Synthesis, Characterization and Application of

CdSe Nanoparticles - PVA, PLA Film

A Major Project Report submitted in partial fulfillment for the award of the degree

Of

MASTER IN TECHNOLOGY

IN

POLYMER TECHNOLOGY

Under esteemed guidance

Of

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Submitted by

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CERTIFICATE

This is to certify that **M.Tech** major project entitled "**Synthesis, Characterization and Application of CdSe Nanoparticles - PVA, PLA Film"**, submitted by **Chansi** for the award of the degree of **"Master of Technology in Polymer Technology**" is a record of the bonafide work carried out by her. Chansi has worked under the supervision and guidance of **Dr. Anil Kumar (Assistant Professor**), and has fulfilled the requirements for the submission of the dissertation .The project work has been carried during the session 2013-2014.

To the best of my knowledge and belief, this work has not been submitted to any other university/institution for the award of any degree or diploma.

No part of this project work has been reproduced elsewhere for any degree or diploma.

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STUDENT DECLARATION

I hereby declare that major project entitled "Synthesis, Characterization and Application of CdSe Nanoparticles - PVA, PLA Film" is a record of original work done by me under the guidance of Dr. Anil Kumar (Assistant Professor), during the session 2013-2014.

I also declare that no part of this report has been previously submitted to any University or any examining body for acquiring any degree.

STUDENT SIGNATURE

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ABBREVIATIONS

1. PVA: Polyvinyl Alcohol
2. PLA : Polylactic Acid
3. PL : Photoluminescence
4. XRD : X-Ray Diffraction
5. SEM : Scanning Electron microscope
6. AFM : Atomic Force Microscope
7. EDX : Energy Dispersive X-ray Spectroscopy
8. FTIR: Fourier Transform Infrared Spectroscopy

ABSTRACT

Semiconducting nanoparticles, whose particle size is in the nanometer range, have very unusual properties. These materials have size dependent physical and chemical properties. The electronic structure of these materials is between the molecule and bulk which lead to drastic change in the physical properties. Blue-shift in the optical absorption spectrum, size dependent luminescence, nonlinear optical effects are some examples of the interesting properties exhibited by these nanomaterial. Monodispersed CdSe nanoparticles of various sizes were prepared .These nanoparticles were used to prepare the nanoparticles polyvinyl alcohol and nanoparticles polylactic acid film.The prepared samples were characterized by UV-Visible Spectroscopy, Photoluminescence spectroscopy (PL) to study the optical properties. The structural and phase analysis was done with the help of X-ray diffraction (XRD).The surface morphology of the formed samples were analyzed by the Scanning Electron microscope and Atomic Force Microscope. Energy dispersive X-ray spectroscopy (EDX) was used for the elemental analysis. These nanoparticle films can be used in electronics by tuning the optical band by controlling the particle size of the nanomaterial.

AIM AND OBJECTIVE

AIM

The aim of the project has been preparation and characterization of CdSe nanoparticle Polyvinyl alcohol film (PVA), Polylactic acid (PLA) film.

PROJECT OBJECTIVE

Synthesis of CdSe nanoparticles: was carried out using solvothermal technique.

Synthesis of CdSe nanoparticles Polymer Film: CdSe nanoparticle Polyvinyl Alcohol, CdSe nanoparticle Polylactic acid film was synthesized by both in-situ and ex-situ method.

Structural Analysis: To know about the crystal structure, crystallinity, and phase transformation the XRD was used.

Optical analysis: Absorption, Band gap of synthesized nanoparticles can be determined by UV-Visible spectroscopy, Photoluminescence Spectroscopy (PL).

Morphological Characterization: Scanning Electron Microscope (SEM) was used to characterize the nanostructure of materials to study about the morphology.

Size Analysis: A size analysis was done by the help of Atomic force microscope (AFM).

Elemental Analysis: Both the nanoparticles formed and the films were characterized for elemental analysis by the EDX.

Chapter 1

INTRODUCTION

Solid materials generally exist either as bulk or as molecules. Recent advances in physics are focused to study the materials whose properties reside in between molecules and that of bulk. A solid exhibit a great variation of the electronic and the optical properties as the size of the particle is reduced below 100 nm, it can be called a nanostructure. These materials can be categorized as two dimensional, e.g. thin films, one dimensional such as quantum wires, zero dimensional e.g. quantum dots. Since the properties of nanoparticles are heavily influenced by the morphology of nanoparticles, methodologies to synthesize nanoparticles with precise control over size and shape have gained importance. These nanomaterials have led to entirely new avenues for applications, especially in electronics, optoelectronics and biology.

A fore-front area of research in the field of nanotechnology is CdSe nanoparticles due to their versatile properties. They are utilized in a wide range of commercial applications ranging from electronics, optical devices, photovoltaic and biomedical imaging [1-2] As group II-VI semiconductor nanoparticles, CdSe nanoparticles exhibit strong confinement of excited electrons and holes, which leads to dramatically different optical and electronic properties compared to bulk CdSe [3]. From the viewpoint of basic science, a nano crystal is in an intermediate state of matter between molecule-like cluster and bulk crystals, and therefore offers a possibility to trace an evolution of electronic and optical properties of the matter from atomic cluster to bulk solids. Many studies were devoted to CdSe nanoparticles due to their high luminescence quantum yield, narrow band gap and a variety of optoelectronic conversion properties compared to bulk CdSe [4-5]. High quality CdSe nanocrystals with uniform monodisperse size and shape are receiving much attention from the industry for the biological labeling reagents. CdSe nanoparticles are prepared by large number of techniques like sol-gel electrostatic deposition, solvent growth, DC magnetron, sputtering and chemical bath deposition. There is need to understand how the optical and electronic properties change with size of nanoparticles of CdSe. Different chemical methods employ cadmium salts as the source of cadmium and

different precursor for the selenium source. The nanoparticles frequently display photoluminescence and sometimes display electroluminescence. It is well known that the quantum confinement effect modifies the electronic structure of nanocrystals when their diameter is comparable to or smaller than the diameter of the bulk exciton [6]. There electronic characteristics are closely related to the size and shape of the individual crystal.

Monodispersed CdSe nanoparticles with sufficient luminescence intensity have been prepared by chemical method in the presence of mercaptoethanol as a capping agent. Size of nanoparticles can be monitored by changing the concentration of the capping agent. Variation in the nanoparticles synthesis was studied by changing the reaction temperature. The optical band gap of nanoparticle has been found to be blue shifted from the bulk value due to quantum confinement of carriers. Polyvinyl alcohol and Polylactic acid were used to prepare the nanoparticle embedded film. Polymer capped nanoparticles were also prepared by the in-situ method. . The resulting nanoparticles have been characterized by X-Ray Diffraction, UV-Visible absorption spectra and Photoluminescence (PL) spectroscopy, FT-IR spectra, Scanning Electron Microscopy, Atomic force microscopy. Characterization of the CdSe nanoparticles embedded polymer film was performed to study the optical properties according to the size variations.

Chapter 2

REVIEW OF LITERATURE

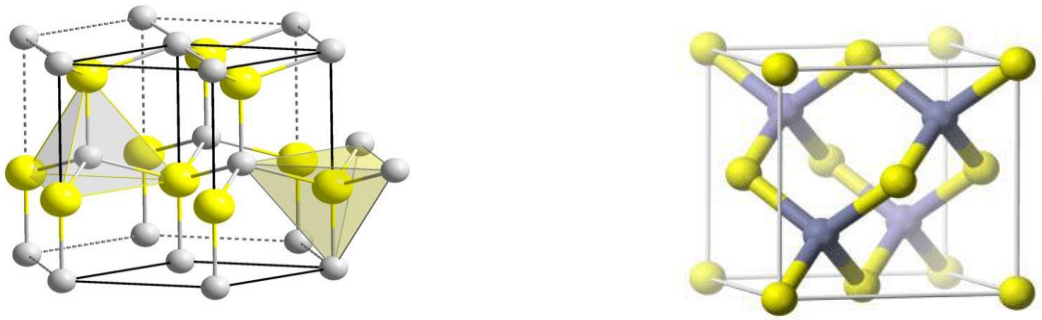
Much attention has been paid to nanoparticle semiconductors during recent time for their remarkable properties, which are different from those of conventional bulk semiconductors. Among the most important II-VI group semiconductors, cadmium selenide (CdSe) has attracted great interest due to its high photosensitivity, its attractive application in photoconducting cells and the dependence of its properties on the particle size. Earlier, CdSe has been prepared through a solid-state reaction between elemental cadmium and selenium at relatively high temperatures. Recently many methods have been introduced to synthesize CdSe nanoparticles, such as solvothermal synthesis, gamma irradiation, hard template, laser ablation and reverse micelles. Successful approaches have been involved for capping the particles with suitable species which help in the stabilization of these particles.

CdSe-derived nanoparticles with sizes below 100 nm exhibit a property known as quantum confinement. . When the electrons in a material are confined to a very small volume they exhibit Quantum confinement. It is size dependent, meaning the properties of CdSe nanoparticles are tunable based on their size. One type of CdSe nanoparticle is a CdSe quantum dot. Semiconductor nanocrystals composed of a CdSe core and a ligand shell is termed as Quantum dot.These ligands play important roles in the stability and solubility of the nanoparticles.

During synthesis, ligands stabilize growth to prevent aggregation and precipitation of the nanoparticles. These capping ligands also affect the quantum dot's electronic and optical properties by passivating surface electronic states [7]. An application that depends on the nature of the surface ligands is the synthesis of CdSe thin films. Photoluminescence properties were also observed to change with ligand moiety. These tiny particles can differ in color depending on their size.

2.1 Cadmium Selenide (CdSe)

Cadmium selenide (CdSe) in bulk form is a solid, binary compound of cadmium and selenium. It is a semiconducting material, with many applications, yet to be explored.



Wurtzite (Hexagonal) Wurtzite (Cubic)

Figure 2.1.1 Structure CdSe Nanoparticles

It shows a variety of colors from transparent to red colors. There are two forms of the CdSe nanoparticles as shown in above Figure 2.1.1 Both the structures differ only in the stacking sequence of the packed layers.

The Wurtzite structure has an ABABAB stacking sequence, while the rock-salt structure has an ABCABC stacking sequence. There is another form known as sphalerite structure which is unstable and converts to the wurtzite form upon moderate heating. The transition starts at about 130 °C, and at 700 °C it completes within a day.

Researchers are concentrating on developing controlled synthesis method of CdSe nanoparticles. They exhibit both physical and chemical properties within an intermediate state of matter, between molecular and bulk. Their unique properties are attributed to two main factors: the large surface-to-volume ratio of atoms and their reduced dimensions in relation to the excitonic radius of the bulk material known as quantum confinement [8]. This quantum-sized effect causes structural and electronic changes that, in turn, induce other novel properties that largely dominate the behavior of these materials and make them different from that of the bulk. The decrease in particle size leads to extremely high surface area to volume ratio which leads to an increase in surface specific active sites for chemical reactions and photon absorption and hence enhances the reaction and absorption efficiency. These unique properties make

semiconductor nanoparticles potentially useful in many areas of applications, which include catalysis, optical recording materials, solar cells, biotechnology, sensor and lasing materials.

2.2 Properties of CdSe nanoparticles

1. Electronic Properties

The energy gap separating the conduction and valence energy bands is the defining feature of semiconductor nanoparticles. The width of the band gap determines the color of light emitted by the semiconductor material. .The gap width is a fixed parameter determined by the material's identity. In the case of nanoscale semiconductor particles with smaller sizes the situation changes. The electronic properties start to change as the diameter of the crystalline material approaches the exciton Bohr diameter. This reduction of particle size, with corresponding increase in the band gap of the nanoparticle is known as the quantum confinement effect.

The band gap becomes energy dependent that is, as the size decreases the absorption edge is blue-shifted (higher energy/lower wavelength) indicating an increase in the band gap. For a spherical quantum dot with radius R, it is predicted that a size dependent contribution to the energy gap is simply proportional to $R^{2,}$ which implies that as the band gap increases the quantum dot size decreases.

This increase in energy is observed in the optical spectra, for a range of nanocrystalline semiconductors, as the particle size decreases there is blue-shift in the band gap. The shifts in the absorption edge of group II-VI semiconductors such as CdSe and CdS can be a large fraction of the bulk band gap and for CdSe can result in turning across a large portion of the visible spectrum.

Nanoparticles shows the presence of the peak which is known as exciton .It is the evidence of the quantum confinement which shows that the spectrum starts to resemble a molecule with discrete absorptions rather than a solid with continuous bonds.

2. Optical Properties

The electrical and optical energies of the band gap are equivalent through the following conversion:

$$E = hc / \lambda$$

E = Band gap energy difference

h = Planck's constant

c = Speed of light

λ= Wavelength of incident light

Thus, the energy difference of the band gap is inversely proportional to the wave length of incident light. Nanoparticles will only absorb light of wavelengths shorter than that determined by the band gap value. For example, CdSe (bulk) has a band gap of 1.73 eV, which corresponds to a wavelength of 716 nm. CdSe begins to absorb light at 716nm and absorbs continuously into the U V (e.g. shorter wavelengths). The band gap increases and the absorbance onset shift to shorter wavelengths as the particle size declines. Thus, onset of absorbance is directly related to particle size.

The influence of particle size on optical properties is not limited to absorbance. Particle fluorescence is also a function of the band gap. Photon absorption creates an excited electron. This electron loses some of its energy to atomic vibrations, which satisfy the second law of thermodynamics. The energy is converted into heat. The electron then decays to ground, emitting a photon. The emitted photon has a longer wavelength than the absorbed photon because of the energy lost to heat. As the band gap decreases, the particle will absorb at longer wavelengths. This will produce a concomitant red-shift in particle fluorescent emission. Because band gap is inversely proportional to the nanocrystal size, larger nanocrystals display red-shifted emission. Additionally, the energy lost to heat decreases in a size-dependent manner.

3. Luminescence Properties

The fundamental parameters to characterize emission properties of a semiconductor are emission color, color purity, brightness, quantum yield (QY) and stability of the emission. Photoluminescence (PL), the behavior of semiconductor nanocrystals is studied by two types of bands they are "band gap" emission and the "deep trap" emission. The band gap is narrow (width determined by the size distribution) and is only slightly red-shifted from the absorption onset.

The deep trap PL is broad and is substantially red-shifted from the absorption onset (typically by 0.5 eV). It has been associated with radiative recombination of localized surface trapped charge carriers and, because of its long lifetime, its intensity is relative to the band gap and PL is enhanced at low temperatures. As the particle size decreases because of high surface-to-volume ratio, surface atoms play a more important role in the emission properties of the nanoparticles. There are unsaturated bonds or dangling bonds, extra free energy in these surface atoms and are more active than those in the bulk. The nanoparticles can easily transform from one phase to another and have a low transformation temperature apply to it.

In general, as a result of the surface states located in the band gap of the nanocrystals there is a low PL quantum yield. These surface states are originated due to presence of the dangling bonds of some of the surface atoms. The ligands on the surface of nanocrystals may remove some or the entire surface trapping states and increase the PL quantum yield of the nanocrystals. Theoretical treatments indicate that surface structure and the nature of the surface states of the nanocrystal determine the efficiency of the electronic passivation provided by the surface ligands.

