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BIOSYNTHESIS OF CdS NANOPARTICLES FROM *CALOTROPIS GIGANTIA LEAF* AND A NOVEL BIOLOGICAL APPROACH

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ABSTRACT

Nanoparticles are of great importance because of their unique physical, thermodynamic and chemical properties, which are different from bulk materials. Nanostructure sulfides have been studied extensively with a view to establish a relationship among size, structure and optical properties. Currently, many workers have focused on cadmium sulphide because of several important properties. Size dependant properties are exhibited by CdS nanoparticles because of high surface to volume ratio. It also possessed high photosensitivity which enables them useful for optoelectronic devices and various other biological applications and because of these applications various part of plants is used in the bulk production of CdS nanoparticles. To Synthesize the Cd $(OH)_2$ precursors from various acids like Succinic Acid and Pthalic acid and Calotropis gigantea Flower Extract. To characterize the various Cd $(OH)_2$ precursors with the help of analytical instruments such as UV-Vis, IR. To Biosynthesize the CdS nanoparticles from various precursors. To characterize the CdS nanoparticles with the help of analytical instruments such as UV-VISIBLE, FT-IR, XRD, SEM with EDAX analysis. To analyze the antibacterial activity of the CdS nanoparticles with both gram positive and gram-negative bacteria such as Gram-positive Staphylococcus aureus, Streptococcus Pyogenes, Gram negative Proteus Vulgaris, Klebsiella Pnemoniae, Escherichia coli.

KEY WORDS

CdS, UV –Visible, XRD, FT-IR and Antimicrobial activity.

1. INTRODUCTION

Integration of principles of "Green Chemistry" in Nano sciences has attracted researchers in recent years. Simple and novel method of semiconductor nanoparticle synthesis is now a great area of interest. Semiconductor nanoparticles show size dependent luminescence, optical and electrical properties which find a number of applications in many areas [1,2]. CdS nanoparticle belongs to the group of Chalcogenides is a II-IV group semiconductor nanoparticle and shows size dependent properties due to its very high surface to volume ratio and quantum confinement at nanoscale. CdS nanoparticles also have very high photosensitivity that makes them a promising candidate for the detection of visible radiations, enhancing efficiency of solar cells, in LEDs, as sample photoconductor in optoelectronic devices3 and a number of biological applications 4. The energy band gap goes on increasing with a decrease in size [5-10]. Due to these unique property's plants are now being established for its large-

scale production. The commonly used capping agents for its synthesis such as mercapto accetate, thiourea, thiophenol etc., are toxic in nature and their large-scale production poses a potential threat to the environment [11]. Thus, here we are describing a simple and novel method for the synthesis of CdS nanoparticle by using glucose as capping agent and studying the effect of capping agent concentration on crystallite size. UV visible spectrum is recorded for studying blue shift and increase in the band gap. CdS nanoparticle shows size dependent properties due to its very high surface to volume ratio and quantum confinement at nanoscale. CdS nanoparticles also have high photosensitivity that makes them suitable for optoelectronic devices and a number of biological applications [12-15]. Because of its various applications and properties in plants and plant products are now being used for its large scale production. Because of these interesting possibilities, there have been some efforts to prepare nanoparticles of CdS. Some researchers focused on the synthesis of CdS nanoparticles by cadmium nitrate and sodium sulfide by co-precipitation method. The capping agents used for CdS nanoparticle synthesis like mercaptoaccetate, thiourea etc., have their own demerits like toxic nature, tedious workup, cost and hazard to the environment. [16-22] Green Chemistry principles are widely used in Nanosciences recent years. Simple, green and novel method of nanoparticle synthesis is now a great area of interest.

