International Journal of Scientific Research in Multidisciplinary Studies
Vol.3, Issue.7, pp.5-10, July (2017)

Research Paper
E-ISSN: 2454-9312
P-ISSN: 2454-6143

Biosynthesis of CdS nanoparticles using *Murraya Koenigii* leaf extract and their biological studies

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Available online at: www.isroset.org

Received 04th Jun 2017, Revised 16th Jun 2017, Accepted 12th Jul 2017, Online 30th Jul 2017

Abstract Nanotechnology is the creation, manipulation and use of materials at the nanometer size scale (1 to 100 nm). At this size there are significant differences in many material properties that are normally not seen in the same materials at larger scales. Although nanomaterials can be produces using a veriety of traditional physical and chemical processes but it is now also possible by biological processes. The present study aims to employ the use of plant as a stabilizing/ reducing agent for the synthesis of CdS nanoparticles with less hazards towards environment, easily scaledup, stable, economically viable and suitable for large scale production. CdS nanoparticles were synthesised by using *Murraya Koenigii* aqueous leaf extract as non toxic and green capping agent. The novelty of this work is the *Murraya Koenigii* leaves present throughout the year and not an seasonal leaves and it has many medicinal properties. In the process of synthesizing CdS nanoparticles using *Murraya Koenigii* leaves extract, observed rapid stabilization of CdS nanoparticles in the solution. Characterization of the particles has been carried out by using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Spectroscopy (TEM), Fourier Transform Infra Red (FTIR) and UV-Visible Spectroscopy. The CdS nanopaticles prepared by using *Murraya Koenigii* leaves extract have potent antibacterial activity and cost wise very cheap and ecofriendly to the environment.

Keywords: CdS Nanoparticles, Bioynthesis, XRD, SEM, TEM, UV-Visible Spectroscopy, antibacterial activity.

I. INTRODUCTION

Nanoparticles have attracted great interest due to their unique physical and chemical properties, which are different from those of either the bulk material or single atoms.[1] Nanoparticles show completely improved properties based on specific characteristics such as size, distribustion and morphology.The field of nanotechnology is one of the most active areas of the research in modern material sciences. Green science pathways are cost effective and do not use toxic chemicals, high pressure and temperatures. [2,3] Due to their unique properties, the precious metal nanoparticles and the semiconductor quantum dot nanopaticles have been increasingly used in the field of biological, biotechnological and biomedial research. In particular, the metal naoparticles are applied to the visualization of cells, subcellualr structures, and the processes in living organism, the delivery of genes, drug and other targeted to proper destinations, the hypothermia treatment of tumors etc. [4-6]

Metal chalcogenides like sulphides, tellurides and selenides are great importance because they are potential materials for optoelectronics applications. It is well known that cadmium sulhide belongs to II-VI group of semiconductor compounds and that it has been widely utilized in making heterojunction thin film solar cells, optoelectronics and wider range of applications. [7-12] Semiconductor nanoparticles such as PbS and CdS have attracted considerable due to their unique properties fromthat of their bulk materials. Cadmium sulphide is traditionally known as yellow pigment called 'aurora yellow'. The search of cadmium for industrial applications revealed that it occurs as sulphide in grenockite mineral, a companion to sphalerite (ZnS). CdS exhibits many remarkanle characterstics including good thermal, mechanical and size-dependant optical properties, hich has potential applications in lasers, light-emitting diodes and optical devices. [13]

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.[14,15] 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. [16,17] 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.

Murraya Koenigii, commonly koown as curry leaves or karipatta in Indian dialects, belonging to Rutaceae which represent more than 150 genera and 1600 species.[18] In tradiational system of medicine, it is used as antiemetic, antidiarrhoeal, dysentery, febrifuge, blood purifies, tonic, stomachic, flavoring agent in curries and chutneys. The oil is used externally for bruises, eruption, in soap and perfume industry.[19] Curry leaf is found to be effective as antioxidant, antidiabetic, antibacteraial, antihypertensive, cyctotoxic and also in the treatment of bronchial respiratory difficulties. In view of its medicinal importance and lack of literature reports on the synthesis of CdS nanoparticles using M. Koenigii leaf extract, an attempt was made to synthesis and characterize CdS nanoparticles using *M. Koenigii* leaf extract as stabilizing/capping agent. We used a simple, novel, ecofriendly, economic and environmentally benign method for the synthesis of CdS nanoparticles by using *Murraya Koenigii* leaf extract. Various spectral studies are carried out for structural determination of synthesized nanoparticles.

