ISSN (Online) 2456 -1304

International Journal of Science, Engineering and Management (IJSEM)
Vol 3, Issue 4, April 2018

Green Synthesis, Characterization of CdO nanoparticles and Zr ion doped CdO nanoparticles assisted Acalyphaindica for photocatalytic application

[1] RajaRajeswari V, [2]Lakshmi K, [3]MuthuChudarkodi R.R
[1][2] Department of Chemistry, Sri SaradaCollege for Women,Tirunelveli
[3] epartment of Chemistry, V.O.Chidambaram College,Thoothukudi

Abstract:- **The synthesis of semiconductor nanoparticles is an expanding research area due to the potential applications in the development of nanotechnologies. The CdO nanoparticles have been synthesized by adding a leaf extract of Acalyphaindica into the aqueous solution of cadmium nitrate. The aqueous Acalyphaindica leaf extract acts as a solvent with multiple roles as promoter, capping agent and reductantfor the synthesis of undopedCdO andZrion doped CdO nanoparticles. Cadmium oxide nanoparticles were characterized by using UV-Visible spectrophotometer, FTIR, AFM, XRD and SEM. The size of the CdO nanoparticles and Zr ion doped CdO nanoparticles were estimated using Debye- Scherrer equation. Photo catalytic degradation was also investigated with Crystal violet dye under UV-irradiation source. The CdO and Zr ion doepdCdO nanoparticles exhibited potential photo catalytic activity towards the degradation of Crystal violet dye. Green synthesis using Acalyphaindicawas found to be the best capping agent for synthesizing nanoparticles.**

Key Words: CdO, nanoparticles, Green synthesis,Acalyphaindica , XRD, SEM, Photo catalytic activity.

INTRODUCTION

Over the past few decades, the use of Nano structured material is becoming more wide spread due to its curious and miraculous application in the areas of chemistry, pharmacy, agriculture, textile sizing, optoelectronics, physics and so on [1]. Among these nanostructures, metal oxide nanoparticles that exhibit the technological importance for solar cell, gas sensor, optical coating and photo voltaic cell[2].Therein, cadmium oxide is a known n-type semiconductor, piezoelectric characteristics and polycrystalline in nature [3]. Cadmium oxide nanostructures are applied in solar cells, gas sensors, transparent electrodes and photodiodes, catalysts, photo catalysts and optoelectronic devices [4]. There are several techniques to prepare these materials such as sonochemical, micro-emulsion, hydrothermal and plant mediated method [1,2,4]. However, currently plan text ract mediated nano material synthesis is getting lot of attention to the several numerous advantages offered by chemical and physical methods[5,6]. Herein, we investigate the cost effective, safe and ecofriendly green synthesis of CdONPs using plant extracts of Acalyphaindica and their characterization has been evaluated. Hence it is proposed that the biosynthesized CdONPs have significant photo catalytic applications.

MATERIALS AND METHODS

The fresh leaves of Acalypha indica L.were collectedfrom Sri Sarada collegecampus, Tirunelveli,Tamilnadu, India. The collected ofAcalypha indica leaves were washed several times with water to remove the impurities. Leaves had been dried in the sun shade for 7 days. After the leaves were dried, it is powdered using mortar. This plant is held in high esteem in traditional Tamil Siddha medicine as it is believed to rejuvenate the body. The plant has also been eaten as a vegetable in Africa and India, but care needs when eating it since it contains several alkaloids as well as hydrocyanic acid

MATERIALS

ISSN (Online) 2456 -1304

International Journal of Science, Engineering and Management (IJSEM)
Vol 3, Issue 4, April 2018

Fig. 1.Acalyphaindica

Cadmium nitrate tetrahydrate ([Cd(NO3)2].4H2O 98%, Analytical grade, Sigma-Aldrich),and dimethyl sulfoxide (DMSO, ACS reagent, 99.9%, Sigma-Aldrich) were used. All chemicals were used as such without any further purification. All the solutions were prepared using deionized water during the synthesis.The collected leaves were washed with deionized water, cut into small pieces. All glass wares were washed with deionized water and acetone and dried in oven before use.

