International Journal of All Research Education and Scientific Methods (IJARESM), ISSN: 2455-6211
Volume 11, Issue 3, March-2023, Impact Factor: 7.429, Available online at: www.ijaresm.com

Sol - Gel Method for Synthesis and Characterization Studies of Cadmium Oxide (Cdo) Nanoparticles

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ABSTRACT

Cadmium Oxide (CdO) nanostructures have been successfully prepared by sol - gel method. The crystallite size and morphology of CdO have been investigated by X-Ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectrum (EDS) and transmission electron microscopy (TEM) techniques. The XRD pattern of average particle sizes of CdO is estimated to be around 16 nm. Furthermore, CdO nanoparticles have the crystallite size in the range ~12-36 nm, as confirmed by TEM. Results obtained indicate that the powder method is a promising low temperature, cheap, and fast method for the production of CdO nanostructures.

Keywords: CdO nanoparticles; Sol – Gel method; Structural; TEM.

INTRODUCTION

Cadmium oxide (CdO) is a white solid inorganic powder. It is non flammable, stable and insoluble in water, II-VI semiconductor with wide band gap energy that is 3.3ev and high excitation energy that is 60 eV [1]. In recent years, noble metal oxide nano particles have been the subject of focused research due to their unique electronic optical, mechanical, magnetic and chemical properties. Cadmium oxide is the topic of interest in these days due to its presence much unique and important morphology likes nanorods, nanoflowers, nanowires, nano dendrites and nanoparticles.

This semiconductor has several favorable properties including good transparency, high electron mobility, strong room temperature, low toxicity, luminescence and photo chemical stability and higher breakdown field strength. General, cadmium oxide crystallizes in two main form, hexagonal wurtzite and cubic CdO blende but the (B4 type) wurtzite structure is obtained only at optimum pressure and temperature [2, 3]. CdO crystallizes in the typical wurtzite hexagonal structure where oxygen and cadmium atoms are spatially arranged in a way that O atoms are arranged in a closed hexagonal structure, while the Zn atoms occupy the centre of the distorted tetrahedron structure [4]. The variety of structures of Nano metric zinc oxide means that CdO can be classified among new materials with potential applications in many fields of nano technology. CdO can occur in one - (ID), two - (2D), and three-dimensional (3D) structures. One dimensional structure make up the largest group, including nano rod -needles, -helixes, - springs and - rings, - ribbons, - tubes - belts - wires and -combs. Cadmium oxide can be obtained in 2D structures, such as Nano plate/Nano sheet and Nano pellets. Examples of 3D structures of zinc oxide include flower, dandelion, snowflakes, coniferous urchin-like, CdO provides one of the greatest assortments of varied particle structures among all known materials [5, 6]. The large specific surface area high pore volume, nano structured properties, low cost and low toxicity of nano CdO [7] make it a promising candidate, particularly in catalysts [8], photo catalysis, electrostatic dissipative coating, transparent UV protection films, and chemical sensors [9-12], gas sensor, solar cells. Moreover, CdO nanoparticles have a tremendous potential in biological applications like biological sensing, biological labeling, gene delivery, drug delivery and nano-medicine [13-16].

One of the main environmental applications of nanotechnology is in the water sector. heterogeneous photocatalysts, one of the advanced oxidation process(AOPS),is a cost-effective treatment methods for the removal of toxic pollutants from industrial waste water sowing to its ability to convert these into safer and products such as CO_2, H_2O and mineral acids [17-18]. Several conventional methods have been used for synthesis of zinc oxide nanoparticles like chemical vapour synthesis [19], laser ablation [20]), solvothermal [21], thermal decomposition [22], and sol-gel method [23]. Here we present a simple Co-precipitation method to synthesize uniform, spherically shaped and pure CdO nanoparticles using zinc Sulphate as a metal precursor and ammonium hydroxide as a precipitating agent. In the present study was report the synthesis of CdO nanoparticles using Co precipitation method and the characterization of CdO nanoparticles using

x-ray diffraction (XRD), transmission electron microscopy (TEM), selected area electron diffraction (SAED) , scanning electron microscopy (SEM), fourier transform infrared spectroscopy (FTIR) energy dispersive spectrum (EDS) are discussed [24 – 26].