If the surface ligands provide good passivation for the surface defects located in the band gap of the nanocrystals, which act as trapping states for the photo generated charges i.e., behaving as a non-radiative relaxation centre for the electron hole recombination shows high PL quantum yield. The efficiency of the electronic passivation provided by the surface ligand is also affected by the optimal surface structure reconstruction of the nanocrystals which minimizes the effect of the atomic

configuration on the surface of nanocrystals. Less surface defects and less surface roughness arises due to slower growth rate in size.Gowth of the crystals is faster at higher temperatures which also lead surface roughening and degradation compete with surface ordering. This induces faster surface degradation, which reduces the luminescence efficiency shortly after maximum efficiency has been reached [9-11].

4. Particle Size

Many model calculations of size quantization effects have been published in the last few years. Efros [12-15] described the first calculation where they used spherical, infinite potential wells which ignored the columbic interactions. Brus *et al* [13] developed the energy levels of the first excited state by considering the coulombic interactions and polarization terms. Most of these models determine the increase of the band gap with decreasing particle size of a particle-in-a-box assumption, with the crystallite size making up the dimensions of the box. The models differ on the complexity of the calculation and the boundary conditions.

Weller [16] made quantum mechanical calculations for higher excited states as a function of particle size, however, the values for the effective masses and high frequency dielectric constants are taken from macro crystalline solids, which results in uncertainty in the accuracy in the calculation.

The most universal particle-in-a-box calculation has been represented by Nosaka whose calculations assume the validity of the effective mass approximation [16]. Other models have also been proposed for particle sizes apart from those based on the particle-in-a-box assumption, by relating the particle size determined by transmission emission microscopy (TEM) and X-ray diffraction (XRD) measurements with the electronic transition occurring in nanoparticles.

2.3 Methods for Preparing CdSe Nanoparticles

There are three distinct approaches for the synthesis and functionalization of nanoparticles.

a) **Bottom-up approach***:* This synthesis involves conversion of a precursor into nuclei that is converted into monodispersed particles through specific reaction such as decomposition, reduction or hydrolysis.
b) **Template-directed approach:** In this approach colloidal spheres serve as physical or chemical templates to coat their surfaces with other materials or, to convert them into a different material with a rich variety of composition, structure, and functionality.
c) **Top-down approach**: The bulk material is added to a hot solvent, melted and sheared to produce colloidal spheres in this method.

2.4 Chemical methods for the synthesis of CdSe Nanoparticles:

1. Colloidal Method

One of the important methods for the preparation of the nanoparticles is the colloidal route. It is carried through the controlled precipitation in presence of the stabilizers in homogeneous solution. The stabilizers role is to prevent the agglomeration of the particles. The synthesis of highly monodispersed colloids was explained in the 1940's by La Mer et al [17-19] who suggested that if seeds (nuclei) could be made to grow in concert into large particles, monodispersed sols could be formed.

Bros, Weller and Henglein [20-22] have used this method to synthesize semiconductor nanoparticles of CdS and ZnS. Their work has contributed significantly towards the understanding of some of the fundamental properties of nanoparticles. It is reported the synthesis of CdS nanoparticles by mixing aqueous solutions of $CdSO_4$ and NH_4S. By altering the nucleation kinetics using pH the subsequent size of the Nano crystalline CdS could be controlled. The repulsion of the electrostatic double layer prevented agglomeration and/or sedimentation. The optical spectra showed a blue-shift in band edge in relation to the bulk material. Structural characterization by high resolution

electron microscopy (HRTEM) and electron diffraction, revealed a zinc blende structure.

Henglein, Weller and co-workers studied the photo physical properties of Nano crystalline CdS [21-22]. The analysis of the size distribution revealed that, a certain size with the greatest oscillator strength was favored, which corresponded to the optical transition in the absorption spectrum.

The fast precipitation of the colloids renders a primary size distribution of small particles, peaking at an initial size m_o

$$\mathbf{m_o\, Cd^{2+} + m_o\, S^{2-} \rightarrow (CdS)\, m_o}$$

Nano crystalline Zn_3P_2 and Cd_3P_2 have been synthesized by the aqueous methods, the injection of phosphine gas (PH_3) into a solution of the relevant metal salt. The nanoparticles show dramatic changes in appearance and electronic properties, as the size of the particles is varied. Bulk Cd_3P_2 is black in color, whereas Nano crystalline Cd_3P_2 displays a wide range of colors, from white for the smallest, to brown for the largest particles.

Wang et al [23] reported a milder synthetic route to MSe (M =Zn, Cd, Sn and Cu) using ethylenediamine as the solvent both in an autoclave and at room temperature (80 -100 GC) with KBH. The reaction at room temperature produced spherical particles of large grain size, while the reaction inside an autoclave yielded nanorods.

The colloidal route is an efficient one for the preparation of nano scale semiconductor particles. However, certain types of semiconductors, such as CdSe, GaAs, InP and lnAs, cannot be synthesized easily via this route. Annealing of the colloidal particle is also a problem as these tend to be a low temperature process. Such aqueous prepared nanoparticles are not sufficiently stable at higher temperature and thus makes annealing of the particle difficult, thereby making the material poorly crystalline. As a result of these difficulties, there have been several modifications to the synthesis via colloidal route.

Chen et al. reported simple solution-phase synthesis, of crystalline and water soluble CdS and CdSe nanorods via the arrested precipitation from their respective inorganic ions in a micellar solution.

Recently selenosulphate and selenourea were identified as the main sources to provide Se^{2-} ions for the preparation of selenides through reactive solution growth. The reaction between selenosulphate and Cd ions is the basis of most commonly used techniques for chemical bath deposition (CBD) of CdSe films.

As a result of the simplicity involved in the synthesis of Na_2SeSO_3, even though it requires long reaction time, it has been used as selenium source together with various cationic precursors to synthesize CdSe nanoparticles via microwave irradiation.

In a very time consuming reaction, Badr and Mahmoud[24] produced selenide analogue by reduction of elemental selenium in polyvinyl alcohol (PVA) photopolymer films in the presence of additional stabiliser, cetyl trimethyl ammonium bromide (CTAB), but the obtained nanoparticles possessed poor optical properties with very broad trapped state emission.

1) Organometallic/Metal-Organic Routes

The early problems associated with the synthesis of nanoparticles via the colloidal route was overcome by a method that makes use of organometallic and/or metal organic compounds under anaerobic conditions. Bawendi et al [7] in 1993, pioneered this method whereby good quality, monodispersed, highly crystalline nanoparticles were synthesized. This method (also called the East Coast Method), involves rapid injection of a volatile metal alkyl (dimethyl cadmium or dimethyl zinc) and a chalcogen source TOP-E (E=Se, Te) mixed in tri-n-octylphosphine (TOP) and injected into hot tri-n-octylphosphine oxide (TOPO), a polar coordinating Lewis base solvent. Nucleation of the CdSe nanoparticles was achieved by the sudden introduction of the concentrated reagents resulting in the abrupt super saturation and formation of nuclei, followed by slower growth and annealing, consistent with an Ostwald ripening process. The nanoparticles were passivated by a monolayer of the solvent ligand and hence could be

isolated/purified by solvent/non-solvent interactions. Purified nanocrystals undergo size selective precipitation to provide powders of nearly monodispersed nanocrystals which can be dispersed in a variety of solvents. The particle size can be controlled by varying the temperature and the time of the reaction. The choice of trioctylphoshine (TOP) as the coordinating solvent allows the reaction to take place above the nucleation temperature and allows the nanocrystals to be soluble in organic solvents or dispersed in a polymer film.

As part of improvement and eliminating the use of toxic materials in the synthesis of nanoparticles, Sapra et al. [23] synthesized CdSe nanoparticles using an inexpensive, harmless olive oil as the coordinating solvent and cadmium oxide as the cadmium source.

2.5 CdSe Nanoparticles Polymer film

Polymer nanoparticles films have been increasingly studied because of their enhanced optical and electronic properties. They are good choices as stabilizers, because they can interact with metal ions by complex or ion-pair formation and, can be designed to certain physical properties of semiconductor nanoparticles.

2.6 Polyvinyl alcohol has following advantages to be used:

(i) High viscosity of the polymer solution would be helpful in controlling the growth of selenide nanoparticles and, prevent particles from aggregating hence; no additional stabilizer would be needed.

(ii) High aqueous solubility

(iii) Applications point of view, the polymer matrix would protect the selenide against photo-oxidation.

Chapter 3

EXPERIMENTAL

3.1 MATERIALS

Two necked round bottom flask, Thermometer, Magnetic bead, Magnetic stirrer, Heater, Argon cylinder (inert atmosphere), Beakers, Test Tubes, Glass rod, Sample Collection Vials, Petriplates.

CHEMICALS

- Cadmium Acetate Dihydrate (CDH Laboratory Reagent)
- Dimethyl Formamide (High Purity Laboratory Chemicals Pvt Ltd.)
- Mercaptoethanol(Aldrich)
- Sodium Selenite Pentahydrate(CDH Laboratory Reagent)
- Acetone (Finar Limited LR Grade)
- Polyvinyl Alcohol (hot) (CDH Laboratory Reagent)
- Potassium Bromide(Aldrich)
- Polylactic acid (High Purity Laboratory Chemicals Pvt Ltd.)

All the chemicals were not further purified, used as such as received.

Instruments for characterization

- Centrifuge
- X- Ray Diffraction (XRD) Bruker D-8
- UV spectrophotometer Perkin Elmer
- Photoluminescence Spectroscope (PL) Horiba Jobin
- Fourier transformation Spectroscope (FTIR) Thermoscientific Nicolet 380.
- Scanning Electron Microscope (SEM) Hitachi S-3700
- Atomic force Microscope (AFM)
- Energy Dispersive X-Ray Spectroscopy (EDX) Hitachi S-3700

3.2 METHODS

Preparation of solutions:

Solution 1

0.5875gm Cadmium acetate dihydrate was dissolved in 50ml of Dimethyl Formamide. (2.2 mM).

Solution 2
0.579gm of sodium selenite pentahydrate was dissolved in 8ml of distilled water.

CdSe nanoparticles of different sizes were prepared varying the concentration of the capping agent.

3.2.1. Preparation of CdSe Nanoparticles

(Sample A)

1. Solution 1 was stirred under inert argon atmosphere.
2. Mercaptoethanol solution 0.125 ml was mixed to the above solution by constant stirring.
3. Solution 2 was added to the solution 1.
4. The resulting solution 3 was stirred for 30 minutes and further refluxed for 3hours at 160^{o}C with constant stirring under inert atmosphere.
5. After 3 hours the reaction was stopped and the precipitation of the nanoparticles was carried out.
6. Size selective precipitation of the formed nanoparticles was carried out by using acetone as the non-solvent.
7. The solution was centrifuged at 15000 rpm for 10 minutes to separate the nanoparticles
8. The resulting precipitate extracted was washed 5 times in methanol.
9. The precipitate was dried and transferred to the vial for further characterization.

(Sample B)

1. Solution 1 was stirred under inert argon atmosphere.
2. Mercaptoethanol solution 0.25 ml was mixed to the above solution by constant stirring
3. Solution 2 was added to the solution 1.
4. The resulting solution 3 was stirred for 30 minutes and further refluxed for 3hours at 160ºC with constant stirring under inert atmosphere.
5. After 3 hours the reaction was stopped and the precipitation of the nanoparticles was carried out.
6. Size selective precipitation of the formed nanoparticles was carried out by using acetone as the non-solvent.
7. The solution was centrifuged at 15000 rpm for 10 minutes to separate nanoparticles.
8. The resulting precipitate extracted was washed 5 times in methanol.
9. The precipitate was dried and transferred to the vial for further characterization.

(Sample C)

1. Solution 1 was stirred under inert argon atmosphere.
2. Mercaptoethanol solution 0.5 ml was mixed to the above solution by constant stirring.
3. Solution 2 was added to the solution 1.
4. The resulting solution 3 was stirred for 30 minutes and further refluxed for 3hours at 160ºC with constant stirring under inert atmosphere.
5. After 3 hours the reaction was stopped and the precipitation of the nanoparticles was carried out.
6. Size selective precipitation of the formed nanoparticles was carried out by using acetone as the non-solvent.
7. The solution was centrifuged at 15000 rpm for 10 minutes to separate the nanoparticles.
8. The resulting precipitate extracted was washed 5 times in methanol.
9. The precipitate was dried and transferred to the vial for further characterization.

Similarly same process was repeated to synthesize the nanoparticles by changing the reaction temperature to 120º Samples were labeled as A1, B1, C1.

Table 3.1 Nanoparticle Samples Prepared

Sample	Capping Agent	Reaction Temperature (ºC)
A	0.125ml	160
B	0.25ml	160
C	0.5ml	160
A1	0.125ml	120
B1	0.25ml	120
C1	0.5ml	120

3.2.2 Preparation of CdSe nanoparticle Polymer Film(ex-situ method)

CdSe nanoparticles samples A, B, C prepared were used to prepare a film with Polyvinyl Alchohol.

Method

1. 4gm of polyvinyl alcohol was weighed and it was mixed with 40gm of water.
2. Above solution was stirred with the magnetic stirrer and heating the solution at 80 º C
3. To the heated solution Cdse nanoparticles were added to it in 5% concentration.
4. The resulting solution was poured in petriplate to form a film.
5. The petriplate was kept in oven at 120ºC for 4hrs and then kept overnight.
6. The resulting film so formed was taken out and was analyzed and further characterized.
7. Same steps 1 to 6 were repeated for all the samples.

4.2.3 Preparation of CdSe nanoparticle Polymer Film(in-situ method)

Another method of preparation of CdSe nanoparticle polymer film is by the formation of CdSe nanoparticles during the film formation stage.

Method

1. .08gm Cadmium Acetate Dihydrate was added to 4ml of water to form 99 milimolar solution.
2. 2gm of Polyvinyl Alchohol was added to above solution.
3. The solution was made 50 ml by adding distilled water to it.
4. After the solution was left for 24 hrs at room temperature to swell.
5. After 24hrs the solution was kept on the mechanical stirrer and heated for half hour at 60 ºC
6. 0.06 gm of Sodium Selenide was added to 1ml of water, the resulting solution formed was added to the Polyvinyl Alchohol solution.
7. The solution was casted on the petriplate and the petriplate was kept in oven and heated at 250º C for 4hrs.
8. The color of the film changes from transparent to yellow and then changes to orange.
9. The film so formed was taken out from the petriplate and then was further characterized.

4.2.4 Preparation of CdSe nanoparticles Polylactic acid film

1. 5gm polylactic acid was dissolved in 50 ml of carbon tetra chloride.
2. The solution was stirred continuously to form a thick solution.
3. CdSe nanoparticle solution prepared in 5% concentration was added to above solution.
4. The resulting solution was stirred continuously and poured in petriplate to form a film.
5. The petriplate was kept in oven at 120º C for 5hrs.
6. The resulting film was taken out and characterized for further analysis.

