

# Research Article Cadmium Sulfide Nanoparticles: Synthesis, Characterization, and Antimicrobial Study

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Group II–VI cadmium sulfide nanoparticles (CdS NPs), a potential alternative to antibiotics, were synthesized using cadmium acetate [Cd(Ac)<sub>2</sub>] as a precursor, sodium sulfate as a reducing agent, and starch as a capping agent through chemical precipitation techniques. The CdS NPs were characterized using different characterizing techniques; X-ray diffraction and selected area electron diffraction patterns verify that the nanoparticles had cubic zinc blende structure with average dimension size of 2.43 nm. Scanning electron microscope and transmission electron microscope images illustrated that the particles were uniformly distributed in cluster form, and FTIR and EDX confirmed the synthesized nanoparticles were pure. The CdS NPs show relevant antimicrobial sensitivity against Grampositive and Gram-negative bacterial strains and the zone of inhibition increases with the increase in the concentration of nanoparticles. In comparison to Gram-positive and Gram-negative bacteria, the synthesized nanoparticles were reported to be more effective toward *Staphylococcus aureus*, Gram-positive bacteria.

# 1. Introduction

A Group II–VI compound [1], an *n*-type semiconductor with a wide-bandgap of 2.4 eV [2], outstanding electronic and optical properties [3, 4], zero-dimensional quantumconfinement [5] CdS NPs have appeared as the most relevant antimicrobial agents [6]. Furthermore, the CdS NPs extensively used for research on different biological application areas such as molecular histopathology [7], biological imaging [8], and advanced disease diagnostics [9, 10]. Besides this, they have tremendous technological operation in fields such as solar cells [11], photoluminescence, photodetector, photocatalysis [4, 5], light-emitting diodes [12], laser [5], high-density magnetic information storage [3], field-effect transistors, biological sensors [6, 13], environmental sensors, and many others in semiconductor industries [3]. Recently, nanometals such as Ag, Au, Cu, Cd, Al, Mg, and Ti. [6, 14] and their derivatives are used for killing microbes as an alternative to antibiotics [15] because nanoparticles have a large surface-to-volume ratio, which increased the chance to expose the positive charge on bacterial cells [16]. Nanoparticles have broad-spectrum antimicrobial activity [17] although their working principle is mysterious [18]. The most acceptable mechanisms for biocidal activity of nanoparticles are (1) bacterial cell membrane disruption and penetration, (2) formation of reactive oxygen species on the surface, and (3) interaction of charged species with the bacterial organelles, proteins, and DNA [15].

In the last few decades, highly stable [19] with outstanding chemical, physical, and structural properties [9, 17] CdS NPs get synthesized using many methods such as solvothermal, vacuum vapor deposition, hydrothermal, photochemical, sonochemical, microwave-assisted, microemulsion, thermal deposition, chemical precipitation [5, 20, 21] from different cadmium precursors such as cadmium chloride [22], cadmium nitrate [23], cadmium acetate [24], and cadmium sulfate [13]. In the present study, CdS NPs are synthesized by chemical precipitation techniques as this technique is simple, less time-consuming, inexpensive, and does not require specialized equipment and organic solvents [5]. Nanoparticles get agglomerated during the chemical precipitation synthesis method; therefore, the starch worked as capping agents to cease agglomeration and oxidation [25]. The structural and morphological characteristics of the synthesized NPs were extensively examined using X-ray diffraction (XRD), selected area electron diffraction (SAED),

transmission electron microscope (TEM), scanning electron mictroscope (SEM), energy-dispersive X-ray spectroscopy (EDX), and Fourier transform infrared spectroscopy (FTIR) techniques. The antimicrobial properties of as-prepared nanoparticles were also investigated at different concentrations against *Staphylococcus aureus*, a Gram-positive, and *Escherichia coli*, a Gram-negative bacteria [26].

#### 2. Experiments

2.1. Materials and Methods. Analytical grade chemicals of cadmium acetate dihydrate  $[Cd(CH_3COO)_2 \cdot 2H_2O]$  (99.0%) as cadmium precursor, sodium sulfide (Na<sub>2</sub>S, 95%) as reducing agents, and starch as capping agent used for the synthesis of CdS NPs. All chemicals and reagents used in the research were parched from Thermo Fisher Scientific Pvt. Ltd., Mumbai, India, and distilled water was used as the solvent throughout the experiment. The reagents were used directly without further purification.