1.1 INDIAN SCENARIO

India's biodiversity is unmatched due to the presence of 16 different agro-climatic zones, 10 vegetation zones, 25 biotic provinces and 426 biomes (habitats of specific species). With only 2.4% of the land area, India already accounts for 7-8% of the recorded species of the world. Over 46,000 species of plants and 81,000 species of animals have been recorded in the country so far by the Botanical Survey of India, and the Zoological Survey of India, respectively. India is an acknowledged centre of crop diversity and harbours many wild relatives and breeds of domesticated animals [23]. Since the beginning of civilization, human beings have worshipped plants and such plants are conserved as a genetic resource and used as food, fodder, fibre, fertilizer, fuel, febrifuge and in every other way. *Calotropis gigantea* is one such plant [24-29]. In ancient Ayurvedic medicine, the plant *Calotropis gigantea* is known as "sweta Arka" and *Caotropis procera* as "Raktha Arka". Both of them are often similar in their botanical aspects and also have similar pharmacological effects. The systematic position, vernacular names, vegetative characters of the plant are given in the following Tables (1.1 -1.3). In the present study henceforth, this plant is referred to as *Calotropis.*


Picture 1.3. ***(Calotropis gigantean)*** **Study locations** Seasonal and locational influences on the phytochemistry, the anatomy and the antimicrobial potential of C*alotropis gigantea* are studied during This Year in Tamil Nadu, India. Thus, here we are describing a simple and novel method for the synthesis of CdS nanoparticle by using Calotropis gigantean as capping agent and studying the effect of capping agent concentration on crystallite size. UV visible spectrum is recorded for studying blue shift and increase in the band gap.

2. MATERIALS

Cadmium Sulphate 8 H_2O, Cadmium Nitrate 4 H_2O, sodium sulphide, Phthalic acid, Succinic Acid was used as the introductory material was supplied by Sigma-Aldrich chemicals. A fresh leaf of Calotropis gigantea Flower was washed thoroughly with double distilled

water, ground and was filtered through Whatmann filter paper was used for further studies. Then the Flower was nicely crushed in mortar pestle, after that was transferred into a centrifuge tube and centrifuged (Optima L-100XP Ultracentrifuge at a speed of 10000 - 11000 rpm for 10 min at 4 to 5°C). After centrifugation, the supernatant was filtered using Whatmann paper and the filtrate was used for the synthesis of CdS.

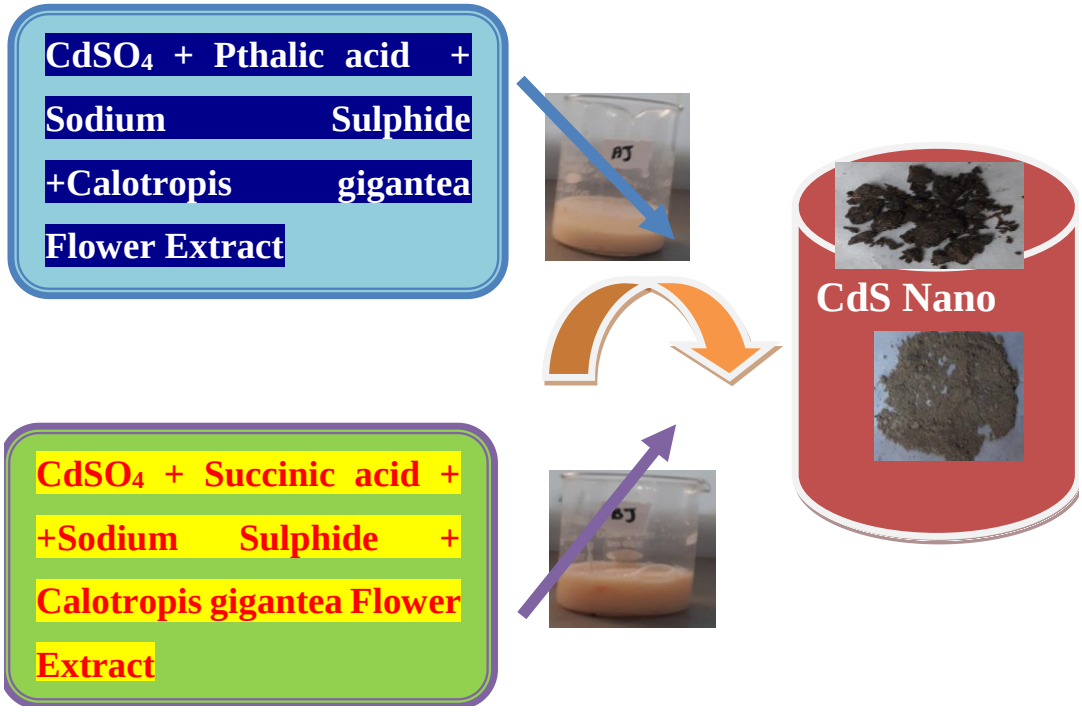
2.1 Preparation of *Calotropis gigantea* leaf extract

