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RESEARCH ARTICLE

Structural, Optical and Antimicrobial Properties of Carbohydrate-Capped Cadmium Sulfide Nanoparticles

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Abstract

Cadmium sulfidenanoparticles (CdSNPs) were prepared through thechemical precipitation process where sweet potato starch and glucosefunctioned as capping agents. Ultraviolet-Visible (UV-Vis) spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy and X-ray diffraction (XRD) techniques were applied for the analysis of prepared CdSNPs. The absorption peaks in the UV-Vis spectroscopy towards the lower wavelength region compared to the bulk CdS (512 nm) indicated the formation of CdS particles in the nanometer scale range. FTIR spectroscopy showed the characteristic absorption peaks of the CdSNPs along with the functional groups present in starch and glucose, suggesting the possible interaction between CdSNPs and capping agents. The XRD revealed crystallographic information such as the average crystallite size and shape of CdSNPs. Using the Scherrer equation, the average crystallite size of CdSNPs was determined to be 11.48 nm and 16.03 nm for starch-capped and glucose-capped CdSNPs (CdS-St andCdS-Gl)respectively. Antibacterial and antifungal activities of the prepared CdSNPs were found to be highly efficient for various pathogenic bacteria and fungi. Hence in the present study, the new synthetic route of CdSNPs was applied, and antimicrobial activities were performed

Keywords: *Chemical precipitation, Cadmium sulfide nanoparticles, FTIR spectroscopy, X-ray diffraction, UV-Visible spectroscopy*

1. Introduction

In recent years, nanoparticles (NPs), having a diameter between 1-100 nm have gained much research concern because of their different attractive optoelectronic, electrical, and antimicrobial properties[1-4]. It has been indeed established that, on decreasing the size of materials to the range of nanometers, their physical, chemical, and biological properties drastically change owing to the large surface area, the existence of electrostatic force and resulting quantum size effects etc. [5]. As a result, the NPs are used these days in advanced applications including energy generation, environmental remediation, food industries and medicine [6-8].

Preparation and characterization of some important semiconducting nanomaterials such as CdO, ZnS, ZnO, CdS, CdSe and CdTe NPs are comprehensively reported in the literature[9, 10]. In this regard, the CdS NPs are one of the most widely studiedbinary chalcogenidesamongst II-IV groups, owing to their wide band gap energy of 2.43 eV in the bulk state and resulting in excellent photosensitivity[11,12]. Different chemical methods comprising

microemulsions, condensation and hydrothermal techniques, the use of ultrasonic radiation [13-17] as well as biological processes [18]are used in the preparation of CdSNPs.However, the use of these methods was limited because of low yields, excessive cost, tedious syntheticprocedures, andthe use of various toxic chemicals. In those methods, various chemicals such as dimethylformamide, thioglycerol, thiourea, sodium dodecyl sulphate, diethylenetriamine, cetyltrimethylammonium bromide, 3-mercaptopropionic acid and thiophenol have been used as capping agents which might cause health and environmental issues[13-19].

Cadmium sulfide nanoparticles(CdSNPs) find wide applications inphotocatalysis, biosensors and targeted drug delivery[20,21], one of their vital applications being their use as novel antibacterial and antifungal agents [22-25]. The electrostatic interaction between positively charged cadmium ions and negatively charged proteins on the cell wall of bacteria might disrupt the latter due to the formation of reactive oxygen species[26, 59] which makes the CdSNPs an effective antibacterial agent. Similarly, the antifungal activity of the CdSNPs has been studied by researchers [23, 24,26-29]. It has been found that when fungi are exposed to NPs for a prolonged period, they may form pits on their cell surfaces causing pores formation and apoptosis [27, 28].

This work aims to synthesize CdSNPs by using non-toxic, less hazardous, and eco-friendly green capping agents such as sweet potato starch (SPS) and glucose. The synthesis was carried out by a simple, less time-consuming, and economic chemical precipitation method[30]. The synthesized NPs were characterized by FTIR spectroscopy, UV-Vis spectroscopy and powder XRD. The synthetic route of CdSNPs was optimized and the antibacterial and antifungal properties ofCdS NPs were also investigated against different microbial strains.