At first, 50 mL of 0.05 M precursor solution of cadmium acetate was mixed with 4 mL of 1% starch solution, and then 50 mL of 0.05 M sodium sulfate solution was run dropwise into the mixture solution. The mixture was stirred for 4 hr at room temperature and left for aging and sedimentation. Finally, the mixture was filtered, and the residues were washed with distilled water a couple of times and dried in a hot oven at 90°C, and then the sample was subjected to characterization.

$$\begin{array}{l} Cd(CH_{3}COO)_{2} \cdot 2H_{2}O + Na_{2}S + Starch \longrightarrow Starch-[CdS] \\ + CH_{3}COO^{-} + Na^{+}. \end{array} \tag{1}$$

2.2. Characterizations Techniques. The structure and morphology of the synthesized nanoparticles were investigated with the help of characterization techniques. The crystalline structure and grain size of the prepared nanoparticles were figured out by using powder X-ray diffraction (XRD) (Rigaku Ultima IV model, using Cu K $\alpha$  radiation of wavelength,  $\lambda = 0.15406$  nm), based on Bragg's Law ( $n \lambda = 2d \sin \theta$ ). However, the average crystalline size "D" of nanoparticles is estimated using Debye–Scherrer's formula:

$$D = \frac{0.94\,\lambda}{\beta\cos\theta},\tag{2}$$

where  $\theta$  is the diffraction angle and  $\beta$  is the full width at half maxima (FWHM) in radians.

The synthesized nanoparticles' surface morphology and shape were evaluated using TEM (Tecnai G2 20 electron microscope) and SEM (JEOL model JSM-7600F), while the elemental composition of the nanoparticles was studied using EDX equipped with SEM.

2.3. Antimicrobial Activity. The agar-well diffusion method was used for the study of the antimicrobial activity of the synthesized samples, where two bacterial strains, namely, *S. aureus* (ATCC), Gram-positive bacteria, and *E. coli* (ATCC), Gram-negative bacteria, were taken from Polytechnic Research Institute of Nepal (PORIN), Kathmandu, Nepal, and then they were grown in the culture tubes in the culture's media of LB Agar (Luria Bertani) and stored at 4°C



FIGURE 1: XRD pattern of CdS NPs and corresponding Lorentzian profile fitting.

temperature for 24 hr. The stock solution of CdS nanoparticles was made at 100 mg/mL in dimethyl sulfoxide (DMSO) and diluted to 50 and 25 mg/mL. Furthermore,  $50 \,\mu$ L of each sample solution and a negative control sample of DMSO were introduced very carefully in the wells labeled in Petri plates, prepared in Muller Hinton Agar, respectively, with the help of the micropipette. Finally, the media plates were left for a while to diffuse with the sample. Furthermore, the diffuse sample was incubated at 37°C in the incubator for 24 hr.

#### 3. Results and Discussion

3.1. Crystalline Analysis. Figure 1 stands for the XRD patterns of as-prepared CdS NPs using Cd(Ac)<sub>2</sub> and the corresponding Lorentzian profile fitting. Four distinct diffraction peaks were reported in the spectrum between  $2\theta$  values of  $20^{\circ}$  and  $80^{\circ}$ , unveiling the crystalline structures. Furthermore, the synthesized nanoparticles consist of well-defined lattice planes with *hkl* values of (111), (220), (311), and (331) corresponding at  $2\theta \sim 26.4^{\circ}$ , 43.8°, 51.9°, and 71.2°, respectively, which were the signature indicators of cubic CdS crystal (ICDD PDF 89-0440) [3]. Using Debye–Scherrer's formula, the average crystalline size of the synthesized CdS NPs is 2.43 nm. And the experimentally analyzed *d*-spacing value of 0.336 nm at a highly intense peak (*hkl* value (111)) closely matches with 0.336 nm of cubic CdS NPs (CDD PDF 89-0440) [27].

The synthesized sample's *d*-spacing or interplanar spacing "*d*" was analyzed at different peak positions using Bragg's conditions [28].

$$n\,\lambda = 2d\sin\theta.\tag{3}$$

The lattice parameters of the nanoparticles were evaluated using Equation 4 [27]:

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2},\tag{4}$$

where *d* is the atomic lattice spacing, *a* is the lattice parameter of the crystal, and *h*, *k*, and *l* are Miller indices.

