

SYNTHESIS AND OPTICAL CHARACTERIZATION OF QUANTUM DOT SEMICONDUCTOR NANOPARTICLES

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Abstract

The manufacture and optical analysis of quantum dot semiconductor nanoparticles have garnered a significant amount of interest due to the unique quantum confinement characteristics that these nanoparticles possess. These features make them exceptionally well-suited for applications in the fields of optoelectronics, biological imaging, and photovoltaics technology. As part of this study, quantum dots were created by the use of a wet chemical method, which enabled the researchers to exercise exact control over the particle size and achieve great crystallinity. X-ray diffraction (XRD) was used to investigate the structural properties, and the results supported the formation of nanocrystalline phases. Transmission electron microscopy (TEM) was used to gain insights into the uniform shape and size distribution of the nanocrystalline phases. A combination of UV-Vis absorption and photoluminescence (PL) spectroscopy was utilised in order to evaluate the optical properties. The results of this analysis revealed variations in bandgap that are dependent on size and tunable emission spectra. It was established that the quantum dots that were synthesised exhibited a high quantum yield and a robust fluorescence, which highlights their potential for applications that include light emission. In addition, Fourier-transform infrared spectroscopy (FTIR) was utilised in order to explore surface functional groups.

Keywords: quantum dot , semiconductor, nanoparticles, Transmission electron microscopy

Introduction

Quantum dot semiconductor nanoparticles have garnered significant attention due to their unique optical and electronic properties, making them highly valuable for applications in optoelectronics, bioimaging, and photovoltaics. The synthesis of these nanoparticles generally involves colloidal methods, such as hotinjection and hydrothermal techniques, which enable precise control over size, shape, and composition. The quantum confinement effect, which arises when the particle size approaches the exciton Bohr radius, leads to size-dependent optical properties, including tunable photoluminescence. Characterization of these nanoparticles involves several techniques, including UV-Vis spectroscopy for absorption studies, photoluminescence spectroscopy for emission properties, X-ray diffraction (XRD) for crystallinity and phase identification, and transmission electron microscopy (TEM) for morphological analysis. Additionally, dynamic light scattering (DLS) is employed to determine hydrodynamic size distribution, while Fourier-transform infrared (FTIR) spectroscopy helps analyze surface functionalization. The optical bandgap of quantum dots can be estimated using Tauc's plot, derived from UV-Vis absorbance data, which provides insights into electronic transitions. Fluorescence lifetime measurements using time-resolved photoluminescence spectroscopy further aid in understanding carrier dynamics and recombination mechanisms. The stability of quantum dots can be enhanced by surface passivation with organic ligands or inorganic shells, reducing nonradiative recombination pathways. These advancements in synthesis and characterization methods contribute to the development of high-performance quantum dots for applications in LEDs, solar cells, and medical diagnostics.

The quantum confinement effect causes quantum dots, which are extremely small semiconductor particles, to display unique optical and electrical properties. Quantum dots are a result of this phenomenon. It is also possible to refer to quantum dots as quantum dots. The discrete energy levels that are produced as a result of their microscopic size, which typically ranges from 2 to 10 nanometres, make it possible to adjust photoluminescence in a manner that is reliant on the particle size. This is because the particle size directly affects the energy levels that are produced. Quantum dots are extremely valuable in a wide range of applications due to the fact that they exhibit these features. Some of these applications include bioimaging, optoelectronics, quantum computers, and photovoltaics.

The production of quantum dot semiconductor nanoparticles is performed by the use of a wide variety of chemical and physical processes, including hydrothermal methods, hot-injection techniques, and colloidal synthesis, amongst others. The size, shape, and composition of the particles can all be precisely controlled using these approaches, which in turn have a direct influence on the optical and electrical properties of the particles. This makes it feasible to exercise precise control over the many aspects of the particles. Additionally, in order to improve the functionality and stability of quantum dots for specific applications, a number of other methods, such as surface passivation and ligand exchange, are implemented on a regular basis.

