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Study of antimicrobial activity of sonochemically synthesized CdS nanoparticles

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ABSTRACT

Wide applications of CdS nanoparticles and their synthesis methods mark this material and method as an interesting area for researchers. In the present investigation, a novel sonochemical route was developed to synthesize CdS nanoparticles in aqueous media by varying pH. The precursors used for the synthesis of nanoparticles are hydrated cadmium acetate (Cd $(CH_3COO)_2$ $2H_2O$) and sodium sulfur (Na_2S) with CTAB as the capping agent. The capping agent and pH play a major role in controlling the size of the nanoparticle. Various techniques were used to characterize chalcogenide nanoparticles such as surface properties characterized by SEM and TEM, structural properties monitored by XRD and EDX. For confirmation of material UV-vis spectroscopy and FTIR analysis were carried out. To study the thermal properties of the nanoparticles in a controlled manner, differential scanning calorimeter (DSC) and thermo-gravimetric analysis (TGA) were used. It is found from the XRD pattern; the as-prepared CdS nanoparticles show cubic crystal structure having an average particle size of about 10 to 15 nm. Disc diffusion method was carried out to study the antimicrobial activity of CdS nanoparticles. The present study concludes that CdS nanoparticles show promising antimicrobial activity against selected bacterial and fungal strains.

Keywords: *Solid dispersion* Nanoparticle, Sonochemical, Cadmium sulfide, pH, CTAB, antimicrobial activity.

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INTRODUCTION

Metal sulfides (ZnS, CuS, HgS and MnS,etc) possess distinctive physical and chemical properties which mark them as potentially investigated material [1-3]. Among group II-VI, CdS is the most promising an intrinsic semiconductor material, which has a direct band gap of about 2.4 eV with the hexagonal wurtzite phase [4, 5]. CdS has a wide variety of applications such as solar cells, humidity sensors, photocatalysis, gas-sensing, nonlinear optical materials, optoelectronics, and so on [6-10]. CdS show three different forms, out of this hexagonal wurtzite crystal structure is found in majority, while zinc blend and a high-pressure rock salt phases are found in the nanocrystalline form [11]. Various methods were used to synthesize CdS nanoparticles in aqueous media such as Chemical Bath Deposition (CBD), sol-gel process, sonochemical method, solvothermal method [12-15], and so on. Recently Chandra et al, reported the preparation and optical studies of nanocrystalline CdS powders in aqueous media using SILAR method and revealed band gap energy of about 2.44 eV [16]. Khan Z et al examined the preparation of cubic CdS nanomaterial by precipitation method. It is also observed that due to confinement, CdS nanoparticles exhibit a significant increase in dielectric properties, especially at low frequencies [17]. Sanjay R. Dhagea et al applied the CBD method to prepare CdS nanoparticles with hexagonal phases to improve the photoluminescence properties and concluded that CdS nanocrystals undergo phase transformations and thermal stability at different annealing temperatures [18]. M. Kristl et al revealed the preparation of nanoparticles of CdS and CdSe by a sonochemical route for different applications in modern optoelectronic devices. They also revealed that with increasing temperature crystal structure changes from cubic to hexagonal [19].

In the present work, high-performance CdS nanoparticles with the desired morphology were prepared through a sonochemical method. The sonochemical method is novel, low cost, and uses harmless chemicals

as compared to other methods. Furthermore, the CdS nan oparticles were carefully investigated under XRD, SEM, TEM, DSC, and TGA characteristics.

MATERIAL AND METHODS

AR grade chemicals were used for the synthesis of CdS nanoparticles throughout and therefore no additional purification was required. For solution preparation, double distilled deionized water was used. An ultrasonicator with a 3.5L capacity, 40 kHz, Athena Technology, India was used for CdS nanoparticle synthesis. To make the solution homogenous, a magnetic stirrer with a 1.5L capacity, Remi, was used.

Synthesis of CdS nanoparticle

In the present study cadmium sulfide (CdS) nanoparticles were synthesized using a low-cost sonochemical route in an aqueous medium. Cadmium acetate dehydrates (Cd $(CH_3COO)_2.2H_2O$), Sodium Sulphide (Na_2S), and CTAB were used as precursor and capping agents respectively. During the synthesis of CdS, the nanoparticle capping agent control the size of the nanoparticle. An aqueous solution of 0.1M (2.66 g) cadmium acetate and 0.1M (0.78 g) Sodium sulfide and a small amount of 0.1 M CTAB were mixed and stirred constantly for 1 hour. NaOH was used to regulate the desired pH of the mixture such as 8, 9, and 10. Later, the mixture was moved into a round-bottom flask and then kept in a sonochemical bath (Ultrasonicator, 3.5 L, 40 kHz, Athena Technology, India) at room temperature. Here, solutions were irradiated to ultrasound irradiation for 60 minutes forms yellow coloredprecipitate at the bottom of the flask. This precipitate was washed by water and ethanol to remove out the impurities. Finally, to develop desired particle size, the precipitate was dehydrated in a furnace at 80°C for 6 to 7 hrs. Fig.1 represents a graphical illustration of the sonochemical fabrication of CdS.

