Available online at www.derpharmachemica.com

ISSN 0975-413X
CODEN (USA): PCHHAX

Der Pharma Chemica, 2017, 9(14):157-162
(http://www.derpharmachemica.com/archive.html)

Synthesis and Characterization of CdS Nanoparticles Using *Artabotrys hexapetalus* Leaf Extract as Capping Agent

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ABSTRACT

Synthesis of nanoparticles can be performed by using different macro to microorganisms such as plants, bacteria, fungi, sea weeds and microalgae. Plant consists of number of natural products like alkaloids, flavonoids, saponins, steroids, tannins and other nutritional compounds. Modern scientific studies have been revealed that the plant extract plays an important role for the preparation of nanomaterials without any hazardous effect. The synthesis of metallic nanoparticle with plant extract is inexpensive, single step and eco-friendly. Cadmium Sulphide Nanoparticles (CdS NPs) were prepared from $CdCl_2$ and Na_2S solutions using Artabotrys hexapetalus leaf extract as capping agent. The nanoparticles obtained were characterized by UV-Visible spectrophotometer, X-ray Powder Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM) and Transmission Electron Microscopy (TEM) analysis. The formation and stability of the nanoparticles in the colloidal solution was estimated by using UV-Visible analysis. Average diameter of CdS nanoparticles was calculated by using Scherrer equation from the XRD analysis and the antimicrobial activity was studied by cup plate method. CdS nanoparticles prepared using A. hexapetalus leaf extract as capping agent were exhibited highest antibacterial activity against the test organism Staphylococcus aureus and highest antifungal activity against Aspergillus niger test organism.

Keywords: *Artabotrys hexapetalus*, CdS Nanoparticles, XRD, TEM, Microbial activity

INTRODUCTION

Integration of principles of 'Green Chemistry' in nanosciences has attracted researchers in recent years. Synthesis of nanoparticles using plant extracts is the most adopted method of green, eco-friendly production of nanoparticles and also has a special advantage that the plants are widely distributed, easily available, much safer to handle and act as a source of several metabolites CdS nanoparticles belong to the group of chalcogenides and it is an II-IV group semiconductor nanoparticles. Due to very high surface to volume ratio and quantum confinement at nano scale the CdS nanoparticles show size dependent properties. CdS nanoparticles also possess very high photo sensitivity that makes them suitable for the detection of visible radiations, enhancing the efficiency of solar cells, and as sample photo conductor in optoelectronic devices [1] and also used in various biological applications [2].

A number of methods are available for the synthesis of nanoparticles out of which chemical [3], electrochemical [4], radiation [5], photochemical methods [6] and Langmuir-Blodgett [7,8] and biological techniques [9] are some of the methods. But they are more expensive and potentially dangerous to the environment and involve the use of toxic chemicals that are responsible for various biological risks. Researchers are exercising the synthesis of nanoparticles at ambient temperatures and neutral pH by novel methods which are cost effective and ecofriendly.

In view of these goals, various routes are employed to synthesize nanomaterials. Among the biological alternatives, plants and plant extracts seem to be the best option. Plants were natural 'chemical factories', contains a wide range of metabolites as plant products/extracts. More stable nanoparticles are synthesized by using plants and the rate of synthesis is faster when compared to microorganisms. In view of these advantages, an attempt was made to synthesize CdS nanoparticles from cadmium chloride and sodium sulfide adopting precipitation method using Artabotrys Hexapetalus as a capping agent. *Artabotrys hexapetalus* (Linn.f) belongs to the custard apple belongs to the family Annonaceae.

The plant is used to treat fever, diarrhoea, dysentery, bruises, cuts, pains, sprain, inflammation, gout, helminthiasis, leprosy, skin disease, wound, ulcers, tumors, menorrhoea, dysmenorrhoea, cough, asthma, bronchitis, flatulence, colic and constipations. The leaves of the plant are simple, alternate [10] or opposite [11], coriaceous [12], glabrous or glabrescent [13], glossy [14] and petiolate [10].