(sample A) **(sample B)** **(sample C)**

Figure 3.2.1 CdSe nanoparticles synthesized

Figure 3.2.2CdSe nanoparticle PVA Film Synthesized by ex-situ method

Figure 3.2.3: CdSe Nanoparticles Polyvinyl Alchohol film formed by in-situ method

Figure 3.2.4: CdSe nanoparticle Polylactic acid film

Chapter 4

CHARACTERIZATION

1. X-ray diffraction (XRD)

X-ray diffraction (XRD) is a common non-destructive method for the analysis of the samples .This method help in revealing detailed information about the chemical composition, atomic spacing and crystallographic structure of both the natural products and the manufactured materials.

The size of the nanocrystal is determined by the help of the XRD using the Scherer formula:

$$D=K\lambda/\beta \cos\Theta$$

D = average dimension of the crystallites normal to the reflecting planes

k = Scherer constant. K varies from 0.89 to 1

β = the integral breadth of a reflection (in radians 2θ) located at 2θ

Value of Scherer constant used is 0.89.

Samples Tested

Samples A, B, C were used in powdered form to take the diffractogram by the help of Bruker D-8

2. Ultraviolet-visible spectroscopy

Ultra-violet spectroscopy is one of the important methods for the analysis of the light. It is known as the absorption spectroscopy. It is operated in UV-visible region (200-800 nm) of the electromagnetic spectrum. In this region of the electromagnetic spectrum, atoms, ions or molecules undergo electronic transitions from ground state to excited state. The chemical structure of a given material characterized by absorbed or the transmitted light. The graphical representation of the amount of light absorbed or transmitted by matter as a function of the wavelength is known as the spectra.

Figure 4.1 Shows the instrumentation and working of the UV-Vis spectroscopy

In this the radiation source (Deuterium lamp – covers the UV – 200-330 nm and Tungsten lamp – covers 330-700 nm) enters the monochromator through the entrance slit. In monochromator beam is collimated, and then strikes the dispersing element at an angle. The beam splits into component wavelengths by the grating or prism. By moving the dispersing element or the exit slit, radiation of only a particular wavelength leaves the monochromator through the exit slit. The beam splitter sends a separate band to a cell (called a cuvette") containing the sample solution and a reference solution. The difference between the transmitted light through the sample (I) vs. the incident light (I) and sends this information to the recorder is measured through the detector.

The spectra helps in the determination of the composition of the organic compound and the elemental analysis of the material .It also help in the determination of the optical band gap (E_g)
Perkin Elmer Spectrophotometer was used for the analysis.

Sample preparation

The samples for UV are prepared by dissolving the powdered sample and the film formed in suitable solvent (Dimethyl formmamide (DMF)) in ratio of 1:20 for the proper dilution and the same solvent is used as the base for taking the readings.

3. **Energy Dispersive X-Ray Spectroscopy (EDX)**

Energy Dispersive X-Ray Spectroscopy (EDX) is an analytical technique used for the chemical characterization or the elemental analysis of the sample. When a high energy electron beam is focused into the sample, x-rays are generated due to secondary electron transitions. These electrons can be analyzed by the EDX technique to determine elements present in the sample. It helps in recording the relative concentration in weight and atomic formula concentration can be obtained. A 2-D image of the sample can be obtained which help in the element detection.

Sample Preparation

Both the powdered samples and the films were analyzed by coating it with conducting material. Hitachi S-3700 variable pressure SEM was used for the analysis.

4. **Scanning electron microscopy**

Scanning electron microscopy is a important tool to analyze the surface of almost any material .It uses a focused beam of the high energy electrons to generate the signals from the specimen.SEM used to analyze the selected point on the sample .The resolution of the image depends on the electron probe and also on the interaction of the sample specimen with electron probe .The combination of the higher magnification, greater resolution, crystallographic information makes it an efficient method.

The semiconducting nanoparticles were imaged using Hitachi S-3700 variable pressure SEM, of resolution 3nm, 2nm, and 500nm with accelerating voltage of 0.3 to 15 kV

5. **FTIR**

FTIR (Fourier Transform Infrared) or simply FTIR Analysis is a simple method for the analysis of both the organic and the inorganic samples. It can be used in the analysis of the solid, liquid and gases. In this method the optical frequency of all the samples are recorded over a period of time. The data collected can be converted into a interference

pattern into a spectrum. The samples were analyzed for the presence of the functional groups.

Sample Preparation

The FTIR spectra of the powdered sample can be taken by the help of preparation of the pellets with the help of Kbr in machine .The film samples and the liquid samples were analyzed directly by changing different assemblies for the each sample type. The samples were analyzed by Thermoscientific Nicolet 380.

6. Atomic force Microscopy (AFM)

Atomic force microscopy is used to provide the 3 D images of the samples on nanoscale.It help in the measurement by the interaction of the probe with the sample. AFM can be used for wide variety of the samples ranging from plastic, metals, semiconductor and the biological samples. It doesn't require the presence of the conducting samples. The limitation is that it doesn't reflect the true surface topography but rather represent the interaction of the probe with the sample surface which is referred to tip convolution.

7. Photoluminescence Spectroscopy

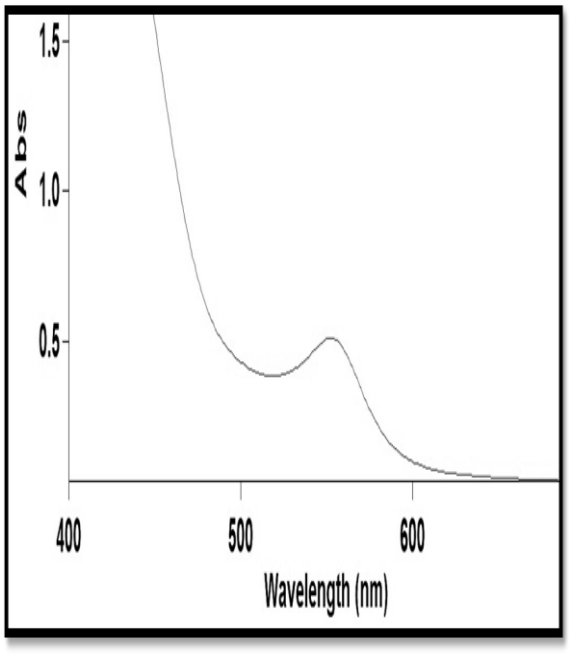
Photoluminescence spectroscopy often referred to as PL is a non-destructive method. It is used for the analysis of the electronic structure. Light in directed to the sample which help in the excitation of the sample also known as the photo excitation. This excess energy is dissipated from the sample in the form of luminescence.

Horiba Jobin Photoluminescence spectroscope was used.

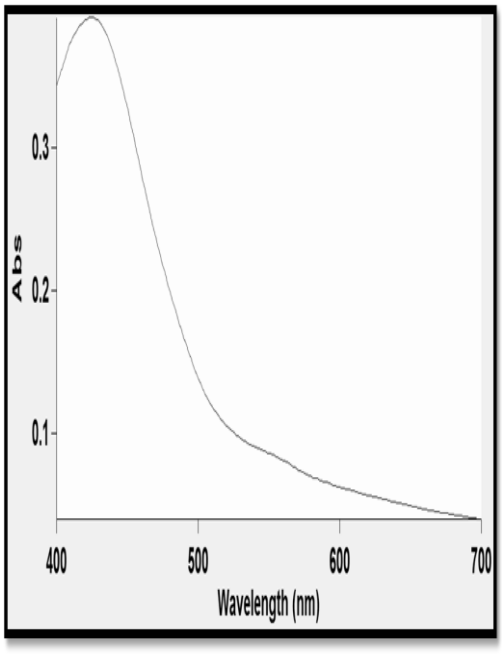
Chapter 5

RESULT AND DISCUSSION

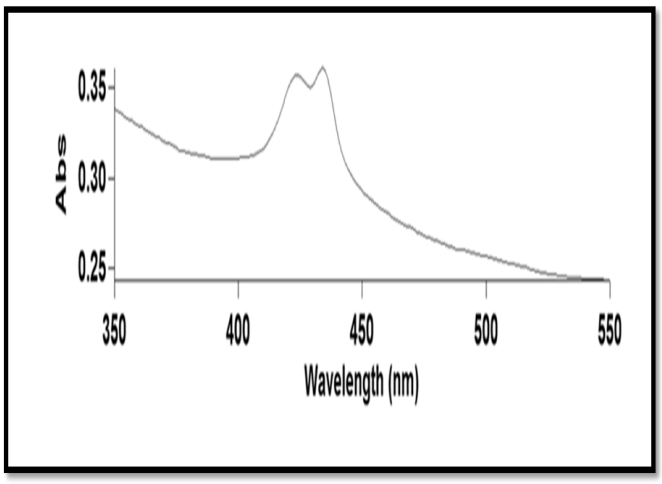
5.1 UV results



Sample (A)



Sample (B)



Sample (c)

Figure 5.1.1 UV Spectra of CdSe Nanoparticles prepared at 160º C

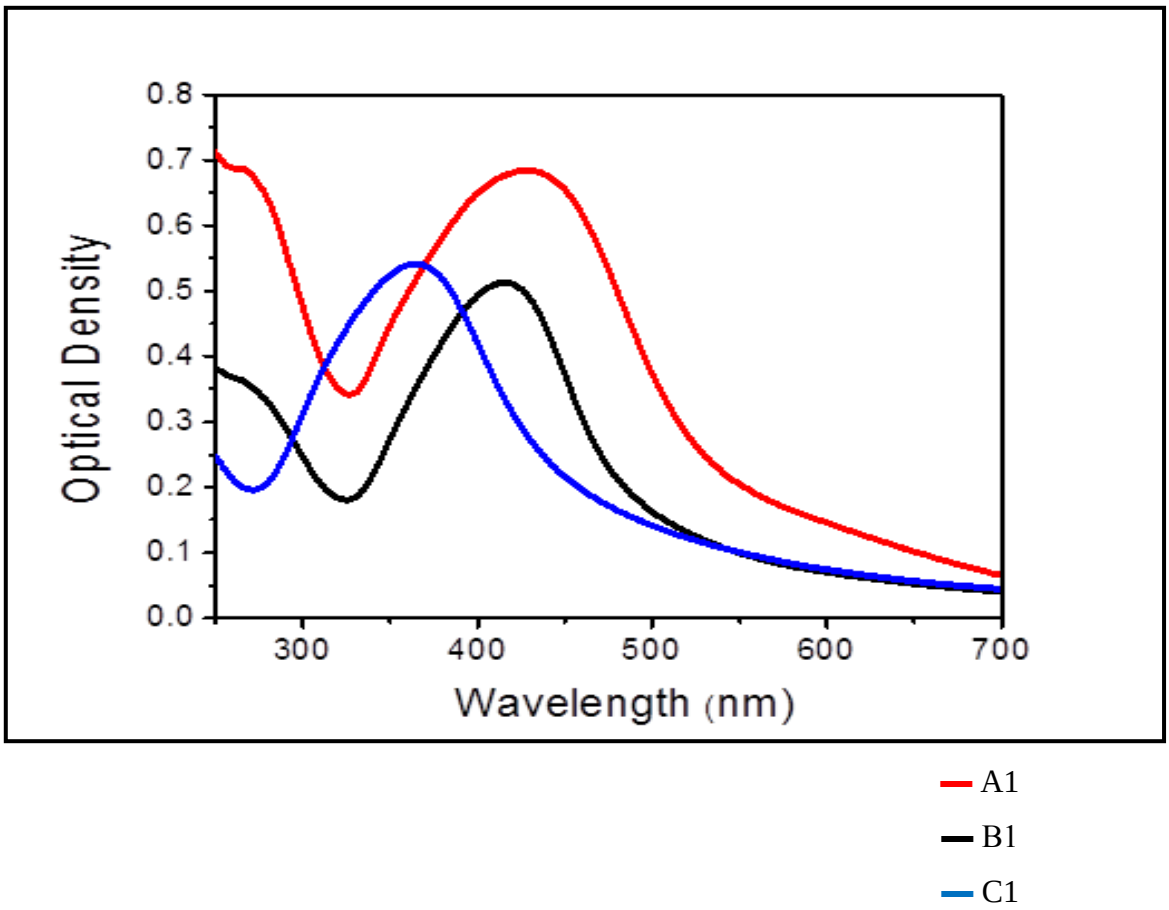


Figure 5.1.2 UV Spectra CdSe Nanoparticles prepared at 120º C

Thus using the relation

$$Eg = hc/\lambda \qquad (1)$$

Where Eg is the optical band gap of the nanoparticle

From the excitonic absorption peak, C speed of light in space (3 x 108m/s)

λ= wavelength of the nanoparticle at various absorption peak.

The size of the CdSe nanoparticles was calculated from the bandgap values, using the Brus equation, which has been simplified to the following equation (Equation 2).

$$Eg = Eg(0) + h^2 / d^2 [1/m_e + 1/m_h] \qquad (2)$$

Where Eg = Optical Band gap of nanoparticles calculated in eV

Eg (0) = bulk Band gap

m_e^* = effective mass of electron

m_h^* = effective mass of holes

d= size of the nanoparticles

h= Planck constant

for Cdse nanoparticle $m_e^* = 0.13\ m_o$, $m_h^* = 0.45\ m_o$ Eg(0) = 1.7 eV

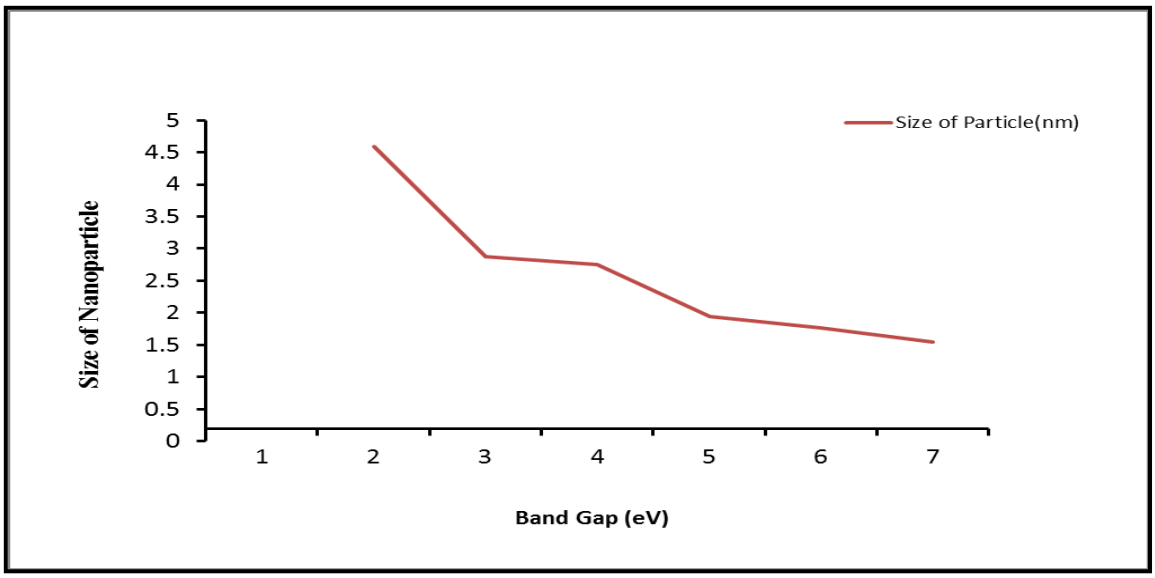
Therefore, the size of CdSe nanoparticles is given by Equation 3.

$$Eg = 1.7 + 3.7/ d^2 \qquad (3)$$

Concurrently, the nanoparticle diameter (size) was calculated using the relation.

