CHAPTER 4

Characterization of Material

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Quantum dots of metal oxide semiconductors exhibit many interesting electronic, optoelectronic and optical properties [1-29]. These properties become more interesting when the size of the quantum dots becomes very small (less than 1000 nm). To explore these properties of our synthesized metal oxide semiconductors quantum dots, the following characterizations have been carried out, which is my contribution to this chapter.

(i) UV-Visible Spectroscopy

(ii) X-ray diffraction study (XRD)

(iii) High Resolution Transmission Electron Microscopy (HRTEM)

In this chapter, the characterizations of the samples by the above mentioned techniques have been explained. The average crystallite size of the quantum dots is estimated by XRD, and HRTEM studies. The variation of band gap that occurs due to variation of size of the quantum dots has been explored by optical absorption spectroscopy.

4.1. UV- Visible spectroscopy

Ultraviolet-visible (UV-VIS) spectroscopy involves the spectroscopy of photons in the UV-visible region. It uses light in the visible and adjacent near ultraviolet (UV) and near infrared (NIR) ranges. In this region of the electromagnetic spectrum,

molecules undergo electronic transitions. This technique is complementary to fluorescence spectroscopy. Fluorescence deals with transitions from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state.

The instrument used in ultraviolet-visible spectroscopy is called a UV-VIS spectrophotometer. It measures the intensity (I) of light passing through a sample and compares it to the intensity (I_o) of light before it passes through the sample The ratio I / I_o is called the transmittance, and is usually expressed as a percentage (%T). The absorbance, *A*, is based on the transmittance as

$$A = -\log (\%T) \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (4.1)$$

Optical absorbance (A) is an important way to explore the different energy states in semiconductor material. This study [10-12] is based on the fact that, if two possible energy states E_1 and E_2 exist in a system as shown in Figure 4.1, then electronic transition from level E_1 to E_2 can takes place when appropriate energy $E_2 - E_1 = \Delta E$ is absorbed and the frequency of radiation has the simple form of

$$\nu = \frac{\Delta E}{h} Hz$$

or, $\Delta E = h\nu$ *joules*. ……(4.2)

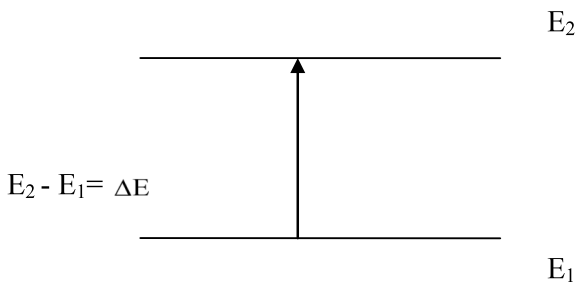

Fig.4.1. Schematic diagram of Electronic excitation between two energy states

That is, absorption wavelength or edge can be written as

$$\lambda_a = \frac{hc}{\Delta E} \quad \text{.....(4.3)}$$

Where 'λ_a' is the absorption edge, 'h' is the Planck's constant and 'c' is the velocity of light.

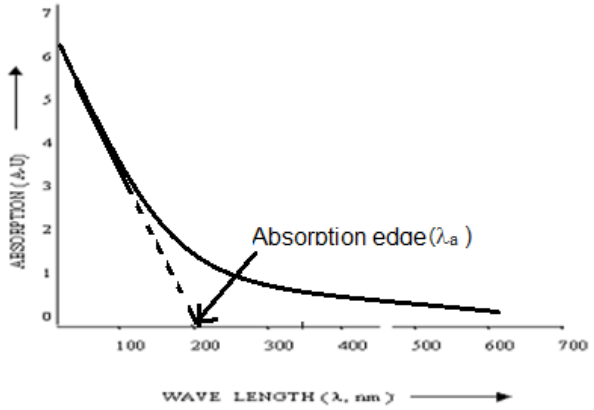

Fig. 4.2. Schematic of UV / Visible absorption spectra

4.2 X-ray diffraction study (XRD)

X-ray diffraction study is an important tool for finding different crystal parameters like crystallite size, d-spacing, diffraction planes, structure, phase, and lattice constants, etc. The intensities and the angles of diffracted X-ray beams are related to atomic arrangement of the crystal.

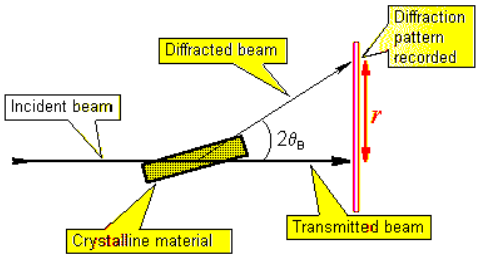

Fig.4.3. X-ray diffraction by sample.

