

RESEARCH ARTICLE

Biosynthesis and Characterization of CdO: Ag NPs using *Moringa* Leaves Extract for Use as Anti-microbial Activity

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ABSTRACT

The current research focuses on synthesizing cadmium oxide (CdO), CdO: Ag nanoparticle (NPs) by green method using the *Moringa* leaves Extract (MLE) and its anti-microbial activity. Using UV-vis spectroscopy to determine the formation of CdO: Ag NPs where the energy gap of the prepared samples was 2.79, 2.67, and 2.46 for CdO, CdO: Ag 3% and CdO: Ag 5%, respectively. The biological substances in responsibility of capping the produced NPs were identified using FTIR. The NPs were further characterized by X-ray diffractograms (XRD). The average grain size for CdO, CdO: Ag (3 and 5%) were 15.322, 15.5, and 36.84 nm, respectively, scanning electron microscope (SEM), energy dispersive X-ray (EDX) and atomic force microscope (AFM), were obtained the average diameter 18.88, 43.48, 99.15 nm for CdO, CdO: Ag 3% and CdO: Ag 5%, respectively. The anti-bacterial activity of green synthesized CdO: Ag(NPS) was investigated against (*Staphylococcus aureus*, *Escherichia coli*, and *Klebsiella pneumonia*) at different concentrations, showing clear inhibition zones from 30 to 38 mm, thereby indicating its inhibitory activity.

Keywords: Biosynthesis, *Moringa oleifera*, Nanoparticles, Plant extract.

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INTRODUCTION

NPs are tiny particles ranging from 1 to 100 nm. Nanotechnology takes a prominent position in science and research, as the wide variety of methods for preparing NPs for nanomaterials in research in the field of materials science energy,¹ medical and biological sciences gave clear facilitation of the use and expansion of nanotechnology.² Nanoscience concerns the entire fields of molecules and substances with fewer than 100 nm. It's also interested in refining these materials' chemical and physical characteristics and studying the events that arise due to their reduced size. Cadmium oxide (CdO) is an N-type semiconductor that refers to an II-VI group. In terms of the crystal structure, it has a cubic crystal structure, face center cubic (FCC) is similar to the design of NaCl,³ cadmium oxide is one of the chemical cadmium compounds that does not dissolve in water or bases, but it dissolves in acids and ammonia salts. It can be obtained by intense heating of cadmium (Cd). It also has interesting properties, such as a large band gap of about 2.2 eV,⁴ low electrical resistivity, and high transmission in the visible and infrared regions. Therefore it was classified among the transparent conductive oxides.⁵ Silver NPs (AgNPs) are being used in an increasing number of industries, including medicine, health care, consumer goods, and industry, as a

result of their distinctive Physical and Chemical properties, such as optical, electrical, and thermal characteristics, as well as solid electrical conductivity and biological properties.⁶ Pure cadmium oxide CdO, Ag doped CdO nanoparticle has received much attention as a metal oxide structure. It may be used in various device applications due to its physical characteristics, including solar cell, optical communication, photo-transistor, gas sensor, low-emissive window, antifungal activities, and catalytic application.⁷ Their significant potential uses, chemical stability, and widespread availability was chosen for this work. Also, they were prepared using a variety of techniques, such as sol-gel, solvothermal, microemulsion method, precipitation method, chemical method and green synthesis. Green synthesis is more efficient than chemical and physical processes since it is quick and straightforward to scale up for large-scale synthesis,⁸ and it is a technique in which various plant components, such as leaves, flowers, pod extracts, etc., are utilized to reduce metal ions into NPs. The horseradish tree, known as the *Moringa* plant, which grows in South Asia, is a significant source of carotene, vitamin C, protein, iron, and potassium. It has been demonstrated that *Moringa* leaves contain more polyphenols, which are known to scavenge free radicals and have a strong antioxidant potential.⁹ XRD, EDX,

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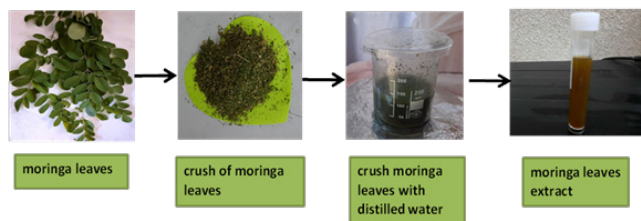


Figure 1: Preparation processes of MLE.

