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Review of Synthesis of Cadmium Oxide Nanoparticles

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Abstract – Cadmium oxide nanoparticles are synthesized by various methods. This article summarizes the literature on the various method of synthesis of cadmium oxide nanoparticles. Investigation has been carried out across the world in order to synthesis and application of cadmium oxide nanoparticles.

Keywords: Cadmium Oxide, Nanoparticles.

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INTRODUCTION

In the recent years, nanocomposite materials have received great interest for both industrial and academic applications. They have unique physical and chemical properties which are different from those of either the bulk materials or single atoms. This is due to the fact that a small amount of the nano-additives could improve the overall performances of the polymeric materials. This is owing to the small size, large specific area, quantum confinement effects, and the strong interfacial interaction of the nanomaterials. Reduction in the dimensionality of such materials from the three dimensional bulk phases to the zero-dimensional nanoparticles can lead to enhanced non-linearity, determined by the quantum size effects and other mesoscopic effects.

Cadmium oxide (CdO) is one of the promising II–VI compounds. CdO powders and films have n-type semiconducting properties and mobility. In addition, CdO has a high optical transmittance in the visible region of the solar spectrum along with a refractive index of 2.49. Many researchers have focused on cadmium oxide (CdO) due to their applications in several areas of research, specifically in optoelectronic and other applications, including solar cells, phototransistors, photodiodes, transparent electrodes and gas sensors. It has been reported that the physical and chemical properties of CdO are relative to its stoichiometry as well as particle shape and size, which, in turn, depend on its preparation methods and preparation conditions .different methods of preparation of cadmium oxide are

1) Hydrothermal Method

2) Green Synthesis Method

3) Laser Technique

4) Precipitation Method

5) Sol gel Method

6) Thermal Decomposition Method

7) Step Calcination Method

Dyes are one of the major groups of pollutants in wastewaters released from textile and other industrial processes. Among various Physical and biological techniques for the treatment of pollutants, precipitation, adsorption, air stripping, flocculation, reverse osmosis, and ultra-filtration can be used for color removal from textile effluents. One of the main environmental applications of nanotechnology is in the water sector. Heterogeneous photocatalysis, one of the advanced oxidation processes (AOPs), is a cost-effective treatment method for the removal of toxic pollutants from industrial waste water sowing to its ability to convert these into safer end products such as CO_2, H_2O and mineral acids. Semiconductor nanoparticles, as heterogeneous photocatalysts, have attracted much interest due to their size tunable physical and chemical properties.

This paper includes comparative study of different methods of synthesis of cadmium oxide nanoparticle and its photocatalytic activity.

1. Hydrothermal Method

K. Karthik et al. (2017), studied CdO nanoparticles (NPs) were synthesized (hydrothermal) with cadmium acetate and ammonium hydroxide. The average crystallite size (D) calculated for the synthesized CdO NPs is 43 nm. The microstrain for

the synthesized CdO NPs is 0.0029. The photocatalytic performance has been evaluated of the CdO NPs for the degradation of methylene blue under sunlight irradiation. CdO NPs were screened for their in vitro antibacterial activity against human pathogens such as Gram negative (Escherichia coli, Pseudomonas aeruginosa, Proteus vulgaris) and Gram positive (Staphylococcus aureus, Enterococcus faecalis, Bacillus subtilis) bacteria has been investigated. From the photoluminescence studies, the band gap is 2.54 eV indicating its efficient photocatalytic activity. Hence, the outcome of this study emphasizing that hydrothermally prepared CdO NPs suitable for optoelectronics (light emitting diode), photocatalytic degradation (waste water treatment) and pharmaceuticals (antibiotics) applications.