2. Material and methods

2.1 Materials

Cadmium nitrate ($Cd(NO_3)_2$), sulphuric acid (H_2SO_4), sodium hydroxide (NaOH), ethanol (C_2H_5OH) and glucose were purchased from Fisher Scientific, India. Starch was extracted from sweet potatoes collected from the nearby area in Tribhuvan University, Kathmandu using standard procedure [31]. All solutions were prepared in distilled water. A Gram-positive bacterial strain viz. *Staphylococcus aureus* (ATCC 25923) and three Gram-negative species viz. *Escherichia coli*(ATCC 25922), *Salmonella typhi* and *Pseudomonas aeruginosa* (ATCC 27853) as well as four fungal strains viz. *Aspergillusniger, Aspergillusflavus, Trichoderma*and*Rhizopus* were used for antimicrobial tests.

2.2 Preparation of solutions

Preparation of SPS solution: Fresh sweet potato rhizomes were taken from Kathmandu and cleaned with distilled water for the removal of all kinds of impurities. Then rhizomes were peeled off, sliced into small pieces, and air-dried. 50 g of the dry sweet potato slices were taken in a dry and clean kitchen blender and ground into a smooth slurry together with distilled water. The slurry was filtered through a finely fabricated cotton cloth to obtain starch suspension which was then settled for 12 h in a refrigerator maintained at 10°C. The supernatant liquid was removed followed by drying the collected sediment at 45°C for 7 h in a hot air oven to get the required SPS. Then, 7 g of thus obtained SPS was mixed with 100 mL of water and10 mL of the SPS solution was used in the synthesis of CdSNPs.

Preparation of glucose solution: 1.8 g of purchased glucose was mixed in 100 mL of water and 10 mL of the glucose solution was used in the preparation of CdSNPs.

2.3*Synthesis of CdSNPs*: Aqueous solutions of $Cd(NO_3)_2$(100 mL, 0.1 mol L^{-1}) were prepared in two separate round bottom flasks. The above-prepared starch and glucose solutions were then added dropwise to the separate solutions containing $Cd(NO_3)_2$ followed by continuous passing of H_2S gas under constant stirring at 500 rpm. The pH level was maintained at 10 by drop-wise addition of ammonia solution. The colour of the solution gradually changed from light to dark yellow. Both mixtures were allowed

toconstantly stir for 24 h for the completion of CdSNPs synthesis. The chemical reactions involved herein are given in equations (1) and (2).

$$Cd(NO_3)_2 + H_2S \xrightarrow{Starch} CdS\downarrow + 2HNO_3 \quad (1)$$

$$Cd(NO_3)_2 + H_2S \xrightarrow{Glucose} CdS\downarrow + 2HNO_3 \quad (2)$$

Theprecipitate was washed several times with distilled water followed by ethanol. Finally, the resulting clear orange-yellow pellets of CdSNPs were obtained by evaporating the solvents at 23°C. Thus, synthesized samples of Starch (St) capped andGlucose (Gl) cappedCdSNPs were denoted asCdS-Stand CdS-Gl,respectively.

2.4 Characterization methods

Fourier-transform infrared (FTIR)spectroscopy was used in the identification of the infrared spectra of capping agents and synthesized NPsusing an IR-Tracer-100 Spectrometer (Shimadzu, Japan) in the wavenumber range of 400-4000 cm^{-1}.

Ultraviolet-visible (UV-vis.) spectroscopywas performedusingChemito UV-Visible spectrophotometer for the analysis of the optical properties of synthesized NPs. The well-dispersed colloidal solution of CdSNPs was used and its absorbance was measured inthe wavelength region of 450-750 nm.

X-ray diffraction (XRD)used was D-2 Phaser Advanced Diffractometer (Bruker, Germany), run at 30 kV and 10 mA with monochromatic Cu-K∝ radiation performed under ambient conditions over the 2θ region of 20° to 80° at the rate of 2°/min. This method was used to determine the size and crystallography of the NPs. The crystallite size (D) was determined by using Scherrer's formula [32]:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

where,

D = the mean size of the ordered (crystallite) domains (in nanometers)

λ = X-ray wavelength (λ= 1.54046 Å for Cu-K∝ radiation)

β = full width at half maximum (FWHM) (in radian)

θ = Bragg's diffraction angle (in degrees)

2.5 Antimicrobial activity:

25 mg of CdSNPs was dissolved in 1 mLof dimethyl sulphonyloxide and screening, as well as evaluation of the antimicrobial activity, was performed [18]. For the antibacterial test, four bacterial species;*S. aureus*, *E. coli*, *S. typhi* and *P. aeruginosa*were cultured in Mueller Hinton Broth and 4 h after the culture, standard McFarland was matched. Thenbacterial strains were swabbed, and wells were made on Mueller Hinton Agar (MHA) plate. 50 µL of the prepared CdSNPs (25 mg/mL) and positive control (Neomycin, 1 mg/mL) were loadedinto the wells followed by incubation for a day at 37°C.