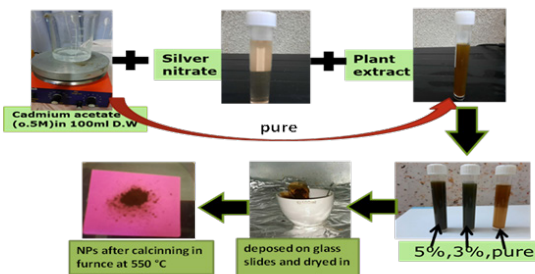


Figure 2: Shown the synthesised of pure CdO and CdO: Ag NPs.

SEM, AFM, FTIR, and UV–vis spectroscopy were used to investigate the NPS structural and optical properties. This study aims to synthesize nano-material from CdO and CdO: Ag by Bio-synthesis because of the importance of this material and its use in broad applications such as cancer treatment and anti-bacterial applications.

EXPERIMENTAL METHOD

Preparation of Pure *Moringa* Leaves Extract (MLE).

Wash the *Moringa* leaves well with distilled water, then drying in a hot air Oven at 40°C; then 10 g of round leaves were mixed with 100 mL of distilled water by a stirrer at 30°C for 12 hours. Finally, the mixture was filtered and kept at 4°C until use¹⁰ Figure 1.

Synthesed of Pure CdO :Ag Nps

Pure CdO NPs were prepared as follows: 100 mL of distilled water was used to dissolve 0.5M of cadmium acetate dehydrate (Cd(CH₃COO)₂·2H₂O). using a magnetic stirrer. The Green Synthesis of CdO NPS was obtained by mixing 10 mL of plant extract with 90 mL of cadmium acetate. This mixture was heated for 20 minutes at 60–80°C antill the color changed from light brown to dark brown. The production of NPs was suggested by the dark brown solution produced. The NPs were created and kept at 4°C till used.¹¹ Cdo: Ag Nps were made using the same method as pure CdO. Just before adding the *Moringa* extract, the predicted amount of dopant silver nitrate (AgNO₃) was included in the mix in terms of (3 and 5) wt% concentration, and the reaction took place at the same temperature. The concentrated CdO: Ag NPs were used to prepare a thin film on a glass slide to study structural characteristics. The glass slides were annealed in the muffle furnace for one hour at 550°C to eliminate organic debris and other impurities (Figure 2).

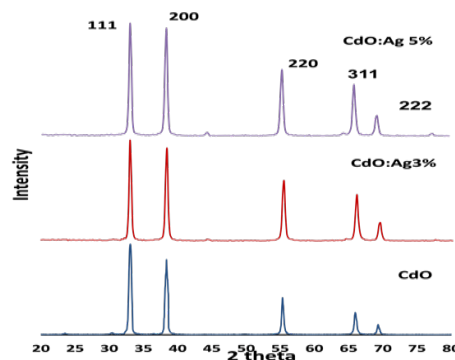


Figure 3: XRD patterns for green synthesized NPs of pure CdO and CdO: Ag.

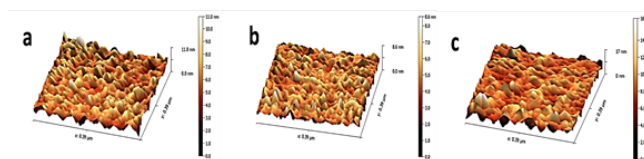


Figure 4: AFM image of green synthesized of (a) CdO; (b) CdO:Ag 3%; (c) CdO:Ag 5% NPS.