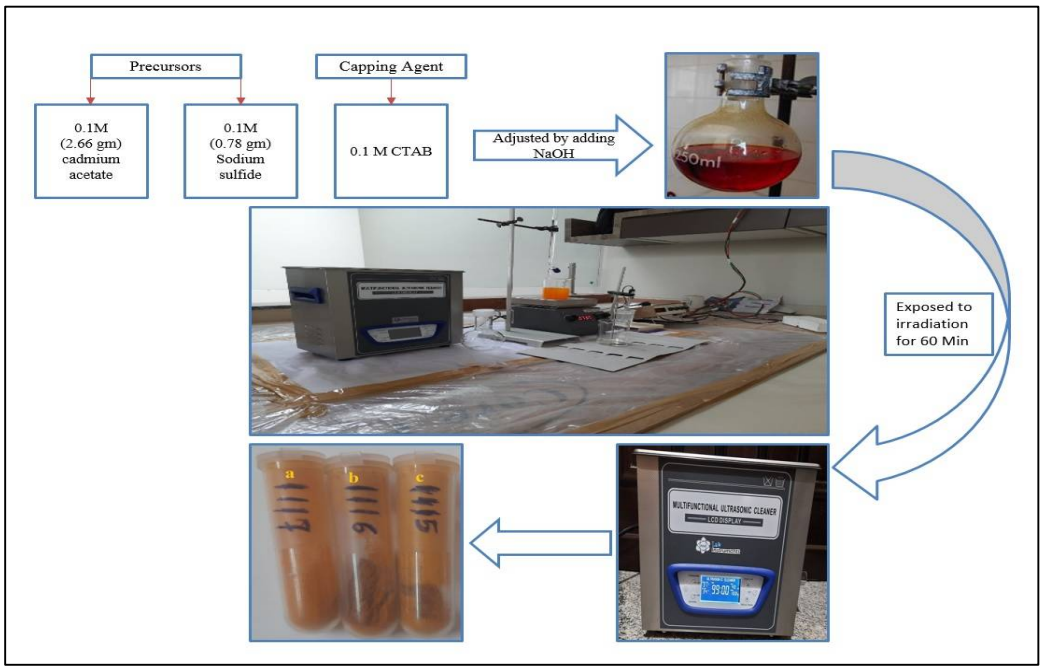


Fig.1 Graphical illustration of the sonochemical fabrication of CdS nanoparticles at different pH such as pH=8, 9, and 10

Characterization

In the present study various characterization techniques were used such as, UV-vis spectroscopy, FT-IR, SEM, EDX analysis, XRD technique, TGA and DSC. For material confirmation, absorption studies were recorded using Shimadzu UV-2450 spectrophotometer and FTIR spectrum was studied in the range of 4000 to 500 cm^{-1} using Shimadzu 8400S spectrophotometer in a KBr matrix. To analyze the structural properties of synthesized nanoparticles, XRD measurements were performed using Bruker D8-Advance XRD with Cu Kα (λ= 0.1542 A°) radiation and 2θ in the range of 20-80° (40 KV). The morphology of the as-prepared samples was studied using SEM and TEM analysis. The surface morphology of samples was characterized by using SEM and EDX techniques (Bruker X-Flash detector 6130 with gold coating). The microstructure of as-prepared nanoparticles was also studied by the TEM Tecnai G^2 microscope (Model JE0IJEMF-200) operated at 200kV.The thermal stability of the samples were carried out using DSC and TGA techniques. Thermogravimetric analysis (TGA) has been carried out with a TGA-55 analyzer (Denmark) from 0 to 800

°C (heating rate 20 °C /min). DSC analysis has been carried out with DSC-7020, Hitachi High Technologies Corporation, Japan analyzer from 0 to 300 °C (heating rate 10 °C /min).

Antimicrobial activity:

Cds nanoparticles were prepared at different pH 8, 9 and 10. Antibacterial activity of such Cds nanoparticles was checked against pathogenic bacteria like Gram positive bacteria viz; *Staphylococcus aureus, Bacillus subtilis, methicillin resistant Staphylococcus aureus* and Gram-negative bacteria like *E. coli, P. vulgaris*. Antifungal activity of Cds nanoparticles was checked against fungi are *A. niger*, *A. flavus* and *Candida albicans*. Antimicrobial assay was performed by disc diffusion. On the surface of nutrient agar (for bacteria) and potato dextrose agar (for fungi), these different pathogens were streaked and discs were dipped in nanoparticle solutions and kept on the plates. Nutrient agar and potato dextrose agar plates were incubated at 37 and 28^0C for 24 and 48h respectively.

Result and Discussion

UV-vis analysis: UV-Vis spectroscopy technique has been used to investigate the optical properties of CdS nanoparticles. Fig.2 shows the UV-Vis spectrum for the CdS nanoparticles by varying pH such as 8, 9, and 10 respectively recorded in the range of wavelength 200-800 nm. The obtained results are in accordance with preceding studies [20]. A strong absorption peak in the UV region was witnessed at wavelength of about 307.50 nm, 305.50nm, and 307 nm respectively. The UV-Vis study confirms the material synthesized was CdS.