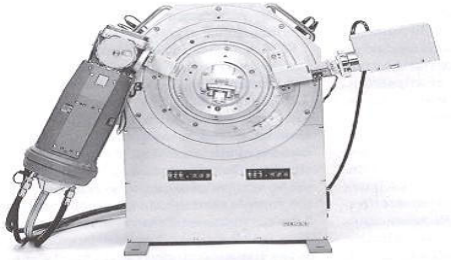

Fig.4.4. A typical X-ray diffractometer.

In this study, X-ray diffractometer detects the X-ray, diffracted by crystal and gives the diffractogram, which is a plot between intensity and diffraction angle. Intensity is given in terms of arbitrary units (A.U) and the angle is in degrees. Figure 4.5 shows a schematic of pattern of such X-ray diffractogram.

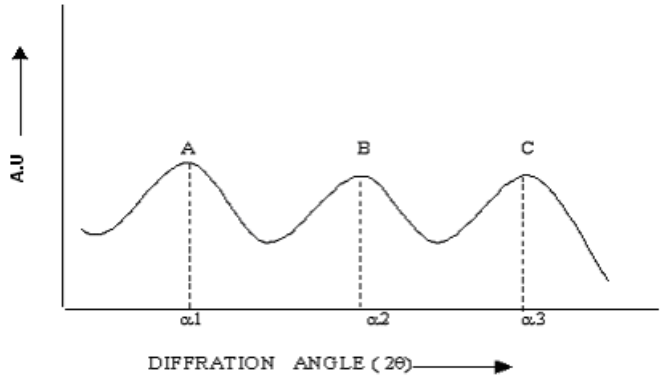

Fig 4.5. Schematic of XRD pattern.

Next we explain how X-ray diffractogram is used in finding various crystal parameters of the quantum dots, prepared in our laboratory.

4.2.1. Sample identification

To identify any specimen, the diffraction angles produced by a particular sample are compared with the standard diffraction angles of the same material. A good match between experimental and standard values of diffraction angles is required to identify the specimen. The most commonly used database for the identification of crystal structures is ICDD (International Center Diffraction Data) system.

4.2.2. Crystallite (grain) size estimations

A crystallite is a single crystal in a polycrystalline aggregate. X-ray diffractogram is extensively used to calculate the average crystallite size (D) using Debye-Scherrer equation [9]. The equation is written as

$$D = \frac{0.9\lambda}{W Cos\theta} \quad \text{.....(4.4)}$$

Where λ is the wavelength of X-ray used, W is the corrected full width at half maximum (FWHM) for instrumental broadening, θ is the glancing angle in radians. The different parameters used in equation (4.4) can be obtained from X-ray diffractogram, in the following way

I. Full Width at Half Maximum (FWHM)

In the example shown in Figure 4.6, the maximum height of the XRD peak is BC and OB is the noise. Half of the maximum height is either AB or AC. That is, the middle point of the peak is A. Two more points P and Q are taken on the plot collinear to point A. Two perpendiculars from points P and Q are drawn on the abscissa, which is cut by the perpendicular lines at point R and S. The segment RS is the full width at half maximum (FWHM) in degrees. Finally, this is converted into radians, which is the required FWHM (W).

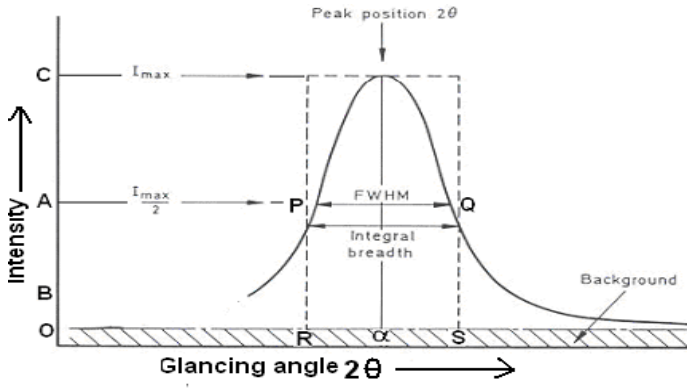

Fig.4.6. Schematic of Single XRD peak.

II. Diffraction angle or glancing angle (θ) and X-ray wavelength

In the diffractogram, the angle axis is calibrated in terms of "2θ / degree". From this, glancing angle "θ" can be obtained. The X-ray wavelength "λ" used in the X-ray diffractometer is 1.541 Angstrom.

In practice, the experimental data obtained from XRD study of any specific specimen may slightly differ from the standard values due to the following reasons.