The bioactive principles of the plant are steroids, terpenoids, saponins and leucoanthocyanin bisabolane, guiane sesquiterpenes, steroids, aporphine tetrahydroherberine alkaloids and long chain hydrocarbons [15].

MATERIALS AND METHODS

A. hexapetalus leaf extract, 2 MLH magnetic stirrer REMI, XRD (make PAN analytical, model no: Xpert pro), UV-Schimadzu, UV-2450 double beam spectrophotometer, FTIR (Schimadzu model no: IR prestige-21), SEM (JEOL-JSM model-66I0LV), with EDS (oxford institute), Transmission Electron Microscopy (TEM). Cadmium chloride (AR grade, Qualigens), sodium sulphide (AR grade, Merck), methanol (Merk). The reagents used were highly purified. The culture media were purchased from Hi-media (India). In order to prepare all the solutions and reagents de ionized water was used.

EXPERIMENTAL SECTION

Preparation of plant extract

Artabotrys hexapetalus leaves were washed for number of times with de ionized water to remove all the dust and impurities. The material was dried, cut to make in to small pieces, crushed to make fine powder. 10 g of powder was taken in to a 250 ml conical flask, added 150 ml water and 10 ml of methanol and then subjected to heat at 100°C of temperature until the volume reduces to half level. Now this mixture was filtered through Whatman No.1 filter paper then cooled to room temperature. The mixture was used for the synthesis of CdS nano particles as a capping agent.

Preparation of cadmium sulphide nanoparticles

The following procedure was adopted for the Synthesis of cadmium sulphide nanoparticles by using Artabotrys hexapetalus leaf extract as capping/stabilizing agent.

A 0.5 ml of Artabotrys hexapetalus leaf extract was taken and 50.0 ml of 0.1 M of $CdCl_2$ solution is added to it in a 250 ml beaker and mixed vigorously. A 50.0 ml of 0.1M Sodium sulphide solution is added drop wise to the mixture of $CdCl_2$ and plant extract solution under magnetic stirring. The contents were kept on a rotatory orbital shaker maintaining at 200 rpm at 30°C for 12 h in dark condition. A visual colour change is observed from brown to orange to yellow confirming the formation of cadmium sulfide nanoparticles.

RESULTS AND DISCUSSION

UV-Visible spectroscopy

A 3.0 ml of this mixture is taken after 12 h to monitor the formation of the CdS nanoparticles by UV-Shimadzu UV-2450 double beam spectrophotometer. From the UV-visible spectroscopic analysis it was observed that the absorption peak of CdS nanoparticles formed by using *A. hexapetalus* leaf extract as capping is at 362 nm as shown in Figure 1.