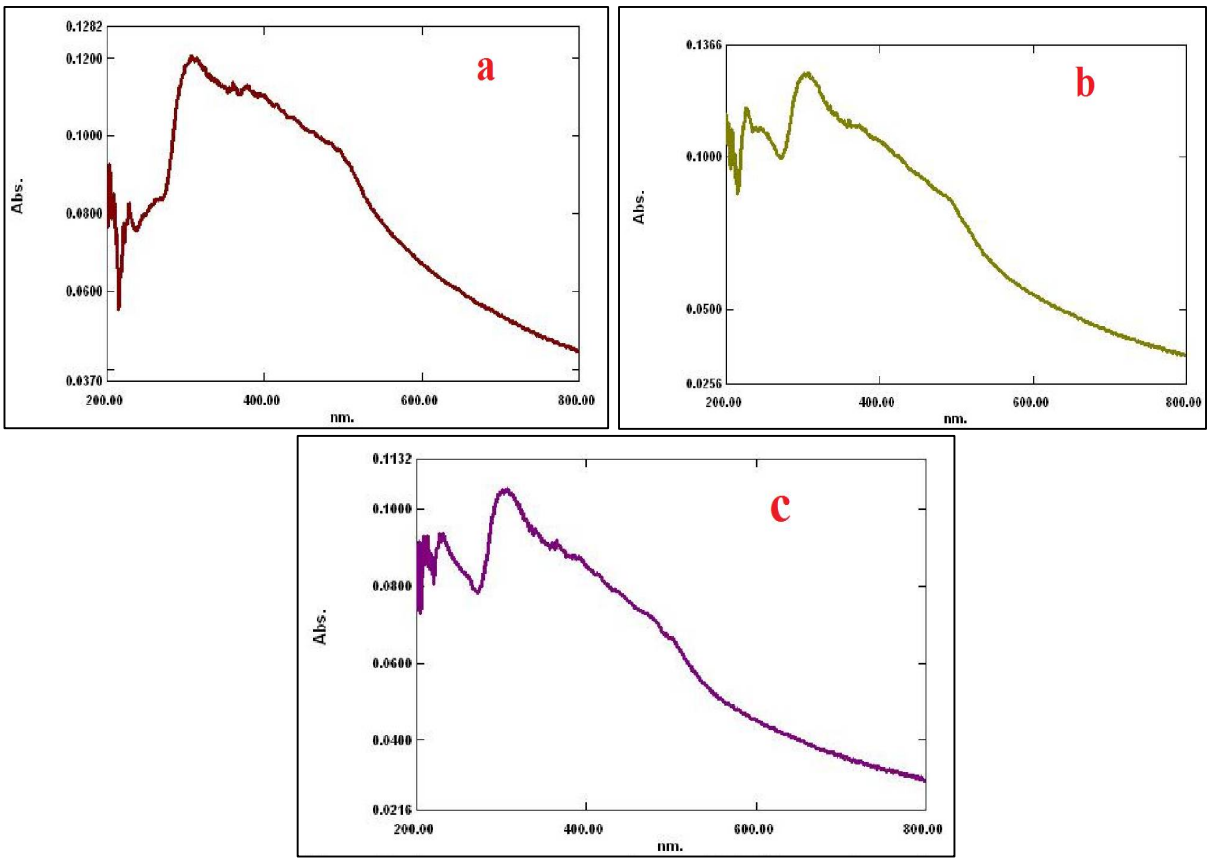


Fig.2 UV-Vis spectrum of CdS nanoparticles prepared at various pH values a) pH=8, b) pH=9, c) pH=10)

FTIR analysis: The purity of the synthesized material has been confirmed by using FTIR study. The identification of the functional groups present in the prepared material has been carried out using FTIR analysis. For FTIR analysis, the KBr pellets were prepared using a mixture of CdS nanoparticles and KBr. Fig.3 illustrates the characteristic spectrum for the CdS nanoparticles by varying pH such as 8, 9, and 10 respectively detected in the range of 4000-500 cm^{-1}. It reveals -OH group, due to absorption of water by the samples, through peak obtained in the range of 3600- 3100cm^{-1}. It also confirms asymmetric stretching of the carbonyl (C = O) group through the peak at 1100-1150 cm^{-1}. As the region 400-480 cm^{-1} was assigned to the metal-sulfur (M-S) bond the peaks recorded in the present study indicate the formation of CdS nanoparticles [21]. The characteristics strong absorption and the peak of Cd-S stretching were revealed in the range of 600 and 700 cm^{-1}[22].

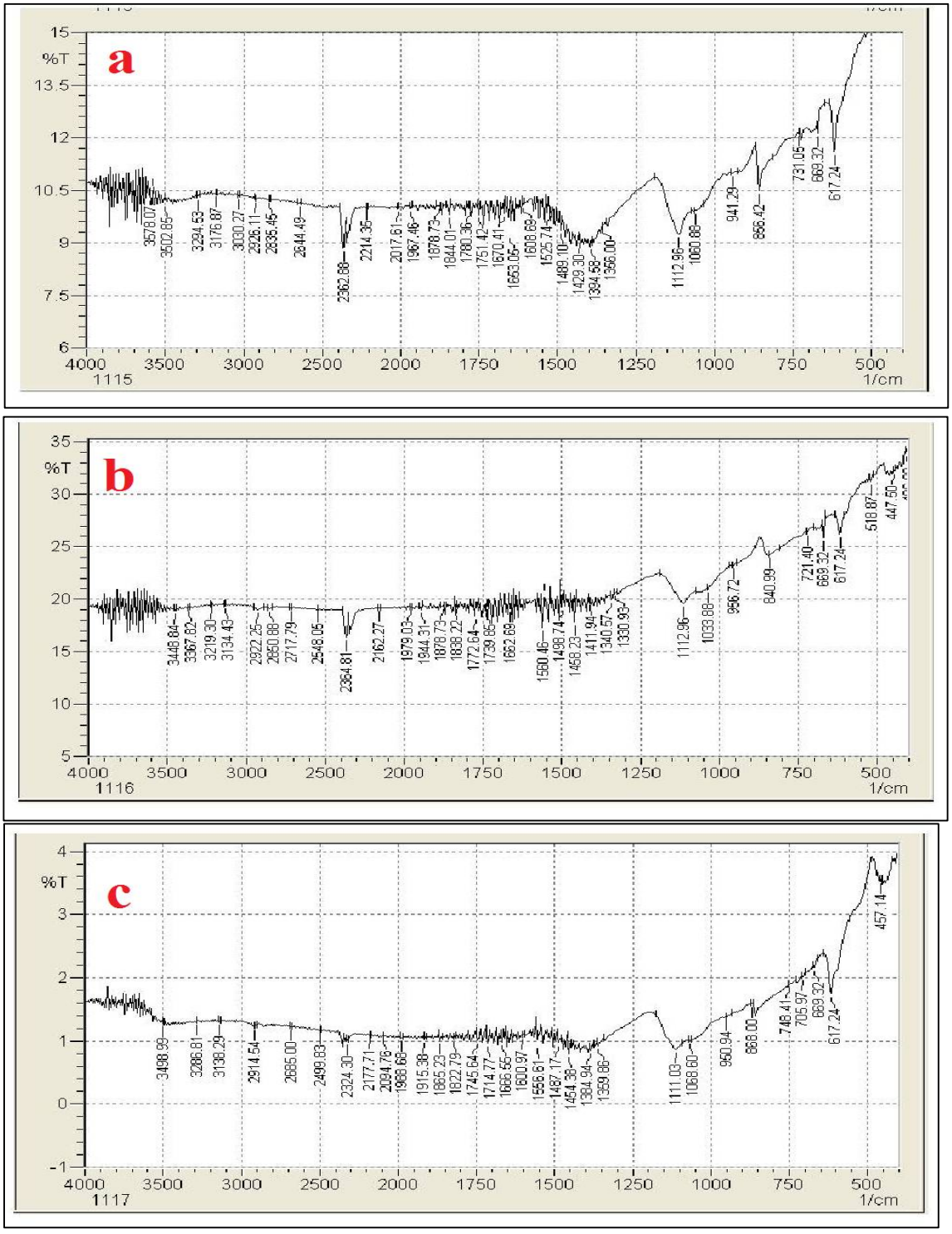


Fig.3FTIR spectrum of CdS nanoparticles prepared at various pH values a) pH=8, b) pH=9, c) pH=10

Structural characterization

X-ray diffraction analysis

XRD pattern for the CdS nanoparticles synthesized at various pH values (8, 9, and 10) were shown in Fig.4a), 4b) and 4c) respectively. CdS nanoparticles were confirmed by comparing obtained data with JCPDS card data. This comparison showed that all the diffraction patterns were indexed to the cubic structure. No other peaks were recognized in the XRD plot, specifying the phase purity of the sonochemically prepared CdS nanoparticles. The high intensity of the diffraction peak marked high crystallinity in the prepared CdS

nanoparticles. Debye-Scherer formula method was used to determine the crystalline size of CdS nanoparticles [23].