In terms of their optical properties, quantum dots are primarily determined by the quantum confinement effect, which is the major factor that governs these features. The occurrence of this phenomenon makes it possible for quantum dots to possess absorption and emission spectra that are dependent on the size of the quantum dots. When it comes to optical qualities, the characteristics of photoluminescence, fluorescence quantum yield, bandgap energy, and carrier recombination kinetics are all quite important. In applications such as light-emitting diodes (LEDs), solar cells, and medical imaging, where traits such as poor luminescence efficiency and high luminescence stability are necessary, these characteristics find broad application.

Characterisation A wide range of spectroscopic and microscopic techniques are utilised in the process of characterisation of quantum dots. These techniques are designed to investigate the structural, optical, and electrical properties of quantum dots. Quantification of absorption spectra and determination of bandgap energy are both accomplished through the use of ultraviolet-visible (UV-Vis) spectroscopy, whilst photoluminescence (PL) spectroscopy is utilised for the purpose of analysing emission properties. In contrast to transmission electron microscopy (TEM), which provides information on particle size and morphology, X-ray diffraction (XRD) provides information about crystallinity and phase composition. In addition, several methods, including dynamic light scattering (DLS) and Fourier-transform infrared (FTIR) spectroscopy, are utilised in order to explore surface chemistry and particle dispersion in colloidal solutions.

A Brief Quantum Dot Semiconductor Nanoparticles

A revolutionary class of nanomaterials, quantum dot semiconductor nanoparticles (QDs) are defined by extraordinary electrical and optical properties that result primarily from quantum confinement phenomena. The size-dependent bandgap of these semiconductor nanoparticles—which typically range from 1 to 10 nm in diameter—allows for tunable emission in the visible and near-infrared regions of the electromagnetic spectrum. Their exceptional photostability, narrow emission bandwidth, and high photoluminescence quantum yield make them ideal for use in optoelectronic devices, biosensing, imaging, and energy harvesting, among other fields. To enhance quantum dots' performance in practical settings, reliable and scalable synthesis methods for controlling their size, composition, and surface chemistry are needed. Optical characterisation techniques are also essential for understanding the electrical and structural properties of QDs, which helps in optimising them for certain technical advancements.

Importance of Synthesis in Quantum Dot Engineering

The method of synthesis is an essential component that plays a significant role in determining the dimensions, morphology, crystallinity, and surface features of quantum dots. These qualities, in turn, have an impact on the optical and electrical properties of quantum dots. The manufacture of quantum dots has been accomplished by the utilisation of a wide range of chemical and physical procedures, such as colloidal synthesis, sol-gel techniques, hydrothermal processes, and chemical vapour deposition. When it comes to these techniques, colloidal synthesis is particularly favoured due to its capacity to produce monodisperse nanoparticles that have a regulated size distribution and an effective surface passivation. Important parameters, such as the temperature of the reaction, the concentration of the precursor, and the use of capping agents, have a substantial impact on the quality and stability of the quantum dots that are produced. Furthermore, the incorporation of dopants and the development of heterostructures, such as core-shell designs, have the potential to further improve their luminous properties and boost their stability in a variety of conditions, thereby making them more relevant for practical applications.

Objective

- 1. To colloidally synthesise CdSe quantum dot semiconductor nanoparticles with controllable size and composition.
- 2. To study the optical characteristics of synthesised quantum dots using absorption and photoluminescence spectroscopy.

Method

The Metters method was used to synthesise quantum dot semiconductor nanoparticles with exact size and optical properties using high-purity reagents. Cadmium oxide (CdO, Fluka, 99%) was used as a precursor, while sulphur powder (S, Riedel, 99%) and selenium powder (Se, Riedel-de Haën, 99%) were used as chalcogen sources for cadmium chalcogenide quantum dots. Oleyl amine (97%) and oleic acid (CH₃(CH₂)₇CH=CH(CH₂)₇COOH, Aldrich, 98%) were utilised as capping agents to stabilise and regulate nanoparticle development. Trioctylphosphine (TOP, Aldrich, 97%) and TOPO (Fuka, 97%) were coordinating solvents that dissolved precursors and improved quantum dot crystallinity. These chemicals in the Metters process produced high-quality quantum dots with specific optical characteristics and enhanced stability, making them appropriate for optoelectronic and bioimaging applications.