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FIGURE 3: SAED pattern of CdS NPs.

TABLE 1: Position, *d*-spacing, *hkl* value, crystal size, and bond length.

$2\theta$ (degree)	$\beta$ (radian)	<i>hkl</i> plane	<i>d</i> (nm)	" <i>a</i> " lattice parameter (nm)	Crystal size (nm)	Average size (nm)	Bond length (nm)
26.5	0.09145	111	0.3360	0.5819	1.6260		
44.5	0.06126	220	0.2034	0.5753	2.5541	2.43	0.2034
51.5	0.05148	311	0.1773	0.5880	3.1232		

The optimized cubic molecular geometry of the synthesized CdS nanoparticles at different lattice planes was drawn using Avogadro's 1.2 version software and represented in Figure 2. The bond length and unit cell volume of synthesized CdS NPs calculated as 2.03469 and 64.0 Å, respectively, and lattice parameters were reported as "*a*" 0.58 nm. The value of *d*-spacing, lattice parameter, average size, and bond length at  $2\theta$ -positions are shown in Table 1.

The SAED techniques were used to investigate the structure of nanoparticles, and Figure 3 stands for the characteristic SAED diffraction ring patterns (111), (220), and (311) of the synthesized CdS NPs, which correspond to the face-centered cubic phase of the reference CdS NPs zinc blende structure (JCPDS no. 89-0440) [29]. Furthermore, the information obtained from SAED data is well supported by the XRD.

3.2. Morphological Analysis. SEM image of CdS NPs synthesized using  $Cd(Ac)_2$  by chemical precipitation is shown in Figure 4. SEM image shows that the primary particles of CdS nanoparticles obtained from the cadmium sources get aggregated into secondary particles because these nanoparticles have tiny dimensions and enormous surface energy.

Similarly, the TEM image of the fabricated CdS NPs using  $Cd(Ac)_2$  is shown in Figure 5. The TEM image insight data about the CdS nanoparticles like morphology, stabilization, and size of the nanoparticles. TEM image revealed that the particles were homogeneously distributed and identical



FIGURE 4: SEM images of synthesized CdS NP.



FIGURE 5: TEM images of synthesized CdS NPs.

in size in assemblage form. The average particle size was evaluated from the TEM image using the ImageJ software, and most of the size fell in the range of 0.5–2.5 nm, which agreed with the size calculated from the XRD using Debye–Scherrer's formula. The histogram of the particle size distribution and corresponding Gaussian fitting for CdS NPs are shown in Figure 6.

3.3. Structural Analysis. FTIR spectrum of Synthesized CdS NPs are reported in the wavelength from 4,000 to 500 cm<sup>-1</sup> in Figure 7. The broad peak appeared at 3,400 and 1,628 cm<sup>-1</sup> signified O–H stretching and O–H bending modes, resulting from the interaction of CdS with water [30]. Significant peaks of synthesized materials (Cd–S bond stretching mode) are found in the fingerprint regions (peaks 570 and 650 cm<sup>-1</sup>) of the spectrum [31]. The O–C=O asymmetrical stretching vibration gives its intense peak at 1,350 cm<sup>-1</sup> [32].

3.4. Elemental Analysis. The purity and composition of synthesized nanoparticles were reviewed using EDX. Figure 8 is the EDX spectra of the CdS nanoparticles synthesized using the precursors of 0.05 M Cd(Ac)<sub>2</sub>, while the optical absorption peak (at 3–4 keV) corresponds to the specific metallic CdS nanocrystallites, and the peaks are because of surface plasma resonance [7]. The weight percentage of Cd and S in the synthesized sample were 54.83% and 9.52%, respectively; however, in the pure CdS molecules, Cd and S's average



FIGURE 6: Particle size distribution histogram and corresponding Gaussian curve fitting.



FIGURE 7: FTIR spectrum of CdS NPs.

atomic mass percentage are 77.8% and 22.19%, [27] respectively, shown in Table 2. Both the XRD and EDX results confirmed that the synthesized CdS NPs are in a pure and stable state in nature.

