

## Effects of 90 MeV carbon ion irradiation on cadmium oxide quantum dots

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**Cadmium oxide (CdO) quantum dots were synthesized in the laboratory by quenching method using CdO powder sintered at 900°C and ethyl alcohol kept at ice-cold temperature. X-ray diffraction studies revealed the NaCl cubic structure of CdO quantum dots. The CdO crystallite size was estimated to be 2.7 nm. Heavy ion irradiation using 90 MeV carbon ( $C^{+6}$ ) ion beam accelerated at 15 UD Pelletron produced enlargement in the size of quantum dots revealed by transmission electron microscopic studies.**

**Keywords:** Cadmium oxide, carbon ion irradiation, quenching, quantum dots.

FABRICATION of quantum dots has emerged as an important area of research in the field of nanotechnology during recent years<sup>1-6</sup>. Cadmium oxide (CdO) is an *n*-type semiconductor which finds applications in photodiodes, photovoltaics, liquid crystal displays, IR detectors, electrodes of storage batteries, phosphors, pigments and ceramic glazes<sup>7</sup>. A major use of CdO is as an ingredient for electroplating baths and in pigments<sup>8</sup>. A number of synthetic routes have been used to synthesize CdO nanoparticles and nanowires<sup>9,10</sup>. We employed a simple technique of sintering CdO powder and quenching it in ethyl alcohol to prepare quantum dots. A thin film of CdO quantum dots was prepared by a simple technique of irradiation using 90 MeV carbon ion beam ( $C^{+6}$ ) available at the Inter University Accelerator Centre (IUAC), New Delhi.

The purpose of this study is to investigate the effect of carbon ion beam on quantum dots of CdO, in view of the novel effects of heavy ion beams on ZnO quantum dots reported by Nath *et al.*<sup>11</sup> and shape transformation (elongation effects) along the beam direction observed in embedded nanoparticles of platinum<sup>12</sup> induced by swift heavy ion (SHI) irradiation.

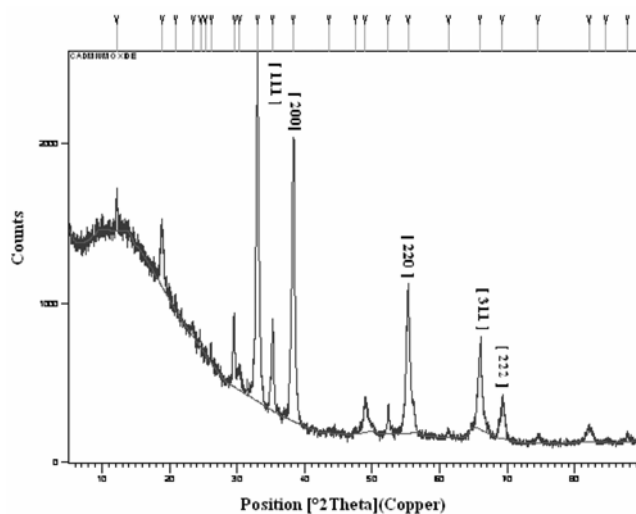
Synthesis of cadmium quantum dots was accomplished by sintering 4 g of CdO powder (purity 99.5%, CDH-make) at 900°C in a muffle furnace for 5 h and then quenching into 7 wt% aqueous ethyl alcohol kept at ice-cold temperature, followed by moderate stirring. The resulting solution contains CdO quantum dots. One part of this solution was used in the preparation of thin films of quantum dots on glass slides. This was done by placing a few drops of the CdO quantum dots solution on a

clean glass slide and stretching it over by another glass slide to cast a very fine film in the micron range. The glass slides were cut into pieces,  $1 \times 1 \text{ cm}^2$  each, for the purpose of irradiation. One of the slides was kept intact as virgin sample for the sake of comparison.

Irradiation experiment was carried out in the material sciences vacuum chamber under high vacuum ( $10^{-6}$  Torr) by mounting the glass-slide specimens on a ladder having four rectangular faces. The fluence of the 90 MeV carbon ion beam ( $C^{+6}$ ), available from 15 UD Tandem Pelletron at IUAC, was varied from  $10^{11}$  to  $10^{13}$  ions/cm<sup>2</sup> in steps. In order to expose the whole target area, the beam was scanned in the *x-y* plane over the mounted sample. The ion beam energy and the thickness of the target film were chosen in such a way that the electronic energy loss of the carbon beam plays the predominant role in creating modifications in the target. SRIM code calculations showed that the 90 MeV carbon ion beam can easily traverse through the thin film of quantum dots solution<sup>13</sup>.

