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Microwave Mediated Synthesis and Characterizations of CdO Nanoparticles

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ABSTRACT

The microwave synthesis route employed for the preparation of cadmium metal oxide nanoparticles. Crystalline $CdCO_3$ has been synthesized at nanoscale through a reaction of cadmium acetate with tetramethyammoniumhydroxide (TMAH) and surfactant polyvinyl alcohol (PVA) at different temperatures and with different aging times. By heating synthesized $CdCO_3$ at different temperatures 400 °C and 700 °C, the CdO nanocrystals are obtained. Debye-Scherrer's formula is used to calculate the particle size and the calculated value is 22.87nm. Band gap energy has been calculated for the prepared $CdCO_3$ and CdO from the UV-Visible spectrum. The band gap value for CdO obtained as 0.0766 eV. While CdO converted into nano-sized particles, the free movements of asymmetric and symmetric stretching are blocked which behavior is observed from FT-IR spectrum. TEM images give the clear morphological appearance of CdO nanoparticles. The present successive simple process can be easily implement to prepare CdO nanoparticles with the low cost of investment in a short time period.

1. Introduction

The cadmium oxide (CdO), is one of the well-known important n-type semiconductor with a direct band gap of 2.5 eV and an indirect band gap of 1.98 eV [1], has promising applications in catalysts, sensors [2], nonlinear materials [3], solar cells [4], and other optoelectronic devices [5-10] etc. 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. Recently, hollow nanostructures of inorganic materials have attracted great research attention because they exhibit a lower density, higher surface area, and distinct optical property, and in most cases, have improved performances for applications in photonic crystals, fillers, vehicle systems, catalysis, sensing devices [11-16].

Many of new synthetic routes for the preparation of ultrafine and nanosized metal oxides to obtain defined properties are under constant investigation and some of them include sol–gel, solvothermal method, metal oxide chemical vapour deposition, coprecipitation, microemulsion, by hydrolysis in polyol medium and decomposition of the precipitates prepared from non-aqueous precipitation routes [17, 18]. The microwave-assisted route is yet another method for the synthesis of metal oxides and has been gaining significance in the synthesis of oxide nanomaterials [19].

The objective of the present work is to synthesize CdO by microwave-assisted method. The particle size and crystalline phase of the catalysts were determined by powder X-ray diffraction (XRD). The TEM technique was used to confirm the formation of single phase material with nanocrystalline particles. The functional group analysis and its band gap properties are evaluated using FT-IR and UV spectrophotometer.

2. Experimental Methods

Two step mechanisms applied for the synthesis of $CdCO_3$ and CdO nanoparticles by precipitation method. In the first step, $CdCO_3$ was prepared followed by the second step of the formation of CdO by decomposition. At stage 1, the respective amount of cadmium acetate was dissolved in 20 mL of methanol in a 100 mL beaker and it was kept over the magnetic stirrer. Now respective molar concentration of TMAH solution was added slowly by drop wise, the total solution mixture is allowed to keep in a constant stirring and then 2 g of polyvinyl alcohol was added as a capping agent and stirring continued again for few minutes. Then this mixture was exposed to microwave using domestic microwave oven (frequency 2.45 GHz) for 30 minutes. After microwave treatment, the precipitate was filtered and washed thoroughly with dilute methanol and dried in air oven at 120°C for an hour. Now the formed product $CdCO_3$ is said to be as a precursor and it leads to the formation of the aimed product CdO. To form the CdO powders the obtained precipitate was calcinated in the presence of air at 400 °C and 700 °C under muffle furnace for two hours. The scheme of the synthesis represented as follows,

$$Cd(OOCCH_3)_2 \xrightarrow[PVA]{TMAH} \text{Nano } CdCO_3 \xrightarrow[400\text{-}700^{o}C]{\Delta} CdO \text{ (Nanosize)}$$

2.1 Instrumental Characterizations

For determination of crystallite size, Scherrer analysis of XRD is commonly used. XRD measurements are performed using a Philips diffractometer of 'X'pert company with mono chromatized Cu $K_0\alpha$ (λ=1.54060Å) radiation. Particle size is determined from FWHM (full width half maxima) of XRD peaks using Scherrer's formula,

$$D = \frac{0.89\lambda}{\beta \cos\theta}$$

Fourier transform infrared spectra (FT-IR) were recorded using Nicolet 6700, Thermo Electronic Corporation, USA made spectrophotometer. A double beam UV-Vis (Jascow-500) spectrophotometer with 1 mm optical path length quart cells was used for all absorbance measurement in the range of 200 nm – 400 nm. The tunneling electron microscopy (TEM) analysis performed using a Philips CM-200 electron microscope with operating voltages 200 KV, resolution 2.4 Å.