During the sample analysis, samples were coated with platinum; oxides were due to unnecessary oxidation of the sample, while the other trace of impurities may be inscribed in the report because they had stemmed from starting reagents or residues of the reactant.

3.5. Antimicrobial Analysis. The antimicrobial properties of synthesized CdS NPs were inspected against clinical pathogenic micro-organisms like *S. aureus* and *E. coli*, as shown in Figures 9 and 10, respectively. The zones of inhibition of the CdS NPs at different concentrations studied against Grampositive bacteria, Gram-negative bacteria, negative control (DMSO), and standard tetracycline antibiotics [4] are interpreted in Table 3. The antimicrobial sensitivity test confirmed



FIGURE 8: EDX of synthesized CdS NPs.

TABLE 2: EDX results showing the elemental composition.

Elements	Symbol	Atomic mass	Atomic mass percentage (%)	Weight percentage in sample (%)
Cadmium	Cd	112.411	77.8	54.83
Sulfur	S	32.065	22.19	9.52



FIGURE 9: Antimicrobial activity of CdS NPs against Gram-positive.



FIGURE 10: Antimicrobial activity of CdS NPs against Gram-negative.

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Micro-organism	Concer	ntration of CdS zone of inhibitio	NPs and	DMSO (negative control)	Standard tetracycline (5 µg/disc)
	25 mg/mL	50 mg/mL	100 mg/mL		
Staphylococcus aureus	5.5 mm	6.5 mm	10.0 mm	0 mm	22 mm
Escherichia coli	5.0 mm	5.5 mm	6.5 mm	0 mm	22 mm

that the synthesized nanoparticles have significant antimicrobial properties.

As the particles are nanodimensional, they have a wide surface area and more surface atoms than their bulk counterpart [6]. The degree of microbial vulnerability of CdS NPs depends on the concentration of nanoparticles. With the increase in the concentration of nanoparticles, a larger number of CdS NPs encounter the bacterial cells, and as a result, it will have more probability of interaction. Nanoparticles with dimensions less than 10 nm will interact with bacteria and generate electronic effects, which increase the reactivity of nanoparticles [33]; therefore, the zone of inhibition increases with the increase in concentration.

The Gram-positive and Gram-negative bacteria have different cell-wall structures. Gram-positive has a thick layer of linear polysaccharide chain membrane on the other hand; Gram-negative has a thin layer membrane [26]. The size of the inhibition action of CdS NPs, for a different strain of bacteria, depends on several variables, including the interaction of the positive electrostatic charge of CdS NPs with the negative charges of proteins present in micro-organisms. The  $Cd^{2+}$  ions of nanoparticles react with the thiol group of protein, to release the reactive oxygen species, which disrupts the cells [34]. When the nanoparticles bind with the protein layer, there will be a disturbance in active transport, enzymatic activity, and dehydrogenase, which causes the inhibition of DNA, RNA, and protein synthesis, which results in cell death [35, 36]. Thus, *S. aureus* has better antibacterial activities with a large zone of inhibition compared with *E. coli*.

## 4. Conclusion

Broad-spectrum, stable, and pure CdS NPs were successfully synthesized using 0.05 M concentration Cd(Ac)<sub>2</sub> by chemical precipitation method. The synthesized nanoparticles were cubic in crystalline structure with an average dimensional 2.43 nm. The morphology and structure of the synthesized nanoparticles were confirmed using the XRD, SAED, FTIR, SEM, TEM, and EDX techniques, and their antimicrobial activities were studied using the agar well diffusion method. Both Gram-positive and Gram-negative bacteria showed sensitivity to the synthesized CdS NPs, and the zone of inhibition increased with increased concentration. Synthesized nanoparticle nanoparticles showed high efficiency toward the Gram-positive bacteria *S. aureus* and reported significant virulence toward both strains of bacteria.

### **Data Availability**

On request, the data used to support the conclusions of this study may be obtained from the corresponding author.

#### **Conflicts of Interest**

The authors declare that they have no conflicts of interest.

# **Authors' Contributions**

Surendra K. Gautam: conceptualization, supervision, and review and editing; Amrit Regmi: manuscript writing, experimentation, data curation, and review and editing; Yamlal Basnet: experimentation, data analysis, and draft writing; Sitaram Bhattarai: data analysis and characterization. All authors read and approved the final manuscript.

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