100 gms of *Calotropis gigantea* plant fresh leaves were washed several times with de-ionized water to remove all the dirt and soil. The leaves were dried and chopped into small pieces, crushed and powdered using mechanical grinder. 10 gms of leaf powder were weighed; 150 ml of de-ionized water; 10 ml of toluene solvent was added to soxelet apparatus and extract was prepared by hydro distillation process. The extract was filtered using Whattman No. 1 filter paper and supernatant was collected and stored for further use [30].

2.2 Synthesis of CdS Nanoparticles

2.2.1. Synthesis of CdS Nanoparticles Using Calotropis gigantea Flower Extract from Cadmium Sulphate P1 Precursor.

CdS nanoparticles production was done by biosynthesis using Calotropis gigantea flower extract as stabilizing/capping agent. For this, Cadmium Sulphate is used for utilizing the cadmium ions; sodium sulphide is used for utilizing the sulphide ions. 10 ml of Calotropis gigantea flower extract was added into 100 ml Cadmium Sulphate (0.1M) solution, Phthalic acid (0.1M) and Sodium Sulphide (100 ml, 0.1M) was added in dropwise, the solution turns into a Pale yellow colour solution, this solution is magnetic stirred continuously for 6 hours at 500 rpm. Filter the precipitates and dried in an oven for 70ºC for 6 hours. The Pale yellow colour residue thus obtained was collected in a previously cleaned, washed and dried silica crucible. It was heated to 400ºC for 2 hrs in a muffle furnace. The Brown coloured Cadmium Sulphide (CdS) nanoparticles thus obtained was collected, preserved and used for further characterization and applications.



Scheme 1. The Synthesis of CdS nanoparticles by Green Method.

3. RESULTS AND DISCUSSION

3.1 STRUCTURAL CHARACTERIZATION OF CdS NANOPARTICLE

Synthesis and crystallite size of nanoparticles were significantly influenced by the concentration of *Calotropis gigantea* capping agent. By using various analytical techniques like SEM with EDX and AFM, UV and IR studies crystallite size it determined and found approximately 20 nm.

3.2 XRD – ANALYSIS

Powder X-ray Diffraction (XRD) is one of the primary techniques used by mineralogists and solid state chemists to examine the physico-chemical make-up of unknown materials.

X-ray diffraction is one of the most important characterization tools used in solid state chemistry and materials science. XRD is an easy tool to determine the size and the shape of the unit cell for any compound. Powder Diffraction Methods is useful for Qualitative analysis (Phase Identification), Quantitative analysis (Lattice parameter determination & Phase fraction analysis) etc. Diffraction pattern gives information on translational symmetry - size and shape of the unit cell from Peak Positions and information on electron density inside the unit cell, namely where the atoms are located from Peak Intensities. It also gives information on deviations from a perfect particle if size is less than roughly 100 nm extended defects and micro strain from Peak Shapes & Widths [26-30].

Figure. 1 shows the XRD patterns of samples synthesized by co precipitation methods with dry temperature *Td* of 400°C for 2 hrs. The spectra are almost equal to the typical XRD spectra of CdS nanoparticles reported from other experiments. In all our XRD patterns nine peaks are observed around 2θ = 32.18, 33.47, 37.62, 48.63, 58.55, 63.52, 69.95, 71.23, 76.24 which correspond to (100), (002), (101), (102), (110), (103), (200), (112) and (201), respectively. For each sample, all observed peaks can be indexed as the hexagonal wurzite structure of CdS with having space group P73*mc*.

The values of lattice parameters calculated from XRD data by using the Raveled refinement analysis are shown in Figures 1 respectively. All available reflections were fitted with the Gaussian distribution. Analysis of XRD patterns Figure 1 (N_1 &N_2) suggested that the lattice parameters for CdS obtained with dry temperature *Td* = 350°C are *a* = *b* = 3.255 Å and *c* = 5.218 Å. There is no significant difference in calculated lattice parameters as dry temperature is increased from 400°C. These results are slightly higher than the standard JCPDS parameter for bulk CdS, *a* = *b* = 3.2498 Å and *c* = 5.206 Å.