1. Calibration error
2. Instrumental error
3. Sudden power fluctuation during experiment
4. External noises.

The X-ray diffraction (XRD) analyses of quantum dot samples are carried out using a PAN analytical X'Pert Pro X-ray Diffractometer with Cu K_α radiation as an X-ray source at 40 kV and 30 mA in a scanning angle (2θ) ranging from 20^0 to 70^0 with scan speed $0.001^0 s^{-1}$. Various XRD data have been calculated using X'Pert High Score software.

Using ICDD (International Center Diffraction Data) sample parameters (Size, lattice constants, crystal planes etc.) are obtained very easily now a days.

4.3 High Resolution Transmission Electron Microscopy (HRTEM):

HRTEM has openes a new door to nano-science research by providing pictorial view of them with very high magnification of 8 lacks or more. This study, gives the information about size, shape and surface morphology of any specimen. High

Resolution Transmission electron microscopy (HRTEM) is a microscopy technique whereby a beam of electrons is transmitted through the specimen. An image is formed from the electrons, transmitted through the specimen which is magnified and focused by an objective lens and appears on an imaging screen, a fluorescent screen is used in most HRTEMs, and also the image is recorded by a sensor such as a CCD camera.

Fig.4.7. Photographs of high resolution transmission electron microscope.

4.4. Conductivity Test

To test the conductivity of the samples, weather p-type or n-type, a simple method called 'thermoelectric effect' have been used [16]. A measurement of the sign of the voltage across a semiconductor specimen, one end of which is heated is a rough and ready way to tell if the specimen is n-type or p-type. For p-type semiconductor, the sign is positive (+ve), and negative (-ve) for n-type semiconductor. In this work, the conductivity of all the prepared samples (ZnO, SnO_2 and Fe_2O_3) are tested by the

method and are found to possess n-type conductivity. The results are verified by "Hall effect" also.

4.5. Characterization of samples

In the next consecutive sections, the characterizations of our prepared semiconductor metal oxide quantum dots are discussed in details.

4.5.1. Zinc oxide (ZnO) quantum dots embedded in PVA (Polyvinyl Alcohol) matrix

ZnO specimen [29] has been characterized by UV/VIS optical absorption spectroscopy (Perkin Elmer Lamda 35 1.24), X-ray diffraction study (Bruker AXS, X-ray source: Cu K_α) and high resolution transmission electron microscopy (HRTEM) (JEM 2100, Jeol).

I. Optical absorption spectroscopy

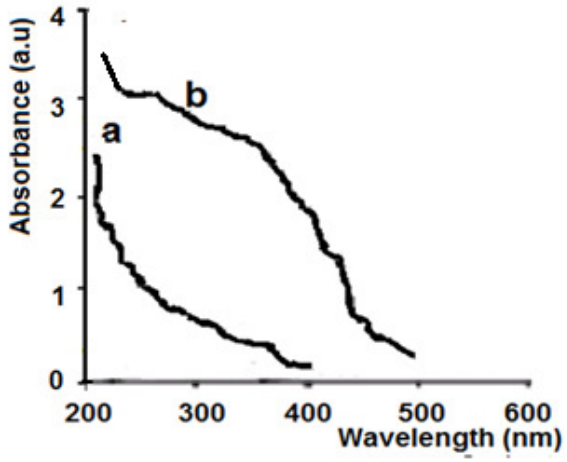

Fig. 4.8. Optical spectra of ZnO a: quantum dot, b: bulk

In the optical absorbance spectra shown in Figure 4.8, a" denotes the spectrum of ZnO quantum dot while "b" denotes that of bulk. It is seen that the band edge absorption in quantum dot is strongly blue shifted at 215 nm with respect to bulk specimen which possesses the absorption edge at around 390 nm. For estimating particle size from blue shift, a theoretical model [7,8] has been proposed, known as hyperbolic band model that is written as

$$R = \sqrt{\frac{2\pi^2 h^2 E_{gb}}{m^*(E_{gn}^2 - E_{gb}^2)}} \quad \ldots\ldots\ldots (4.5)$$

Where, R is the quantum dot radius, E_{gb} is the bulk band gap, E_{gn} is the quantum dot band gap, h is Planck's constant, m* is the effective mass of the specimen. The bulk band gaps at room temperature and the effective mass [6.7] of ZnO is listed in the Table 4.1.

Name of the samples	Bulk Band gap	Values of effective mass	Structure
ZnO	3.20 eV	2.45×10^{-31} Kg	wurzite

Table 4.1. Bulk band gap and effective masses of ZnO

The data obtained from the analysis are put in Table 4.2.

Absorption edge in quantum dot	Energy gap in quantum dot	Bulk absorption edge	Bulk band gap	Enhancement in band gap due to size quantization	Quantum dot size
215 nm	5.2 eV	390 nm	3.20 eV	2 eV	12 nm

Table.4.2. Data of ZnO/PVA specimen revealed from optical absorption spectroscopy.