EXPERIMENTAL PROCEDURE

Materials

Cadmium acetate (Merk) and ammonia solution (Merk, 98%) were used for the synthesis of CdO nanoparticles. Cadmium acetate were of analytical grade and used as received without further purification was used for the synthesis. Double distilled water was used for all the experiments.

2.2 Synthesis

The initial solution was prepared dissolving 0.1 mol of cadmium acetate in 20 ml of ethanol, which was later mixed with 175 ml of deionized water. The pH of the solution was maintained at 8 by adding a liquid ammonia solution dropwise. The resulting product was filtered and washed with double distilled water and ethanol until it became free from impurities. The precipitate was irradiated for 5 minutes in a household microwave oven (radiation frequency - 2.45 GH_Z, Power up to 1 $_KW$) with convection mode, giving a white product. Additionally, the resulting powders were thermally treated at 300°C in a muffle furnace for 4 hours.

2.3 Characterization

The resulting powders were analyzed by X-ray diffraction (XRD) using a Bruker AXS D8 Advance instrument diffractometer with monochromatic CuKα1 wavelength of 1.5406 Å. The samples morphology was observed by scanning electron microscopy (SEM), using a JEOL 5600LV microscope at an accelerating voltage of 10 kV. The microstructure was studied by transmission electron microscopy (TEM) and selected-area electron diffraction (SAED) in a Tecnai G20-stwin operated at 200 kV. The Fourier transform infrared spectra (FT-IR) of the samples were recorded by using a Nicolet 5DX FTIR spectrometer.

RESULTS AND DISCUSSION

X-Ray Diffraction analysis

There are various methods which have been used to produce nanoparticles powder method is one of the advantage of improved compositional homogeneity. Since the recant constituents are mixed at a molecular level. Moreover it is a simple method. Nano phase materials are synthesized have to be characterized the particle size and morphology.

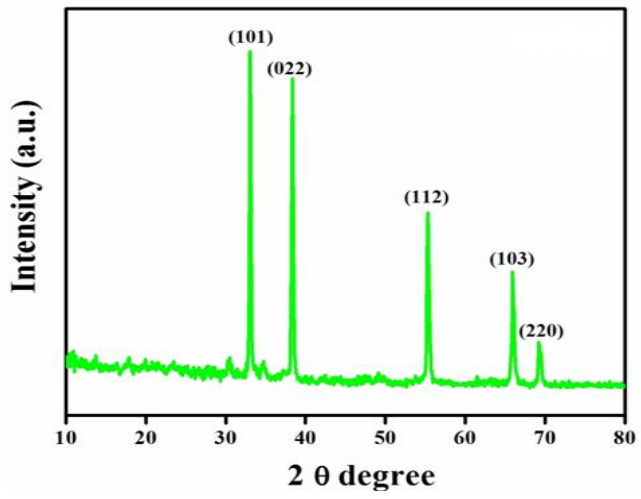

Fig.1 XRD pattern of CdO nanoparticles synthesized by sol-gel method

- X-ray diffraction patterns (XRD) are generally used to determine the mean size of nanoparticles by using Scherer's equation.
- The grain sizes estimated from (SEM) observations were different from those done by means of Scherer's equation. This equation assumes that all the crystallites are of the same size, but in an actual specimen, the size range and distribution affectβ.

- Energy-dispersive X-ray spectroscopy (EDS) is an analytical technique used for the elemental analysis or chemical characterization of a sample. The fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum.

X-Ray Diffraction Analysis Of Cdo Nano particles

X-ray diffraction, based in wide angle elastic scattering of X-rays, has been the most important technique for determining the structure of materials characterized by long range order typical applications are in physics, material science, geology, mineralogy, ceramics etc. The wavelength of x-ray is of order of the distance between neighboring atoms in a crystal and exhibits interference and diffraction effects. X-ray are reflected beam interference positively to give a strong diffracted beam is represented by Bragg's law and is given by $2d \sin\theta = n\lambda$

Where, n is integer describing the order of reflection, λ is the wavelength of X-rays, d is the inter planar spacing and θ is the Bragg's angle at which maximum in diffracted density occurs. The diffracted method .if the material under study is crystalline, well-defined peaks can be observed while amorphous system show a instead of well-defined peaks.