Table 5.1.1 Calculated Band gap and Diameter (size) of nanoparticle calculated

Samples	Wavelength(nm)	Band Gap (Eg in eV)	Size of Particle(nm)
A	**550**	**2.25**	**4.59**
B	**450**	**2.75**	**2.88**
C	**425**	**2.90**	**1.75**
A1	**460**	**2.68**	**1.94**
B1	**430**	**2.87**	**1.77**
C1	**380**	**3.25**	**1.54**



Graph 5.1.1 Size of Nanoparticle vs. Band Gap

Optical property of the CdSe nanoparticle is studied through the absorbance excitation peaks .The electronic state of these nanoparticles can be well studied through the valence and conduction band and with the band gap between these bands known as the optical band gap . The band gap calculated through the relation as reported in Table 5.1.1 help in the determination of the size of the nanoparticles.

It is clearly seen in above results (Table 5.1.1) that as the size of the nanoparticle shrink ,the absorbance peak is shifted to shorter wavelengths. This also result in increase in the optical band gap .The value of band gap of the synthesized nanoparticles is large as compared to the bulk CdSe nanoparticle. The increase in band gap is due to increase in the surface to volume ratio.

Important feature of CdSe nanoparticle is their color. The nanoparticles though synthesized from the same material but show different color as the size of the nanoparticles changes. The coloration of the nanoparticles so formed is dependent on the energy level. Quantitatively speaking, the band gap determined the energy (and hence colour) of the fluorescence light which is inversely proportional to the size of the nanoparticle.

(2) Photoluminescence (PL) results

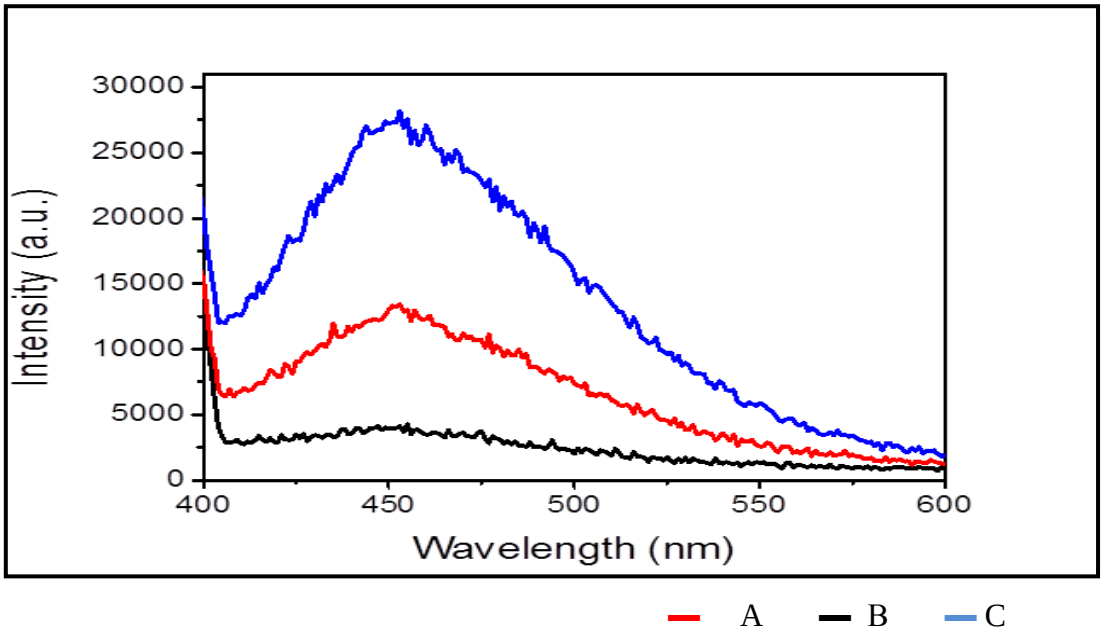


Figure 5.2.1 PL spectrum of Nanoparticles formed at 160º C

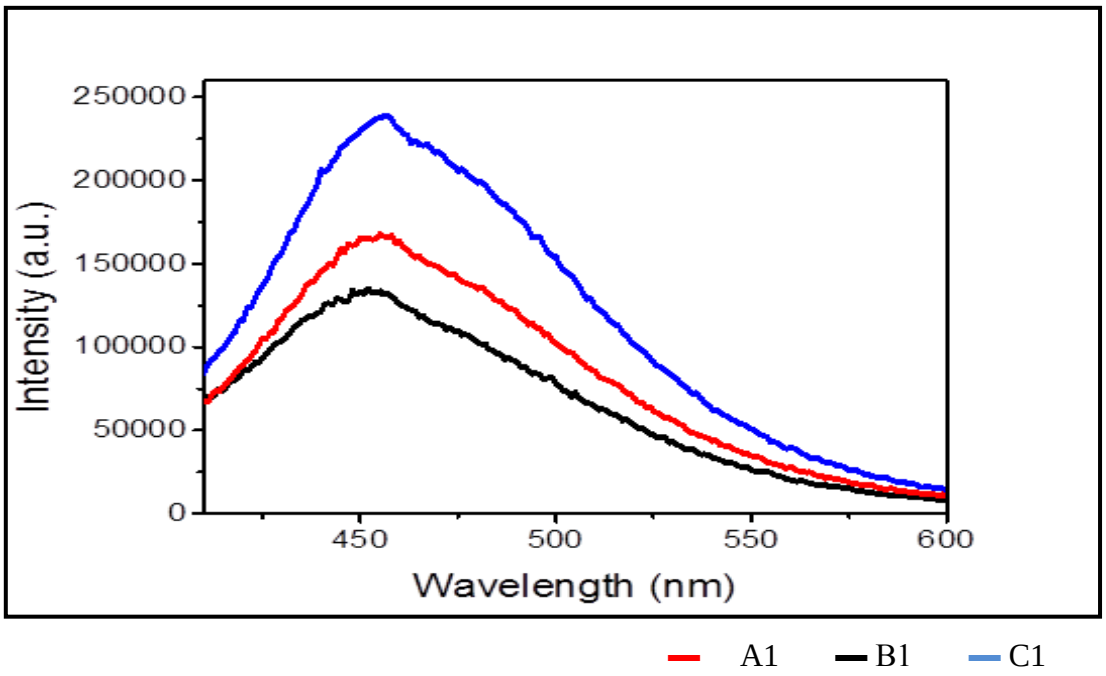
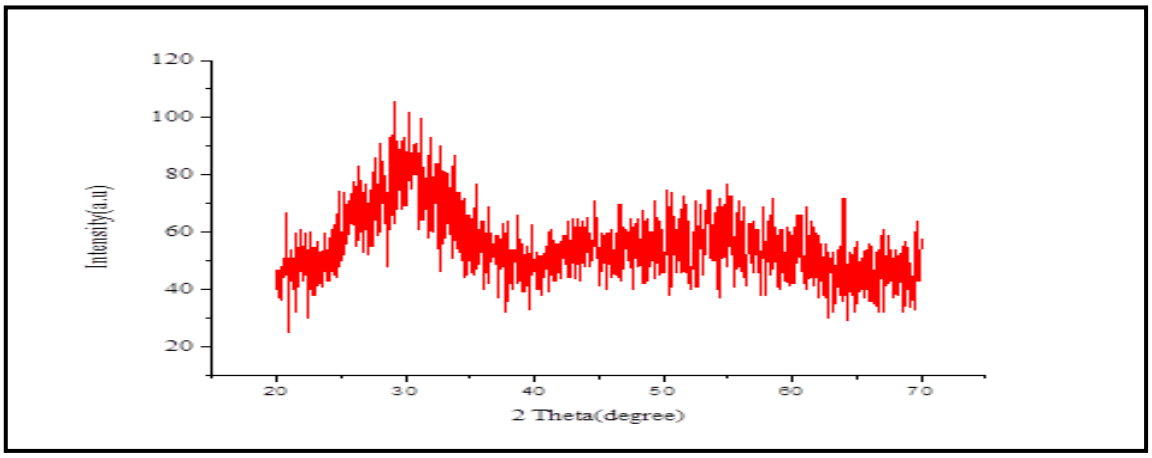


Figure 5.2.2 PL spectrum of Nanoparticles formed at 120º C

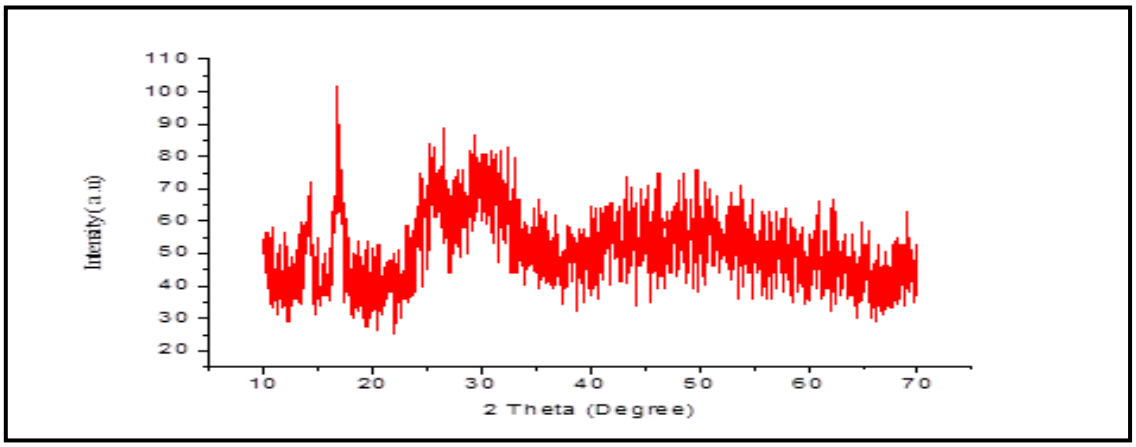
The PL spectra of the CdSe nanoparticle samples are as shown in Figure (5.2.1, 5.2.2).The peak is seen around 450nm. It is clear from the above results that As the concentration of the capping agent is increased the size of the nanoparticles gets reduced. The PL spectrum is shifted a little to lower wavelength as the size of the nanoparticles gets reduced.

There is a presence of the single peak for each and every sample.The capping agent worked efficiently is seen in these results .As there are no multiple peak present due to the defects. The capping agent work as the passivizing agent which reduces the surface defects of the semiconductor nanoparticles. The capping shell fill up gap on the surface of the nanoparticle and it will prevent the recombination of the trapped electrons inside the selenium vacancy with a hole in valence band of the CdSe nanoparticles.

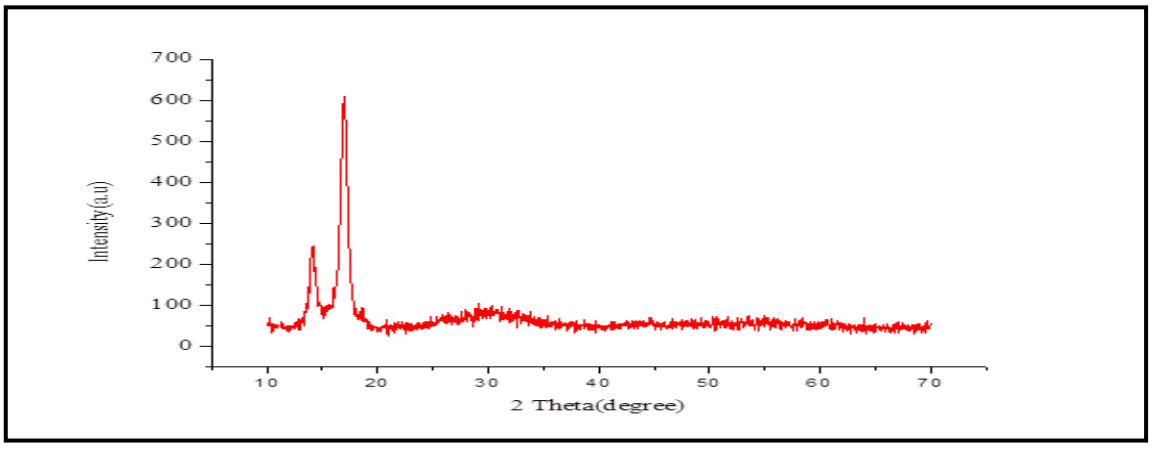
5.3 XRD RESULTS



(A)



(B)



(C)

Figure 5.3.1 Xrd graph (A) Sample A (B) sample B (C) sample C

Structural and phase analysis of prepared sample has been carried out using X-ray diffractometer (XRD) with Cu-Kα (1.5418 A^0)

The size of the nanocrystal is determined by the help of the XRD using the Scherer formula:

$$D=K\lambda/\beta \cos\Theta$$

D = average dimension of the crystallites normal to the reflecting planes
k = Scherer constant. K varies from 0.89 to 1
β = the integral breadth of a reflection (in radians 2θ) located at 2θ
λ=1.5418 A^o
Value of Scherer constant used is 0.89

Table 5.3.1 Nanocrystal size calculated by XRD data

Samples	2Θ	B=integral breadth	D=size of nanoparticle(nm)
A	**31.6**	**0.1**	**15.67**
B	**18.1**	**0.2**	**7.21**
C	**17.6**	**0.2**	**7.19**

It is clear from the result that the nanoparticles of smaller size are formed. The size of the nanoparticles gets reduced as the concentration of the capping agent is increased; this is in agreement of the results obtained by the help of other analysis techniques**.**

(4) SEM Results

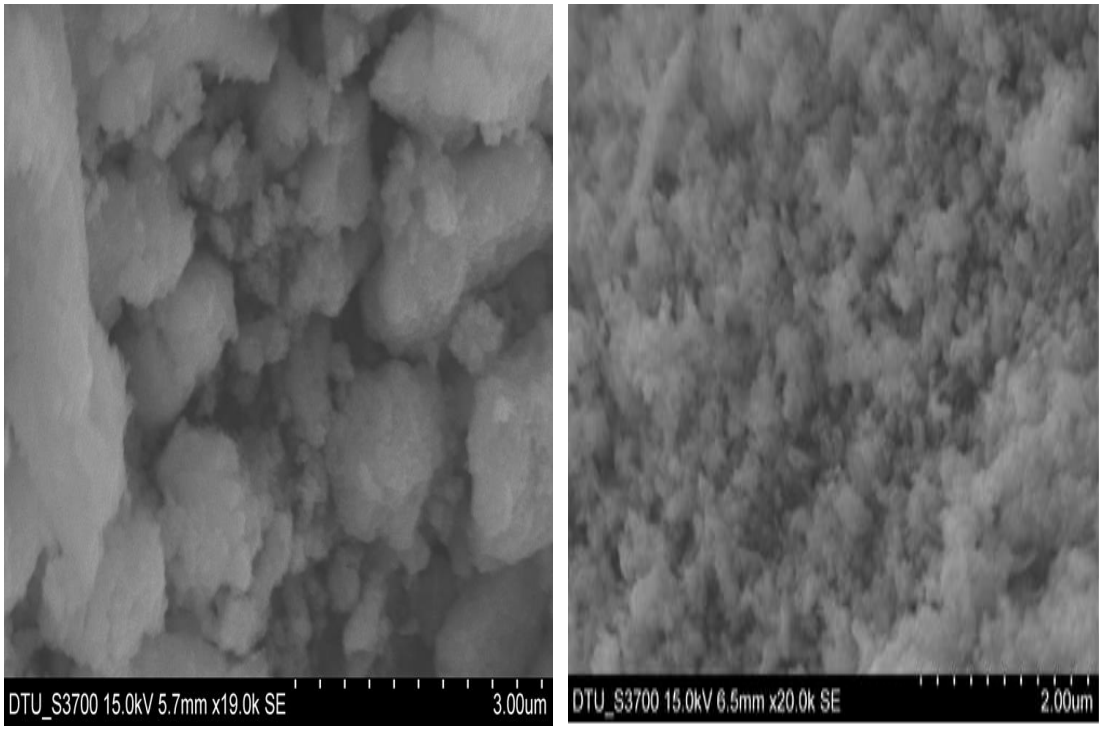


Figure 5.4.1 SEM Images of CdSe Nanoparticle (Sample A)