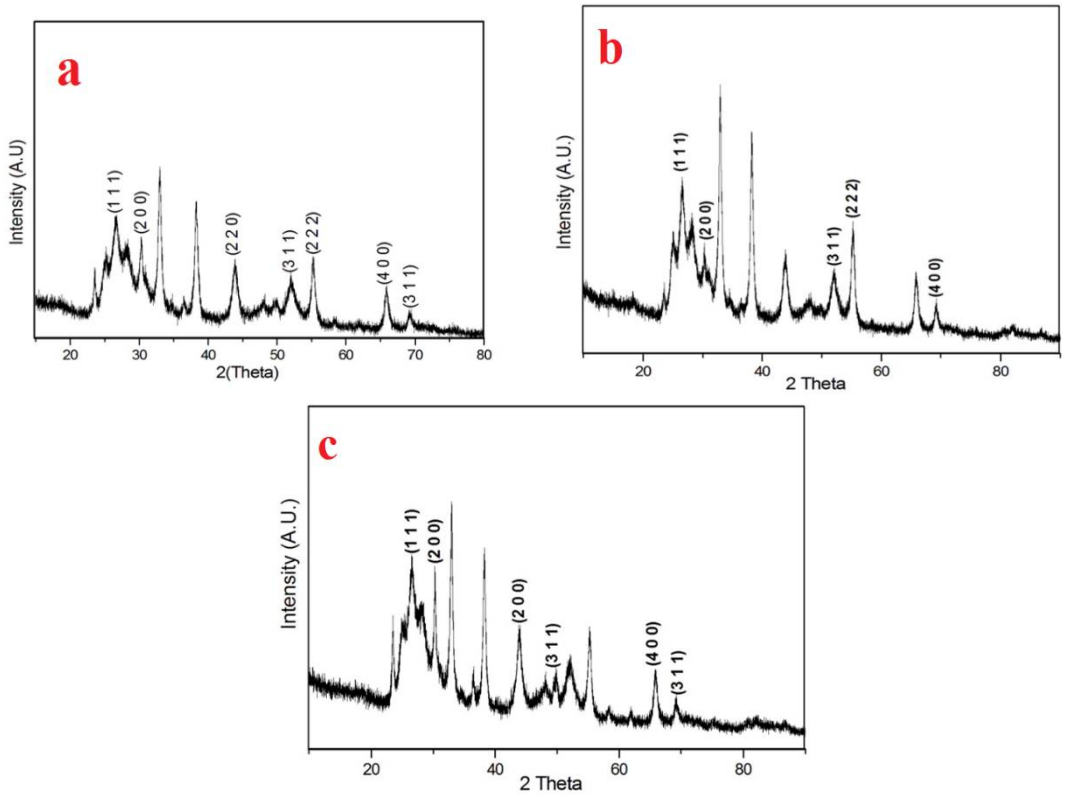


Fig.4 XRD Pattern of CdS nanoparticles prepared at various pH values a) pH=8, b) pH=9, c) pH=10

Fig.4a) shows distinct, noticeable XRD peaks of CdS nanoparticles at 2θ = 26.61°, 30.37°, 43.91°, 52.06°, 55.25°, 65.92° and 69.29° which corresponds to the (1 1 1), (2 0 0), (2 2 0), (3 1 1), (2 2 2), (4 0 0) and (3 3 1) planes respectively. It matches with JCPDS card No. 80-0019 and confirms the cubic structure of CdS nanoparticles. The peak broadening specifies unique nanocrystalline form of CdS nanoparticles with very small size.

Fig.4b) shows distinct, noticeable XRD peaks of CdS nanoparticles at 2θ = 26.59°, 30.38°, 43.89°, 52.08°, 55.24° and 69.26° which corresponds to the (1 1 1), (2 0 0) , (2 2 0) , (3 1 1), (4 0 0) and (3 3 1) planes respectively. It matches with JCPDS card No. 75-1546 and confirms the cubic structure of CdS nanoparticles. The peak broadens in g reveals the distinctive nanocrystalline form of the as-prepared nanoparticles which are very small in size.

Fig.4c) shows distinct, noticeable XRD peaks of CdS nanoparticles at 2θ = 26.86°, 30.27°, 43.90°, 52.05°, 55.23°, 65.90° and 69.26° which corresponds to the (1 1 1), (2 0 0), (2 2 0), (3 1 1), (2 2 2), (4 0 0) and (3 3 1) planes respectively. It matches with JCPDS card No. 10-0454 and confirms the cubic structure of CdS nanoparticles.

Table 1 Average grain size of CdS nanoparticles prepared at various pH values a) pH=8, b) pH=9, c) pH=10 [23]

Sample	$d_{(hkl)}$ at a) 2θ = 26.61 b) 2θ = 26.59 c) 2θ = 26.86	Lattice Parameter 'a' in Å	No. of Cell Per particle	Volume in $(Å)^3$	Average Grain Size in (nm)
a	3.354	5.811	6.262	196.22	12.66
b	3.361	5.820	6.296	197.13	10.35
c	3.360	5.818	6.226	196.93	14.93

Morphological characterization

SEM analysis

The surface morphology of CdS nanoparticles has been studied by using SEM images recorded using scanning electron microscopy as shown in Fig.5 a), b) and c) respectively. SEM images revealed a collection

of irregular particles and were randomly agglomerated. Moreover, SEM images show the particle size achieved nano form of the material. CdS nanoparticles produced by aqueous precipitation method showed the presence of spherical particles [24]. The particle size obtained was 20-50 nm, which showed a high correlation with XRD data.