3.3 Particle Size Calculation

From this study, considering the peak at degrees, average particle size has been estimated by using Debye-Scherrer formula Particle Size prediction by the Debye-Scherrer formulae

$D = 0.9\lambda / \beta\cos\theta$

$\lambda = 1.5406 \times 10^{-10}$m

β = Full width at half maximum (radian)

Calculation of d-Spacing

The value of d (the interplanar spacing between the atoms) is calculated using

Bragg's Law: $2d\sin\theta = n\lambda$

$$d = \frac{\lambda}{2\sin\theta} \quad (n=1)$$

Wavelength λ = 1.5418 Å

Dislocation density (δ) is calculated with the crystalline size.

$$\delta = \frac{1}{D^2}$$

Table- 1 Simple peak indexing N1

Peak position 2 θ	1000 x Sin^2 θ	1000 x Sin^2 θ/ 40	Reflection	Remarks
47.92	165.4	4.1	200	$2^2+0^2+0^2=2$
55.03	260	5.5	210	$2^2+1^2+1^2=5$
66.13	360	6.4	211	$3^2+0^2+0^2=6$

Table - 2 The grain size of Cds Nanoparticles.

S. No.	Compound	Size of the particle (D) nm	Dislocation density (δ)
1	N 1	65.06	0.0163
2	N 2	58.64	0.0192

Moreover, a careful analysis of peak positions suggestive a small shifting in its value toward a lower 2θ with increasing dry temperature, indicating a presence of compressive strain in the samples. It is also shown that for the entire samples the reflection peaks become sharper and the full width at half maximum (FWHM) are slightly decreased with increasing dry temperature.

Using the Scherrer peak broadening method, the average crystallite sizes obtained are ~ 80 nm.

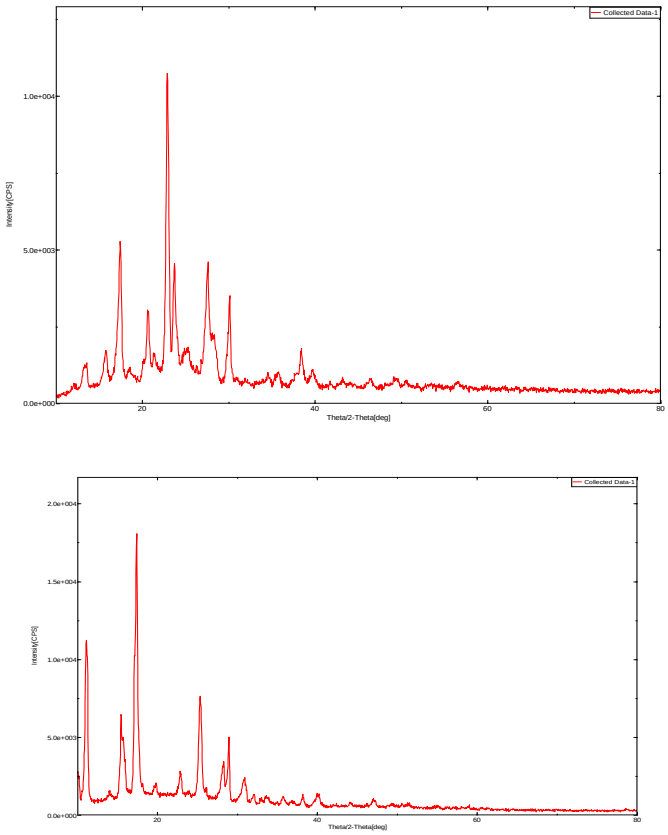


Figure – 1 XRD graph for CdS Nanoparticles N1 & N2

3.4 SCANNING ELECTRON MICROSCOPE

The Morphology of synthesized CdS Nanoparticles was investigated by scanning electron microscopy (SEM). In Figure (2,3) shows the SEM image of CdS synthesized from green method and Spherical shape. Figure 3 shows the elemental composition is analyzed by Energy dispersive X –ray spectroscopy (EDS). As seen from the SEM images, green CdS nanoparticle has lower particle size. Particles were found to be around 100 nm but were in agglomerated form [31-35].