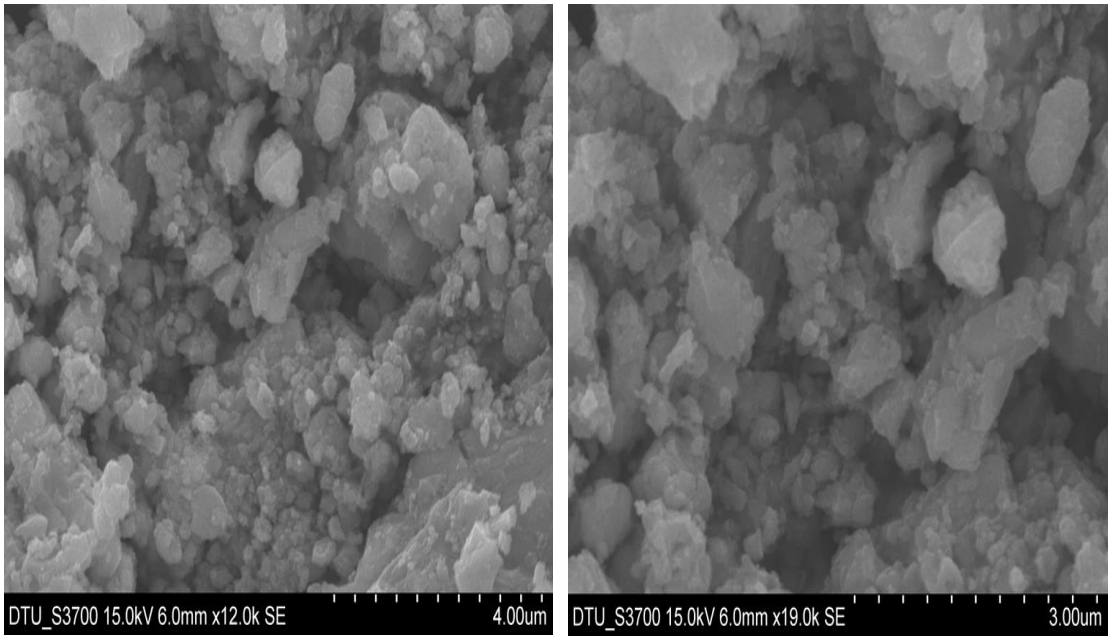


Figure 5.4.2 SEM Images of CdSe Nanoparticle (Sample B)

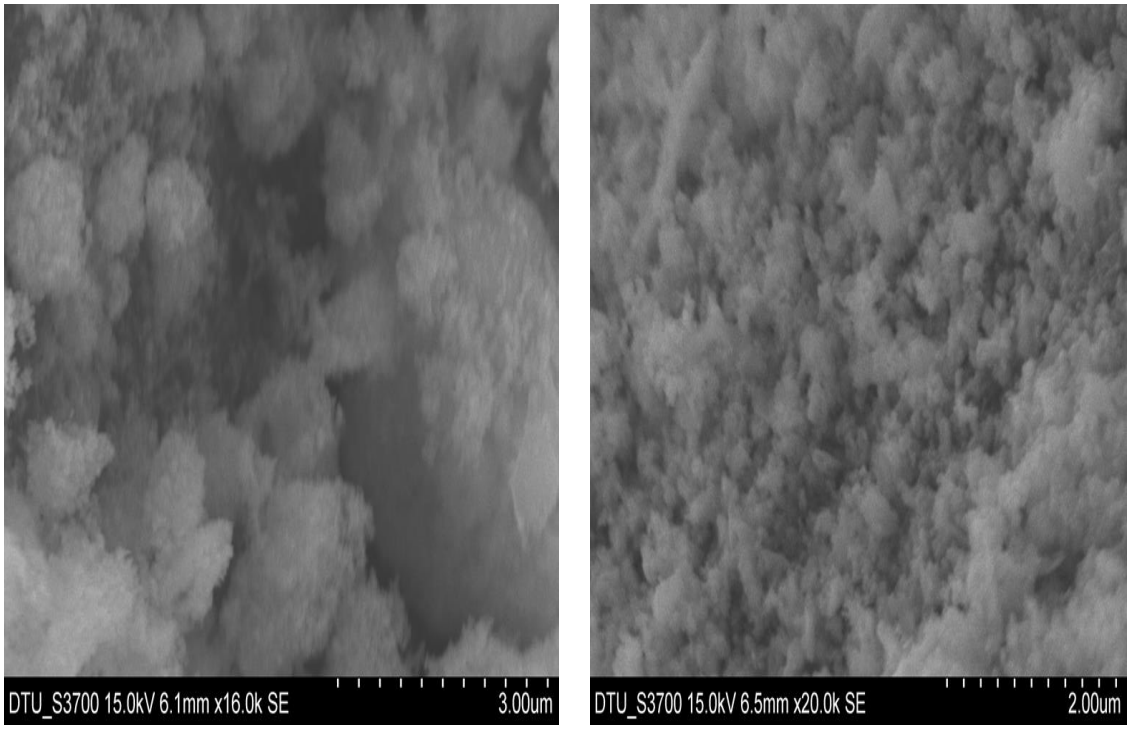


Figure 5.4.3 SEM Images of CdSe Nanoparticle (Sample C)

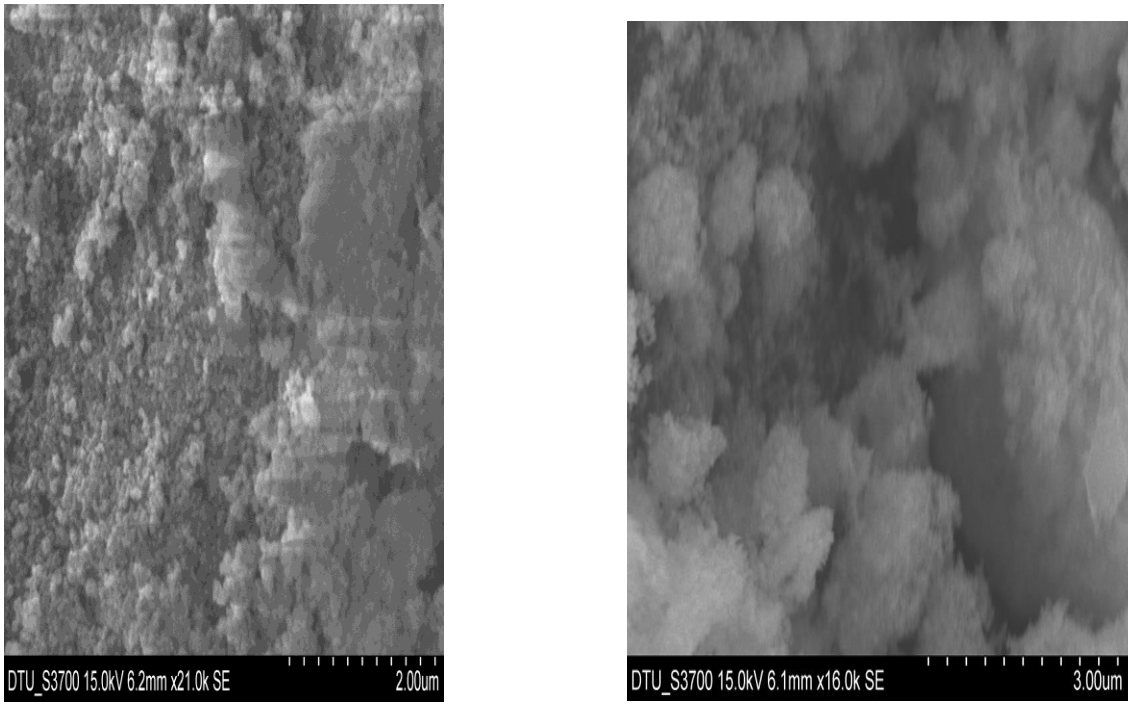


Figure 5.4.4 SEM Images of CdSe Nanoparticle (Sample A PVA film)

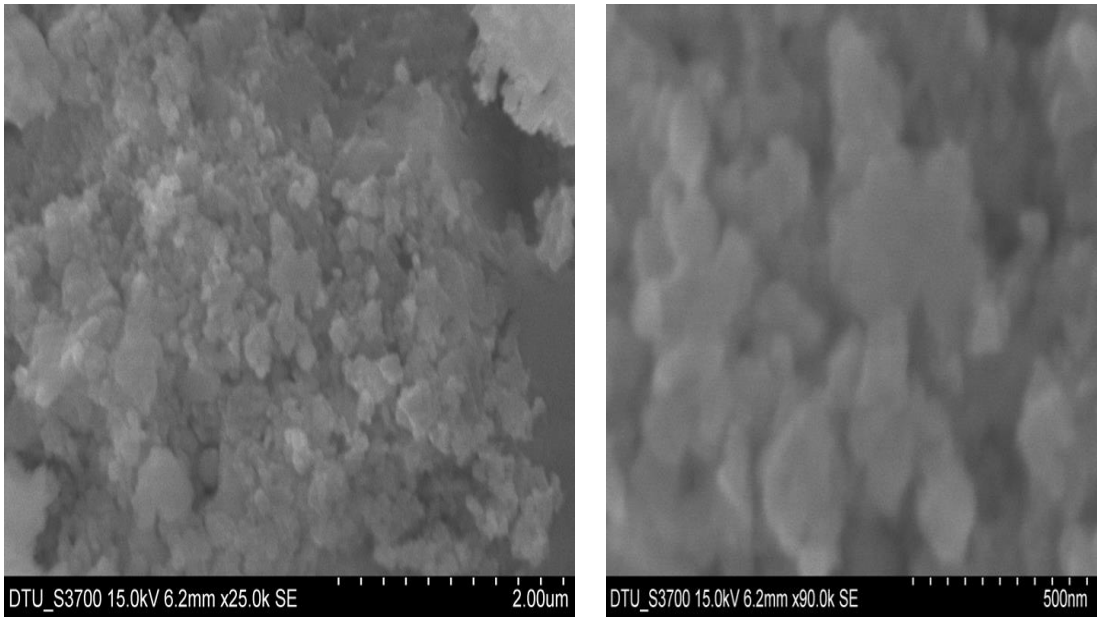


Figure 5.4.5 SEM Images of CdSe Nanoparticle (Sample B PVA film)

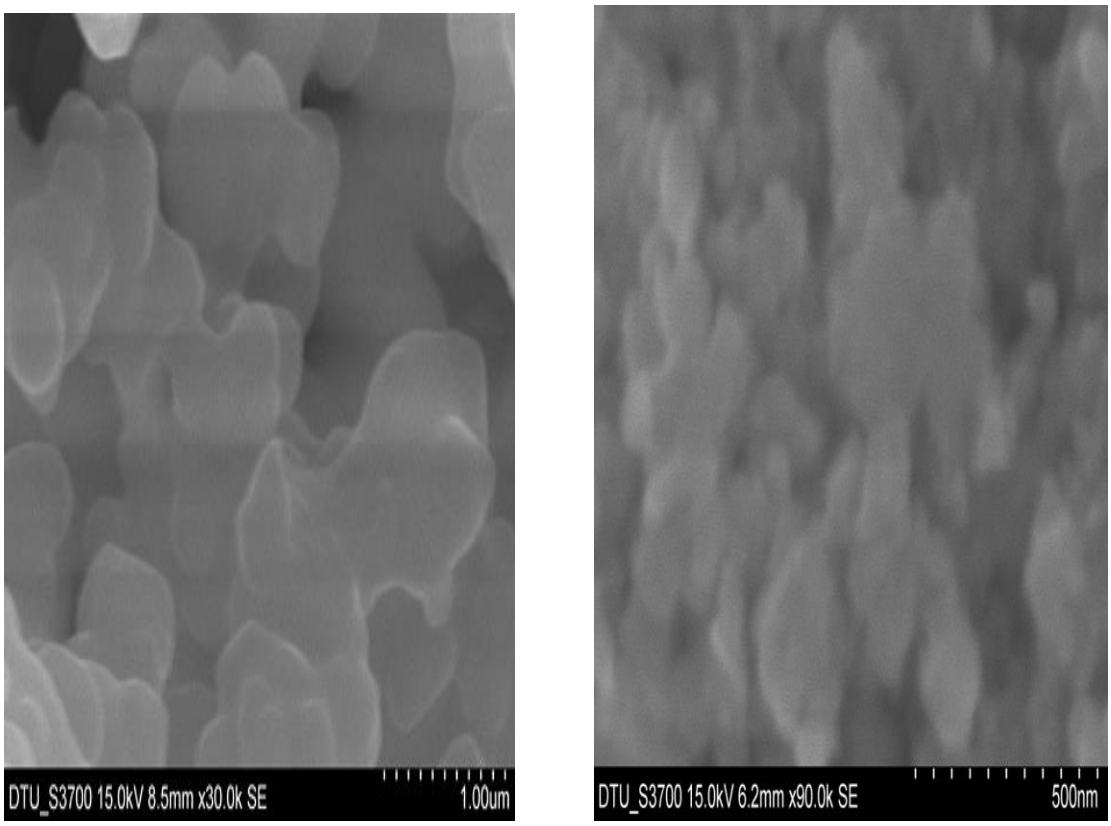


Figure 5.4.6 SEM Images of Cdse Nanoparticle (Sample C PVA film)