X-ray diffraction (XRD) studies were carried out at the Sophisticated Analytical Instruments Facility (SAIF) set up by Punjab University, Chandigarh using X'Pert PRO (PANalytical, The Netherlands) using Cu-K $\alpha$  radiation. High Resolution Transmission Electron Microscope (HRTEM, Hitachi H 7500) at SAIF was used for revealing morphology and measurement of quantum dot diameters. The samples for TEM analysis were prepared by ultrasonication of CdO quantum dots in ethanol prior to loading on carbon-coated copper grids under ultrahigh vacuum.

XRD spectrum of CdO nanoparticles using a Cu-K $\alpha$  radiation source of  $\lambda = 1.5406 \text{ \AA}$ , is shown in Figure 1. The spectrum shows some prominent peaks at  $2\theta = 32.99^\circ$ ,  $38.29^\circ$ ,  $55.27^\circ$ ,  $65.94^\circ$  and  $69.26^\circ$ . These peaks correspond to Miller indices (111), (200), (220), (311) and (222) respectively. All the prominent peaks in XRD spectrum



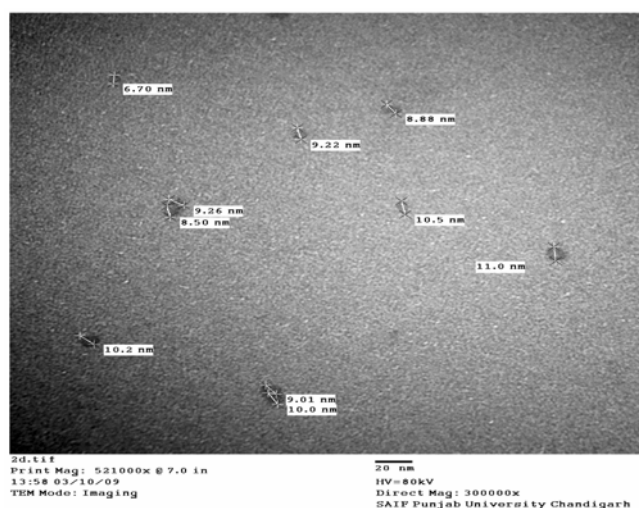
**Figure 1.** X-ray diffraction spectrum of cadmium oxide (CdO) nanocrystals showing some prominent peaks.

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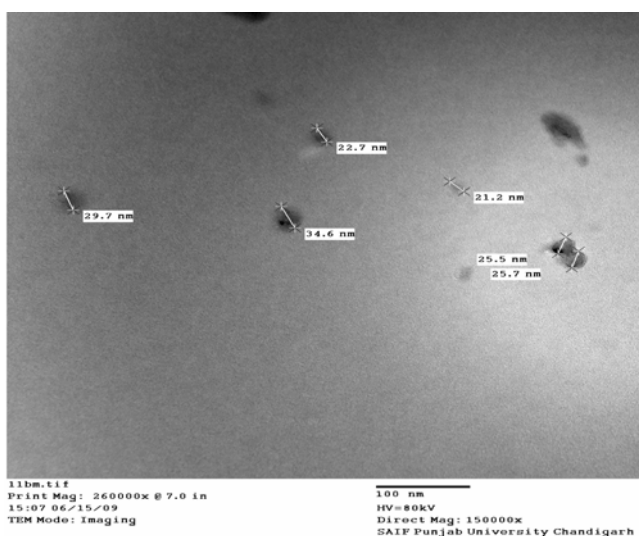
can be indexed<sup>14</sup> to standard ICSD card no. 029291. The most prominent peak at  $2\theta = 32.99^\circ$  corresponds to *hkl* (111) with cell constant  $a = 0.4569$  nm and  $d = 0.2714$  nm. It clearly shows that CdO nanoparticles crystallize in rocksalt or NaCl cubic structure.

The average size  $D$  of the CdO crystallites is calculated using the Debye Scherrer's formula<sup>15</sup>:  $D = 0.9\lambda/\beta \cos\theta$ , where  $\lambda = 1.5406$  Å is the wavelength of the X-ray radiation used,  $\beta$  the full width at half maximum of the diffraction peak (0.0612),  $K$  the shape factor is assumed to be 0.9 and  $\theta$  is the Bragg diffraction angle of the most prominent XRD peak. Substituting appropriate values in the formula, the CdO crystallite size was calculated to be 2.7 nm.