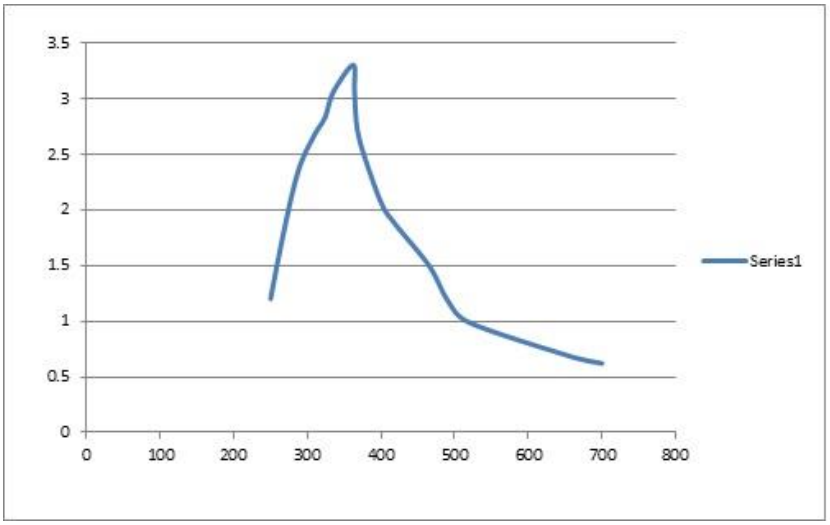

Figure 1: UV-vis spectrum for *Artabotrys hexapetalus* capped CdS nanoparticles

Fourier Transform Infrared (FTIR) Spectroscopy

The dried CdS nanoparticles mixed with KBr were characterized with FTIR. The FTIR spectrum could be explained by various peaks obtained in the sample. The FTIR spectra of the crude plant extract were also recorded for comparison. The absorption peak at 3319.72 cm^{-1} was assigned to –OH group of the plant material. The strong absorption band at 1606.90 cm^{-1} was assigned for C=C stretching of the particles. The weak absorption band at 1393.77 cm^{-1} can be attributed to C=O stretching. The broad peak at 1099 cm^{-1} could be assigned to the ester group present in the extract with CdS formation at 601.35 cm^{-1} (Figures 2 and 3).

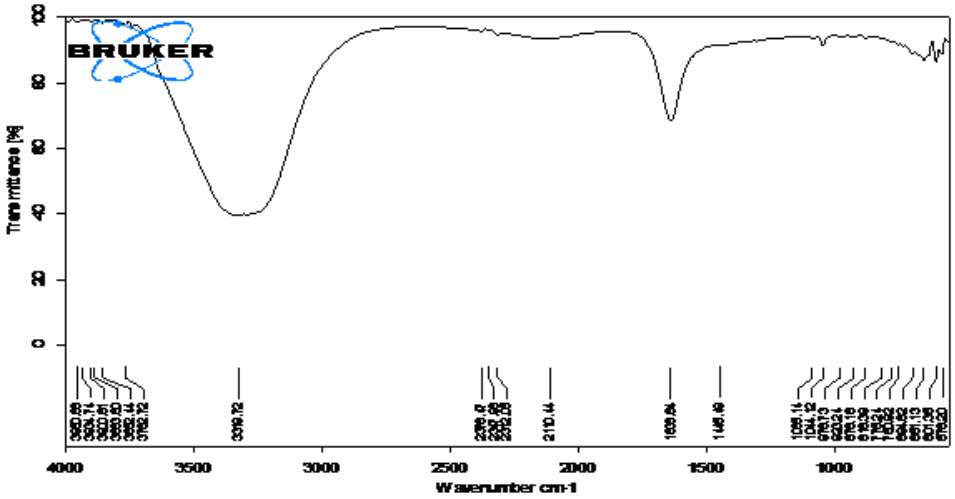

Figure 2: FTIR spectrum for plant extract

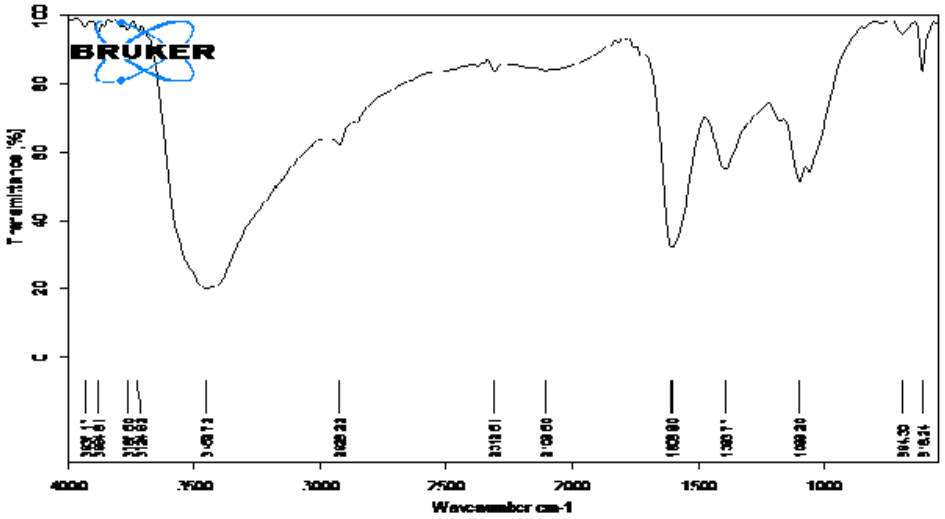

Figure 3: FTIR spectrum for CdS nanoparticles formed by using *Artabotrys hexapetalus* as capping agent