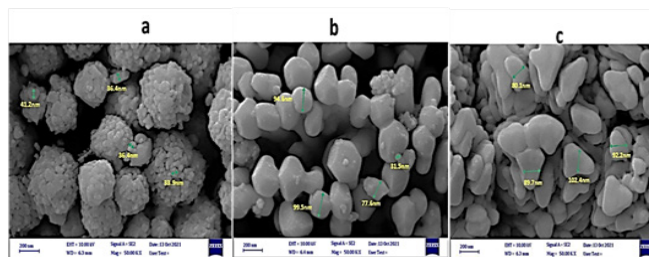


Figure 5: SEM images of CdO and CdO:Ag, as follows : (a) CdO (b) CdO: Ag 3% (c) CdO: Ag5% NPS.

RESULT AND DISCUSSION

XRDAnalysis

The materials were evaluated at scales ranging from 20 to 80° using an X-ray diffractometer with “copper-filtered monochromatic CuK radiation” of = 1.540, Shimadzu, Japan. Figure 3 displays the XRD pattern of pure CdO NP doped with concentrations of (3 and 5) at wt% of Ag, which was prepared by using the Biosynthesis method. The Figure 3 shows that every prepared particle has a polycrystalline reflection pattern, with the majority of reflection occurring in the plane of (111) at an angle of 2 = (33.07–33.04°). Each sample’s crystalline phase was determined by comparing its XRD patterns with the (JCPDS) card when the measured XRD patterns were compared to the standard (JCPDS) card No. (5–0640), the pure cadmium oxide NPs phase is found to have crystal structure with a face-centered cubic (FCC) shape,¹² as shown in Table 1.

The diffraction patterns of a CdO Sample displayed robust and sharp diffraction peaks. They were related to a CdO phase. And appears small peaks of Ag in CdO doped with (3 and 5) at wt% of Ag samples at $2\theta = (44.4, 64.4, \text{ and } 77.6^\circ)$, this agrees with Noha.¹³ The strong peaks show that the particles were well-crystalline. Ag addition causes slight offset shifting in the diffraction angle to the less 2θ with increasing peak intensity and grain size, indicating the enhancement of particle crystallinity. This agrees with Velusamy. *et al.*¹⁴ The crystallite size (D) (was calculated from the)Scherer relation (1).¹⁵

$D = K\lambda / \beta \cos\theta$ (1) where (K) is a quantity that fluctuates depending on the crystallites' solid form, (λ) is the wavelength of XRD it's 1.5418 \AA , (β) is the Full Width at Half the Maximum intensity (FWHM) and (θ) is Bragg's angle, the mean XRD crystallite size values for CdO and CdO: Ag are given in Table 1. It was found that the crystallite size of the CdO that had been produced was 15.322 nm. At the same time, with 3%wt of Ag, there was a slight increase in the size of NPs, the crystallite size was 15.5 nm, and then the grain size increased again when doping with 5% wt of Ag to become 36.84 nm. Due to the decrease of FWHM and decrease in the concentration of CdO with the addition of Ag, where the ionic radius of (Ag^+) is greater than that of (Cd^{+2}), this agrees with Saravana Kumar *et al.*¹⁶ The Dislocation density (δ) measures the number of defects in a crystal. It's calculated from relation (2).¹⁷

$$\delta = 1/D^2 \text{ (lines / m}^2\text{)} \dots\dots\dots (2),$$

where (D) is the grain size.

Table 1 demonstrates how decreasing crystal lattice defects cause a drop in dislocation density (δ) and an increase in crystalline size. This agrees with Saravana Kumar *et al.*¹⁶

Atomic Force Microscopy (AFM)

Three Dimensional (3D) profiles of AFM, WORKSHOP, TT-2, and the USA, were used to give information on the morphology of materials' surfaces, such as the grain size and the average surface roughness.¹⁸ Figure 4 (a-c) shows the surface morphology nano-particle of pure CdO and CdO doped with 3 and 5 wt% of Ag concentration. The images show that the samples have spherical and conical shapes. In Table 2, the AFM's parameters have been listed. The surface morphology analysis revealed that raising the Ag concentration led to an increase in grain size and average roughness, in addition to a rise in r.m.s, which agrees with the XRD study results.