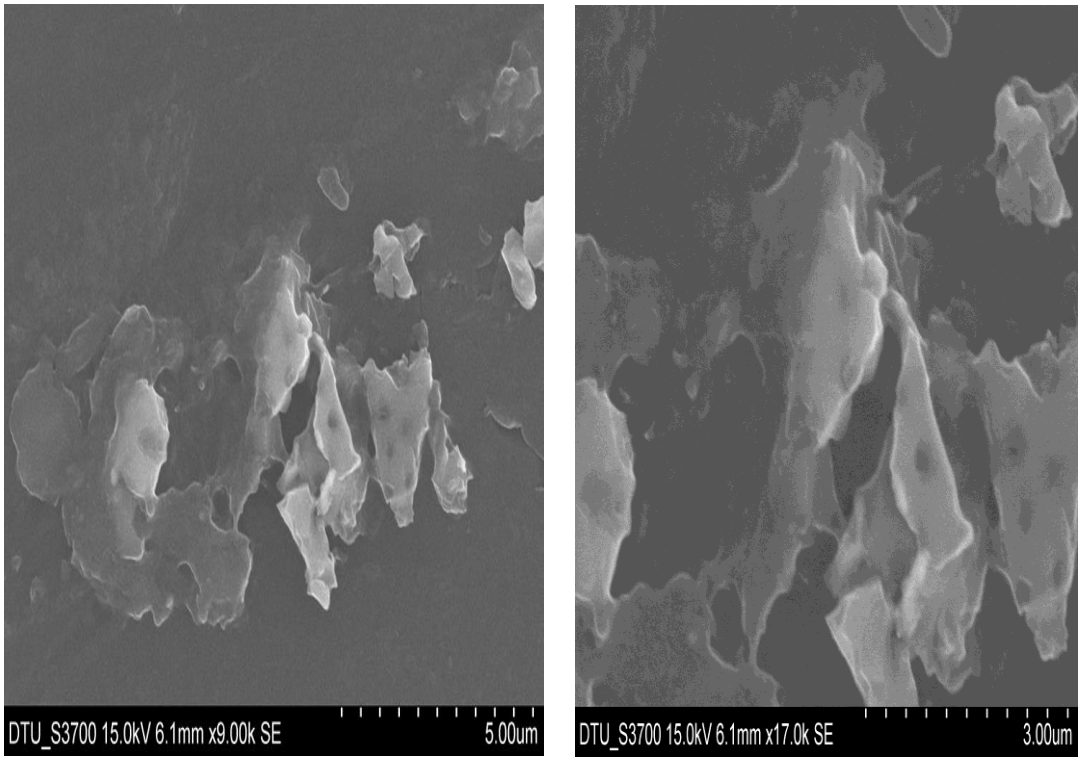


Figure 5.4.7 SEM Images of CdSe Nanoparticle PVA film formed by in-situ method

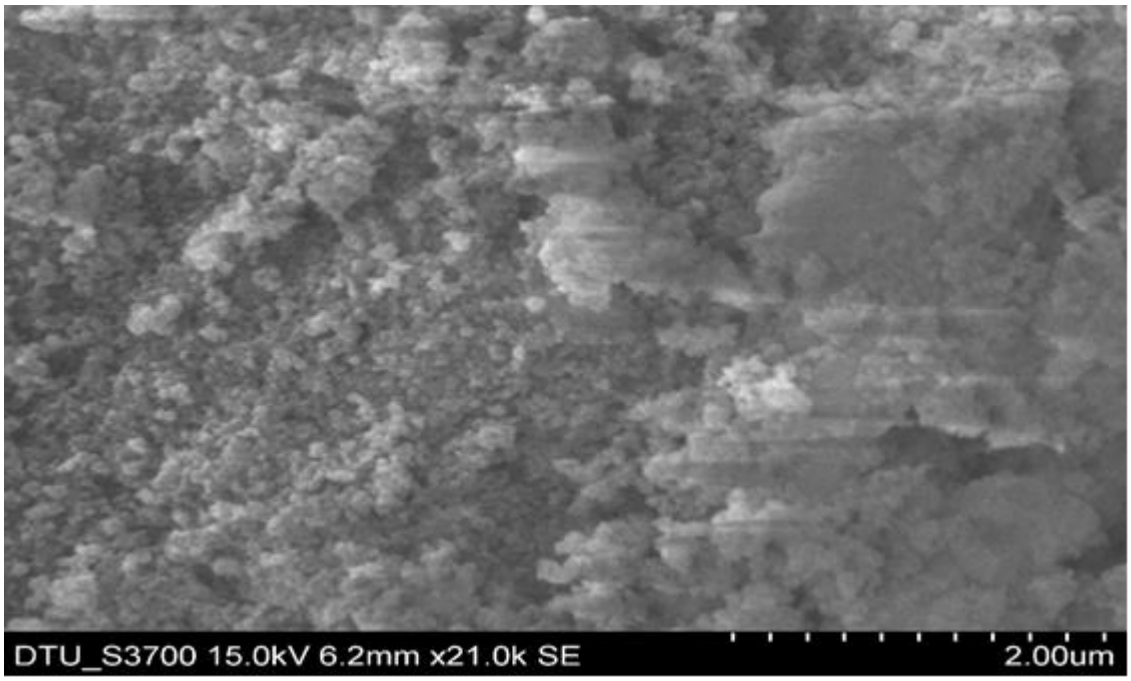


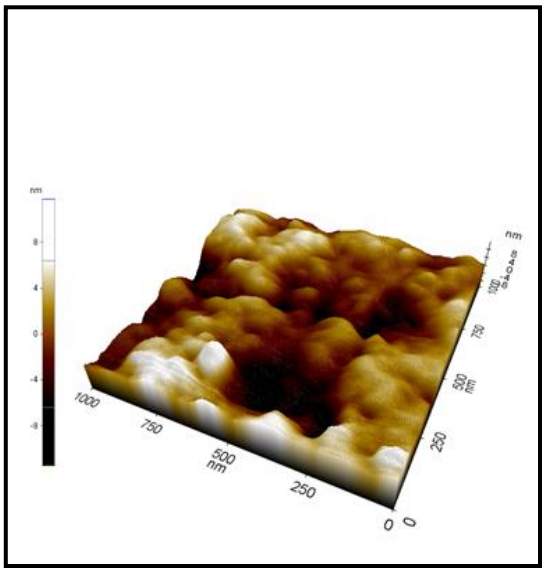
Figure 5.4.8 SEM Images of CdSe Nanoparticle PLA film

The surface morphology of the powdered nanoparticle sample , CdSe nanoparticles polyvinyl alcohol film formed by sample A,B,C and CdSe nanoparticles polylactic acid film were analyzed by the help of scanning electron microscope(SEM).The samples were studied at 15 kV with proper magnification.(Figure 5.4.1-5.4.8)

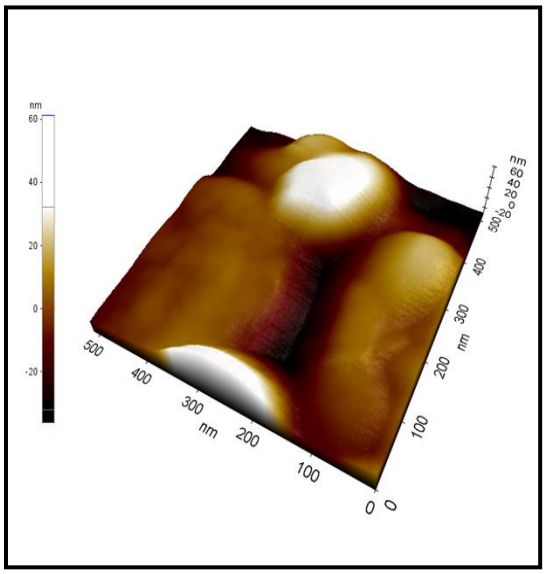
The morphology of the formed CdSe nanoparticles is not uniform and contains small irregular nanoparticles. SEM images show that the particles are not properly separated, they are generally agglomerated. It is clear from the images that the particles are formed which is visible. The images taken under magnification of 500 nm show the separation of the nanoparticle. It is clear from the results that the films formed by the ex-situ method show the proper dispersal of the nanoparticle on the surface of the film. Whereas the nanoparticle polymer film formed by the in-situ method show clear morphology but the aggregation of the nanoparticles in particular region and the seeded growth of the nanoparticle is seen. The structure and the surface could be well studied if the magnification was improved further.

Surface morphology of the Cdse nanoparticle polymer film clearly indicates that the nanoparticles are not well dispersed and rather they are accumulated.

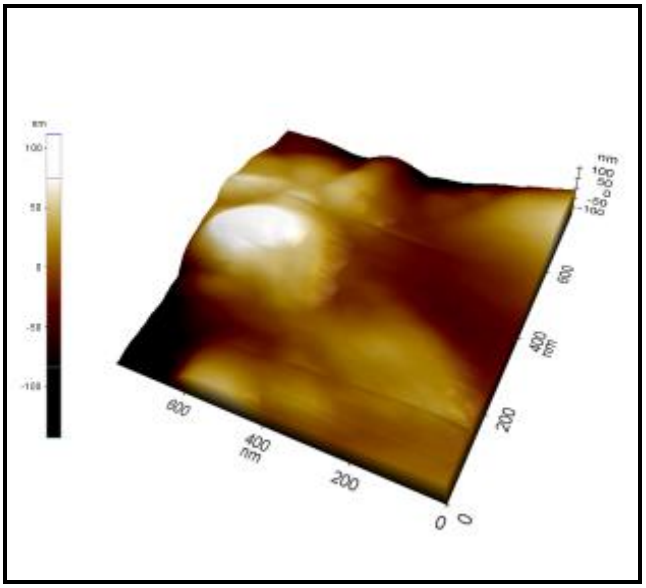
(5) AFM results



(Sample A)

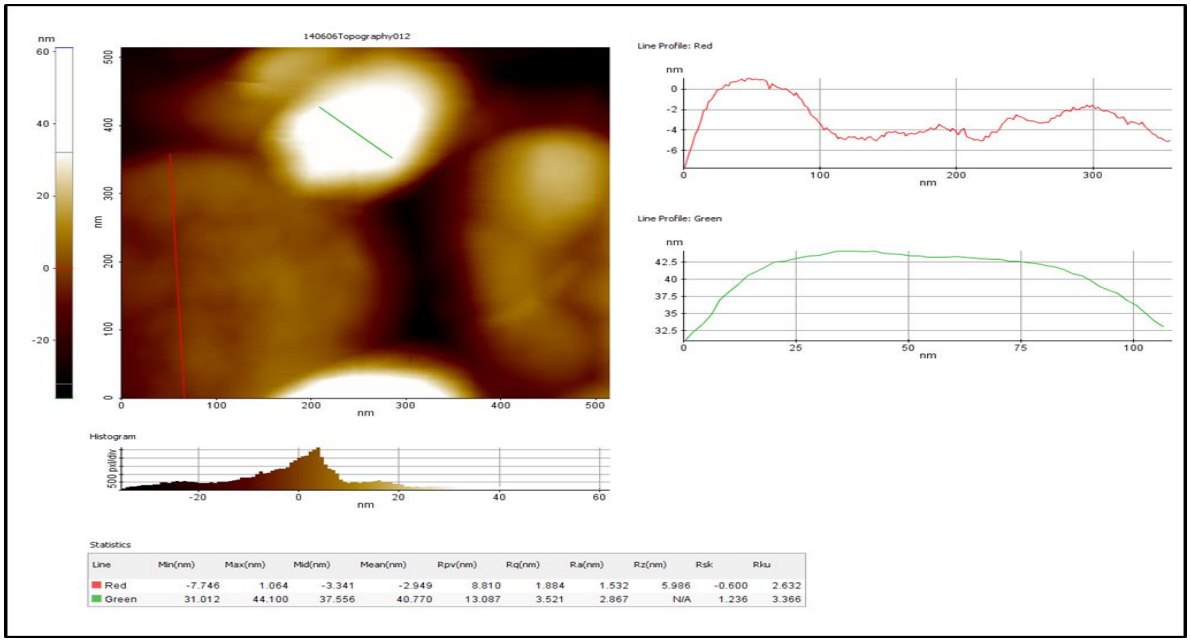


(Sample B)

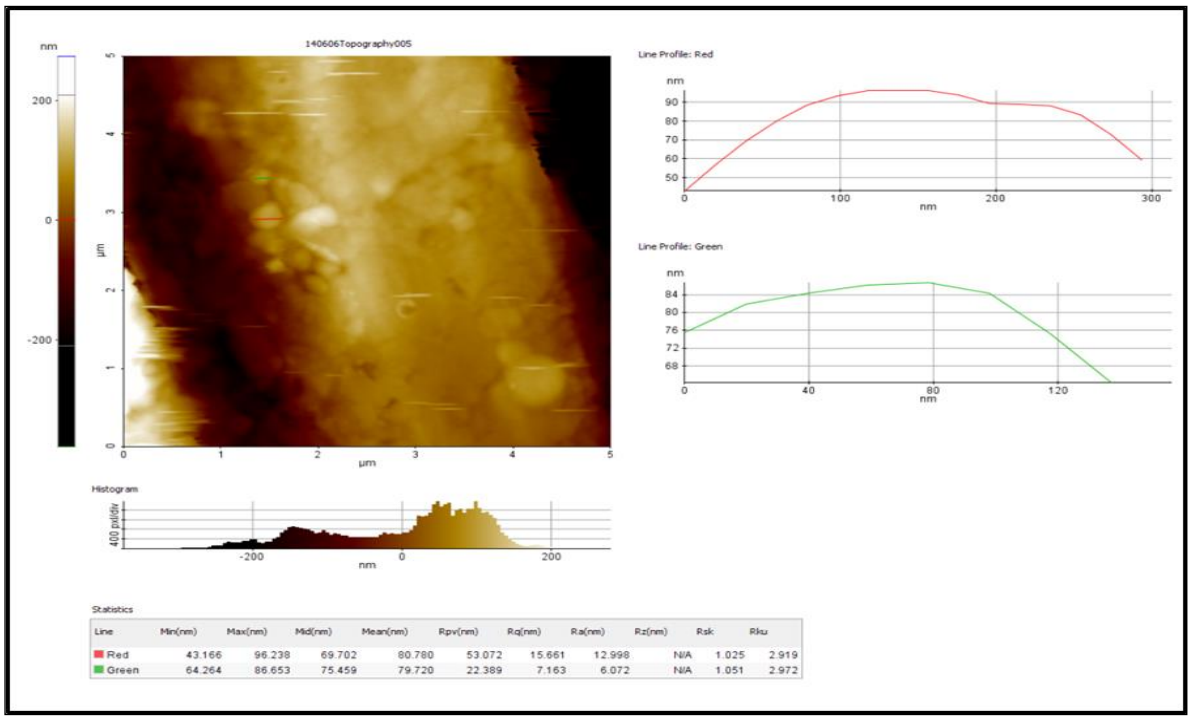


(Sample C)