Partical Size Determination

The XRD pattern of nanometer size particles are quite striking because of the size dependent and structure-specific features observed. Therefore XRD techniques are widely used for the particle size determination and structure of nanoparticles. The tin oxide nanoparticles of XRD pattern have the observed diffraction peak point at (101, 022, 112, 103, and 220) agree well with the **cubic structure** of cadmium oxide **(JCPDS file no.46-1088).** The average crystalline size has been approximately **18 nm** if the line broadening only due to small crystalline size, the size of the crystallite can be estimated from the Scherer's formula,

$$D = \frac{K\lambda}{\beta\cos\theta}$$

Where, θ is the Bragg's angle , λ is the wavelength of the X-rays, D is the mean dimension of the crystallite size of powder sample, β is the mean width at half maximum of the diffraction peaks on the 2% scale in radia K is a constant approximately equal to 0.9 (the wide of reflection peaks and so a correction for instrumental broadening is usually applied in this method).

(SEM) ANALYSIS OF CdO NANO PARTICLES

The surface morphology of tin oxide nanoparticles is studied by scanning electron microscope. Fig. 2 Shows the SEM image CdO nanoparticles with magnification of 5000. The instrumental parameters, accelerating voltage, spot size and magnification are used to obtain SEM image.

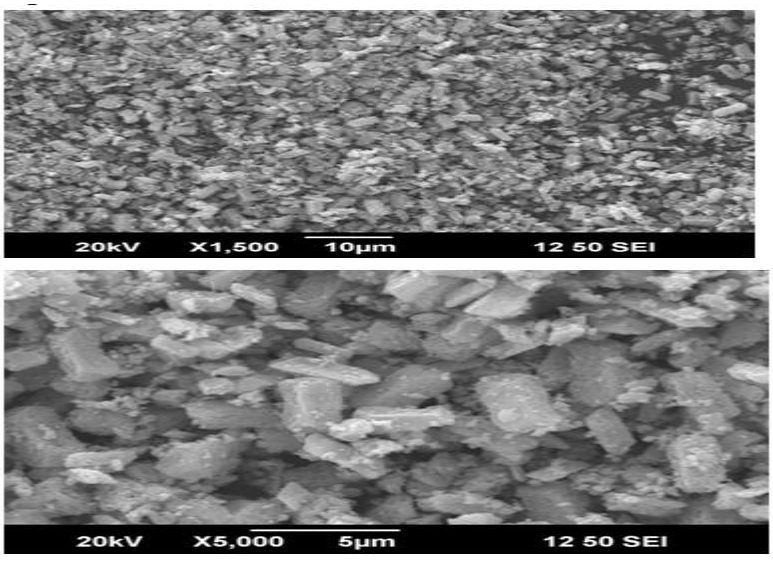

Fig.2 SEM analysis of CdO nanoparticles by synthesized sol-gel method

The nanoparticles have **cubic shape** with the sizes smaller than 100 nm. It shows the microstructure of the electrochemical reduction, which runs the cadmium oxide nanoparticles that reveals the presence of agglomerations and non agglomeration. The appearance of some particles has a regular shape and the distributions are in uniform, and it is due to the partial solubility of surfactant in the solvent under the given experimental conditions at room temperature.