II. X-ray diffraction study

From X-ray diffraction study (Figure 4.9) average particle size (crystallite size) is calculated by using Debye-Scherrer formula [9] $D = \frac{0.9\lambda}{WCos\theta}$, 'λ' is wave length of X-ray (0.1541 nm), 'W' is FWHM (full width at half maxima), 'θ' (theta) is the glancing angle and 'D' is particle diameter (crystallite size).Considering all the peaks[2] (2θ in degree) in the X-ray diffractogram , the average crystallite (quantum dot) size has been assessed and found to be 11 nm. Further, by analyzing the X-ray diffractogram with the help of ICDD (International Center Diffraction Data) it has been revealed that ZnO quantum dots are wurzite in structure.

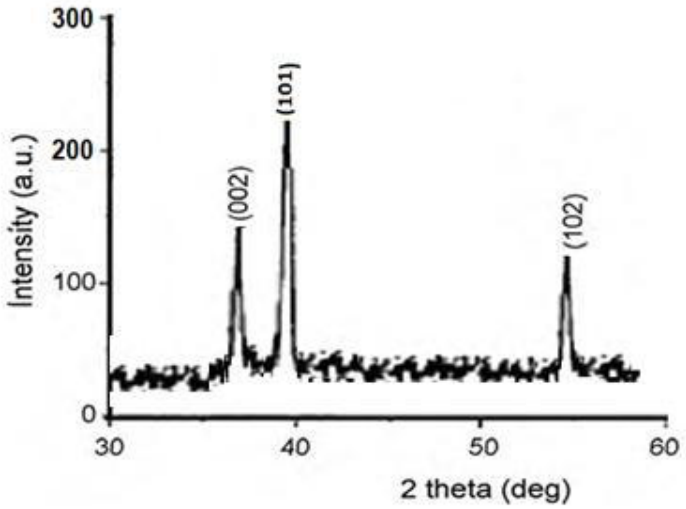

Fig 4.9: XRD Spectra of ZnO Quantum dots embedded in PVA

III. High Resolution Transmission Electron Microscopy (HRTEM)

HRTEM photograph of ZnO quantum dots[29] is shown in Figure 4.10 which provides the surface morphological view of the particles. The sample size and shapes are put in the Table 4.3.

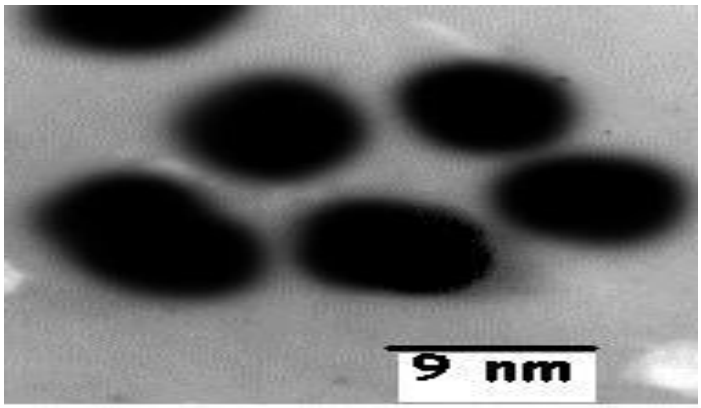

Fig.4.10. HRTEM Photographs of ZnO

Average particle size	Particle Shape
11 nm	Elliptical

Table. 4.3. Data of ZnO/PVA sample obtained from HRTEM study

All these characterizations infer that sizes of ZnO quantum dots are within 12 nm.

4.5.2 Zinc oxide (ZnO) quantum dots embedded in PVP (Polyvinyl Pyrrolidone) matrix

ZnO specimen[28] has been characterized by UV/VIS optical absorption spectroscopy (Perkin Elmer Lamda 35 1.24), X-ray diffraction study (Bruker AXS, X-ray source: Cu K_α) and high resolution transmission electron microscopy (HRTEM) (JEM 2100, Jeol).

II. Optical absorption spectroscopy

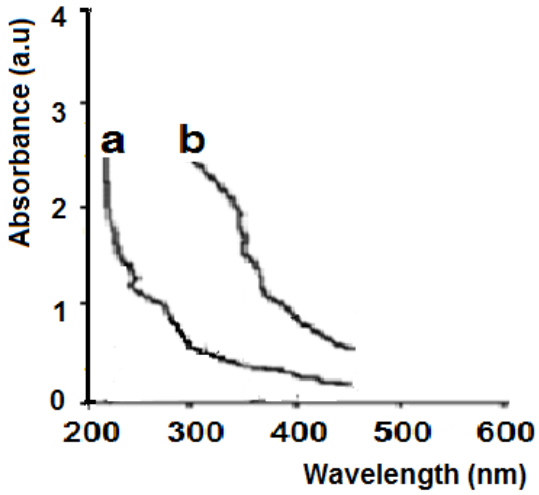

Fig. 4.11. Optical spectra of ZnO embedded on PVP a: quantum dot, b: bulk

In the optical absorbance spectra shown in Figure 4.11, "a" denotes the spectrum of ZnO quantum dot[28] while "b" denotes that of bulk. It is seen that the band edge absorption in quantum dot is strongly blue shifted at 210 nm with respect to bulk specimen which possesses the absorption edge at around 390 nm. For estimating particle size from blue shift, a theoretical mode [7,8] has been proposed, known as hyperbolic band model that is written as