A.K. Barve et al. (2014), stuided nano-sized cadmium oxide was synthesized by a simple hydrothermal method using $CdC^{l}{}_{2}$. H_2O as a metal precursor in the presence of polyethylene glycol and urea. The advantage of hydrothermal method is that it requires very less time for synthesis, easy to operate and gives high yield. The photocatalytic activity of CdO nanomaterial was studied using methyl red dye as an organic pollutant. Organic dyes are the main pollutants in wastewaters released from textile and other industrial processes. The photocatalytic activities of CdO nanoparticles were evaluated by measuring the degradation of Methyl red (MR) in water under the UV region. The degradation percentage were found to decrease with increase in concentration of the organic pollutant. Their result shows the the band gap of semiconductor materials increases with the decrease in particles size, which leads to the shift of the absorption edge toward high energy effect. The absorption band of the CdO nanoparticles have been shows a blue shift due to the quantum confinement of the exactions present in the sample compare with bulk CdO particles. The particle shows spherical nature and particle size found to be 60-70 nm. Methyl red was chosen as the model organic pollutant. According to the principles of CdO photocatalysts , the energy gap are generated on the surface of Cadmium oxide when are irradiated in photon light which can either exceeding or equaling the band gap energy, an electron may be promoted from the valence band to the conduction band (e^{-}_{cb}) leaving behind an electronic vacancy or “hole” in the valence band (h+vb). If charge separation is maintained, the electron and hole may migrate to the catalyst surface where they participate in redox reactions with the adsorbed species. CdO nanoparticles has been synthesized by hydrothermal method using very easy, cheap and convenient process.

2. Green synthesis

H. Nagabhushan et al. (2016), studied the synthesis of CdO nanostructures via green combustion route using green tea extract as fuel. The whole process takes only minimum time to produce CdO nanoparticles. Due to strong green emission peak, the CdO nanostructures can be utilized in the industry of high quality monochromatic laser.

N. Thovhogi et al. (2016), studied the first time synthesis of CdO nano-crystals by green chemistry process based on Hibiscus Sabdariffa flower extract. Hibiscus Sabdariffa is a shrub belonging to the family Malyaceae with red flowers in a form calyces. In this plant phenolic compound is present. These phenolic compounds found in the flower includes organic and phenolic acids, such as citric acid, hydroxycitric acid and hibiscus acid.

3. Laser Technique

Ayman M. Mostafa et al. (2017), explained colloidal spherical nanoparticles of cadmium oxide (CdO) have been prepared by using Nd:YAG pulsed laser ablation [1064 nm, 7 ns, 10 Hz, and 80 mJ] of cadmium sheets target immersed on deionized water. The optical properties of prepared CdO nanoparticles were investigated by using UV-Visible spectrophotometer. The structure of CdO nanoparticles is confirmed to be crystalline and has hexagonal structure. In this method, nanomaterials could be produced with or without the presence of stabilizer. The produced nanomaterials from PLAL technique are completely pure without any byproduct or toxic chemicals compared to others. The formation of nanomaterials by PLAL could be produced by two ways;

1- Traditionally PLAL process; direct ablation process of bulk metals in liquid media to produce colloidal particles in nanoscale.

2- Modified PLAL process; Laser induced process of colloidal particles in liquid media prepared by chemical wet methods to enhance particle size or shape. Cleaned Cd sheets were immersed in 10 ml if deionized water. The target was irradiated with the second harmonic of a pulsed Nd:YAG laser operating at 1064 nm with pulse repetition rate 10 Hz, pulse width 7 ns, and laser energy 80 mJ/pulse. During that process, the solution was stirred by rotational moving to prevent shielding from produced nanoparticles. CdO nanospheres with average particle size 24nm were successfully synthesized by laser ablation in liquid environmental. The optical characterization was studied using UV-VIS spectrophotometer.

Ibrahim R. Agool et al. (2016), Nanoparticles NPS of cadmium oxide CdO were generated by laser ablation of a solid target (cadmium) in polyvinylpyrrolidone (PVP) solution. CdO colloidal nanoparticles have been synthesized by laser

ablation Nd: YAG (1064 nm, 100 pulses, pulse energy= 400 mJ). Laser ablation of cadmium Cd solid target immersed in polyvinylpyrrolidone, which includes the laser source Nd:YAG laser system type HUAFEI, λ=1064nm wavelength with maximum energy per pulse of 400 mJ, pulse width of 9 ns, repetition rate of (1Hz) and beam diameter of 2.3 mm. The ablation process was typically done for (1 min) at room temperature. Advantage of this method is convenient for synthesis of CdO nanoparticles in normal laboratory conditions and low cost.