During our preliminary studies, some interesting results have been obtained on irradiation of CdO quantum



**Figure 2.** TEM micrograph showing variable size of CdO quantum dots in virgin sample.



**Figure 3.** TEM micrograph showing enlargement in the size of CdO quantum dots irradiated at a fluence of  $1 \times 10^{11}$  ions/cm<sup>2</sup>.

dots using 90 MeV carbon ion beam. The mean diameter of CdO quantum dots shown in the TEM micrograph (Figure 2) of virgin (unirradiated) sample was 9.33 nm. The irradiation effect produced an agglomeration of quantum dots due to intense heating by the carbon ion beam, resulting in the enhancement of the quantum dot size. There was no linear relationship between the mean diameter and the fluence of carbon ion beam used in the experiment. The CdO sample irradiated using minimum fluence of  $10^{11}$  ions/cm<sup>2</sup> displayed a mean diameter of 25.6 nm (Figure 3), an enhancement nearly three times that of the mean diameter of its virgin sample. The mean diameter for the CdO irradiated with a fluence of  $10^{12}$  ions/cm<sup>2</sup> was determined to be 32.5 nm; whereas the one irradiated at a maximum dose ( $10^{13}$  ions/cm<sup>2</sup>) had a mean diameter of 32 nm. This shows that the diameter attains a plateau value after irradiation using  $10^{12}$  ions/cm<sup>2</sup>. Mean diameter was determined by counting on the average 20–25 quantum dots in several grids. There was a random distribution in the size and shape of the quantum dots in the virgin and irradiated samples. The statistical counting error ( $1\sigma$ ) in diameter measurement was of the order of 20%.

Ion beams interact with matter in various ways, but the predominant loss of energy is through electronic stopping power  $(dE/dx)_e$ . Using SRIM-2008.04 code<sup>16</sup>, electronic and nuclear stopping powers for the 90 MeV carbon ion beam have been estimated to be 8.818 and 0.475 keV/ $\mu$ m respectively. The range of the 90 MeV carbon ion beam was estimated to be 66.37  $\mu$ m. Hence, the energy deposited in the CdO quantum dots thin film was predominantly of electronic origin.

Heavy ion irradiation effects have been studied on ZnO quantum dots using 100 MeV  $\text{Cl}^{+9}$  ion beam, with fluence varying from  $10^{11}$  to  $10^{13}$  ions/cm<sup>2</sup>, as in our experiment. Enhancement in the size of the quantum dots with ion beam fluence has been reported<sup>11</sup>. SHI irradiation<sup>17–20</sup> of metallic nanocrystals embedded in  $\text{SiO}_2$  has been carried out for Co, Au, Sn and Pt. Several models have been developed to explain the complex interaction between SHI and matter. The thermal spike model<sup>21</sup>, which predicts the formation of a molten track around the ion path, is one plausible explanation for shape change of metallic nanocrystals. However, further studies are required to find a proper explanation of the observed phenomenon.

The CdO quantum dots (nanoparticles) represent NaCl cubic structure with crystal growth along the (111) plane. The crystallite size was determined to be 2.7 nm. The range of the 90 MeV carbon ion beam was estimated to be 66.37  $\mu$ m. Carbon ion beam irradiation resulted in the enhancement of mean diameter of CdO quantum dots, almost by a factor of three compared to its virgin sample.

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## A simple method for estimation of sulphur in nanoformulations by UV spectrophotometry

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**We have recently reported the development of nano-sulphur with remarkable fungicidal properties, much better than the conventional sulphur. The nanosulphur could substitute the toxic conventional synthetic fungicides. The lack of estimation protocols for active ingredient in nanoformulations and exploitation of nanosulphur as a new technology for fungal management necessitates the need for estimation protocols to ensure the quality and quantity of sulphur in nanoformulations. Therefore, a method was developed for the estimation of sulphur in its nanoformulations using UV–visible spectrophotometry. Thirty-four samples of nanosulphur were analysed to validate the method. The percentage estimation of the active ingredient in all the samples was above 80.**

**Keywords:** Nanoformulations, polyethylene glycol, synthetic fungicides, UV spectrophotometry.

THE application of synthetic pesticides has caused threat to non-target organisms and the environment due to their overuse<sup>1</sup>. Since the release of xenobiotic results in the increase of environmental risk, the goal should be to use such compounds carefully so that they cause least negative impact on the environment into which they are released<sup>1</sup>. To remove harmful effects on the non-target organisms, encapsulation of the active ingredient with other materials such as a polymer can allow sensitive ingredients to be physically enveloped into a protective matrix in order to protect core materials from adverse reactions due to factors like air or light<sup>2</sup>.

Sulphur is a widely used element in different applications such as fertilizer, pharmaceutical, anti-microbial agent, insecticide, fungicide, high-density charger in lithium ion battery as well as for rubber and fibre industries<sup>3,4</sup>. Various methods were reported<sup>5</sup> for the preparation of nano elemental sulphur (nano-S<sup>0</sup>). Most of these

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