$$R = \sqrt{\frac{2\pi^2 h^2 E_{gb}}{m^*(E_{gn}^2 - E_{gb}^2)}} \quad \ldots\ldots\ldots (4.6)$$

Where, R is the quantum dot radius, E_{gb} is the bulk band gap, E_{gn} is the quantum dot band gap, h is planck's constant, m* is the effective mass of the specimen. The bulk band gaps at room temperature and the effective mass [6,7] of ZnO is listed in the Table 4.4.

Name of the samples	Bulk Band gap	Values of effective mass	Structure
ZnO	3.20 eV	2.45 x 10^{-31} Kg	wurzite

Table 4.4. Bulk band gap and effective masses of ZnO

The data obtained from the analysis are put in Table 4.5.

Absorption edge in quantum dot	Energy gap in quantum dot	Bulk absorption edge	Bulk band gap	Enhancement in band gap due to size quantization	Quantum dot size
210 nm	5.3 eV	390 nm	3.20 eV	2.10 eV	10 nm

Table.4.5. Data of ZnO/PVP specimen revealed from optical absorption spectroscopy.

II. X-ray diffraction study

From X-ray diffraction study (Figure 4.12) average particle size (crystallite size) is calculated by using Debye-Scherrer formula[9] $D = \frac{0.9\lambda}{WCos\theta}$, 'λ' is wave length of X-ray (0.1541 nm), 'W' is FWHM (full width at half maxima), 'θ' (theta) is the glancing angle and 'D' is particle diameter (crystallite size).Considering all the peaks[2] (2θ in degree) in the X-ray diffractogram, the average crystallite (quantum dot) size has been assessed and found to be 9 nm. Further, by analyzing the X-ray diffractogram with the help of ICDD (International Center Diffraction Data) it has been revealed that ZnO quantum dots are "wurzite" in structure.

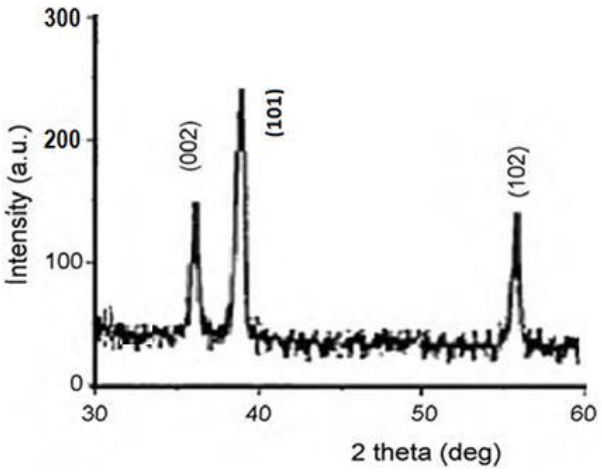

Fig 4.12: XRD Spectra of ZnO embedded in PVP

III. High Resolution Transmission Electron Microscopy (HRTEM):

HRTEM images of PVP film (4.13.i.a) and ZnO quantum dots (4.13.i.b) are shown in Figure 4.13. It is evident in the HRTEM image (4.13.i.b) that ZnO crystallites (quantum dots) are circular in shape with sizes within 10 nm. Fig (4.13.ii) shows the Scanning Electron Microscope (SEM) image of ZnO quantum dot surface [28]. ZnO sample sizes assessed from these three studies are well matching, which is a distinct advantage over earlier reports [2,3] . All these characterizations infer that sizes of ZnO quantum dots (diameter) are within 10 nm.