Scanning Electron Microscope (SEM) Study

The (SEM) of the ZEISS model, Sigma VP, with a magnification of 20,00 to 50,00 XK, and an electron strength of EHT of 10,00Kv, was used to provide information about topography and micro-graphs of green *Moringa* synthesized CdO, CdO: Ag NPs are shown in Figure 5 (a-c). Through the Figure 5, it can be noted that the surface of the NPs is very smooth. There are no tiny particles seen in the SEM scan. And the particle is spherical for the CdO sample, ranging from 33.9 to 41.2 nm. In contrast, the shape noticed rhombic and pyramidal shapes in addition to spherical when silver is added to cadmium oxide. The particle size was from 31.5 to 99.5 nm for CdO doped

Table 1: (hkl) plane, (FWHM) value, (D) value, and (δ) value of CdO and CdO: Ag.

Ag concentration wt%	hkl	2theta AS	2theta TM	FHM	D(nm)	$\delta = 1/D^2$
CdO					15.545	
	111	33.03	33.07	0.5568	14.976	0.00414
	200	38.31	38.35	0.5866	15.443	0.00446
	220	55.30	55.36	0.606	Average of D=15.322 nm	0.00419
3%					15.3	0.00427
	111		33.06	0.5654	15.399	0.00422
	200		38.35	0.5705	15.8	0.004
	220		55.34	0.5928	Average of D=15.5nm	Average of $\delta=0.00416$
5%					38.502	0.00067
	111		33.04	0.2248	36.71	0.00074
	200		38.34	0.2652	35.325	0.0008
	220		55.33		Average of D = 36.84nm	Average of $\delta=0.00074$

Table 2: Average diameter and roughness for green synthesized NPs of pure CdO and CdO: Ag as a function of dopant concentration.

Ag concentration wt%	Average diameter (nm)	Average roughness (nm)	r.m.s roughness (nm)
Pure	18.88	2.89	4.05
3%	43.48	3.307	5.345
5%	99.15	18.77	29.51

Table 3: shows the proportion of the composition of the prepared materials.

EDX Composition	Cd Wt%	C Wt%	O Wt%	Ag Wt%
Pure	94.8	1.7	3.5	0
3%	93.2	1.4	4.7	0.7
5%	94.2	1.2	3.7	0.9

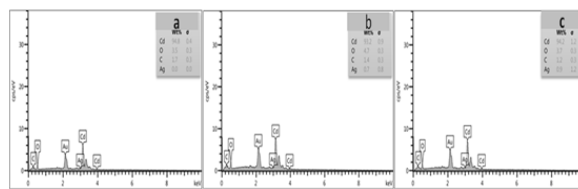


Figure 6: EDX spectrum of CdO and CdO:Ag as follows : (a) CdO (b) CdO: Ag 3% (C)CdO: Ag5% NPS.

3%wt of Ag sample, while the particle size for CdO doped 5% wt of Ag sample was from 80.1 to 102.4 nm. This means that the particle size increase with the increasing concentration of Ag. This agrees with Mannarsamy Anitha *et al.*¹⁹

The component content of the produced samples is explained by energy-dispersive X-ray analysis (EDX) spectra, which demonstrate that silver ions were efficiently inserted into the CdO lattice. Figure 6 and Table 3 show the EDX results. As indicated by the result, no distinctive peaks of contaminants or other precursor chemicals were found.

Table 4: FTIR bonds of CdO: AgNps produced by *Moringa* leaf extract.

Band type	Cdo	Cdo: Ag3%	Cdo: Ag5%
CdO	422.38–460.96	420.45–439.74	420–437.81
Aromatic hydrocarbons and alkynes groups for silver. C-O	1200–1369	1200–1369	1200–1369
Alkynes and esters for silver. C-H	1369.37	1371.29	1371.29
Small bond H2O	1562.23–1587.31	1560.30–1587.31	1558.38–1590
carbonyl stretching C=O	1643.24	1645.17	1645.17
Esters and chlorophyll	1741	1747.39	1747.39
Bending C=H	2972.10	2970.17	2970.17
Alcoholic and phenol O-H	3419.56	3423.41	3427.27