Similarly, for the antifungal tests, four fungal species;*A. niger*, *A.flavus*,*Trichoderma*and*Rhizopus* were cultured inPotato Dextrose Broth for 3 days at 28°C. These strains wereswabbed,and then wells were made on the Potato Dextrose Agar plate. 50µL of the preparedCdSNPs (25 mg/mL) and positive control (cycloheximide, 25 mg/mL) were loadedinto the wells. It was subjected to incubation for 3 daysat 28°C. The inhibitions in both above-mentioned tests were observed on the following day and zones of inhibition were measured.

3. Results and discussion

3.1 Structural characterization

Figure1depicts the FTIR plots of starch-capped and glucose-capped CdSNPs (CdS-St andCdS-Gl) powder. The broad absorption band around 3270 cm^{-1} (i.e., 3280 cm^{-1} forCdS-Stand 3265 $cm^{-1}$for CdS-Gl) is assigned to the O-H stretching mode of starch, glucose,and adsorbed water. Such an observation was also reported earlier for sweet potato and cassava starch [33]and bare CdS [34].

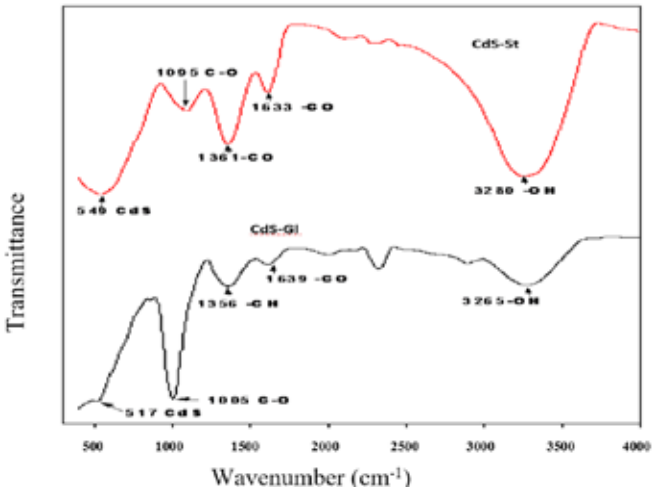

Figure 1. *FTIR spectra of starch and glucose-capped CdSNPs(CdS-St and CdS-Gl) in the range of 400-4000 cm^{-1}*

On moving towards the lower wavenumber region, the weak absorption peaks located at 1633 cm^{-1} (CdS-St) and 1639 cm^{-1} (CdS-Gl) were found which could be attributed to C=O stretchingof carbonyl functional groups present in starch or glucose or adsorbed atmospheric carbon dioxide in CdS [33-35]. On moving further towards the lower wavenumber region; the peaks at 1361 cm^{-1} (CdS-St) and 1356 cm^{-1} (CdS-Gl) were recorded, representingthe anti-symmetric bending of -CH_3 corresponding to starch or glucose [36,37]. The C-O andS-O stretching vibration of starch/glucose or sulphate respectively give its broad and weak peaks at 1095 cm^{-1} (CdS-St) and 1005 cm^{-1}(CdS-Gl)[38]. These observations convincingly support the template role of starch/glucose which prevents the agglomeration of CdSNPs. Moreover,a peak near 500-550 cm^{-1}suggestedthe metal-Sulphur (M-S) bond, indicating the synthesis of CdSNPs [39-41]. The small and weak absorptions at 549 cm^{-1}(CdS-St) and 517 cm^{-1}(CdS-Gl)areassociated with the characteristic peak of CdSNPs which correspond to the findings observed by P. S.Khewet al.[42], X.Lu et al.[43], X.Lu et al. [44], T. P. Martin et al. [45] and Z. Qiaoet al.[46]. Hence the presence of major functional groups of starch and glucose-capped CdSNPswas assessed by FTIR analysis. The data of FTIR analysis are tabulated in table 1.