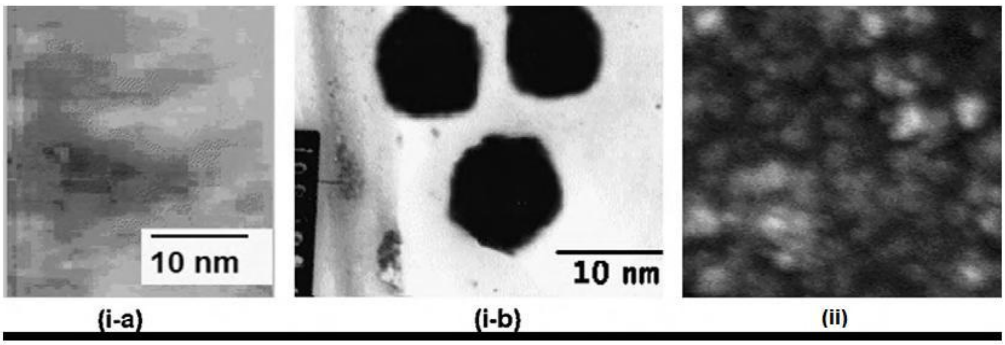

Fig4.13: (i-a) HRTEM images of PVP matrix. (i-b) ZnO quantum dots embedded in PVP. (ii) Image Of ZnO quantum dot surface

4.5.3 Tin oxide (SnO_2) quantum dots embedded in PVP matrix

SnO_2 specimen[22] has been characterized by UV/VIS optical absorption spectroscopy (Perkin Elmer Lamda 35 1.24), X-ray diffraction study (Bruker AXS, X-ray source: Cu K_α) and high resolution transmission electron microscopy (HRTEM) (JEM 2100, Jeol).

I. Optical absorption spectroscopy

In the optical absorbance spectra shown in Figure 4.14, denotes the spectrum of SnO_2 quantum dot (prepared sample) [22]. It is seen that the band edge absorption in quantum dot is strongly blue shifted at 230 nm with respect to bulk specimen which possesses the absorption edge at around 340 nm .Blue shift is a distinct signature of quantum dot formation [1,2,4] . For estimating particle size from blue shift, a theoretical model [6,7] has been proposed, known as hyperbolic band model[5] that is written as

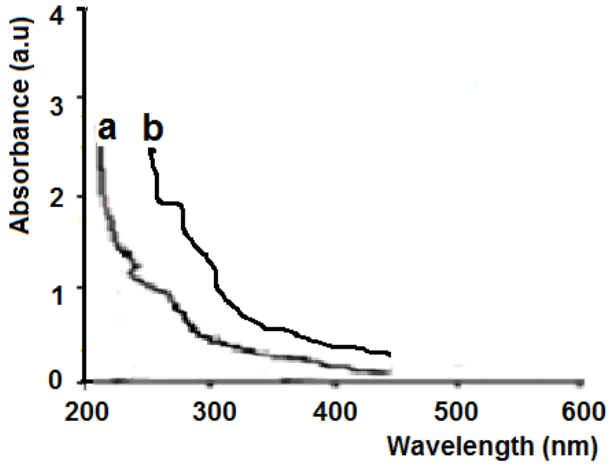

Fig. 4.14: UV/VIS absorption spectra of SnO_2 a. quantum dots embedded on PVP

$$R = \sqrt{\frac{2\pi^2 h^2 E_{gb}}{m^*(E_{gn}^2 - E_{gb}^2)}} \quad \text{……..(4.7)}$$

Where, R is the quantum dot radius, E_{gb} is the bulk band gap, E_{gn} is the quantum dot band gap, h is planck's constant, m* is the effective mass of the specimen. The bulk band gaps at room temperature and the effective mass[6,7] of SnO_2 is listed in the Table 4.6.

Name of the samples	Bulk Band gap	Values of effective mass (m*)	Structure
SnO_2	3.6 eV	2.51 x 10^{-31} Kg	Rutile structure

Table 4.6. Bulk band gap and effective masses of SnO_2

The data obtained from the analysis are put in Table 4.7.

Absorption edge in quantum dot	Energy gap in quantum dot	Bulk absorption edge	Bulk band gap	Enhancement in band gap due to size quantization	Quantum dot size
230 nm	4.4 eV	340 nm	3.6 eV	0.8 eV	13 nm

Table.4.7. Data of SnO_2/PVP specimen revealed from optical absorption spectroscopy

II. X-ray diffraction study

From X-ray diffraction study (Figure 4.15) average particle size (crystallite size) is calculated by using Debye-Scherrer formula[9] $D = \frac{0.9\lambda}{W Cos\theta}$, 'λ' is wave length of X-ray (0.1541 nm), 'W' is FWHM (full width at half maxima), 'θ' (theta) is the glancing angle and 'D' is particle diameter (crystallite size). Considering all the

peaks[2] (2θ in degree) in the X-ray diffractogram, the average crystallite (quantum dot) size has been assessed and found to be 12 nm. Further, by analyzing the X-ray diffractogram with the help of ICDD (International Center Diffraction Data) it has been revealed that SnO_2 quantum dots are rutile in structure.