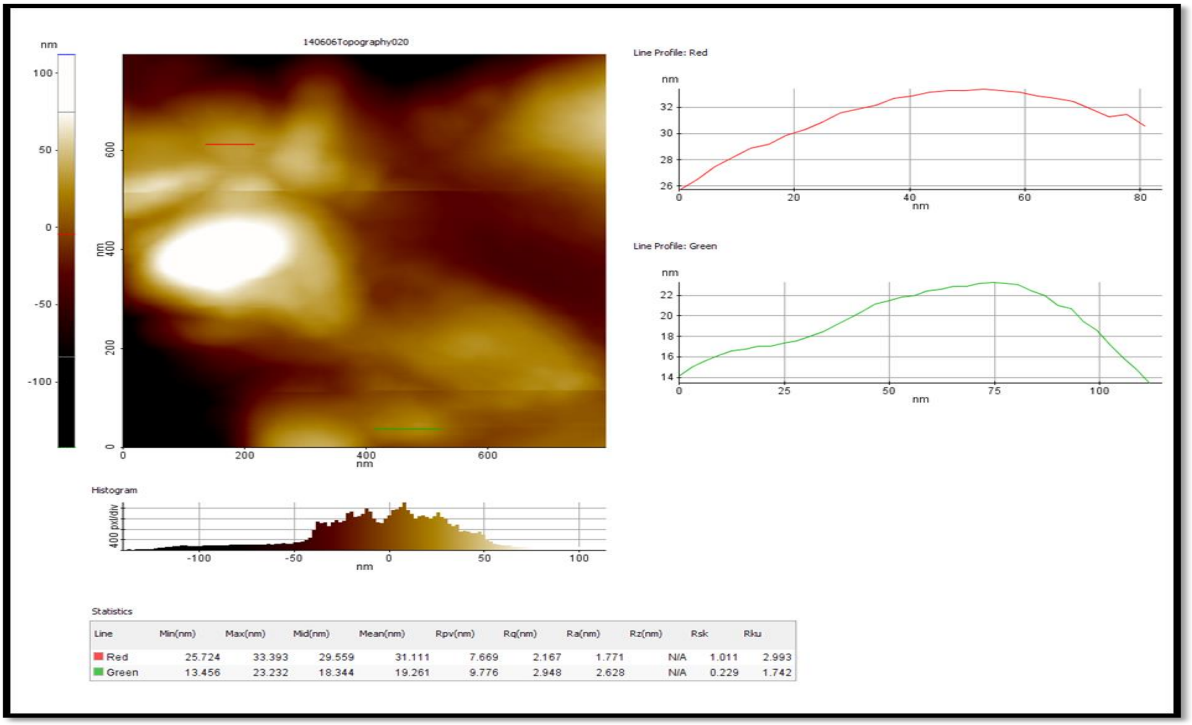
Figure 5.5.1 AFM Images of CdSe Nanoparticle



(a) Sample A PVA film



(a) Sample B PVA film



(c) Sample C PVA film

Figure 5.5.2 AFM Images of CdSe Nanoparticle PVA film

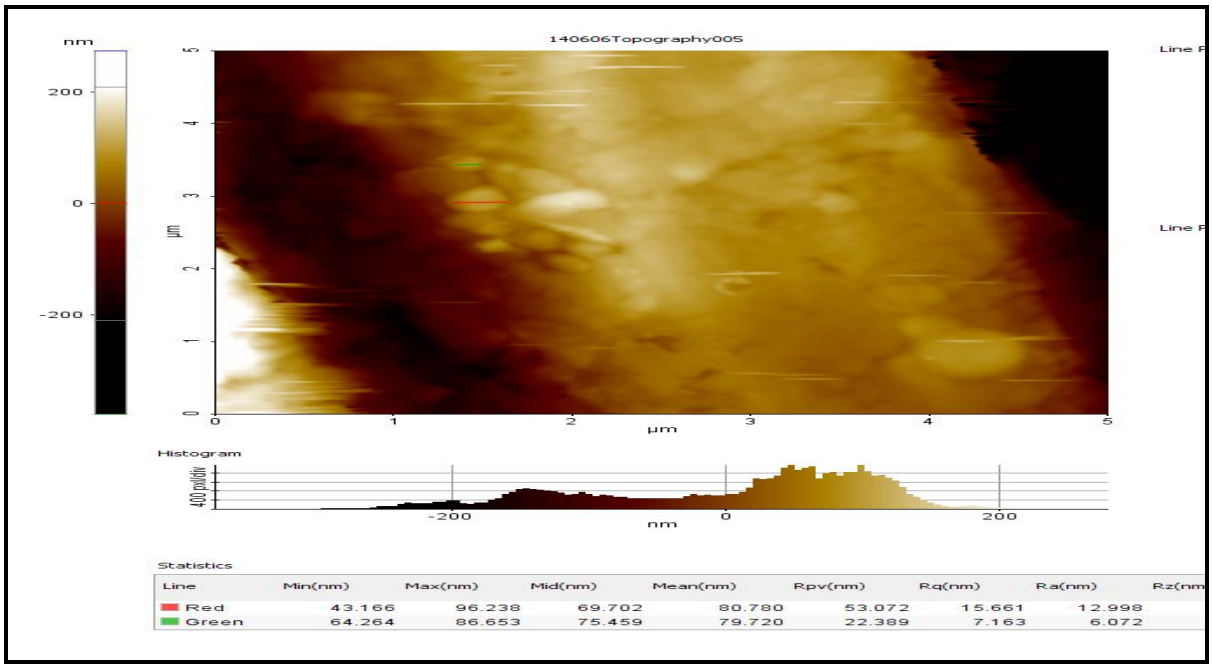
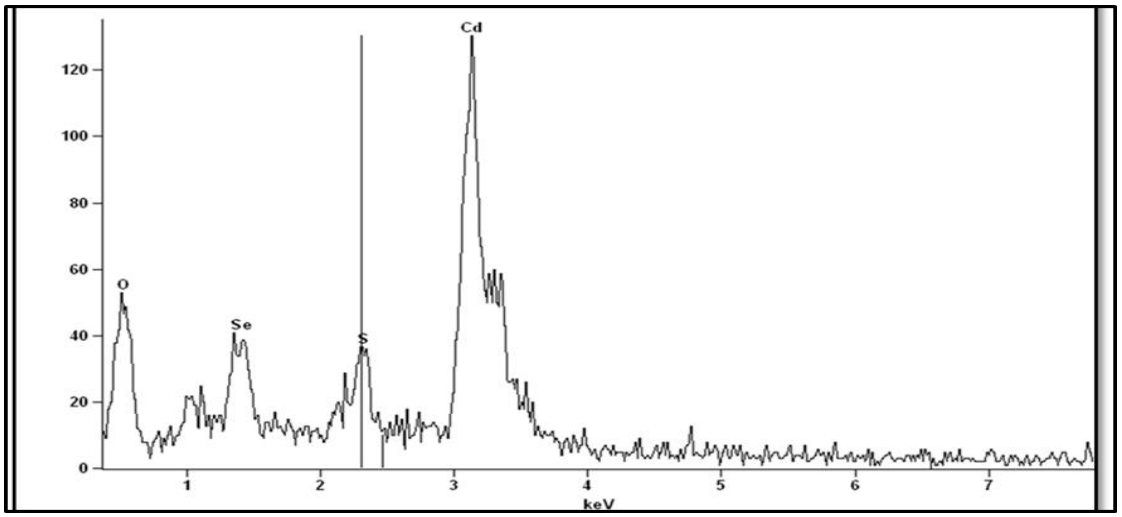


Figure 5.5.3 AFM Images of CdSe Nanoparticle PVA film (in-situ)

Atomic force microscopy was used for the analysis of the sample films formed. The images of all the samples show that there is a presence of the nanoparticles in the films both formed through the in-situ and the ex-situ method (Figure 5.5.1-5.5.3). The film matrix was found to have some nanoparticles which are embedded in the finely grained matrix.(Figure5.5.2 a-c). There is a presence of white color in the yellow background show the presence of the nanoparticles. The sample films formed by the ex-situ method shows the presence of the nanoparticles which are smaller in size as they are added externally and as there is a presence of the capping agent in these samples the size of the particles is more controlled as the surface defects of these particles have been removed. While nanoparticles formed through the in-situ method show that nanoparticles are more clustered to the particular area (Figure 5.5.3). The size of the nanoparticles formed externally is smaller as compared to the nanoparticles formed internally .The internal formed nanoparticles are bigger; they are generally greater than 50nm. The size ranges from 50- 100nm.The nanoparticles films can be tuned properly. The size resolution of the AFM machine is 5nm. Under the mentioned instrumental limitations, it can be concluded from both the SEM and AFM images that, the CdSe nanoparticles/aggregates of dimensions less than ~100 nm are formed.

(6) EDX results

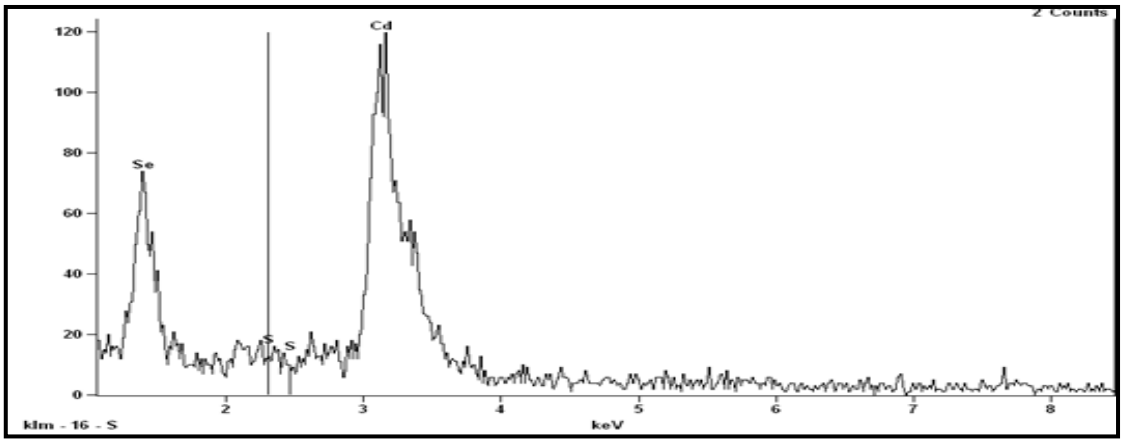


Acc.Voltage: 15.0 kV Take Off Angle: 77.9 deg.

Quantitative Results for: Base(394)

Element Line	Net Counts	Int. Cps/nA	Weight %	Weight % Error	Atom %	Atom % Error	Formula
O K	275	---	14.27	+/- 2.39	50.98	+/- 8.53	O
S K	273	---	2.75	+/- 0.33	4.90	+/- 0.59	S
S L	258272	---	---	---	---	---	
Se K	13	---	---	---	---	---	
Se L	480	---	8.91	+/- 1.04	6.45	+/- 0.75	Se
Cd L	2715	---	74.08	+/- 4.45	37.67	+/- 2.26	Cd
Cd M	0	---	---	---	---	---	
Total			100.00		100.00		

(a) CdSe nanoparticle Sample A PVA Film

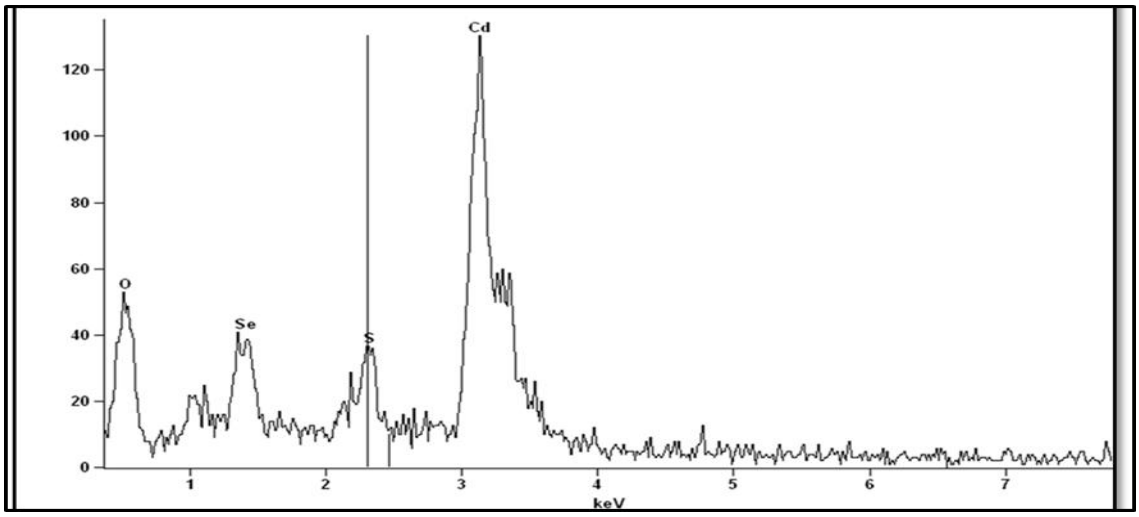


Acc.Voltage: 15.0 kV Take Off Angle: 78.2 deg.

Quantitative Results for: Base(393)

Element Line	Net Counts	Int. Cps/nA	Weight %	Weight % Error	Atom %	Atom % Error	Formula
O K	462	---	19.77	+/- 1.33	61.63	+/- 4.14	O
S K	38	---	0.34	+/- 0.27	0.53	+/- 0.42	S
S L	369926	---	---	---	---	---	
Se K	9	---	---	---	---	---	
Se L	780	---	12.65	+/- 1.25	7.99	+/- 0.79	Se
Cd L	2781	---	67.25	+/- 4.59	29.85	+/- 2.04	Cd
Cd M	0	---	---	---	---	---	
Total			100.00		100.00		

(b) CdSe nanoparticle Sample B PVA film

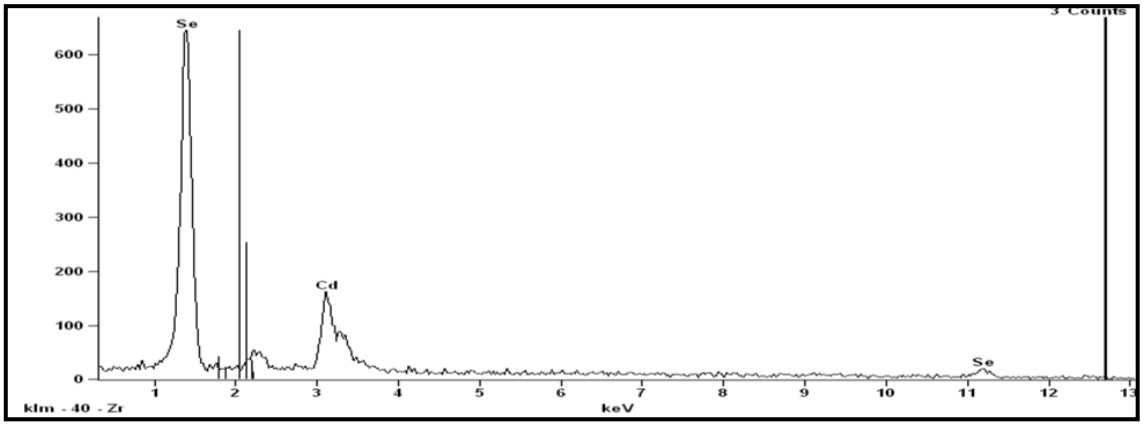


Acc.Voltage: 15.0 kV Take Off Angle: 77.9 deg.