S. C. Singh et al. (2009), studied cadmium hydroxide/oxide nanocomposite material is synthesized by pulsed laser ablation of cadmium metal in double distilled water. Laser ablation of cadmium metal in pure water produces 18.6 nm average sized particles of $Cd(OH)_2$/CdO nanocomposite, which converted into CdO nanocrystals with 15.9 nm average size after annealing at 350 C for 9 h. High purity cadmium target placed on the bottom of glass vessel containing 30 mL double distilled water, was allowed to irradiate with focused output of 1,064 nm from pulsed Nd:YAG laser operating at 35 mJ/pulse energy, 10 ns pulse width, and 10 Hz repetition rate for 1 h. As synthesized colloidal suspension was brown in color and found stable for 1 week. Solution was centrifuged at 4,000 rpm and obtained residue was dried at 60 C in oven for 24 h. Annealing of the sample at 350 C for 9 h converts most of the CdO nanocrystals, which is also verified by DTA data. Produced CdO nanocrystals after annealing have 2.7 eV band gap, very close to the CdO bulk band gap indicating that there is no quantum confinement effect, which is effective below the size of 10 nm. This method can provide an alternative, pollution free method.

4. Precipitation Method

D. Durga Vijaykarthik et al.(2014), explained cadmium oxide (CdO) nanoparticles were prepared by precipitation method using Cadmium acetate and ammonia solution. In this the antimicrobial activities of different concentration of the CdO nanoparticles were tested by treating *E.coli* cultures with CdO nanoparticles. Cadmium Oxide nanoparticles show effective antimicrobial activity. Different organisms were used to observe the zone of inhibition of CdO nanoparticles. The average crystalline size of CdO nanoparticles is found to be 39.73 nm. Scanning Electron Microscope (SEM) Study indicates the amount of Cadmium and Oxide present in the CdO Nanoparticle is 84.02% & 15.98% and no characteristic peaks of impurities or other precursor compounds are observed. The energy band gap of Cadmium Oxide nanoparticle is 2.15 eV. The optical absorbtion peak is calculated as 73.33%. Prepared CdO nanoparticles showed good antimicrobial activity by inhibiting their growth.

M. Tabatabaee et al.(2013), studied nano-sized cadmium oxide was synthesized by a simple method using $Cd(NO_3)_2.4H_2O$ as a reagent in the presence of polyethylene glycol (PEG 2000). The crystal structure consists of face centered cubes and the entire d-line patterns match the reported values. The size distribution and morphology of the sample was analyzed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Energy dispersive spectrometry (EDS) attached to SEM was employed to perform the elemental analyses of the nanostructured materials. Prepared CdO crystals were grown in face-centered cubes over the range (15-25 nm). By the co-precipitation method used, $Cd(OH)_2$ was formed in a basic medium.

Sathish Reddy et al.(2010),prepared cadmium oxide by precipitation method. First ethanol was dried using type 3A molecular sieves before use. NaOH pallets were pulverized into fine powder under a dry use nitrogen flow. In a typical experiment first solution prepared using 0.03M $CdSO_4$, 0.06M CH_3COOH and 40 mg CTAB as surfactant in 1 dm3 of double distilled water. The second solution was prepared by 0.09 M NaOH pallets and 25ml 70% ethanol in 1 dm3of double distilled water. Then first solution was added to second solution with continues stirring. The obtained precipitate was filtered by using Whatmann filter paper and dried at 800C in hot air oven about 1hour. Then dried precipitate was transferred to silica crucible and ignited at 4000C for about 4hours. Then obtained powder was washed with ethanol three to four times to remove impurities present in the particles .Then these are characterized using XRD, UV-Visible absorption Spectroscopy, and applied for fabrication of carbon paste electrode for determination of DA and AA. Advantage of this method is convenient for synthesis of CdO nanoparticles in normal laboratory conditions, in low cost.

5. Sol gel Method

A.M. El Sayed et al.(2014), synthesized CdO nanoparticles by using a sol-gel process. First, 0.7 M solution was prepared by dissolving 16.54 g of a high-purity cadmium nitrate [$Cd(NO_3)_2$,, MW5236.42, Nova Oleochem Limited] and 8.825 g of oxalic acid ($C_2H_2O_4$) in 100 ml double distilled (DD) water with a magnetic stirring for 2 h. The obtained sol was held in an oven at 100_C for 8 h, then cooled to 70_C and stirred to obtain the gel.