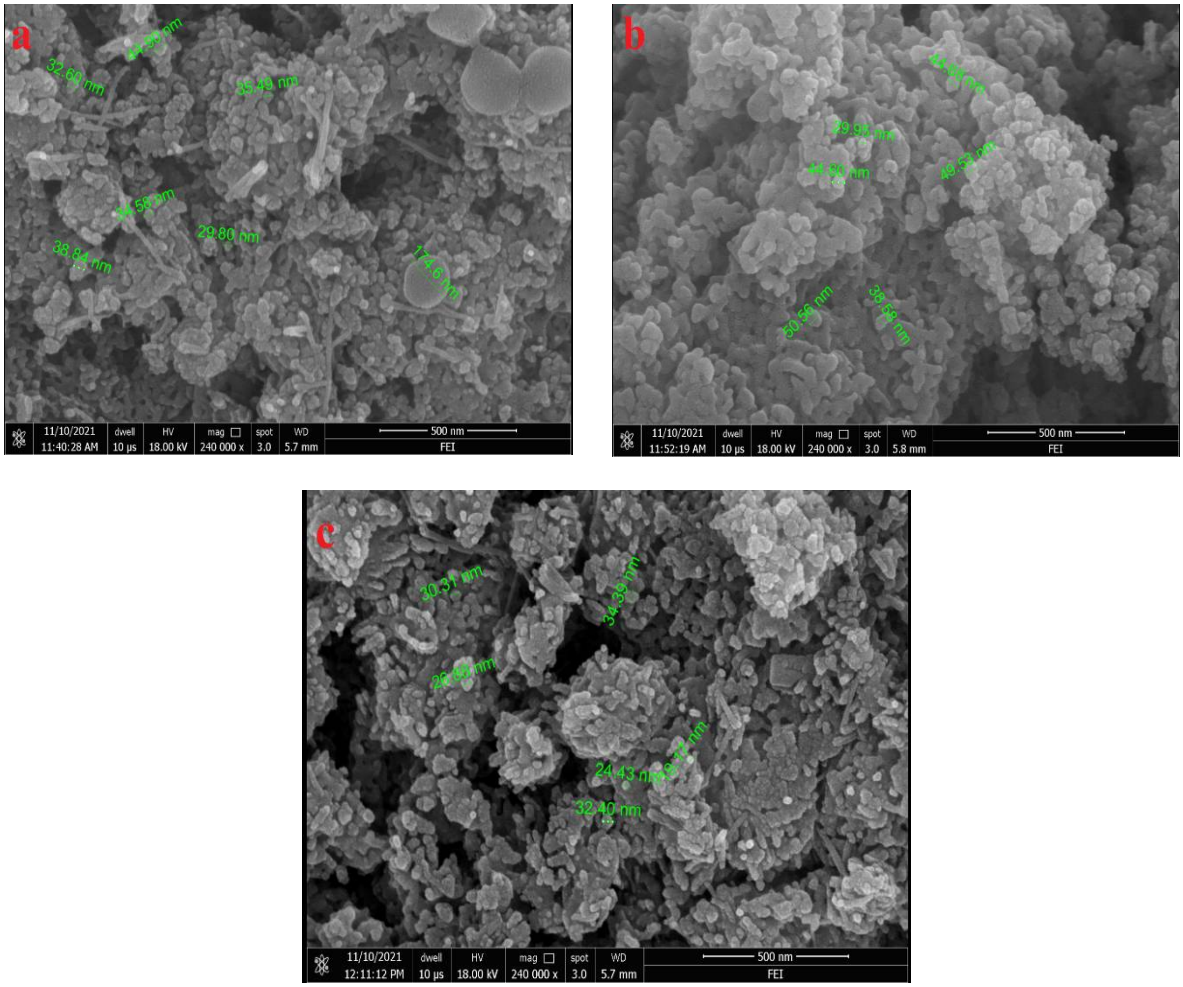
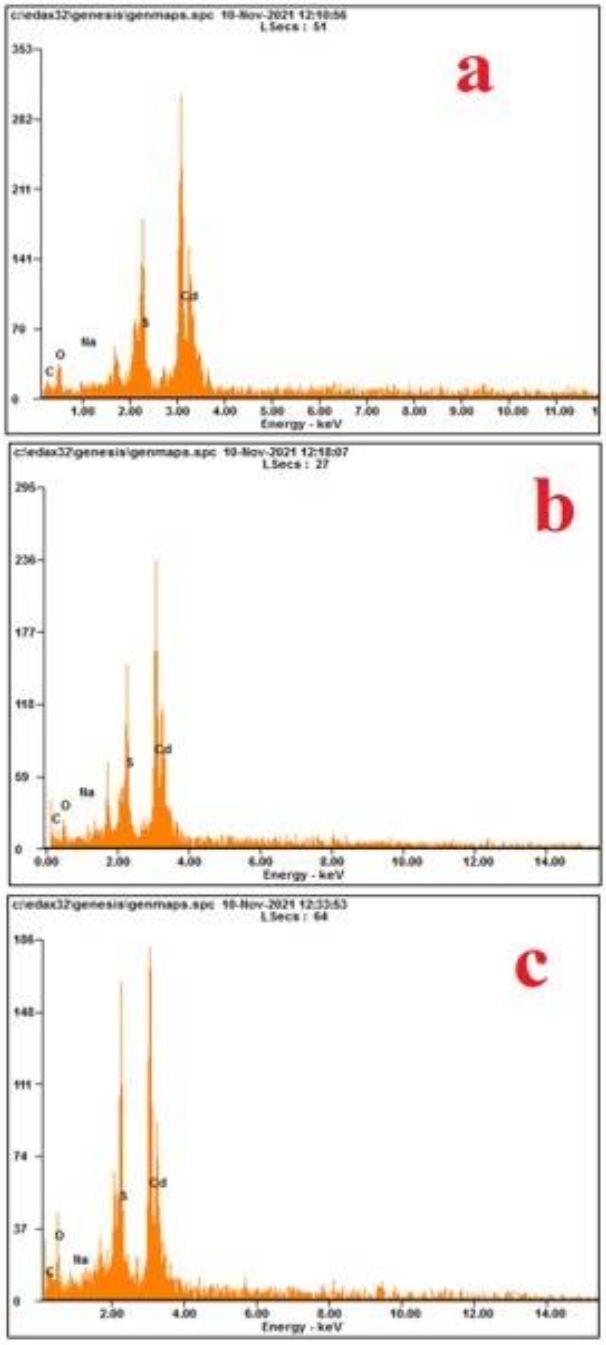


Fig.5 SEM micrographs of CdS nanoparticles prepared at various pH values a) pH=8, b) pH=9, c) pH=10

Compositional characterization

EDX analysis

The elemental composition of sonochemically prepared CdS nanoparticles has been calculated using EDX spectroscopy. The EDX analysis of CdS nanoparticles prepared varying pH values such as pH=8, 9, and 10 respectively, revealed Cd and S atoms as the elementary components in the prepared samples [25] shown in Fig.6. The low-intensity peak was identified due to precursors and carbon film used as a sample holder during EDX measurements.