II. MATERIAL AND METHODS

All the chemicals were of analytical grade and purchased from Merck (India) and used without further purification. The culture media were purchased from Hi-Media (India), de-ionized water was employed for preparing all the solutions and reagents. The XRD measurements were carried out using Bruker D8 Advance X-ray diffractometer. The x-rays were produced using a sealed tube and the wavelength of x-ray was 0.154 nm (Cu K-alpha). The x-rays were detected using a fast counting detector based on Silicon strip technology (Bruker LynxEye dtector), Scanning electron microscopy (SEM, Japan) and the IR spectra of the sample were recorded using Perkin Elmer FTIR. Plant extract of *Murraya koenigii* leaves was prepared by hydrodistillation process using Soxelet Apparatus.

Preparation of *Murraya Koenigii* leaf extract

100 gms of *Murraya Koenigii* 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 hydrodistillation process. The extract was filtered using Whattman No. 1 filter paper and supernatant was collected and stored for further use.

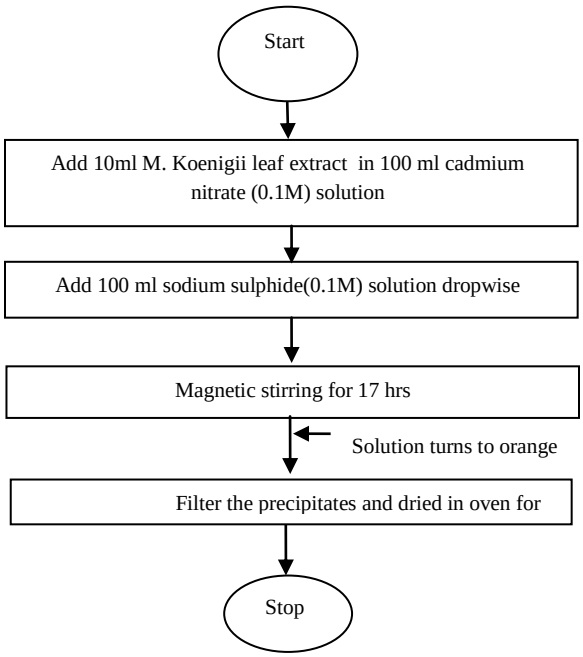
Synthesis of Cadmim sulphide (CdS) nanoparticles by *Murraya Koenigii* leaf extract

CdS nanoparticles production was done by biosynthesis using *Murraya Koenigii* plant leaf extract as stabilizing/capping agent. For this, cadmium nitrate is used for utilizing the cadmium ions, sodium sulphide is used for utilizing the sulphide ions. 10 ml of *Murraya Koenigii* leaf extract was added into 100 ml cadmium nitrate (0.1M) solution and sodium sulphide (100 ml, 0.1M) was added in drop wise, the solution turns into an orange colour solution, This solution is magnetic stirred continuously for 17 hours at 500 rpm and kept it in oven for 7 hours at 100 ºC. Filter the precipitates and dried in oven for 70ºC for 6 hours.

Synthesis can be summarized in the equation given below:

$$Cd(NO_3)_2 + Na_2S \xrightarrow{M.\ Koenigii} CdS + 2NaNO_3$$

Flow Chart



Characterization of CdS nanoparticle: X-Ray Diffraction was performed by using Rigaku Miniflex X ray diffractometer which utilized CuKα (1.54 Å) as target material.[20] XRD data were used to determine the lattice parameter, crystallite size and phase identification.

Crystallite size was calculated by applying Debey-Sherrer's equation [21]:

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

Where, d is the crystalline size, λ is the wavelength of the CuKα (1.54 Aº), θ is the angle between the incident beam and the reflecting lattice plane and β is the full width at half maxima (FWHM) of the diffraction peak (in radian).

Lattice constant was calculated by applying the formula :

$$a = d\,(h^2+k^2+l^2)^{1/2}$$

Where, a is lattice constant, d is interplanar spacing and h, k, l are lattice planes.
Interplanar spacing was calculated by applying the formula:

$$d = \lambda/2 \sin\theta$$

Where, θ is the angle between the incident beam and the reflection lattice planes and λ is the wavelength of the CuKα (1.54 Aº).