Green synthesis of CdONanoparticles

5g powder of Acalypha indica L. Leaves were transferred into 250mL beaker containing 100mL deionized water. The mixture were refluxed at 80-90 °C for 20 minutes and cooled at room temperature followed by filtered through ordinary filter paper. Then, result ant filtrate was again filtered through WhatmannNo.1.The filtered extractis stored in refrigerator at 4°Candused for synthesis of CdONPs.2.0g of Cadmium nitrate tetra hydrate was added in 100ml of the AcalyphaindicaL. water extract solution. The solution was mixed homogeneously using magnetic stirrer at400rpm for60min.Aftertime of period the color of solution turns to yellow. The solid deposit was purified by centrifugation at 4000rpm for 30min. Itwasthen dried inovenat 300°C[7]. There sulted powder was obtained and packed for characterization purposes.

Photocatalytic Measurement: Nanosized CdO particle is a good photocatalyst to degrade organic contaminants, such as crystal violet dye. The dye solution was prepared by dissolving 10mg powder of crystal violet dye in 100ml distilled water. 0.1g Zr ion doped CdO nanoparticles was added to 100ml of prepared crystal violet dye solution and the mixer was stirred magnetically for 1 h in shadow before exposing to sunlight. Then the colloidal suspension was placed in a closed chamber and irradiated with sunlight. The reactions were observed one by one in every time interval of 10 min for 1hr. Finally, the rate of dye decomposition was monitored by taking 4ml samples from each set and recording the UV-Vis spectra in the wavelength after centrifugation andfiltration [8].

Ultraviolet-Visible Spectra Analysis: To study the effect of cadmium nitrate solution’s concentration on CdO nanoparticles formation, the concentration of cadmium nitrate solutionwas varied from 0.06M-0.19M.The reduction of cadmium ions and leaf extract lead tothe formation of nanoparticles at ambient temperature. Duringthis reaction, synthesis of cadmium oxide nanoparticlesreduced by Acalyphaindicaleaf extractmade the color of leaf extractchange.

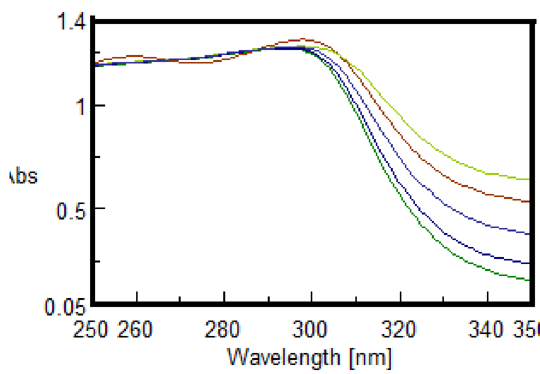

Fig. 2UV-Visible spectra of CdO nanoparticles synthesizedusing Acalyphaindicaleaf extract by varying precursors concentration.

This change of color was recorded by means of the UV-Vis spectrophotometer. Fig.2. shows the UV-Visible spectra of the synthesized Cadmiumoxide nanoparticles using Acalyphaindicaleaf extract by varying the concentration of cadmium nitrate.Absorption spectra ofCdO nanoparticles had an absorbance peak at near 300 nm [9].

FTIR analysis:

FTIR was used to recognize the possiblebiomolecules responsible for the reduction of the Cd ions and capping of the bioreducedCdO nanoparticles synthesized bythe leaf extract [10]. Fig.3 &Fig.4 show the FTIR spectra of undopedCdO nanoparticles and Zr ion doped CdOnanoparticles which represent thediverse functional groups of the adsorbed biomolecules onthe surface of the CdO nanoparticles. The absorption peak ataround 825cm−1 confirmed the binding of –C-Cl group,the peak at 1416 cm−1 corresponding to the C–N stretching modeof the aromatic amine group. The peak at 1623 cm−1 can beassigned to the amide I band of the proteins and

ISSN (Online) 2456 -1304

International Journal of Science, Engineering and Management (IJSEM)
Vol 3, Issue 4, April 2018

aromaticrings. The peak observed at 2365 cm−1 confirmed to the C–C vibration. The peaks observed at 2853cm-1 and 2925 cm−1indicated the secondary amines and C–H stretching vibrationmodes in the hydrocarbon chains (CH aliphatic). Finally, 3396cm-1 and 3478cm−1 was related to O-H and N–H stretching vibrations. The variationsin the peak positions indicated, presumably, some metabolitessuch as polyphenols, flavonoids, alkaloids, and terpenoidswhich are abundant in leaf extract and produce the CdOnanoparticles.