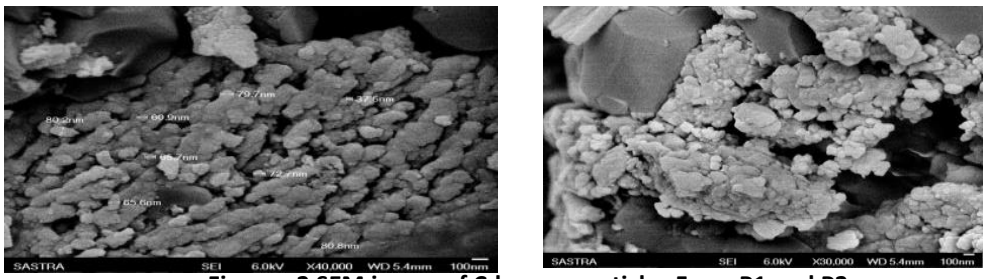


Figure - 2 SEM image of Cds nanoparticles From P1 and P2

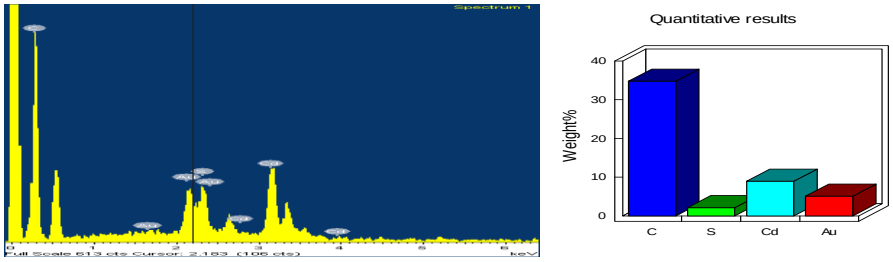


Figure - 3 SEM With EDX image of CdS nanoparticles From P2

3.5 FT- IR SPECTRA ANAYSIS

The FT IR spectrum of CdS Nanoparticles is recorded in the spectral range of 4000–400 cm-1 and it is shown in Figure 4 (a,b&c). The strong broad absorption band centered around 3400 cm-1 is assigned to the OH stretching vibrations of hydroxyl groups of the plant material. while the band around 1450 cm-1 is assigned to the asymmetric stretching vibrations of water molecules associated with synthesized CdS Nanoparticles. The absorption peak at 951 cm-1 corresponds to metal-oxygen stretching of Nanoparticles. The formation of absorption band 508 cm-1 is attributed to CdS stretching vibrations. It has been reported that the intense IR bands around 500, 1000 and 1400 cm-1 are the characteristic bands of CdS. The sharp peaks at 1118 cm-1 are of C-H bending vibrations with CdS formation at 617 cm-1. Hence, the FTIR spectrum also confirms the formation of CdS because of the existence of the characteristic bands of CdS.