This gel was aged for 18 h, then it was calcined at 400_C for 3 h to obtain CdO nanoparticles. CdO nanoparticles were characterized by X-ray diffraction (XRD) . It has been demonstrated that adding the sol-gel synthesized CdO nanoparticles into the PVC films has analysis induces significant modifications in both the optical and dielectric properties. XRD analysis indicated that the crystal structure of the CdO is cubic with Fm3m space symmetry group while PVC has a typical semicrystalline nature. The refractive index of the pure PVC is 1.48 and increased significantly with increasing the CdO content and the optical dispersion constants were also changed.

V. S. Vadgama et al(2017), synthesized CdO nano particles successfully by Sol-Gel technique. The analytical reagent (AR) grade chemicals cadmium nitrate tetra hydrate (Cd(NO3)2 .4H2O) and aqueous ammonium hydroxide (NH4OH) were used as precursors to synthesize CdO nano particles. In a typical process, 15.42 gm of Cd(NO3)2 . 4H2O and 5 ml of aqueous ammonia was dissolved in 100 ml of distilled water under stirring. Thus, the colloidal gel suspension was obtained and then this gel suspension was separated from the liquid by filtration and washed several times with distilled water and dried at 50°C for 4 hours. Finally, the dried samples were calcined at temperature of 600°C to obtained cadmium oxide. From powder XRD, it is confirmed that CdO nano particles have cubic crystal structure with unit-cell parameters: a=b=c=4.694 Å. The average crystallite size is found to be nearly ≈ 31 nm from Scherrer's formula. Presences of O–H, Cd–O vibrations are confirmed by FT-IR. TEM images indicate the spherical type of morphology with the size ranging between 50 to 100 nm in the view field studied. highly stable nature of the material with only 10% weight loss due to moisture collected.

6. Thermal Decomposition Method

Divine Mbom Yufanyi et al.(2014) prepared cadmium oxide nanopartcle by thermal decomposition method. In this method HMTA (4 mmol, 0.5608 g) was dissolved in 15 mL of ethanol (sonication for 20 min at room temperature). Cadmium nitrate (2 mmol) in 10 mL of ethanol was added drop wise under magnetic stirring. The mixture was stirred for a further 2 h. The white precipitate formed was filtered, washed several times with ethanol and dried in a desiccator over silica gel. A sample of the dry precursor (0.5 g) was ground, placed in a ceramic crucible and calcined at 500 °C (CdO-500). The crucible was placed in the furnace, heated to the desired calcination temperature, and calcination in air continued for 2 h. The sample was allowed to cool down to room temperature in the furnace. The reddish-brown powder obtained could easily be re-dispersed in water and ethanol. The CdO nanoparticles obtained is mesoporous, has a surface area of 58.4 m2/g and an average pore diameter of 4.7 nm. The low-temperature synthetic technique is simple and cost effective and it can be extended to the synthesis of other metal oxide nanoparticles.

7. Step Calcination Method

A.S. Aldwayyan et al.(2013), synthesized CdO nanparticle by one step calcinations process. A mixture of 2,9-dimethyl-1,10-phenanthroline (50.0 mg, 0.24 mmol) in dichloromethane (5 ml) and CdI2 (65.4 mg, 0.24 mmol) in methanol (10 mL) was placed in a round bottom flask and stirred for 4 h at room temperature. The solution was concentrated to about 1 mL under reduced pressure. Addition of 40 mL of n-hexane caused the precipitation of white powder, which was filtered and then dried under vacuum to 108 mg (yield 94% based on Cd). 0.5g dmphen-CdI2 was calcinated directly at 800 oC for 120 min, the calcinations process was stopped upon no organic function group vibrations was detected by IR, white powder CdO was formed at the end of the process. This report has shown the synthesis of CdO nanoparticles using organometallic dmphen-CdI2 complex through one step calcinations process at 800 oC. From XRD, SEM and TEM data obtained of the nanoparticle size were ~ 50 nm. Advantage of this method is convenient for synthesis of CdO nanoparticles in normal laboratory conditions and low cost.