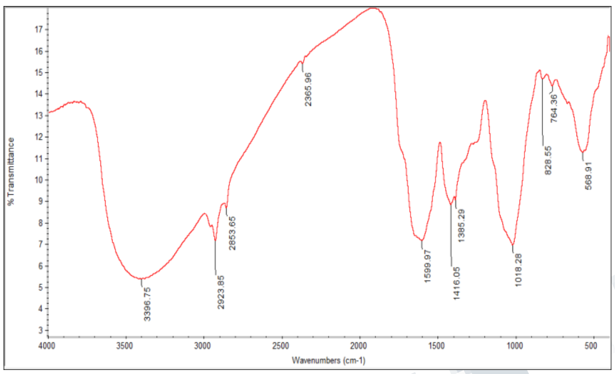

Fig.3FT- IR spectrum of undopedCdO nanoparticles synthesized using Acalyphaindicaleaf extract

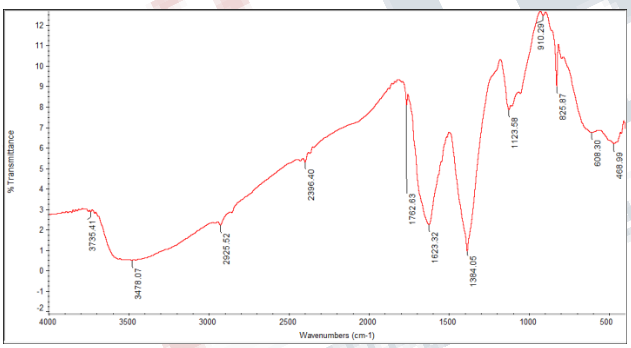

Fig. 4FT- IR spectrum of Zrion dopedCdOnanoparticles synthesized using Acalyphaindicaleaf extract

SCANNING ELECTRON MICROSCOPE:

The formation of CdO nanoparticles as well as their morphological dimensions by the SEM study demonstratedthat the average size of particles was 34 nm forundopedCdOnanoparticlesand 23.44 nm forZr ion doped CdOnanoparticles and it was shown in (Fig.5and Fig.6)[11].From the SEM micrographs shown in the Fig.5 the nanoparticles of undopedCdOhad surface with agglomerated particles having crystallinestructure. As shown in Fig.6 .the Zr ion doped CdO nanoparticles were found to have continuous surface with crystalline like nanoparticles.

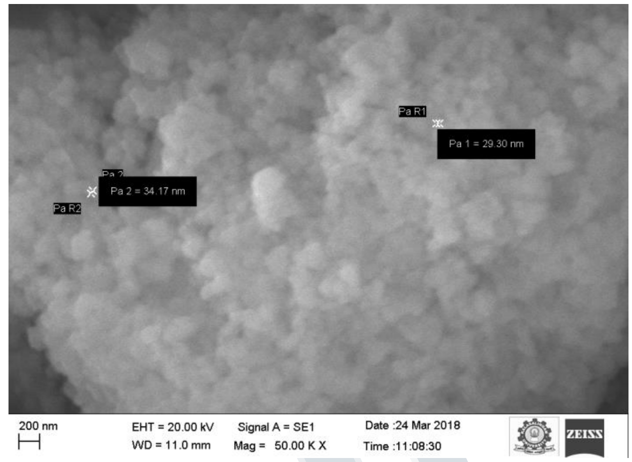

Fig.5SEM image of undopedCdO nanoparticles synthesizedusing Acalyphaindicaleaf extract