Quantitative Results for: Base(394)

Element Line	Net Counts	Int. Cps/nA	Weight %	Weight % Error	Atom %	Atom % Error	Formula
O K	275	---	14.27	+/- 2.39	50.98	+/- 8.53	O
S K	273	---	2.75	+/- 0.33	4.90	+/- 0.59	S
S L	258272	---	---	---	---	---	
Se K	13	---	---	---	---	---	
Se L	480	---	8.91	+/- 1.04	6.45	+/- 0.75	Se
Cd L	2715	---	74.08	+/- 4.45	37.67	+/- 2.26	Cd
Cd M	0	---	---	---	---	---	
Total			100.00		100.00		

(c)CdSe nanoparticle Sample C PVA film



Element Line	Net Counts	Int. Cps/nA	Weight %	Weight % Error	Atom %	Atom % Error	Formula
Se K	310	---	---	---	---	---	
Se L	9375	---	61.24	+/- 1.11	69.22	+/- 1.26	Se
Cd L	3218	---	38.76	+/- 2.54	30.78	+/- 2.02	Cd
Cd M	0	---	---	---	---	---	
Total			100.00		100.00		

(d) **CdSe nanoparticle PVA film (in–situ)**

Figure 5.6.1 Elemental analysis by EDX

The elemental composition of the samples are determined by the help of the EDX.The samples A, B, C polyvinyl alcohol films were analyzed as shown in figure 5.6.1(a-c).It is clear from the images that there is presence of cadmium, selenium and small percentage of sulphur and oxygen .As these films are formed by the ex-situ method there is presence of the capping agent mercaptoethanol which is confirmed by the presence of the sulphur in the EDX analysis. Presence of oxygen is seen through these analysis which is due to polyvinyl alcohol present in the film sample and due to presence of capping agent.

Figure 5.6.1.(d) show the EDX analysis of the film formed by the in-situ method. The elemental analysis of this film shows the presence of cadmium and selenium.

(7) FTIR Results

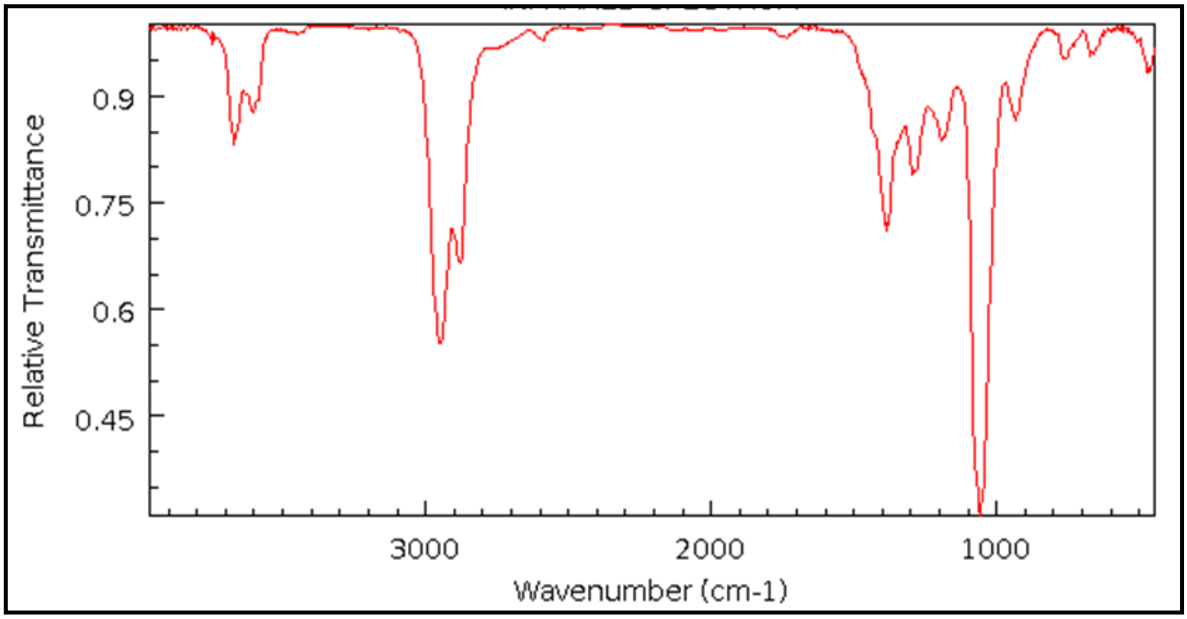


Figure 5.7.1 FTIR Spectra for pure mercaptoetanol

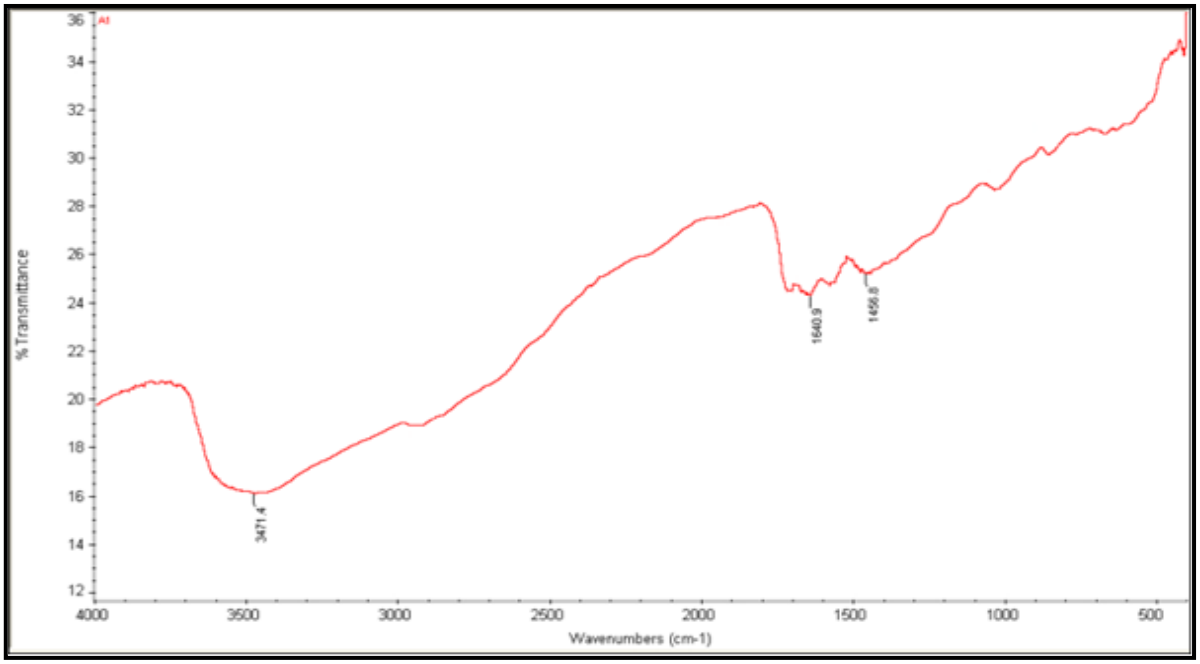


Figure 5.7.2 FTIR spectra for Synthesized CdSe Nanoparticles Capped with mercaptoethanol

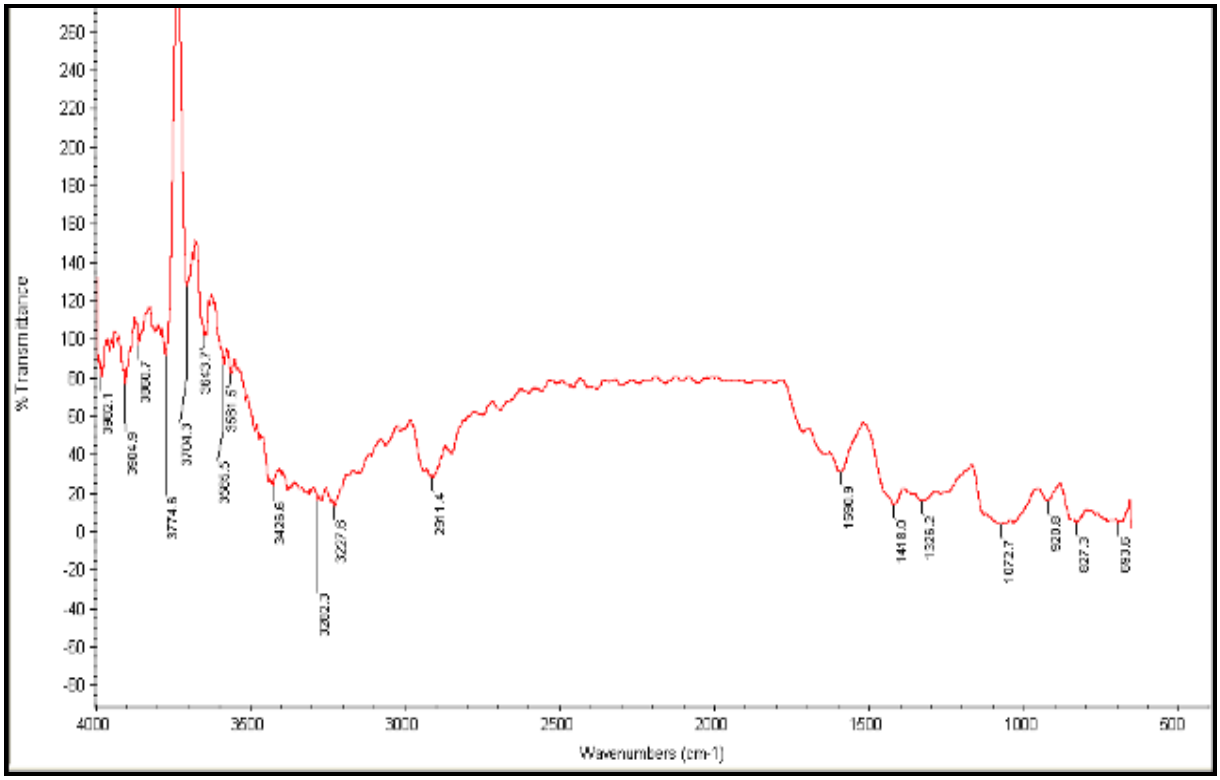


Figure 5.7.3 FTIR spectra for Synthesized CdSe Nanoparticles PVA film formed by in -situ method

The FTIR Spectrum of pure mercaptoethanol and CdSe nanoparticle capped with mercaptoethanol showed similar peaks. This indicates that the mercaptoethanol is attached to the surface of CdSe nanoparticles. . A peak is observed between ranges 3000-3500cm^{-1} represents OH stretching vibrational peak. Weak thiol peak is observed around 2500cm^{-1}. A CH-CH bending vibration is seen around 1400- 1600 cm^{-1} . It is believed that the mercaptoethanol present covers the surface defects of the CdSe nanoparticle formed and it improves the optical properties. It also helps in control the growth of the nanoparticles.The FTIR analysis of the sample film formed with polyvinyl alcohol and CdSe nanoparticles show that there is interaction between the nanoparticles and the film sample.

Application of CdSe nanoparticles Polymer Film

CdSe nanoparticles dispersed in polymer film show many unique properties which are useful and can be explored in wide areas. These areas include optical recording material, sensors, biotechnological application.Thus making it an important class of material for optics, photonic and biological industries of next century.

Optical properties of these nanoparticles as seen through the photoluminescence make these material important components for the integration with the devices. These material can convert light and electricity in tunable manner based on the crystal size ,this is a significant improvement over the silicon based devices.These improvements have led to imperative applications of these nanoparticles polymer in photovoltics,LED's,lasers.

These nanoparticles show a great enhancement in properties as comparison to the organic dyes. Organic dyes are susceptible to poor yield due to interaction with themselves or with solvent. The pasivated nanoparticles when protected show a better yield. Another disadvantage of these dyes is photobleaching and loss of the fluorescence ,it is reduced in these material as same passivating layer, that enhances quantum yield, also protects particles from external interactions .Emission band width of dyes is large which makes it difficult to separate different colors whereas it is narrow in these nanoparticles embedded polymer film.All these properties make these materials application diverse in biological field like biosensing,cellular labelling and therapeutics.

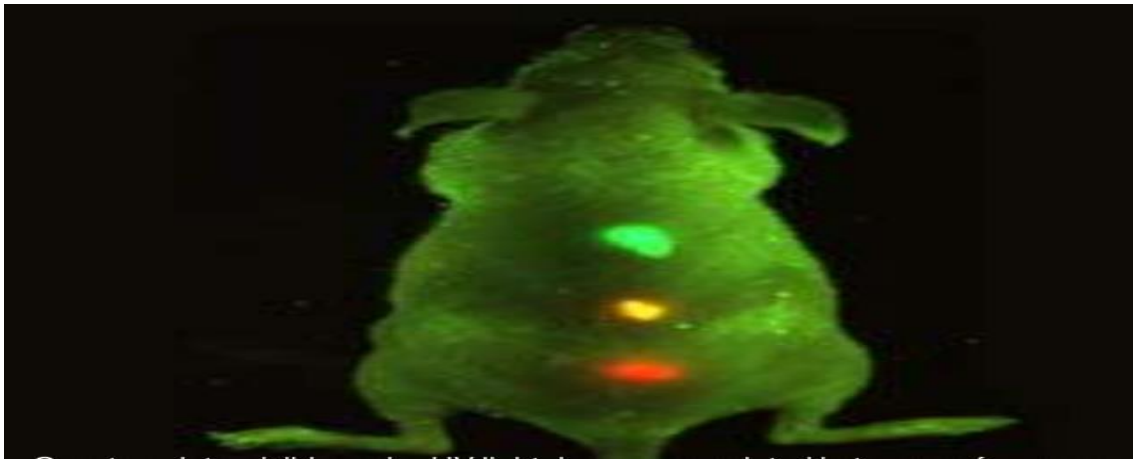


Figure 5.7.4 CdSe nanoparticle visible under UV showing tumor cell of mice

Conclusion

CdSe nanoparticles of variable sizes were synthesized by the solvothermal route. The study shows that the smaller size nanoparticle can be prepared by increasing the capping agent concentration. These nanoparticles were studied for the optical property by analyzing the UV-Visible spectra. The optical band gap of the nanoparticle increases as the size of the nanoparticles gets reduced. The peak is blue shifted as the concentration of the capping agent is increased. The exact mechanism for the formation of CdSe nanoparticles is still unclear, but it is reasonably concluded that the appropriate ratio of solvents volume may play the significant role for the formation of CdSe nanoparticles.The nanoparticles polymer film was synthesized by dispersing these nanoparticles in the PVA and PLA as matrix. The films synthesized were analyzed by the help of Atomic Force Microscope (AFM) and Scanning electron microscope (SEM). It is clear that the nanoparticles are dispersed on the surface of polymer film. PVA film formed was synthesized show a good morphology as compared to the PLA film. The PVA film formed with adding nanoparticles externally by the help of ex-situ method show that there is a better control of the size of the nanoparticles and the stability is more as the capping agent passivizes the surface defect present in the sample and hence the optical property could be tuned properly whereas the nanoparticles polymer film prepared by the in-situ method show that the particle size control is less though it could be control by changing the concentration of the precursors. The surface defects arise more in the in-situ film which restricts the practical use of these films. The nanoparticle PLA film though formed by the ex-situ method by the addition of the particles externally, but the film formed is not stable and especially at higher temperatures which shows that further study is required to increase the stability of the nanoparticle PLA film by the addition of additives.

Future Prospect

An easy way to manipulate the nanoparticles for their application is by incorporation of these particles inside the processable polymer film. This study will help in understanding the formation of the CdSe nanoparticle and the compatibility with the polymers. A wide variety of the colors are obtained from the single material by just altering the capping agent concentration thus changes the particle size. With these feature it is possible to study the behavior of changing particle size in application in biosensensing and biolabelling by combining them with the biodegradable polymers. These nanoparticles when combined with the photostable polymers can be used for the catalysts in photochemical reaction. Optics, catalysis, bioelectronics are some of the major areas where these CdSe nanoparticles polymer film could be well explored.

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