(EDS) ANALYSIS OF CdO_2 NANOPARTICLES

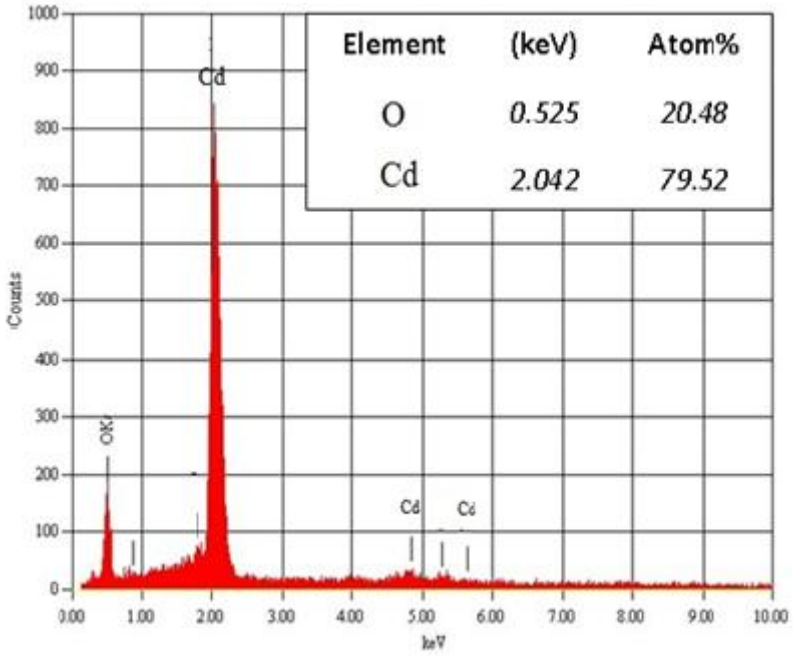

Fig.3. EDS analysis of CdO nanoparticles by synthesized by sol-gel method

The composition of the obtained CdO was analyzed by means of energy dispersive spectrum (EDS) as shown in Fig. 3. The EDS result showed the presence of Cd and O by the appearance of Cd and O peaks.

Transmission Electron Microscopy (TEM)

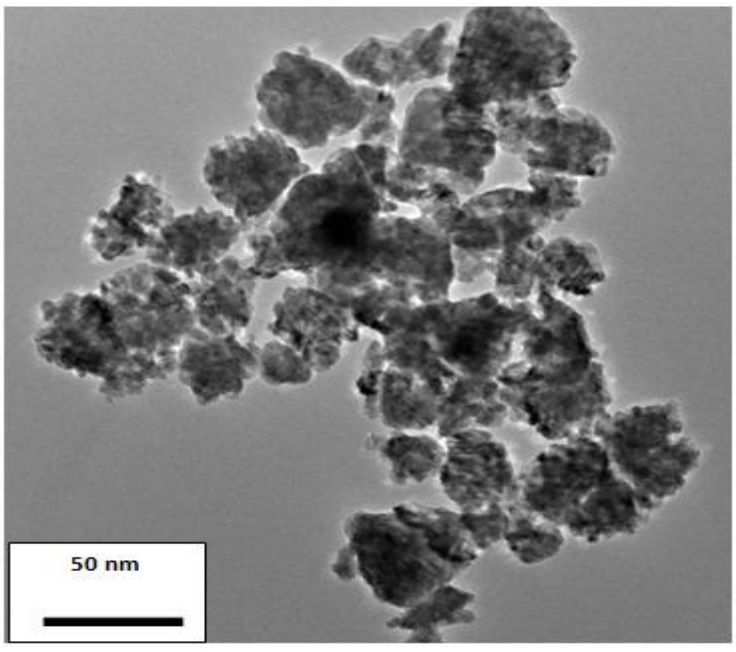

Fig.4. TEM analysis of CdO nanoparticles by synthesized by sol-gel method

Transmission electron microscopy (TEM) images have been investigated. Particle size and nanostructure of **CdO** nanoparticles have been examined through transmission electron microscope are shown in Fig 4. The particle sizes of the cadmium oxide samples are consistent with the results of XRD analysis. The pattern implies that the prepared CdO_2 nanoparticles are some particles nearly spherical shape and some particles agglomeration shape in the range of about 16 - 28 nm. The SAED pattern of CdO_2 nanostructures further confirms the crystalline nature of the sample. Fig 4 shows an electron diffraction pattern representing well-defined quasicontinuous diffraction rings. It is visible that the (011), (021), (211) and (131) planes were clearly distinguished as observed in XRD patterns. The SAED pattern of the high resolution TEM image conformed the nanoparticles corresponds to Cubic structure.

CONCLUSION

The sample have been investigates to the annealed at 400°C temperature. The Cadmium Oxide (CdO) nanoparticles synthesized by sol-gel method have been investigated. The Powder x-ray diffraction pattern of the nanocrystalline CdO_2 possessed cubic structure with average crystallite size in the range of 18 nm for annealed temperature at 400°C. SEM results are presence of the agglomerated with crystalline nature with composed by the agglomeration and non agglomeration of the smaller crystallites. The EDS result showed the presence of Cd and O by the appearance of Cd and O peaks. The TEM pattern implies that the prepared CdO nanoparticles are some particles Needle shape and some particles spherical shape in the range of about 16 - 28 nm. This is the simple synthesis method; it has used for gas sensor and diode applications.

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