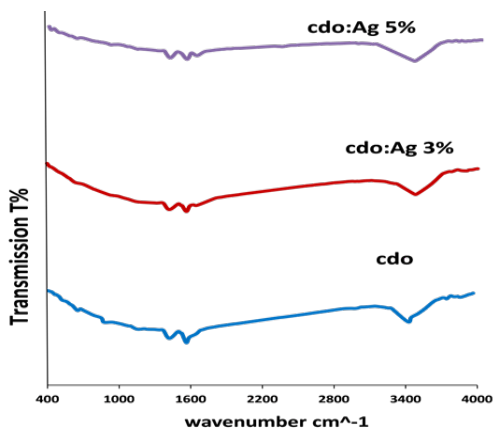


Figure 7: Fourier transform infrared spectra analysis of green synthesized CdO, CdO: Ag NPs prepared by precipitation methods from *Moringa* extra leaves.

Table 5: Energy band gap of Green synthesized for nano-particle of CdO and CdO: Ag (3, and 5)%wt.

Material	Wavelength (nm)	The band gap (eV)
Cdo	444	2.79
Cdo: Ag 3%	464	2.67
Cdo: Ag 5%	504	2.46

FTIR Analysis

The Green synthesized CdO, CdO: AgNPs was identified using FTIR spectroscopy, Shimadzu 8400, Japan. The FTIR spectra of CdO and CdO: Ag NPs were between 400 to 4000 cm⁻¹. Figure 7 and 8 displays the conclusion drawn from the FTIR spectra of green-produced CdO and CdO: Ag NPs.

The presence of CdO-related peaks at 422–460 cm⁻¹ supports the catalyst’s pure formation²⁰ The stretch and vibration of the C-O bond associated with aromatic hydrocarbons are likely responsible for the area between 1200 and 1369 cm⁻¹. Transmittance at 1369 and 1371 cm⁻¹ has been generated by the weak stretching of C-H wagging in C-CH alkynes. Due to the tableting process, unbound H₂O molecules are responsible for the narrow band between 1558 and 1590. The C=O carbonyl stretching vibration caused the medium transmittance trough at 1643,1645 cm⁻¹, which verifies the existence of amide (I) and is a feature of β -sheet protein, and has resulted in stabilized NPs.²² A C=O fragment of esters linked to chlorophyll is responsible for the medium stretching and vibration at 1741, 1747 cm⁻¹. It is possible to link C-H to the faint peak between 2970 and 2972 cm⁻¹. Stretching of an outer band within the spectrum of 34419, 3423, 3427 cm⁻¹ and vibration of the O-H bond provide evidence of the existence of phenolic and alcoholic chemicals.¹¹⁻¹⁶ The peaks at 1200–1369, 1369–1371, and 1741–1747 cm⁻¹ show the formation of silver NPs by alkynes, esters, and carboxylic groups, as well as the stability of those particles by the amide group.²¹ In the Table 4, all summits are mentioned .

Optical Properties

The UV spectrophotometer, Shimadzu 8400, Japan, absorption spectrum was used to evaluate the optical characteristics of Green produced for CdO and CdO: Ag CdO NPS. The spectra were drawn across a wavelength range of 350 to 800 nm Figure 8 displays the absorption spectrum for green synthesized nanoparticle of pure CdO and CdO: Ag. From the Figure, an increase in the absorbance was noticed, and the absorbance

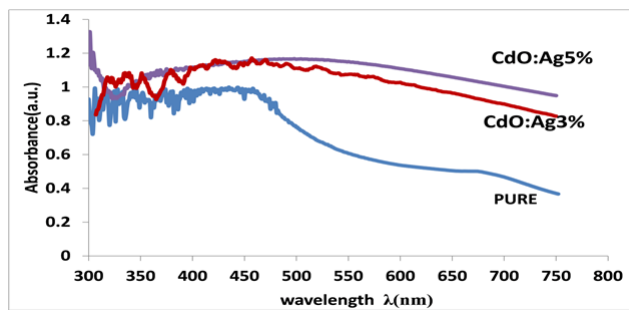


Figure 8: Absorption spectra of Green synthesized for nano-particle of CdO and CdO: Ag (3, and 5)%wt.