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Results and discussion

Optical Characterization Techniques for Quantum Dots

Spectroscopic techniques, which reveal the electronic structure and excitonic behaviour of quantum dots, are the primary means by which the optical properties of quantum dots are evaluated. Analysis of quantum confinement effects and estimation of particle size can be accomplished through the use of ultravioletvisible (UV-Vis) absorption spectroscopy. This technique involves analysing shifts in the absorption edge. One of the most common uses of photoluminescence (PL) spectroscopy is the investigation of emission properties. These variables include peak wavelength, intensity, and quantum yield, all of which are essential for applications in light-emitting diodes (LEDs) and bioimaging. In order to determine the surface functional groups and ligand interactions that have an effect on the solubility and stability of quantum dots, Fourier-transform infrared spectroscopy (TEM) are utilised. In addition, X-ray diffraction (XRD) and transmission electron microscopy (TEM) are utilised in order to validate the crystalline structure and morphology, respectively. Additionally, time-resolved photoluminescence measurements are utilised in order to gain an understanding of the carrier dynamics and recombination processes. A comprehensive study of these optical properties makes it possible to precisely optimize quantum dots for a wide range of technological applications as a result of this understanding.

Applications of Quantum Dot Semiconductor Nanoparticles

The extraordinary optical and electrical capabilities of quantum dots have made it possible for them to be utilised in a wide variety of scientific and industrial fields. Quantum dots are widely used in the field of optoelectronics, specifically in quantum dot-based light-emitting diodes (QLEDs), display technologies, and solar cells. This is primarily owing to the outstanding colour purity and efficiency in energy conversion that quantum dots possess. When it comes to biomedical applications, quantum dots serve as highly sensitive fluorescent markers for bioimaging, drug administration, and biosensing. These quantum dots offer substantial advantages over typical organic fluorophores due to their increased brightness and photostability. Quantum dots have the ability to improve light absorption and the separation of charge carriers in photovoltaic applications, which ultimately leads to increased solar cell efficiency. Furthermore, the fact that their electrical properties can be altered has made it possible to experiment with new possibilities in the field of quantum computing and single-photon emission devices. Continuous advancements in synthesis and characterisation techniques are driving the development of next-generation quantum dot technologies, which in turn is supporting the commercialisation of these applications across a variety of industries.

Synthesis of CdSe Quantum Dots

The procedure involved making small adjustments to a method first described by Peng et al. in order to synthesise luminescent CdSe QDs of various colours. The 100 ml three-necked flask was prepared by adding 1.2 ml of oleic acid and 0.12 g of CdO. The flask was fitted with a thermometer and a N2 inlet, and it was subjected to reflux while being vigorously stirred magnetically with a Teflon-coated magnetic stir bar. To generate a reactive Cd species, the liquid in the reaction vessel is heated to 1250 C. Optical observation reveals a colour shift from turbid violet-red to pale yellow, and finally to a clear, colourless homogenous solution. This happens because the CdO is entirely dissolved and the oleic acid is complexing

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with Cd. Typically, the red colour will fade away after around fifteen minutes. After the red colour had faded at 130°C, 2 millilitres of Oleyl amine and 2.0 grammes of TOPO were added to the reaction vessel. The reaction temperature was set between 200 and 220°C. Inject 3.0 ml of Se in TOP (0.12 gm Se into 3.0 ml TOP to coordinate Se) into the flask under vigorous stirring as soon as the temperature reaches 200 °C, after which the heat mantle is removed. The requisite size of the nanocrystals is determined visually by looking at the colour of the liquid, and samples of the reaction mixture are taken at intervals corresponding to that size into a cold organic solvent (e.g., hydrophobic toluene or hexane). Depending on the precise temperature, the mixture's colour changed from transparent and colourless to green, yellow, orange, or red. The capping substance now stabilises the CdSe NCs against aggregation, as they are prepared. The NCs are made to precipitate when nonsolvent methanol is added, since it is miscible with the initial dispersing solvent and destabilises them. Centrifugation is used to collect NCs that have precipitated from non-reacting precursors. The mixture is then washed with chloroform 2-3 times before being redistributed in toluene or hexane.