3. Results and Discussion

The nano particles CdO and $CdCO_3$ are synthesized and characterized by XRD, FTIR, UV and SEM spectral studies. The important parameters, viz. FWHM, particle size, structure of compounds, the value of energy gap, presence of the compounds and impurities are discussed.

Regarding the nanoparticle size determination, the broadening nature of the XRD peaks itself will be a good substantiation for the nanosize of the particle, the higher the 'β' value and lower the 'θ' value, the lower will be the size of the nanoparticles. Therefore the broadening nature and the position of the peaks furnishes are outline idea about the nanosize of the particle. The XRD pattern images Fig. 1 and Fig. 2 show the characteristic

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peaks of $CdCO_3$ and CdO crystalline structure. The rhombohedral crystalline nature of $CdCO_3$ is matched with the JCPDS file 85-0989 with plane of (110), (211), (110), (200), (332), (211), (310) and face centred cubic symmetry structure of CdO with the plane of (111), (200), (220) has been matched with the JCPDS file 65-2908. Debye-Scherrer's formula is used to calculate the particle size, and the calculated value is obtained as 22.87 nm.

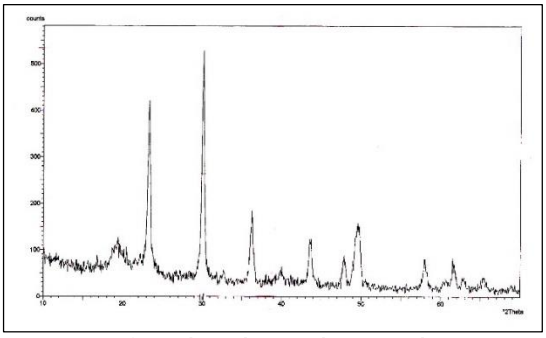

Fig. 1 XRD pattern for synthesised intermediate nano $CdCO_3$

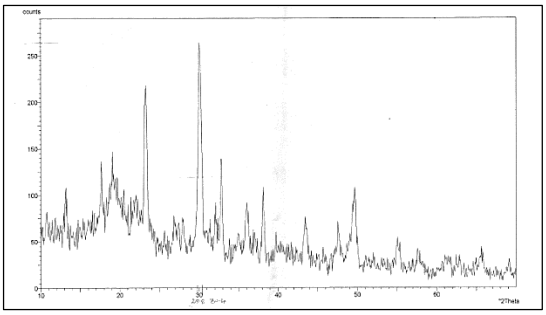

Fig. 2 XRD obervation for synthesised nano CdO particles

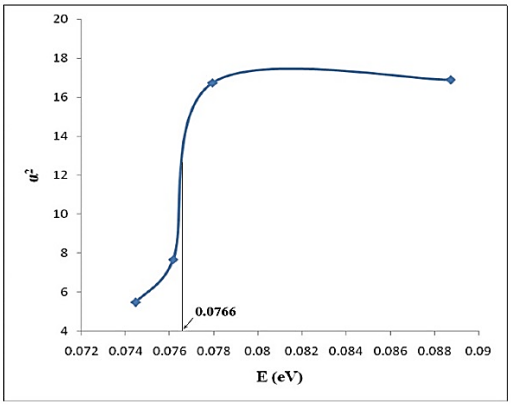

Fig. 3 A plot of α^2 versus E for band energy calculation

The energy band gap is measured with the help of absorption spectra and a graph of α^2 versus hv is plotted. The extrapolation of the straight line to $\alpha^2=0$ gives the value of the energy band gap of prepared materials. The UV-Visible spectra are recorded for all the nano powder samples prepared by precipitation method. Cadmium oxide is an n-type degenerate semiconductor with high electrical conductivity. Due to its large linear refractive index (n_0=2.49) it is a promising applicant for optoelectronic applications and other applications including solar cells, photo transistors, photodiodes, transparent electrodes and gas sensors. Because of possibilities of these interesting applications, there have been some efforts to calculate the band cap energy for the prepared $CdCO_3$ and CdO from the UV-Visible spectrum, which is obtained by plotting of E (eV) verses α^2 as shown in Fig. 3. The band gap value for CdO found to be as 0.0766 eV. If the particle size decreases, the corresponding CdO band gap will be reduced and it will enhance its own semi conductive property [8, 20]. Therefore it is most evident support for the confirmation of CdO nanoparticle formation.