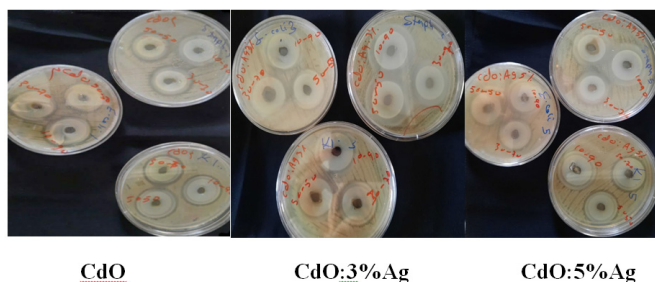


Figure 9: Antibacterial activity of green Synthesis CdO, CdO: Ag 3% and CdO: Ag5%NPs for bacterial strains (*S. aureus*, *E. coli*, and *K. pneumonia*).

Table 6: Anti-bacterial activity of green Synthesis CdO, CdO: Ag3% and CdO: Ag5% NPs for Bacterial strains (*S.aureus*, *E. coli*, and *K. pneumonia*).

Name of the bacteria	CdO			CdO: Ag3%			CdO: Ag5%		
	10-90	30-70	50-50	10-90	30-70	50-50	10-90	30-70	50-50
<i>Escherichia coli</i>	36	35	32	34	31	32	30	32	32
<i>Staphylococcus</i>	34	35	31	38	35	36	36	33	33
<i>Klebsiella pneumonia</i>	36	35	33	35	34	33	35	35	33

peaks were creeping towards the right (red region) with the rise in silver concentration, which leads to a decrease in the energy gap with increasing attention. These numbers represent the visible portion of the electromagnetic spectrum.²³ These changes can be attributed to donor levels reacting inside the energy gap and near the conduction band, resulting in the absorption of low-energy photons. Consequently, an apparent increase in absorbance value also led to a creeping absorption edge. This agrees with Ahmed KH. Ibraheem.²⁴ These levels are increased by increasing deflection rates, thereby reducing the width of the gap.²⁵ Using basic absorption (electronic stimulation of the conduction band from the valence band), it is possible to identify the nature and magnitude of the band gap. Planck's law ($E_g = 1240/\lambda$)²⁶ may be used to obtain the band gap value E_g .

Table 5 displays absorption edge for CdO and CdO: Ag NPs. The band gap values are in the range (2.79, 2.67, and 2.46) eV. for pure CdO and CdO (doped with 3 and 5) %wt of Ag, respectively.

Evaluation of Anti-bacterial Agents using the “Zone of Inhibition”

The anti-bacterial sensitivity test was performed on gram-positive bacteria *Staphylococcus aureus* and gram-negative and *Escherichia coli* and *Klebsiella pneumonia* to check the efficacy of green synthesized CdO, CdO: Ag NPs with three concentrations of *Moringa* leaf extract in CdO: Ag solution, using the agar well method. It was noticed that there was a cleared zone around the experimental wells that inhibited the development of bacteria after 24 hours of incubation at 37°C, as shown in Figure 9 and Table 6. It is clear that Green synthesized CdO: Ag NPs have good inhibitory properties against *Klebsiella* and *Staphylococcus* and less inhibitory capacity against *E. coli* plate; this agrees with Shilpashree Mayachar K *et al.*²⁷ and with Shukla M *et al.*²⁸ Further, the green synthesized NPs show higher antibacterial activity at the concentration of 10-90 for CdO: Ag 3% and lower activity at the concentration of 50-50 for pure CdO.

CONCLUSION

In this study, pure NPs of CdO and CdO doped with Ag were prepared successfully by the green synthesis method. The synthesized NPs' morphological, structural, compositional, and optical characteristics were thoroughly studied using various methods. The production of CdO with a face-centered cubic (FCC) crystal structure was confirmed by XRD analyses. The produced NPs' grain sizes were estimated. And

found in the range of (15.32–36.84) nm. The roughness of the surface increases from 2.89 nm to 18.77 nm with an increase in Ag concentration. The optical energy bandgap decreased from 2.79–2.46 eV as the concentration increased, while the absorption increased with the Ag concentration. These results can be used in different applications such as solar cells applications and medical application.

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