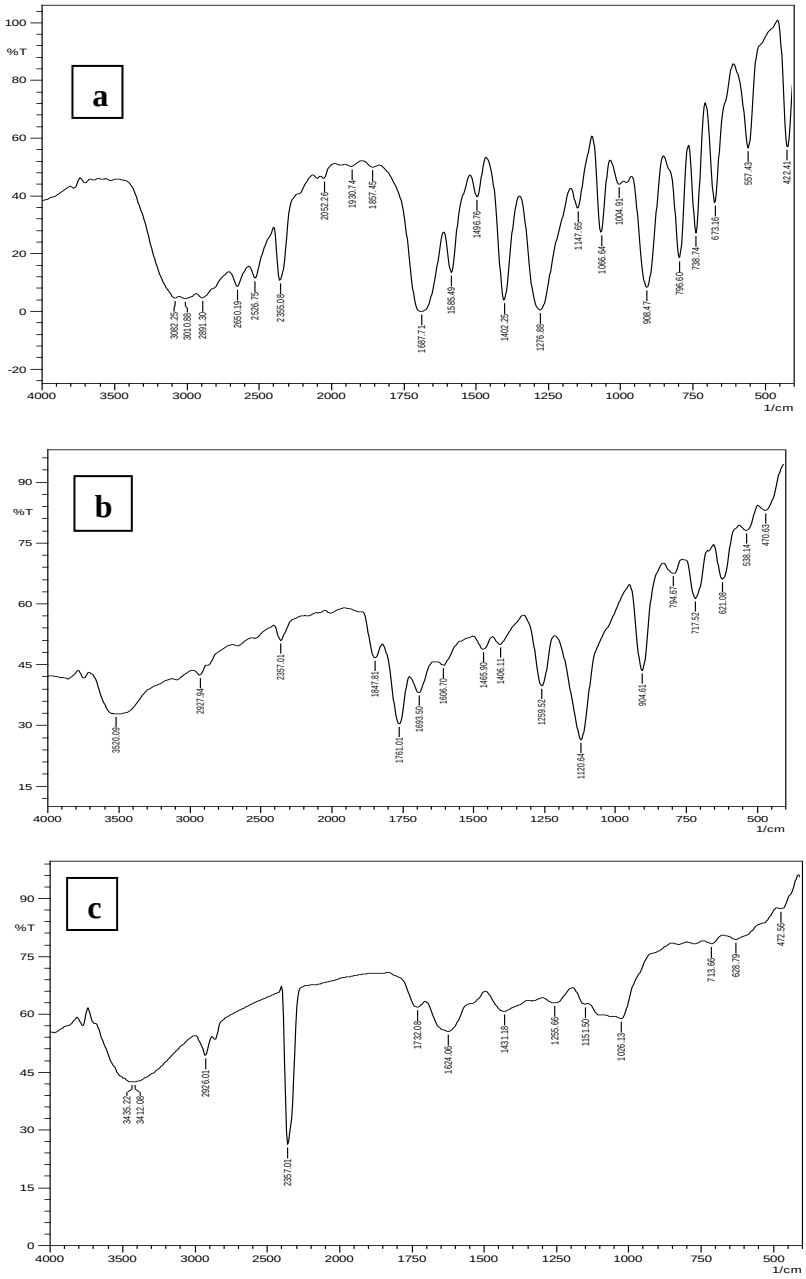


Figure – 4 (a) FT IR Spectral graph for CdS Nanoparticles N1 (b) Precursor P1 (c) Calotropis gigantea Flower Extract.

3.6 UV-VIS Spectroscopy

Fig. 5(a,b) shows the UV–Vis spectrum of CdS NPs biosynthesized from the leaves of Calotropis gigantea Flower Extract. CdS NPs displays an absorption band at 472 nm, assigning to the inter band transitions of core electrons of Cd metal and CdS nanocrystals.

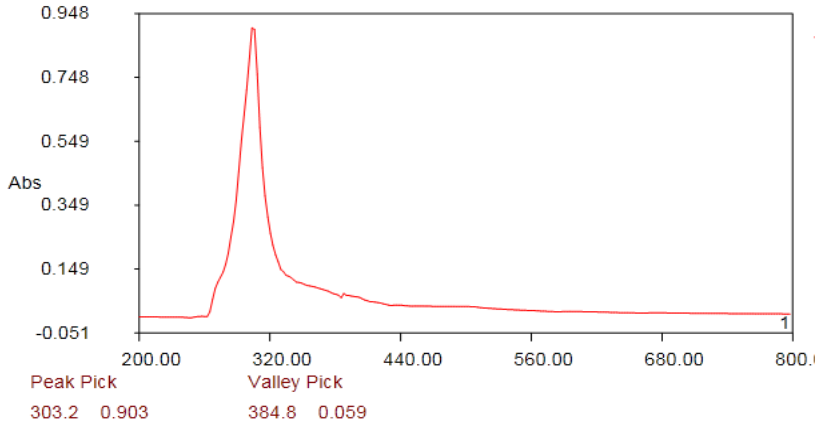


Figure – 5(a) UV-Visible Spectra of Precursor P1.