Element	Wt%	At%
CK	12.37	32.12
OK	20.68	40.33
NaK	01.47	01.99
SK	10.61	10.33
CdL	54.87	15.23
Matrix	Correction	ZAF

Element	Wt%	At%
CK	12.07	31.29
OK	21.47	41.77
NaK	00.95	01.29
SK	10.84	10.52
CdL	54.66	15.14
Matrix	Correction	ZAF

Element	Wt%	At%
CK	14.12	32.04
OK	27.32	46.55
NaK	00.97	01.15
SK	10.35	08.80
CdL	47.24	11.46
Matrix	Correction	ZAF

Fig. 6EDX micrographs of CdS nanoparticles at various pH values a) pH=8, b) pH=9, c) pH=10

Morphological characterization

TEM analysis

SAED patterns and TEM micrographs were used to study the structure of sonochemically prepared samples at different pH. Fig.7a) shows TEM micrographs of the CdS nanoparticles. TEM analysis shows that the sonochemical method is the most potent because due to its various benefits as it produces a stable low-cost product with good uniformity. The mean particle size obtained was 20-50 nm. It is consistent with the SEM data. The prepared CdS nanoparticles were obtained without any aggregations of bulk particles and it reveals the effect of pH on the nanoparticle surface. Fig. 7b) noticeably shows lattice fringe pattern and size of CdS nanoparticles. The lattice spacing between [1 1 1] planes of CdS nanoparticles has been calculated and found to be 235.13 pm. Fig.7c) illustrates the SAED pattern and the well-ordered hexagonal-like spot

arrays confirmed the development of the hexagonal structure of CdS nanoparticles [25]. Alternatively, the deviation of pH played a major role in the preparation of hexagonal CdS nanoparticles.

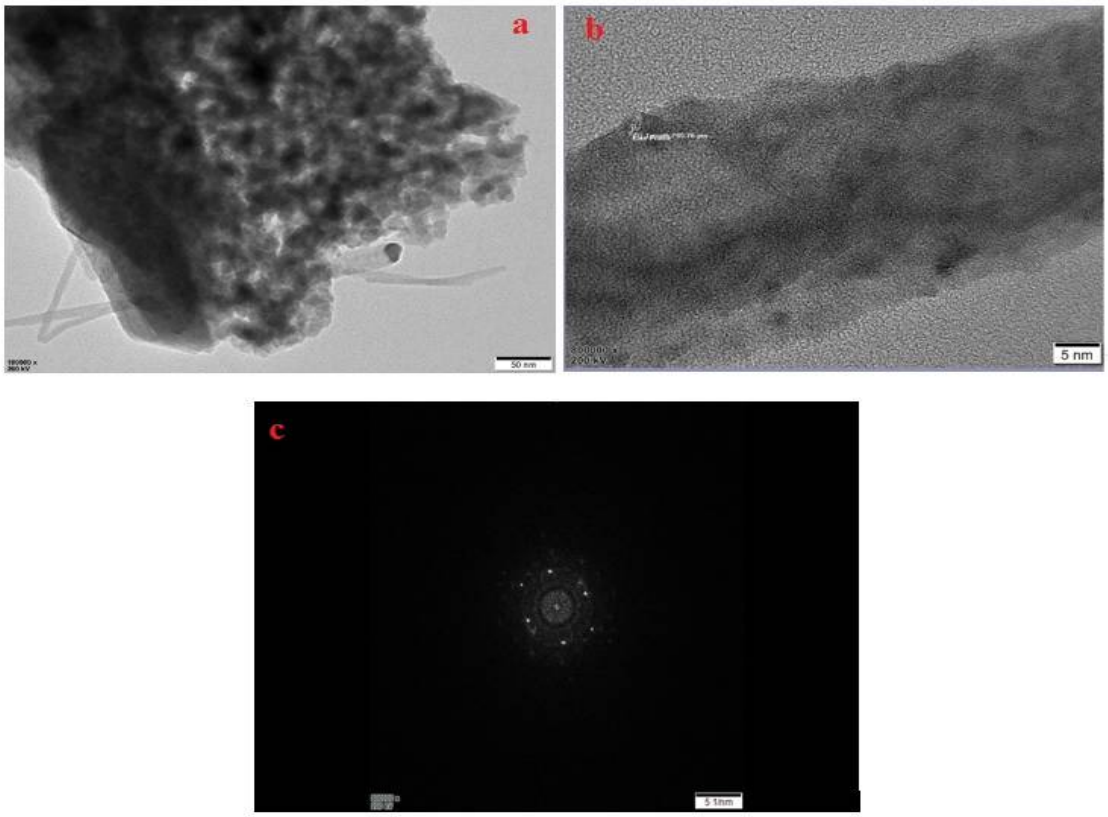
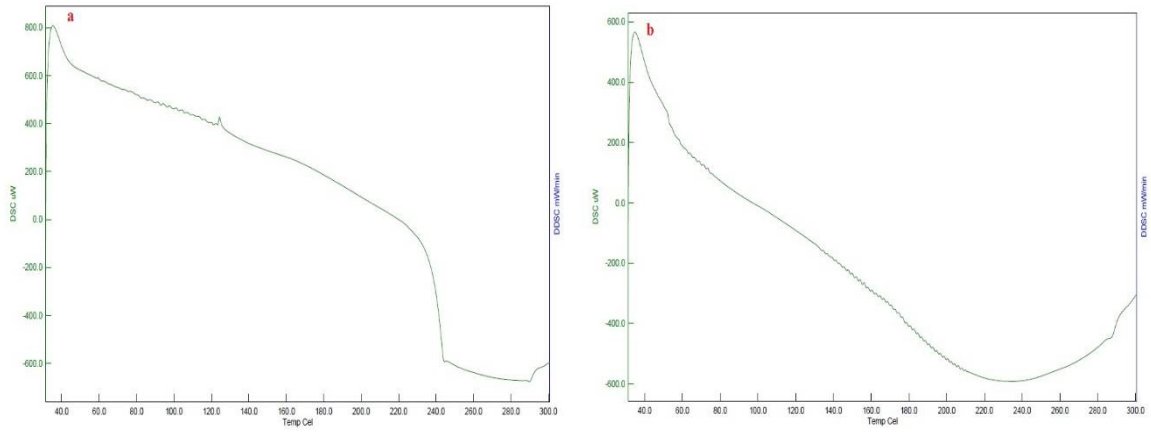


Fig. 7a), b) TEM images c) SAED pattern of CdS nanoparticles

Thermal Characterization

DSC analysis

DSC is a potent analytical technique used to analyze various physical and thermal properties of materials. DSC analysis prefigures curve for the CdS nanoparticles and has been analyzed from 40°C to 300°C. Initially, a 6 mg sample was taken for this analysis. Fig.8 illustrates the DSC curve for CdS nanoparticles. It is observed from the DSC curve, that lower peaks attribute to the change of phase from amorphous to crystalline nature and the peak at 50°C also revealed the dehydration on the surface of the material [25]. It also exhibits a peak around 240°C and the analysis is observed up to 300°C only.