3.2 Optical properties

The UV-visible spectrum of starch-capped and glucose-capped CdSNPs (**CdS-St** and **CdS-Gl)** is shown in figure2.

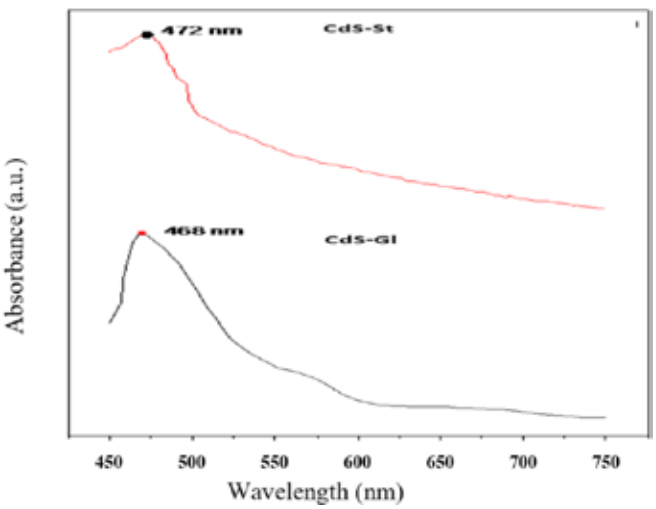

Figure 2.*UV-visible spectra ofstarch and glucose-cappedCdSNPs (**CdS-St** and **CdS-Gl**)*

The spectrum depicts a well-defined, blue-shifted absorption peak.The spectrum shows a well-defined absorption peak at 472 nm (2.63 eV) for CdS-Stand 468 nm (2.65 eV) for CdS-Gl. It was observed that the binding energy of the excitonofCdS-Stand CdS-Glwas slightly increased as compared to bulk CdS, which showsabsorbance at 512 nm (2.42 eV) as suggested byS.Muruganandamet al.[47]. This is attributed to the higher binding energy of the electron-hole pair

Table 1.*FTIR peaks of FTIR spectra of starch and glucose-capped CdSNPs (**CdS-St** and **CdS-Gl)**.*

Absorption (cm^{-1})		Functional Group	Intensity of Peak	Remarks and References
CdS-St	**CdS-Gl**			
3280	3265	O-H stretching	Strong, Broad	Starch, Glucoseor Water[33, 34]
1633	1639	C=O stretching	Strong	Starch, Glucose [33-35]
1361	1356	-CH bending of CH_3	Weak	Starch, Glucose [36, 37]
1095	1005	C-O stretching	Small	Starch or Glucose[38]
549	517	Cd-S stretching	Small, weak	CdSNPs[39-46]

by correlating with the report of M.Maleki et al.[48], suggesting the synthesis of CdSNPs. The quantum confinement of photo-generatedelectron-hole pair is associated with the nanometer scale particle size [49]. In this way,the formation of CdSNPs was confirmed by UV-visible spectroscopy and this result agreeswith the FTIR results explaining the presence of spectra associated withthe synthesizedCdSNPs.

3.3 Morphological characterization

X-ray diffraction (XRD) resultof powderedstarch and glucose-capped CdSNPs (CdS-StandCdS-Gl) is shown in figure3.

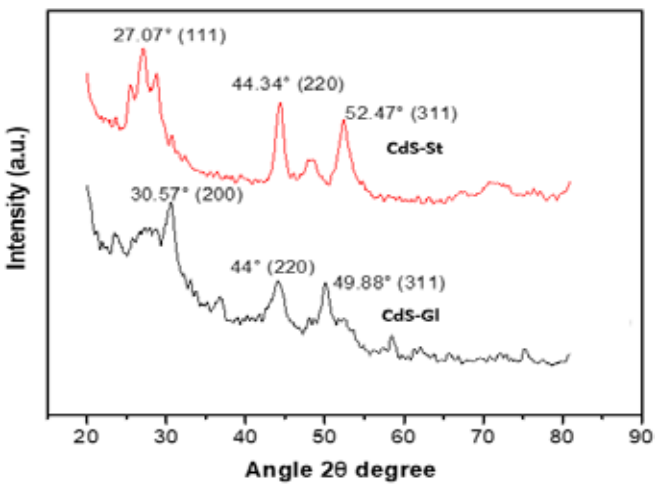

Figure 3. *X-ray diffraction graph of starchand glucose capped CdSNPs (**CdS-St** and**CdS-Gl**) in the region of 2Θ degree of 20°-80°*