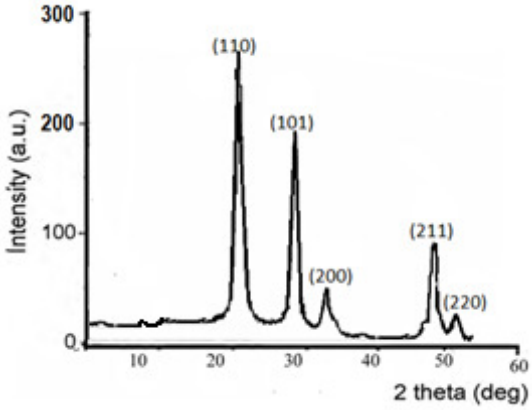

Fig. 4.15: XRD spectra of SnO_2 quantum dots

III. High Resolution Transmission Electron Microscopy (HRTEM)

HRTEM images of PVP film (4.16.c) and SnO_2 quantum dots (4.16.a,b) are shown in Figure 4.16. It is evident in the HRTEM image (4.16.a) that SnO_2 crystallites (quantum dots) are circular in shape with sizes within 12 nm[22].

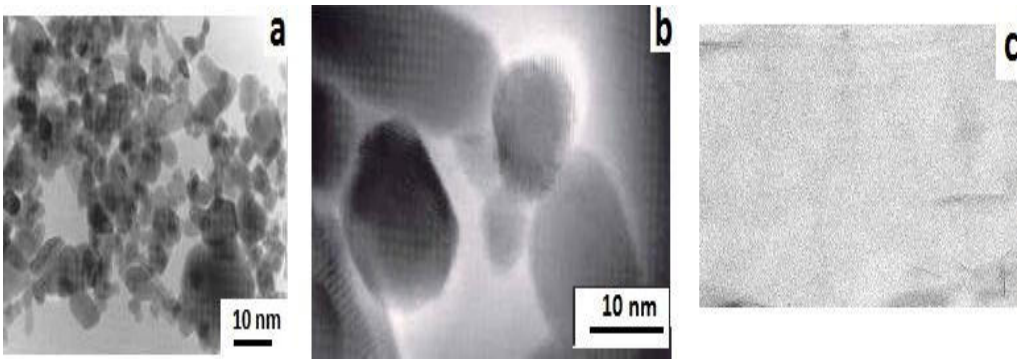

Fig. 4.16: HRTEM images of SnO_2 sample

SnO_2 sample sizes assessed from these three studies are well matching. This matching occurs due to the formation of well uniformed and circular shaped quantum dots by using PVP matrix [2] . All these characterizations infer that average size of SnO_2 quantum dots (diameter) are within 13 nm.

4.5.4 Iron oxide (Fe_2O_3) quantum dots embedded in PVP matrix

Fe_2O_3 specimen [23] has been characterized by UV/VIS optical absorption spectroscopy (Perkin Elmer Lamda 35 1.24), X-ray diffraction study (Bruker AXS, X-ray source: Cu K_α) and high resolution transmission electron microscopy (HRTEM) (JEM 2100, Jeol).

I. Optical absorption spectroscopy

In the optical absorbance spectra shown in Figure 4.17, denotes the spectrum of Fe_2O_3 quantum dot (prepared sample) [23] . It is seen that the band edge absorption in quantum dot is strongly blue shifted at 290 nm with respect to bulk specimen which possesses the absorption edge at around 650 nm.. For estimating particle size from blue shift, a theoretical model [6,7] has been proposed, known as hyperbolic band model that is written as

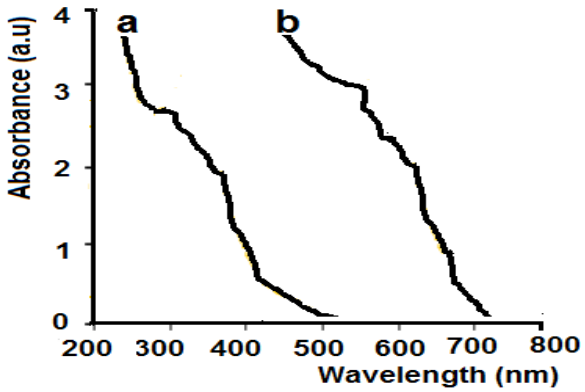

Fig. 4.17: UV/VIS absorption spectra of Fe_2O_3 a. quantum dots embeddedon PVP b. bulk

$$R = \sqrt{\frac{2\pi^2 h^2 E_{gb}}{m^*(E_{gn}^2 - E_{gb}^2)}} \quad \text{……..(4.8)}$$

Where, R is the quantum dot radius, E_{gb} is the bulk band gap, E_{gn} is the quantum dot band gap, h is planck's constant, m* is the effective mass of the specimen. The bulk band gaps at room temperature and the effective mass[6,7] of Fe_2O_3 is listed in the Table 4.8.