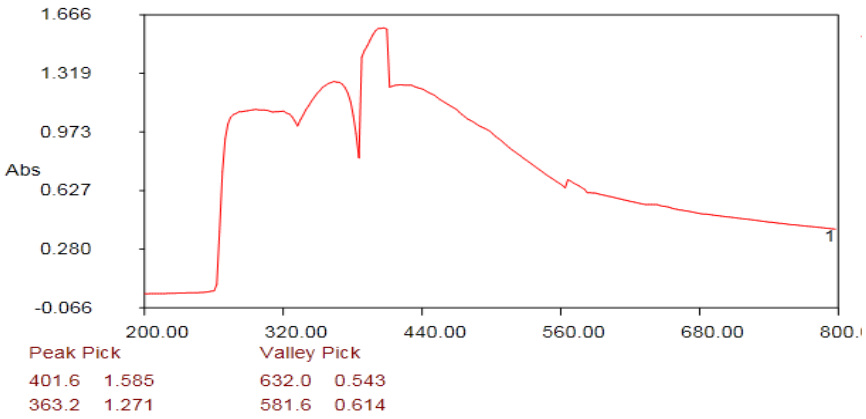


Figure – 5(b) UV-Visible Spectra of CdS nanoparticles N 1

3.7 Band Gap Energy Value of CdS nanoparticles in the Powder Form using a UV/Vis Spectrometer.

The measurement of the band gap of materials is important in the semiconductor, nanomaterial and solar industries. This note demonstrates how the band gap of a material can be determined from its UV absorption spectrum. The values are shows in the given table (3-4). With similar experimental conditions and accessories, band gap energy values for various Precursor and Nanopowder nanomaterials can be calculated. With this, the quality of CdS also can be determined compared precursor. Various other semiconductor nanomaterials can also be subjected to the experiment method matched theoretical value.

Band Gap Energy (E) = h * C / λ

h= Planks Constant = 6.626 X 10^{-34}

C = Speed of light = 3.0 X 10^{8}

λ = Cut off wavelength

Where 1 eV = 1.6 X 10^{-9} (J)

Table – 3 Shows Band Gap Energy for various Precursors

Precursor	λ	E	eV
P1	303.2	6.5560	4.09754
P2	296.0	6.7155	4.19721

Table – 4 Shows Band Gap Energy for CdS Nanoparticles

CdS Nano	λ	E	eV
N1	384.8	5.1658	3.2286
N2	399.2	4.9794	3.1121

3.8 POROSITY

Porosity is one of the most important rock properties in describing porous media. It is defined as the ratio of pore volume to bulk volume of a rock sample. The values are shows in the given table (3-4). Table (5) The porosity of a rock is the fraction of the volume of space between the solid particles of the rock to the total rock volume. The space includes all pores, cracks, vugs, inter- and intra-crystalline spaces. The porosity is conventionally given the symbol f and is expressed either as a fraction varying between 0 and 1, or a percentage varying between 0% and 100%.

$$\emptyset = \text{pore volume / bulk volume}$$

When core is saturated with water, volume of absorbed water is equal to pore volume of core sample.

$$\text{weight of absorbed water} = \text{wet weight} - \text{dry weight}$$

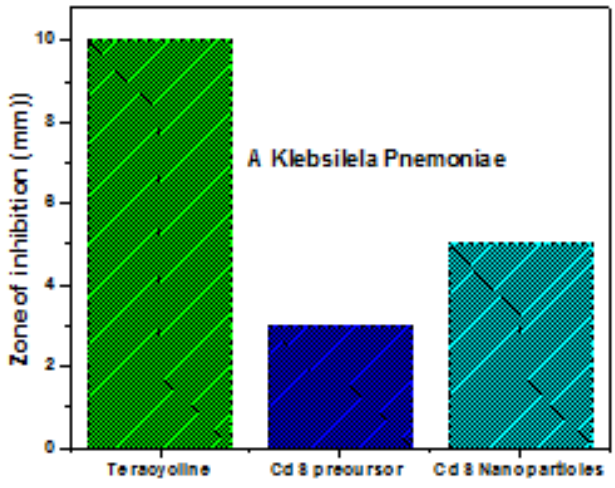
$$\text{Volume of absorbed water} = \text{pore volume} = \text{weight of water} * \text{density}$$