Scanning Electron Microscopy (SEM)

SEM analysis was carried out by using JEOL-JSM 66Iolv SEM machine. A very little amount of the sample is placed on a carbon coated copper grid to prepare thin films of the sample; extra solution was removed using blotting paper. The film on the SEM grid was allowed to dry under mercury lamp for 5 min. The SEM micrographs showed crystalline mass structure as shown in the Figure 4.

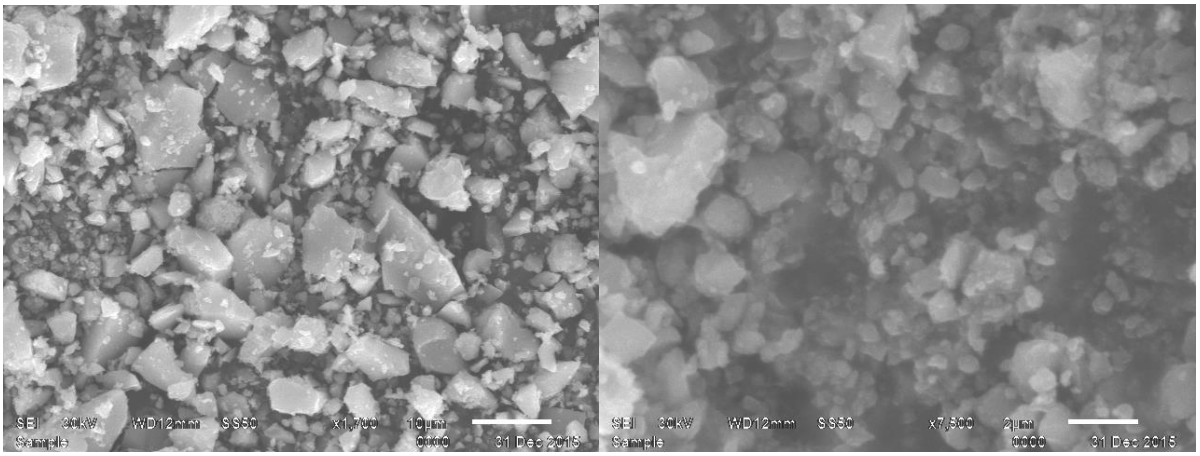

Figure 4: SEM images for CdS nanoparticles synthesized using *Artabotrys hexapetalus* leaf extract as capping agent

Energy dispersive X-ray spectroscopy (EDS)

Energy dispersive X-ray analysis is a technique to analyze near surface elements and estimate their proportion at a different position, thus giving an overall mapping of the sample. Figure 5 reveals the EDS spectra of the synthesized CdS nanoparticles, the presence of Cd and S peaks confirmed the formation of pure CdS with no other elemental purity. Other peaks in the figure correspond to carbon, oxygen, and sulphur, which were due to the sputtering coating of the glass substrate on the EDS stage and were not considered in the elemental analysis of Cd.

