and 100 mg/mL) in Muller Hinton agar. The sterilized agar media were poured into Petri dishes and allowed to solidify, and the media microbial suspension were spread on the surface using loop. DMSO was used as a solvent for the cultures. The resulted plates were put in the incubator at 37 oC for 48 hours. The results are provided in Table 2. As seen from Table 2, the antibacterial activity of the test compounds gave excellent activity. The prepared compounds show activity against S. aureus is nearly close to that of the reference in the 50 and 100 mg/mL concentrations. The activities were excellent against S. pneumonia for 50 and 100 mg/Ml. Furthermore, same activity was observed against E. coli and K. pneumonia for both concentrations of 50 and 100 mg/mL since the drug gave resistance to moderate activity against them.

4 CONCLUSIONS

Two CdO nanoparticles have been synthesized using a simple, non-cost-effective, eco-friendly method using two different precursor salts's CdSO4 and CdCl2. The result clearly indicates that the properties of the prepared compounds varied and depended on the type of salt used as a precursor. The formations of two CdO nanoparticles were confirmed using XRD, FE-SEM micrographs, TEM, and DLS. However, the XRD analysis showed that the average size for the CdO nanoparticles was 55.36 nm and 45.69 nm for CdO NPs obtained from CdSO4 and CdCl2, respectively. In addition, the FE-SEM image indicates that the nanoparticles of CdSO4 are found to be cylindrical in shape with an average size of nanoparticles at 77.16 nm, and of CdCl2 found to be spherical in shape with an average size of nanoparticles at 109.57 nm.

Anti-bacterial evaluation of the prepared CdO exhibits a significant inhibition against the four types of bacteria Staphylococcus aureus, Streptococcus pneumonia, Klebsiella pneumonia, and Escherichia coli, compared to the reference.

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