The absorption spectrum was recorded by using Camspec M550 Double Beam Scanning UV- Visible spectrophotometer and the energy band gap was calculated by employing direct formula [22]:

$$EG^0\ (eV) = 12397.8 / \lambda_{max}\ (Å)$$

Where, EG^0 is an energy band gap and λ_{max} is the wavelength at which maximum absorption was shown corresponding to (111) and (220) planes, respectively. It was observed that broadening in peaks at high concentration of glucose occur due to decrease in crystallite size. The shoulders in the (111) diffraction peaks may result from the X-ray irradiation on the sample as the cubic CdS are a metastable phase.[23] X-ray irradiation may induce a phase transition from cubic phase to hexagonal phase of CdS to some extent resulting in the appearance of the shoulders.

Bacterial activity: Antibacterial activities of *Murraya Koenigii* capped CdS nanoparticles was determined. The disc diffusion method was used to perform antibacterial activity procedure. Four different bacterial strains were collected as test organisms to check the antibacterial activity of synthesized nanoparticles.

Tested bacterial strains

Sr. No.	Gram Positive	Gram Negative
1	*Basillus subtilis*	*Escherichia Coli*
2	*Staphylococcus aureus*	*Salmonella typhi*

The bacteria were sub cultured in nutrient agar medium and the culture of each bacterium was preserved on the same medium at 40°C. The cultures were sub cultured periodically on the same medium at 37°C ± 2°C. 20 µL of each of the solution was adsorbed on sterile Whattmann filter paper (6mm) discs and kept for few hours under vacuum to remove the solvent. Grown cultures were stored in TSB (Trypticase Soya Broth) maintaining the pH 7.3 in 0.5% agar at 4°C in the screw capped tubes. Trypticase soya agar plates were inoculated with standard suspension of different bacterial cultures containing 10^7c.f.u/mL using sterile cotton swabs to obtain a confluent lawn. The prepared discs were placed on the surface at different positions and plates were incubated at 37±2°C for 24 hrs. The results were recorded in tables.

III. RESULTS AND DISCUSSION

Structural characterization of CdS nanoparticle:
Synthesis and crystallite size of nanoparticles were significantly influenced by the concentration of capping agent. By using various analytical techniques like SEM with EDX and TEM, UV and IR studies crystallite size it determeined and found approaximately 20 nm.

XRD analysis: The XRD data for samples having different capping agents has been shown in Fig. 2a & b. The various diffraction peaks appeared at 26.62, 42.62 and 51.86° confirmed the presence of cubic phase of CdS nanoparticles according to JCPDS file no. **10-454.** From XRD data calculated crystallite size is about 4 nm for the highest peak at 26.65°. (Fig. 1)

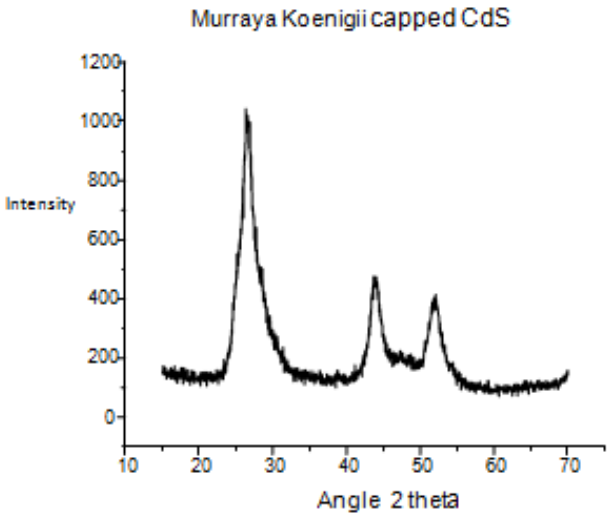


Fig. 1 XRD of *Murraya Koenigii* capped CdS

XRD data (*M. Koenigii*) for crystallite size

Peak	2Theta	Beta	D (nm)
1	26.65	2.12	3.12
2	43.93	1.24	6.89
3	51.94	1.51	5.84

SEM Measurement:

The figure depicts the SEM images obtained from drop coated films of CdS nanoparticles synthesized 5ml of sample on the grid, extra solution was removed using a blotting paper and then the films on the SEM grid were allowed to dry by putting a mercury lamp for 5min. It is clear that the particles are crystalline in nature as shown in Fig.:2 The SEM image of the CdS nanoparticles corresponding to the XRD pattern in Fig.:1 it is clear that the prepared CdS nanoparticles have regular cubic shape and uniform size, with an average size of 20 nm and one can see some coalesced nanoparticles with a size of about 40 nm. [24]