Name of the samples	Bulk Band gap	Values of effective mass	Structure
Fe_2O_3	2.2 eV	2.49 x 10^{-31} Kg	wurzite

Table 4.8. Bulk band gap and effective masses of Fe_2O_3

The data obtained from the analysis are put in Table 4.9.

Absorption edge in quantum dot	Energy gap in quantum dot	Bulk absorption edge	Bulk band gap	Enhancement in band gap due to size quantization	Quantum dot size
290 nm	3.4 eV	650 nm	2.2 eV	1.2 eV	15 nm

Table.4.9. Data of Fe_2O_3/PVP specimen revealed from optical absorption spectroscopy

II. X-ray diffraction study

From X-ray diffraction study (Figure 4.18) average particle size (crystallite size) is calculated by using Debye-Scherrer formula[9] $D = \frac{0.9\lambda}{W Cos\theta}$, 'λ' is wave length of X-ray (0.1541 nm), 'W' is FWHM (full width at half maxima), 'θ' (theta) is the glancing angle and 'D' is particle diameter (crystallite size). Considering all the

peaks[3] (2θ in degree) in the X-ray diffractogram, the average crystallite (quantum dot) size has been assessed and found to be 14 nm. Further, by analyzing the X-ray diffractogram with the help of ICDD (International Center Diffraction Data) it has been revealed that Fe_2O_3 quantum dots are "wurzite" in structure.

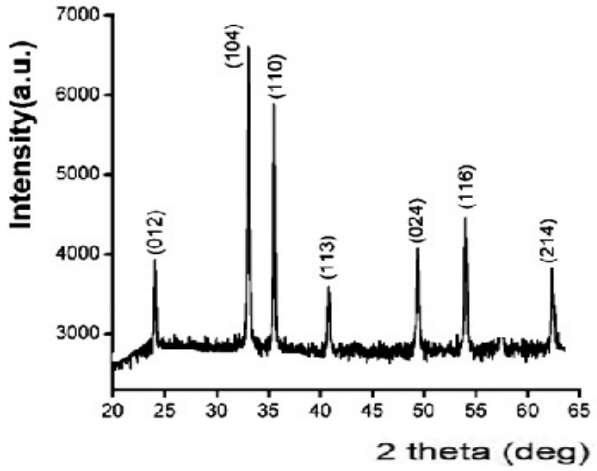

Fig. 4.18: XRD spectra of Fe_2O_3 quantum dots

III. High ResolutionTransmission Electron Microscopy (HRTEM)

HRTEM images of Plane-view(4.19.a) and cross sectional view of (4.19.b) of Fe_2O_3 quantum dots are shown in Figure 4.19. It is evident in the HRTEM image (4.19.a) that Fe_2O_3 crystallites (quantum dots) are circular in shape with sizes within 15 nm. [23]

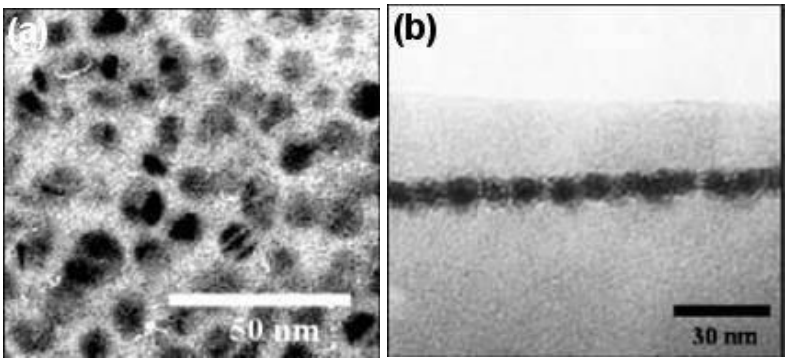

Fig. 4.19: HRTEM images of Fe_2O_3 sample

Fe_2O_3 sample sizes [23] assessed from these three studies are well matching. This matching occurs due to the formation of well uniformed and circular shaped quantum dots by using PVP matrix instead of PVA (polyvinyl alcohol) matrix[2] . All these characterizations infer that sizes of Fe_2O_3 quantum dots (diameter) are within 15 nm.

4.6 Conclusion: The prepared quantum dots have been characterized by various techniques, namely UV/VIS spectrometry, XRD and HRTEM. The characterizations infer that ZnO quantum dots are within 12 nm and 10 nm on PVA and PVP respectively. Further, it has been inferred that ZnO quantum dots are uniformly arranged on PVP rather than on PVA. In case of SnO_2 quantum dots, the size is within 13 nm while the size of Fe_2O_3 is within 15 nm. the structures of ZnO, Fe_2O_3 the quantum dots are wurtzite while the structure of SnO_2 is rutile.

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