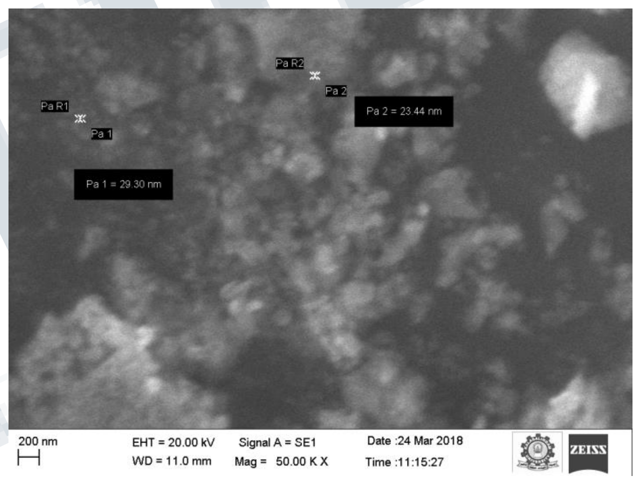

Fig.6 SEM image of Zr ion doped CdO nanoparticles synthesized using Acalyphaindicaleaf extract.

XRD:

Structural parameters of CdO nanoparticles and ZriondopedCdO nanoparticles synthesized using Acalypha indicaleaf extracts were calculated from the XRD pattern. The average grain size (D) was attained by using Debye-Scherrer Equation(1). was well matched with the standard JCPDS Card No. 36-1451. The crystallite size of CdO nanoparticles obtained were estimated to be1.5nm [12].

$D = k\lambda / \beta\cos\theta$---------(1)

Where D is average crystalline diameter in nanometer (nm), k is Scherrer constant equal to 0.94, λis wavelength of the X-ray radiation used and its equal to 2.3476 Å, βis the corrected line broadening of the nanoparticles, and θis the Bragg angle. X-ray diffraction pattern of undopedCdO nanoparticles was shown in Fig.6.It shows the grain size of

ISSN (Online) 2456 -1304

International Journal of Science, Engineering and Management (IJSEM)
Vol 3, Issue 4, April 2018

the Zr ion doped CdO nanoparticles is 1.5 nm obtained from the FWHM of peak corresponding to 2θ= 38.34160.

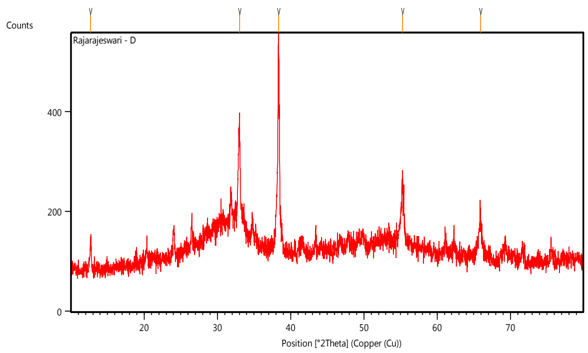

Fig.7 XRD pattern of undopedCdO nanoparticles synthesized using Acalyphaindicaleaf extract

AFM

The SynthesizedundopedCdOnanoparticles and Zr ion doped CdO nanoparticles were characterized using AFM offered a three dimensional visualization (Fig.8 and Fig.9.) with a scanning area of 2.461 pm2 between 0 m X 1.56 µm and 0 m Y 1.56 µm. Uneven spherical nanoparticles were found to be distributedover an irregular surface. The size of undopedCdOnanoparticlesis in the range of 25 nm -190nm.

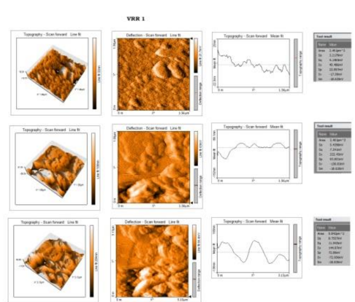

Fig.8AFM image of undopedCdO nanoparticles synthesized using Acalyphaindicaleaf extract

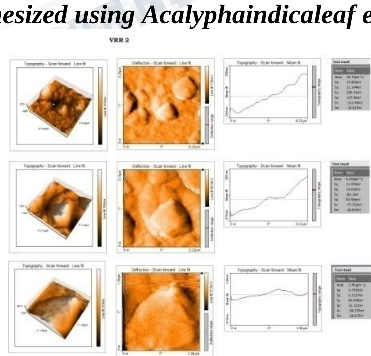

Fig.9AFM image of Zr ion doped CdO nanoparticles synthesized using Acalyphaindicaleaf extract.