The diffraction peaks for CdS-Stwere obtained at 2θ values of 27.57°, 44.34° and 52.47° corresponding to (111), (220) and (311) miller reflection planes while the diffraction peaks for CdS-Glwere obtained at 2θ valuesof30.57°,44.00°and49.88°correspondto(200), (220) and (311) miller reflection planesrespectively [50-52]. These outcomes comply with the peaks of pure CdS crystals, corresponding to the JCPDS file number 10-454 [53]. It is quite interesting to note that all the investigated samples have quite similar XRD patterns confirming the presence of the cubic (Zinc blende phase) structure, in agreement with Marin Jet al.[54] and Birajdar et al. [55]. The average crystallite size was calculatedwith the help of Scherrer's equation using the diffraction intensity of the peak that has the highest intensity[32]. The average crystallite size of CdSNPs was determined to be 11.48 nm and 16.03 nm for CdS-Stand CdS-Gl,respectively. It revealedthat CdS-Gl were agglomerated to a greater extent as compared to CdS-St.However, both capping agents restrict the particle size in the nanometer range, showing promising results in stabilizedCdSNPssynthesis[56].These findings are summarized in Table 2.Moreover,thediffraction peaksare broadened as compared to bulk CdSdue to the reduced crystallite size, extensive surface defects and powdered form of CdSNPs, as reported previously by R.Banerjee et al.[57] and H. L. Lee et al. [58].XRD analysis, accordingly, correlates with the results of UV-visible and FTIR spectroscopy which support the synthesis of CdSNPs.

Table 2. *Crystallite phase and average crystallite size obtained from X-ray diffraction data.*

NPs	2Θ (degree)	hkl	Crystallite phase	Average crystallite size (nm)
CdS-St	25.57 44.34 52.41	111 220 311	Cubic	11.48[50-55]
CdS-Gl	30.57 44.00 52.47	200 220 311	Cubic	16.03[50-55]

3.4 Antimicrobial activities

3.4a. Antibacterial activities

A comparison of positive and negative controls of the inhibition zone of CdS-St and CdS-Glis presented infigure4. The antimicrobial activities of the synthesized CdSNPs (CdS-S**t** andCdS-Gl) were studied by agar well diffusion assay against four bacterial species. Two species; *S. typhi*and *S. aureus* were found to be inhibited by the NPs while the rest of the bacteria were resistant.Therefore, active transport, enzymaticaction and formation of nucleic acidsare arrested in the periplasmic space, causingapoptosis [60].The inhibitions by the CdSNPs were observed to be lesser thanthe positive control whichis summarized in table 3.

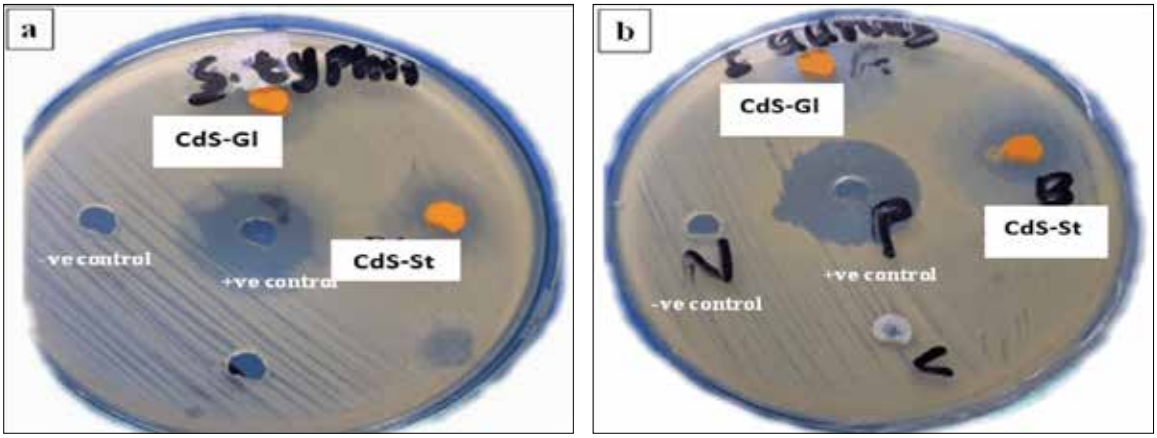

Figure 4.*Photographs showing antimicrobial tests of CdSNPs against (a) S. typhiand (b) S. aureus*