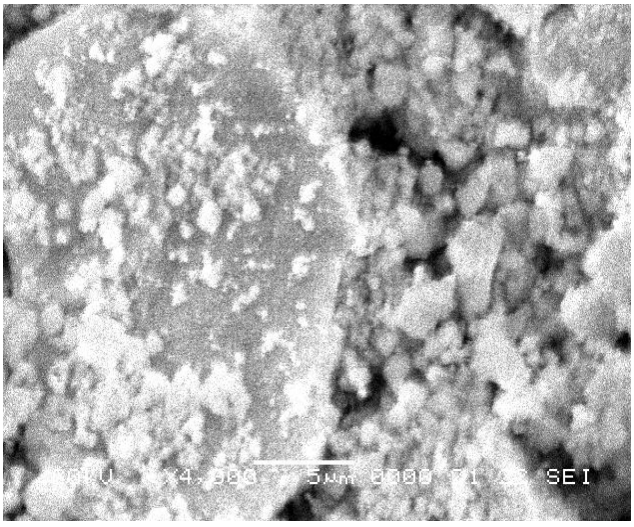


Fig. 2: SEM image of *Murraya Koenigii* cappped CdS nanoparticles

Energy dispersive X-Ray spectroscopy

Further analysis of CdS particles by Energy Dispersive X-Ray spectroscopy confirmed that the presence of signal charactersitcs of Cd and S. Other peaks in the Figure-3 corresponds to Sodium, oxygen, sulphur were due to sputter coating of glass substrate on the EDS stage and were not considered in elemental analysis of Cd and S (Fig.: 3)

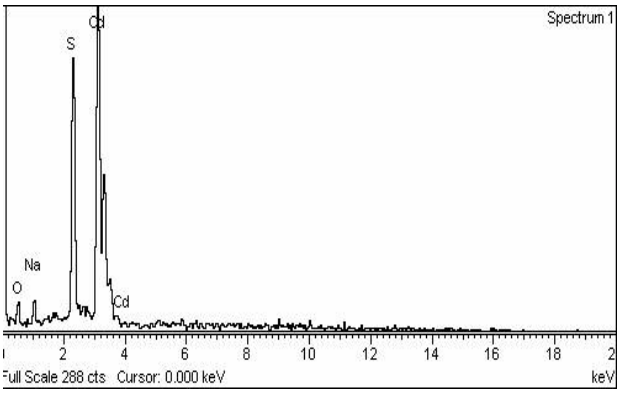


Fig. 3: EDX pattern of *Murraya Koenigii* doped CdS

TEM measurement:

The TEM image of the CdS nanoparticles corresponding to the XRD pattern in Fig 1, and SEM in Fig. 2, the particle size distribution was shown in Fig. 4a&b from TEM, the average size appears to be around 20nm. These particles are single crystalline as revealed by the high resolution electron image. The particles are cubic in shape.

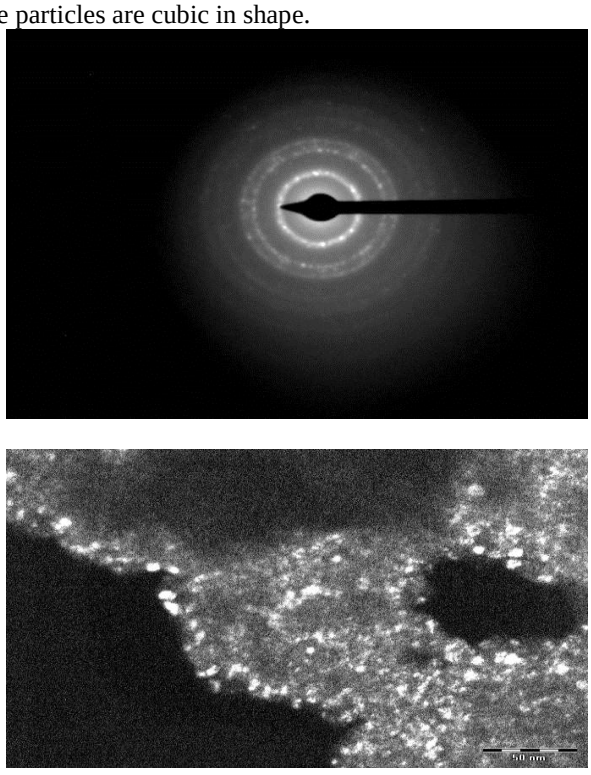


Fig. 4a&b: TEM image of *Murraya Koenigii* cappped CdS nanoparticles

UV-Visible Spectrum:

Characterization was confirmed by measuring the optical properties of CdS nanoparticle. An absorption spectrum was obtained by UV-Visible spectroscopy in the range of 200-700 nm. (Fig. 5) The UV-Visible spectrum of the synthesized

nanpaticle is in accordance with the reported compounds. The absorption spectrum was shown λ_{max} at 480 nm by nanoparticle that confirmed a blue shift of about 50 nm as compared to bulk whose λ_{max} 530 nm.