The surface of Zr iondoped CdO nanoparticles were found to be rock like with irregular square nanoparticles with size in the range of 145 nm-338 nm.

.Photocatalytic activity: The UV visible absorbance values of pure crystal violetdye solution shows at590nm. The characteristic absorbance value at 590nm was used to track the photocatalytic degradation process. From Fig.10it can be clearly noticed from the recorded values that no significant changes of the concentration of Crystal violet dye after 3hirradiation, which indicated that pure Crystal violetdye solution, cannot be easily degraded by UV light. The degradation efficiency of pure crystal violet dye within 3h irradiation time was about 41% and it was shown in Table.2. The crystal violet dye degradation in presence of bio synthesized CdOnanoparticles was verified by the decrease of the peak intensity during 60min exposure in solar light shown in Fig.11. The dye degradation (%) was calculated by using the following equation (2) and its variant with the time of sunlight exposure was shown in Fig.11.

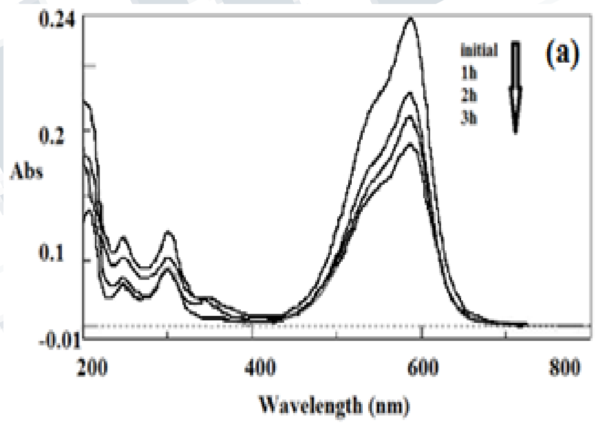

Fig.10.UV-Visible spectrum of pure Crystal dye

Efficiency=[1-C/C0]x100----- (2)

Where C0is the initial concentration of crystal violet and C is the concentration of the dye solution at the selected irradiation time. From the absorbance spectra, it was observed that the maximum degradation efficiency of crystalviolet dye within 60 min irradiation time was about 81%for undoped CdOnanoparticles.The results showed that CdO increases efficiency of degradation. Thus, the undoped CdO nanoparticles possess much higher photocatalytic activity than pure dye. The absorbance values and degradation efficiency of undopedCdO nanoparticles synthesized from Acalyphaindicaleaf extractis shown in Table.1.

ISSN (Online) 2456 -1304

International Journal of Science, Engineering and Management (IJSEM)
Vol 3, Issue 4, April 2018

Table.1.Absorbance values and degradation efficiency of undopedCdOnanoparticles synthesized using Acalyphaindicaleaf extract

Time (min)	**Absorbance**	**Efficiency (%)**
0	0.6640	-
10	0.4945	25
20	0.3584	46
30	0.3251	51
40	0.2859	57
50	0.2354	65
60	0.1271	81

Table.2. Degradation efficiency of pure Crystal violet dye

Time(h)	Degradation (%)
0	-
1	24
2	32
3	41

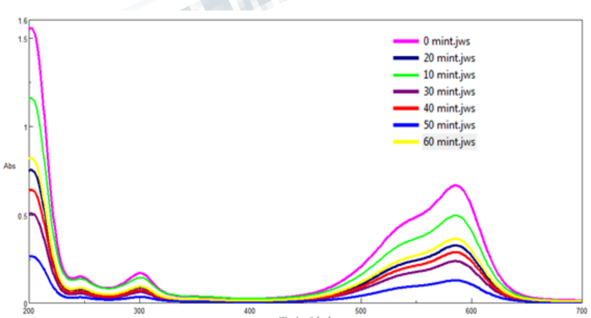

Fig.11. UV-Visible absorption spectra of Crstalvioletdye in the presence of undopedCdO nanoparticles synthesized using Acalyphaindicaleaf extract