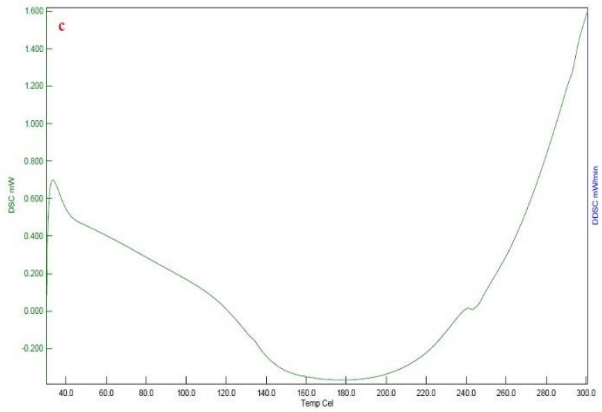


Fig. 8 DSC curve of CdS nanoparticles prepared at various pH values a) pH=8, b) pH=9, c) pH=10

Thermo gravimetric analysis (TGA)

The thermal stability of prepared nanoparticleshas been measured by TGA technique. It revealed the transformation in weight as the sample is heated at a fixed rate [26]. TGA was carried out between 0 and 800°C. The nanoparticle sample was initially stable up to 50°C because water molecules started to remove and decomposition took place between 150-500°C with a mass loss of 25%. Fig.9 shows, the TG curve for CdS nanoparticles. Weight loss of the sample indicates the preparation of CdS nanoparticles due to thermal decomposition. TGA curve shows an endothermic peak at 450°C.

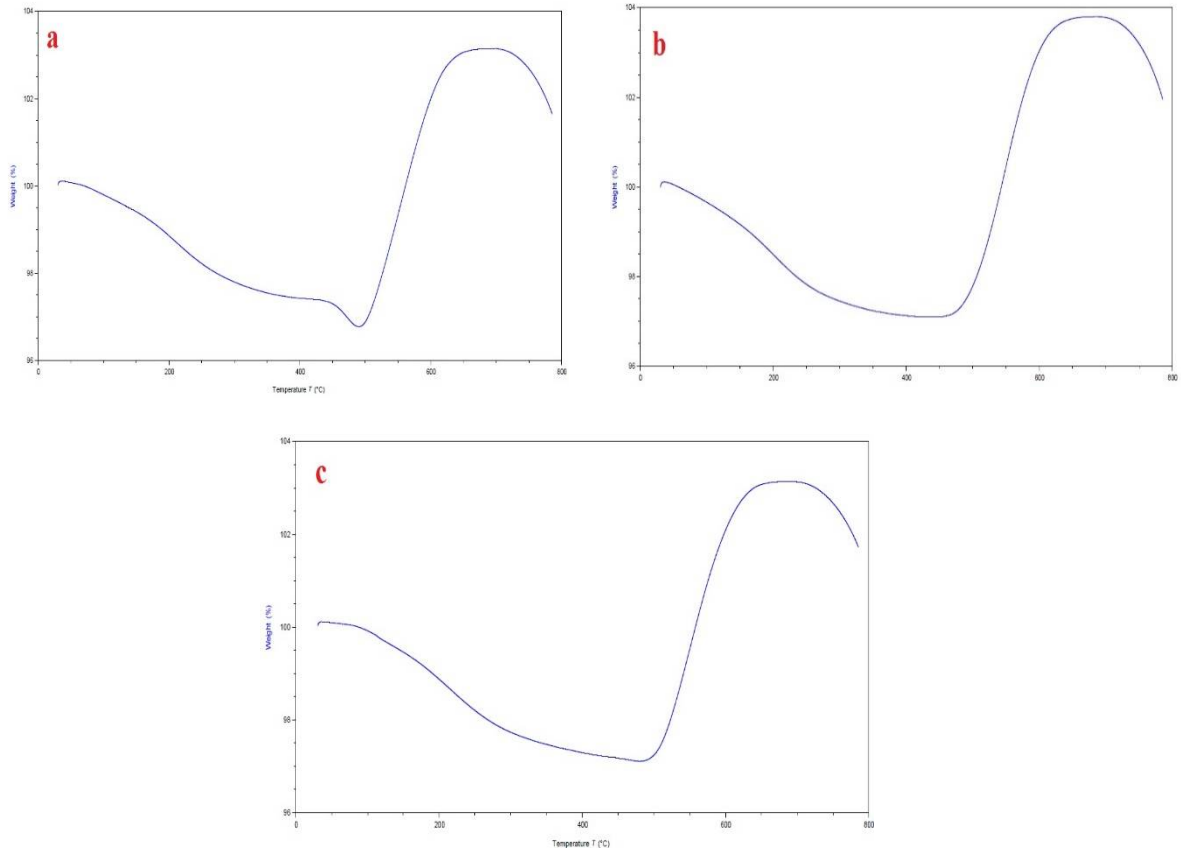


Fig. 9 TGA curve of CdS nanoparticles prepared at various pH values a) pH=8, b) pH=9, c) pH=10

Antimicrobial Activity

The antimicrobial activity of the CdS nanoparticles has been evaluated against some Gram-positive bacteria, Gram-negative bacteria, and fungi using the standard zone of inhibition (ZOI) microbiology assay or agar diffusion assay (Disc diffusion) method [27, 28]. In this method, the CdS nanoparticles showed significant antimicrobial activity on different bacterial and fungal strains. Table 2 signifies the comparative bacterial and fungal activities. Fig.10 shows the antimicrobial activity of CdS nanoparticles prepared at

varying pH such as 8, 9, and 10 against different bacterial and fungal strains (a-h). This investigation illuminates the best antimicrobial activity of CdS nanoparticles against different bacteria and fungi.