The FT-IR spectra was taken by using Nicolet 6700 model to analyses the functional groups present in the nanomaterials with 400 cm^{-1} to 1500 cm^{-1}. In the case of FT-IR spectrum two most intense peaks and many minor are obtained for both pure $CdCO_3$ and synthesized nano particles of $CdCO_3$. For pure $CdCO_3$, the peaks obtained at 1396 cm^{-1} and 3526 cm^{-1} (major peaks), 1440 cm^{-1}, 1518 cm^{-1}, 1747 cm^{-1}, 2306 cm^{-1}, 2378 cm^{-1} and 2924 cm^{-1} (minor peaks) for the asymmetrical stretching and vibrational stretching respectively. While this compound converted into nano-sized particles, the free movements of asymmetric and vibrational stretchings are blocked. These are indicated by missing of peaks at 3525 cm^{-1} and other minor peaks in the FT-IR spectrum of synthesized $CdCO_3$ nanoparticles in from pure components. From Fig. 4, the same trend obtained for CdO nanoparticles where the peaks misplacements and decrease in intensity of peak obtained in the ranges of 3421-3464 cm^{-1}, 2926 cm^{-1}, 2308 cm^{-1}, 1745 cm^{-1} and1122 cm^{-1}. These all data from the FT-IR spectrum gives an additional support to the formation of nano particles of $CdCO_3$ and CdO. Fig. 5 shows the TEM images of CdO and it clearly indicates the uniform cubic structure of synthesized cadmium oxide nanoparticles.

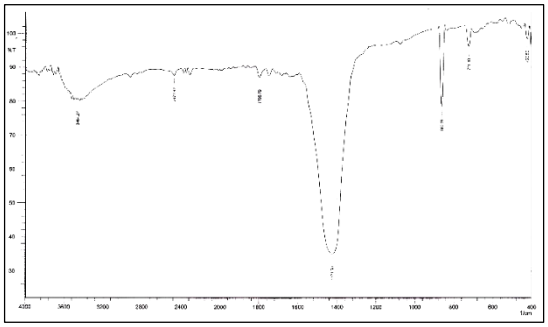

Fig. 4 FT-IR spectrum of synthesized CdO nanoparticles

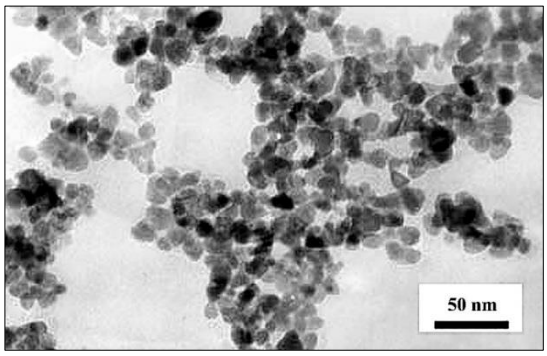

Fig. 5 TEM images of synthesized CdO nanoparticles

4. Conclusion

The present study indicates that microwave assisted precipitation method can be successively employed for the preparation of nano $CdCO_3$ as well as CdO nanocrystals. Particle size of CdO is found to be 22.87 nm by using Debye-Scherrer's formula and band gap value as 0.077 ev. The spectral characterizations and microscope studies clearly point out the cubic crystalline nature of CdO. Thus from the above successive reports, this method can easily implement to prepare CdO nanoparticles with the present simple procedure and expected to be applicable in the fabrication of other nanosized particle.

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