CONCLUSION

Cadmium oxide and Zr ion doped cadmium oxide nanoparticles were synthesized (simple and cost effective)using aqueous leaf extract of Acalyphaindica. The UV spectra of CdO nanoparticles and Zr ionDoped CdO nanoparticles exhibited absorption peaks at 300nm. From the XRD pattern the particle size was found to be 1.5 nm for Zr ion doped CdO nanoparticles. SEM analysis confirms that the size of the particles was 34 nm for undoped CdO and 23.44 nm forZr ion doped CdO nanoparticles. The photocatalytic study concludes that these bio-CdO nanoparticles have greater efficiency to degrade crystal violet dye under solar irradiation. Therefore they can find application in water purification and textile industries.

ACKNOWLEDGEMENT

The authors are thankful to Department of Science and Technology (FAST TRACK and FIST) New Delhi for using Jasco UV-Visible Spectrophotometer at V.O. Chidambaram College.

REFERENCES

[1] Shukla M, Kumari S, Shukla S, Shukla RK ,Potent antibacterial activity of nanoCdO synthesized via microemulsion scheme. J Mater Environ Sci 3(4): 678-685,2012.

[2] Thema FT, Beukes P, Gurib-Fakim A, Maaza M ,Green synthesis of monteponiteCdO nanoparticles by Agathosmabetulina natural extract. J Alloys and Compounds 646: 1043-1048,2015.

[3] Heidari A, Brown C ,Study of composition and morphology of cadmium oxide (CdO) nanoparticles for eliminating cancer cells. J Nanomed Res 2(5): 00042,2015.

[4] Thovhogi N, Park E, Manikandan E, Maaza M, Gurib-Fakim A, Physical properties of CdO nanoparticles synthesized by green chemistry via Hibiscus sabdariffa flower extract. J Alloys and Compounds 655: 314-32,2015.

[5] Pande SN, Bharati KT, Wakchure SK, Ghotekar SK, Gujrathi DB, et al. ,Green synthesis of silver nanoparticles by carallumafimbriata L. and its characterization. IndJou App Res 5: 749-750,2015.

[6] Ghotekar SK, Pande SN, Pansambal SS, Sanap DS, Mahale KM, et al. ,Biosynthesis of silver nanoparticles using unripe fruit extract of Annonareticulata L. and its characterization. WJPPS 4: 1304-1312,2015.

[7] Ajay Savale, Suresh Ghotekar, ShreyasPansambal and OnkarPardeshi, Green Synthesis of Fluorescent CdO Nanoparticles using Leucaenaleucocephala L. Extract and their Biological Activities, J BacteriolMycol Open Access, 5(5): 00148,2017.

[8] MuthuChudarkodi R.R, and Rajalaxshmi A, Green Synthesis, Characterization of ZnO nanoparticles and Ceion doped ZnO nanoparticles assisted Sesbania Grandiflora for photocatalyticapplication, Research Journal ofMaterialSciences, Vol. 4(2), 1-6,March,2016.

[9] Javad KarimiAndeani and Sasan Mohsenzadeh, Phytosynthesis of Cadmium Oxide Nanoparticles from Achillea wilhelmsii Flowers, Journal of Chemistry Volume, Article ID 147613, 4 pages,2013.

[10] Vani. P, Manikandan. N and Vinitha. G, A Green strategy to synthesize environment friendly metal oxide nanoparticlesforpotential Applications A Review, Asian Journal of Pharmaceutical and Clinical Research, 03 March,2017.

[11] D. DurgaVijaykarthik, M. Kirithika, N.Prithivikumaran, N. Jeyakumaran, Synthesis and characterization of Cadmium Oxide nanoparticles for antimicrobial activity, Int. J.NanoDimens. 5(6): 557-562, Special issue,2014.

[12] A.K.Barve, S.M.Gadegone, M.R. Lanjewar and andR.B.Lanjewar, Synthesis and Characterization of CdO Nanomaterial and their Photocatalytic Activity, International Journal on Recent and Innovation Trends in Computing and Communication ISSN: 2321-8169 Volume: 2 Issue: 9,2017.