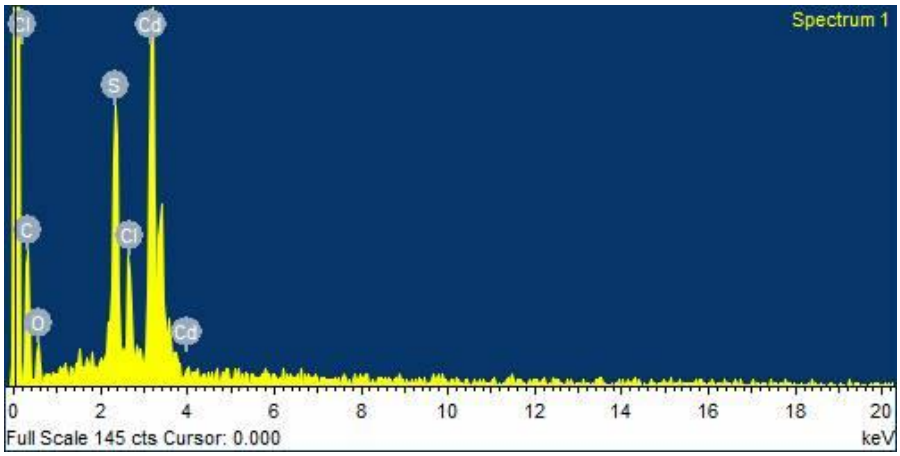

Figure 5: Energy dispersive X-ray spectrum for CdS nanoparticles formed using *Artabotrys hexapetalus* as capping agent

X-ray diffraction (XRD) spectrometer

Figure 6 shows the XRD analysis of CdS nanoparticles synthesized by using Artabotrys hexapetalus leaf extract as capping agent. It is used to identify the size of the respective nanoparticles. The diffraction peaks appeared at 11.74, 15.35, 26.66, 37.04, 44.02, 47.88, 52.03, 78.90 and 86.2514 as shown in the Figure. The average crystallite size according to Scherrer equation calculated using the highest peak of the 26.66 is found to be 2.5 nm and the lowest peak of the 78.90 is found to be 3.8 nm.

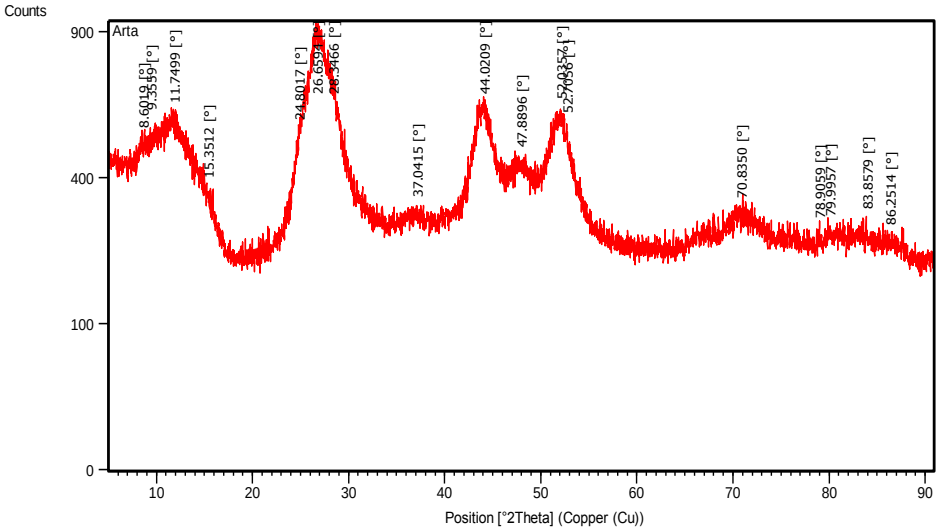

Figure 6: X-ray diffraction plot for *Artabotrys hexapetalus* capped CdS nanoparticles

Transmission Electron Microscopy (TEM)

TEM analysis reveals the size and morphology of the CdS nanoparticles synthesized by using Artabotrys hexapetalus leaf extract as capping agent. The sample was prepared as a thin foil so that the electron beam can penetrate through it. A JEOL model was used for TEM measurements. Figure 7 reveals that the particles obtained are spherical in shape and the bar marker represents 10 nm size.