SUMMARY OF LITERATURE

Sr. No.	Method of synthesis	Raw Materials	Result	Remark	Authors Name
1	Hydrothermal Method	Cadmium acetate, Ammonia solution, Ethanol, Methylene blue.	The microstrain for the synthesized CdO NPs is 0.0029. particle size found to be 43 nm.	This synthesized CdO nanoparticles shows efficient photocatalyic and antimicrobial activity.	K. Karthik et al. (2017)
2	Hydrothermal Method	Cadmium chloride, Ethylene glycol and Urea, Methyl red dye	The particle shows spherical nature and particle size found to be 60-70 nm.	This is very easy, cheap and convenient process.	A.K. Barve et al. (2014)
3	Green Synthesis	Cadmium nitrate, Green tree extract.	Highly crystalline CdO nanostructures with different morphology (spherical and raspberry like) have	This fast and low cost method may be suitable for the preparation in few minutes of other crystalline	H. Nagabhushan et al. (2016)

			been obtained by the green synthesis procedure adopted without high calcination temperature.	metal oxide nanostructures without post-synthesis heating.	
4.	Green synthesis	Hibiscus Sabdariffa flower extract, Cadmium nitate.	The optical band gap values of the CdO nanoparticles were deduced to be 2.1 eV and 2.7 eV for indirect and direct transitions respectively.	The biosynthesis of high purity and crystalline CdO nanocrystals by green novel and environmental friendly process	N. Thovhogi et al. (2016),
5.	Laser Techniquie	Cd metal granulated sheets,	Synthesized nanoparticles has crystalline and hexagonal structure and has diameter approximately 24 nm.	In this method, nanomaterials could be produced with or without presence of stabilizers.	Ayman M. Mostafa et al. (2017)
6.	Laser Techniqu e	Cadmium , polyvinyl , Pyrrolidone	Average grain size of CdO hasn't exceeded 88 nm.	This method is convenient for synthesis of CdO nanoparticles in normal laboratory conditions and low cost.	Ibrahim R. Agool et al. (2016),
7.	Laser Techniqu e	cadmium metal , double distilled water	21.6 nm sized CdO nanocrystals were obtained with 2.7 eV band gap	Nanocrystals synthesized by this method have chemical contamination free surfaces, which can be used for biological applications.	S. C. Singh et al. (2009),
8.	Precipitation Method	Cadmium acetate , ammonia	average crystalline size of CdO nanoparticl	Prepared CdO nanoparticles showed	D. Durga Vijayakarthik et al.(201
			es is found to be 39.73 nm , energy band gap 2.15eV	good antimicrobial activity by inhibiting bacteria growth.	4)
9.	Precipitation Method	cadmium nitrate tetrahydrate, polyethylene glycol	particle size of nano-sized CdO is ~15–25 nm	It is a simple method in which CdO crystals were grown in face-centered cubes	M. Tabatabaee et al.(2013)
10.	Precipitation Method	Cadmium sulphate , Sodium hydroxide , Graphite powder, Acetic acid (99.955 %), Ethanol (99.5%) ,Hydrochloric acid, CTAB, Ascorbic acid and Dopamine , Acetate buffer	from XRD data obtained CdO nanoparticle size was 47.8 nm	Method is low cost and convenient to synthesis cadmium oxide nanoparticle on laboratory condition.	Sathish Reddy et al.(2010),
11.	Sol-Gel Method	cadmium nitrate , oxalic acid	XRD analysis indicated that the crystal structure of the CdO is cubic with Fm3m space symmetry group	With incresed CdO content in PVC, refractive index of PVC increases.	A.M. El Sayed et al.(2014)
12	Sol-Gel Method	cadmium nitrate tetra hydrate, aqueous ammonium hydroxide, ammonia	CdO nano particles have cubic crystal structure with unit-cell parameters. spherical type of morphology with the size ranging between 50 to 100 nm	highly stable nature of the material with only 10% weight loss due to moisture collected.	V. S. Vadgama et al(2017)

13.	Thermal Decomposition Method	HMTA, ethanol, Cadmium nitrate, silica gel	The CdO nanoparticles obtained is mesoporous, has a surface area of 58.4 m2/g and an average pore diameter of 4.7 nm	It is simple and cost effective method	Divine Mbom Yufanyi et al., (2014)
14	Step Calcination Method	2,9-dimethyl-1,10phenanthroline, dichloromethane, Cd I_2, methanol	From XRD, SEM and TEM data, obtained nanoparticle size were ~ 50 nm.	Method is low cost and convenient to synthesis cadmium oxide nanoparticle on laboratory condition.	A.S. Aldwayyan et al.(2013)

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