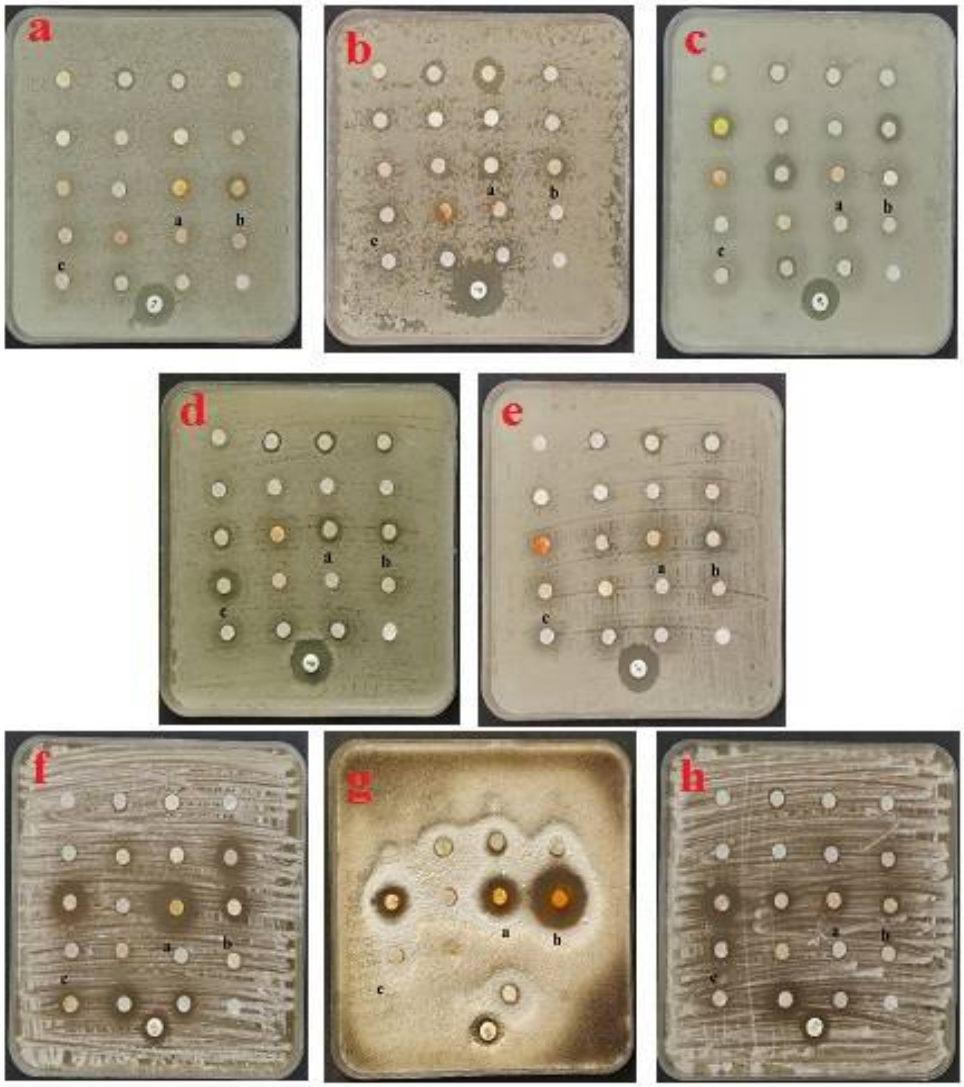


Fig. 10 Antimicrobial activity of CdS nanoparticles prepared at varying pH=8, 9, and 10 against different bacterial and fungal strains (a-h) using agar diffusion assay (Disc diffusion) method

Table 2 Study of the zone of inhibition (mm) of CdS nanoparticles prepared at varying pH=8, 9, and 10 against different bacterial and fungal strains [27, 28, 29]

Type of Bacterial / Fungal strains (Microorganisms)	Fig.10	Bacteria / Fungi	Zone of inhibition (mm)		
			CdS @ pH=8	CdS @ pH=9	CdS @ pH=10
Gram-positive bacteria	a	*Staphylococcus aureus* (NCIM 2079)	6.56	12.70	-
	b	*Bacillus subtilis* (NCIM 2063)	11.89	11.87	10.52
	c	Methicillin Resistant *Staphylococcus aureus* (MRSA) (ATCC 25923)	-	-	-
Gram Negative bacteria	d	*Escherichia coli* (NCIM 2109)	11.35	9.47	13.82
	e	*Proteus Vulgaris* (NCIM 2172)	-	13.11	6.84
Fungi	f	*Aspergillus niger* (NCIM 1028)	24.55	12.13	-
	g	*Aspergillus flavus* (NCIM 1028)	18.85	25.14	-
	h	*Candida albicans* (NCIM 3471)	11.29	10.68	10.67

CONCLUSION

The present investigation reveals a low-cost sonochemical synthesis of CdS nanoparticles at different pH in aqueous media. The as prepared CdS nanoparticles was characterized using spectroscopic techniques such as UV-Visible spectroscopy, FTIR, furthermore microscopic techniques such as XRD, SEM, EDX, TEM, and thermal techniques such as TGA, DSC were also used. The maximum wavelength at 305-307 nm obtained during the UV-vis study confirmed the presence of CdS nanoparticles. The existence of the cadmium-sulfur bond was confirmed by FTIR analysis. The cubic structure of CdS nanoparticles was revealed by XRD. The typical size of prepared samples was about 20-50 nm confirming it as a nanoparticle. EDX investigation confirmed only the presence of Cd and S atoms as the elementary components. TEM micrographs and SAED patterns revealed the crystal-like form of the CdS nanoparticles. Thermal techniques (TGA, DSC) analysis revealed high crystallinity and thermal stability of the as-prepared samples. Wide applications of CdS nanoparticles make it a versatile candidate for antimicrobial activity, solar cells, etc. Antimicrobial activity of different bacterial and fungal strains was studied in the present work. CdS nanoparticles synthesized at pH8, 9, and 10 were studied using antimicrobial activity against different bacterial and fungal strains using the agar diffusion assay (Disc diffusion) method. This study revealed CdS, as the most promising nanoparticle.

DECLARTION OF COMPITING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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