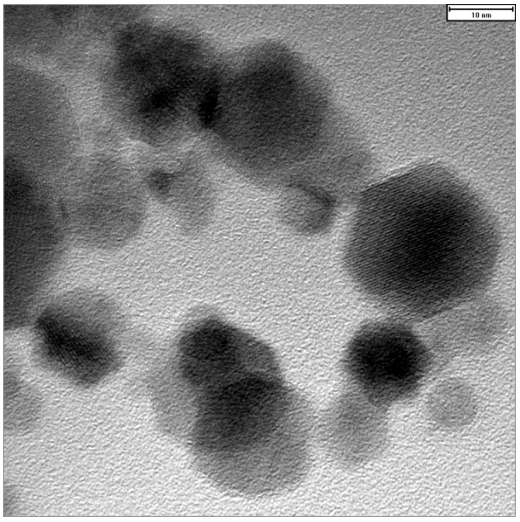

Figure 7: Transmission electron microscopy image for CdS nanoparticles synthesized using *Artabotrys hexapetalus* leaf extract as capping agent

Microbial studies

Antibacterial activity

The antibacterial activity was examined against bacterial culture (*Staphylococcus aureus*) using standard zone of inhibition assay. Nutrient agar medium was sterilized by moist heat sterilization using an autoclave (121°C; 15-20 lb for 20 min). 60 sterile petri plates were used for the assay to get triplicate values. Molten agar medium was inoculated with microbial suspension and poured into the plates (temperature of the medium for inoculation is 35-40°C.) After solidification of the medium, cups were made aseptically using a stainless borer. 50 µl of a sample of the extracts and an antibiotic solution of 50 µl were placed in the cups and the plates were kept in the refrigerator for diffusion for a period of 1 h. The plates were then kept in the incubator for one day at 37°C. The inhibition zone diameters were then recorded and the diameters were compared against those obtained for the standard antibiotic. The antibacterial activity of leaf extract does not show any inhibition and the CdS nanoparticles prepared by using leaf extract as capping agent shows the inhibition against bacteria, and the zone of inhibition is about 3.5 cm (Figure 8).

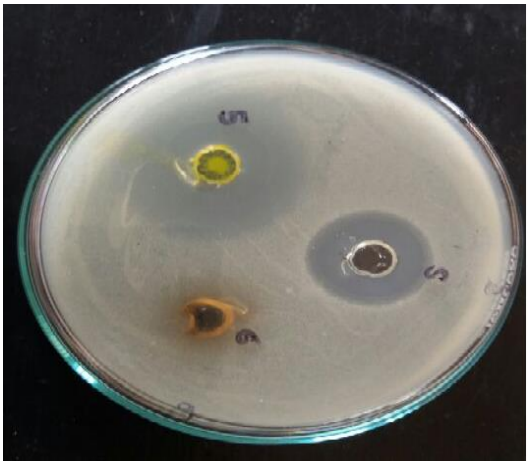

Figure 8: Antibacterial activity of CdS nanoparticles

Antifungal activity

The antifungal activity was examined against fungal culture (*Aspergillus niger.)* using standard Zone of Inhibition (ZOI) microbiology assay. Potato dextrose agar medium was sterilized by moist heat sterilization using an autoclave (121°C; 15-20 lb for 20 min). 60 sterile petri plates were used for the assay to get triplicate values. Molten agar medium was inoculated with microbial suspension and poured into the plates (temperature of the medium for inoculation is 35-40°C.) After solidification of the medium, cups were made aseptically using a stainless borer. 50 µl of a sample of the extracts and an antibiotic solution of 50 µl were placed in the cups and the plates were kept in the refrigerator for diffusion for a period of 1 h. The plates were then kept at room temperature for 3 days. The inhibition zone diameters were then recorded and the diameters were compared against those obtained for the standard antibiotic. After 3 days, the inhibition zone diameters were recorded and the diameters were compared with a standard antibiotic. The antifungal activity of leaf extract does not show any inhibition and the CdS nanoparticles prepared by using leaf extract shows the inhibition against fungus, and the zone of inhibition is about 2.9 cm (Figure 9).

Figure 9: Antifungal activity of CdS nanoparticles

CONCLUSION

CdS nanoparticles were prepared using *A. hexapetalus* leaf extract as capping agent. The particles synthesized were characterized by UV-Visible spectro photometer, XRD, FTIR, SEM and TEM. The particles were found to be crystalline in shape and spherical in structure. The average sizes of the particles are 2.5 to 3.8 nm. It is to be considered as ecofriendly and cost effective method because no chemical reagent was used as capping agent [16,17].

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