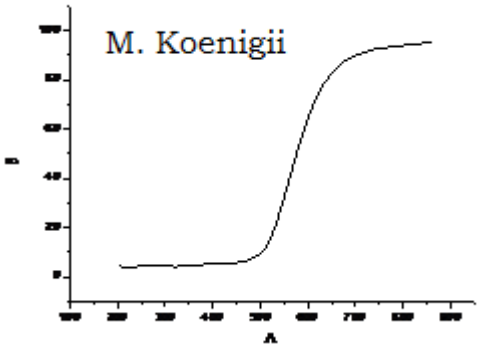


Fig. 5 UV-Vis spectrum of *M. Koenigii* doped CdS **[**A= wavelength nm, B= Abs (%)]

IR spectrum:

Further, characterization was confirmed by Fourier transformation Infrared spectrum of CdS nanoparticle. An absorption spectrum was obtained by IR spectrum in the range of 650-4000 cm^{-1}. The IR spectrum of the synthesized nanpaticle is in accordance with the reported compounds.

Biological activity

The results are presented in **Table-1**, indicate that all selected compounds pertains antibacterial activity against four bacteria taken for study. against four bacteria i.e. two gram-positive *Staphylococcus aureus & Basillus subtilis* and two gram-negative *Escherichia coli* & *Salmonella typhi* starims. CdS NPs exhibited good to excellent antibacterial activity against all the tested bacterial strains rangimg from 17mm to 21mm zone of inhibition and results were comparable with standard ciprofloxacin compounds.

Table 1: Antibacterial activity of CdS nanoparticles from *Murraya Koenigii* leaves extract

Tested organisms	Zone of inhibition (in mm)	Ciprofloxacin (in mm)
Basillus Subtilis	18	26
Staphylococcus Aureus	19	23
Escherichia Coli	17	22
Salmonella Typhi	21	24

IV. CONCLUSION

The work incorporates a study on the synthesis of CdS nanopartticles and is carried out by green method using *Murraya Koenigii* leaf extract which act as a capping agent or stabilizing agent to reduce cadmium sulfide to nanosize particles. A simple, economic, green and ecofriendly route of cadmium sulphide nanoparticle synthesis has been developed by using as *Murraya Koenigii* capping agents. The size calculated by XRD data of the *Murraya Koenigii* capped nanopaticles is near about same as SEM and TEM data obtained of 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, concentration of capping agent has significantly effect and can be seen in crystallite size and a blue shift was seen in $\lambda_{max.}$ Further, CdS nanoparticles has potent antibacterial activity against all tested bacterial strains and hence, it is valuable in the field of medicine and drug development.

V. ACKNOWLEDGEMENT

Authors are thankful to Mr. Ankit Sharma, HOD, Department of Microbiology & Biotechnology, Advance College of Science & Commerce, Vikram University, Ujjain for providing microbiological facilities to carry out antibacterial activity fo the synthesized CdS nanoparticles.

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Author's Profile

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Dr.Preeti Jain, Professor and the Head of Department of Chemistry, Medi-Caps University, Indore, has been associated with the institute since the year 2000. She is M.Sc. (Organic and Inorganic Chemistry both) and Ph.D. (Environmental Chemistry). She has published 32 research papers in National/International Journals and Conferences. Ten books have been published under the discipline of Engineering Chemistry (Theory and Practical both), EEES (Environment) and one international Environmental Nanotechnology Book published by LAP Publishing House Germany. She is also a fellow member of International congress of chemistry and environment and also a member of editorial board of SCI indexed journal Research Journal of Chemistry and Environment • . She has been an active participant in International conference at Thailand in the year 2010 where her research paper was appreciated very much. A national symposium on Climate change and Environment management was organised under the convenor ship of Dr.Preeti Jain in Medi-Caps University during August 2011. A national workshop on Good Laboratory Practices and Environmental Issues was also held under the guidance of Dr.Preeti Jain (Convenor) at Medi-Caps University during 22-23 January 2014. She is the cultural head of the activities being carried out at Medi-Caps University. She has headed the exam team which was involved in conducting various exams (RGPV, other competitive exams) centered at Medi-Caps. She is the advisor of the major activities regarding first year engineering curriculum. She has been very encouraging towards the faculties for research projects and academic development. Her contribution in the establishment of advanced laboratory for the engineering courses as well as researc purposes is consistent.