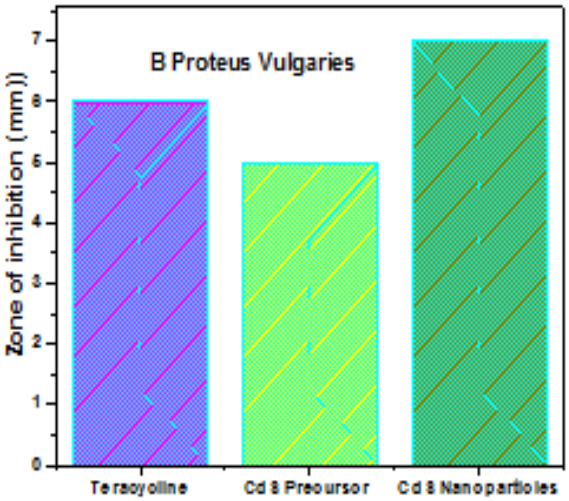
Table – 5 Shows the Porosity of the Precursor and CdS nanoparticles.

COMPOUND	POROSITY %
P1	0.235
P2	0.250
N1	0.125
N2	0.065

3.9 ANTIBACTERIAL ACTIVITY OF CDS NANOPARTICLES

The antibacterial activity of CdS Nanoparticles was studied against a ***Klebsilela*** **and** ***Proteus Vulgaries*** bacterial pathogen. The selection of the organisms was based on their roles for causing infections such as diarrhea in both children and early-weaned piglets. CdS nanoparticles at a concentration of 10 mg/ml showed inhibitory effect against the growth of both ***Klebsilela*** **and** ***Proteus Vulgaries***. The clear zone of inhibition around the discs was the evidence of antibacterial activity, which is presented in **Figure 5 (A-C).** Results showed that **CdS Nanoparticles** had good antibacterial activity against ***Klebsilela*** **and** ***Proteus Vulgaries***.





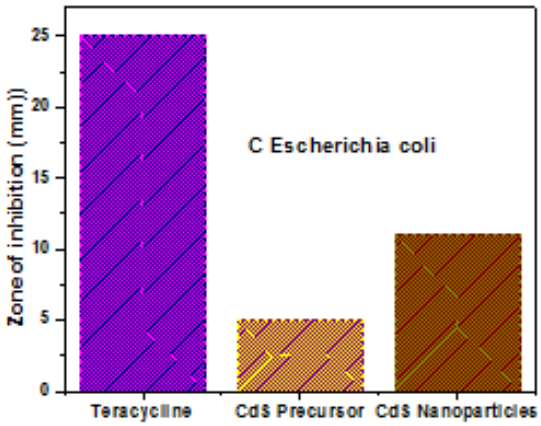


Figure 5(A-C) The diameter zone of inhibition (ZOI) of Drug, $Cd(OH)_2$ Precursor and CdS nanoparticle impregnated disks in presence of *Klebsilela* and *Proteus Vulgaries* and *Escherichia coli* microorganisms.

4.CONCLUSIONS

In this paper the work incorporates a study on the synthesis of CdS nanoparticles and is carried out by a green method using Calotropis gigantean flower extract which acts as a capping agent or stabilizing agent to reduce cadmium sulfide to nanosize particles. A simple, economic, green and eco-friendly route of cadmium sulphide nanoparticle synthesis has been developed by using as Calotropis gigantean flower extract capping agents. The size calculated by the XRD data of the Calotropis gigantean flower extract capped nanoparticles is near about same as SEM and TEM data obtained from the nanoparticle size. This method is eco-friendly for commercial scale production as it does not involve the use of hazardous and toxic capping agents. Further, the concentration of capping agent has a significant effect and can be seen in crystallite size and a blue shift was seen in λmax. Further, CdS nanoparticles have potent antibacterial activity against all tested bacterial strains and hence, it is valuable in the field of medicine and drug development. A simple Green method of CdS nanoparticles synthesis was developed by using glucose as a capping agent. This method is eco-friendly for commercial scale production as it does not involve the use of hazardous and toxic capping agents such as thiophenol, thiourea and mercaptoaccetate. Further, capping agent concentration had a significant effect can be seen in crystallite size and a blue shift was seen in λ max.

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