Table 3.*Summary of the antibacterial properties shown by CdSNPsagainst S. typhiand S.aureus, methods used as per literature works [59-60].*

Name of bacteria	CdSNPs	Size of the zone of inhibition (mm)		
		NPs	+ve control	-ve control
S. typhi	CdS-St	15 mm	25 mm	-
	CdS-Gl	10 mm	25 mm	-
S. aureus	CdS-St	8 mm	26 mm	-
	CdS-Gl	8 mm	26 mm	-

3.4b. Antifungal activities

The zone of inhibition of CdSNPs compared to the positive and negative controls is presented in figure5. The antifungal activity of two synthesized CdSNPs, CdS-Stand CdS-Gl were studied by well diffusion method against four fungal strains. Among them, only the fungal strain *A. flavus* was found to be inhibited by the CdSNPs, whereasthe rest of the fungi were found to be resistant.The inhibitions by the CdSNPswere observed to be lesser than the positive control whichis summarized in Table 4. As compared to antibacterial activity, the mechanism of antifungal activity by the CdSNPs is quite different. The fungal strains produce an excess of nucleic acid through the stress response of fungal hyphae against the CdSNPs leading to cell death [27, 28].

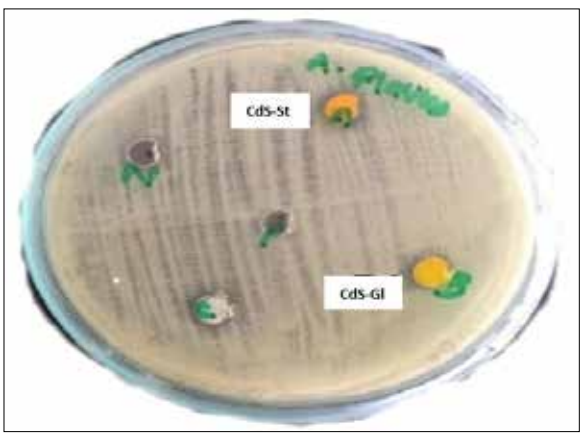

Figure 5.*Photographs showing an antifungal test ofCdSNPs against A.flavus*

Table 4. *Antifungal properties shown by CdSNPsagainst A. flavus[23].*

CdSNPs	Zone of Inhibition		
	NPs	+ve control	-ve control
CdS-St	6 mm	15 mm	-
CdS-Gl	6 mm	15 mm	-

4. Conclusions

This research work is encircled in an easy, inexpensive, and environmentallyfriendly way for the synthesis ofcarbohydrate-cappedCdSNPs. The work was completed under the chemical precipitation method for the synthetic procedure. The following conclusions have been drawn:

- Anew synthetic route of CdSNPs was applied.
- In FTIR analysis, the peak in the 500-550 cm^{-1}absorption region confirmed thecharacteristic peak associated withCdSNPs.Moreover, it contains the peaks associated with the

functional groups of starch and glucose which suggested that the particles were well capped by capping agents.

- UV-visible spectroscopy showed a well-recognized, blue-shifted absorption peak at 472 nm and 468 nm for starch and glucose-capped CdSNPs, respectively.
- XRDplotshowed the crystal structure inthe nanometer scale range. The average crystallite size of CdSNPswas determined to be 11.48 nm and 16.03 nm for starch-capped and glucose-capped CdSNPs, respectively. The XRDpattern also suggested the crystal structure of the latticewas in cubic phasewhich was marked by (111), (200), (220) and (311) miller indices of the lattice.
- CdSNPs were found to be highly effective for two bacterial strains*S. typhi*and *S. aureus* and a fungal strain*A. flavus*.

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