An organometallic pyrolysis process was developed for the preparation of CdSe nanocrystals. Organometallic precursors are introduced into a TOPO or long-chain alkyl amine-type hot coordinating solvent in this synthesis process. The experimental section demonstrates how this technology has developed over the past decade to create a wide range of high-quality materials in colloidal solutions, including II-VI semiconductor nanocrystals (e.g., CdSe) and CdTe. For the purpose of creating CdSe nanocrystals, it is necessary to combine the precursor reagents in a controlled environment in order to ensure a continuous and uniform chemical reaction.

Every atomic species that would eventually make up the NC is first introduced to a reactor as a precursor in a generic synthesis. A molecule or complex that contains one or more atoms species that represent the building blocks of NC is called a precursor. To initiate the nucleation and subsequent growth of the NCs, the precursors are added to the reaction flask, where they undergo reactions or breakdowns to produce the reactive monomers. It is the reactor's liquid that supplies the energy needed to crystallise the nanoparticles and decompose the precursors, whether by thermal collisions, a chemical reaction between the liquid and the precursors, or a mix of the two. Due to its inexpensive cost, reduced toxicity, and nearly excellent quality, CdO has been utilised as a Cd precursor. When it comes to controlling the size and form of colloidal NCs during growth, the presence of organic molecules in the reactor, commonly referred to as "surfactants," is the most important factor. In their molecular structure, surfactants have both a hydrophilic (containing a polar or charged functional group) and a hydrophobic (containing, at its most basic, one or more hydrocarbon chains) component. In instance, there are numerous instances where the application of certain molecules that act as "terminating" agents is sufficient to regulate the proliferation of NCs. These molecules control the reaction conditions to guide the formation of nanostructures through the dynamic surface coordination. Carboxylic, alkyl thiol, phosphine, phosphine oxide, phosphate, phosphonate, amide, carboxylic acid, and aromatic nitrogen compounds are some examples of molecules that possess functional groups with electron donor atoms and can be used as surfactants. Surfactants are case-specific; for example, a molecule that adheres too tightly to the quantum dot surface would be useless since it would inhibit the nanocrystal's growth.

For his senior honours thesis, the author conducted trials in which he heated cadmium oxide in a mixture of 90% pure trioctylphosphine oxide (TOPO) and Oleic acid (OA) under an argon blanket at 340°C until

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the colour transformed from a dark redbrown to an almost colourless state. At the same time, trioctylphosphine (TOP) was added to selenium powder and agitated under argon until the selenium dissolved. After the Cd-TOPO solution was almost colourless, its temperature was reduced to 270°C. After these steps were completed, the cadmium solution was injected with the selenium solution, and the reaction started right away; portions were taken out at different intervals. After being rinsed with methanol and toluene, the reaction product could be kept in a number of different solvents, including hexane, dichloromethane, or toluene.

The images were captured using a 120 kV Joel-JEM-1230 electron microscope, which is used for transmission electron microscopy. Featuring a 4*4 kccd camera from Gatan, the UltraScan 4000SP. The X-ray diffraction (XRD) patterns were captured at room temperature with CuK 1 radiation using an X'Pert Philips Materials Research Diffractometer. The absorption spectra were captured with a diode array spectrophotometer manufactured by Hewlett-Packard, model HP-8453.

Optical spectroscopy of pure CdSe nanocrystals:

Optical band gap correlation with particle size and absorption spectra The absorption spectra of pure CdSe semiconductor nanocrystals (SNCs) in their solutions provide useful information like: (a) the nanocrystals' size, which is determined by the maximum wavelength (λ max) of the first excitonic band; (b) the nanocrystals' size distribution, as shown by the profile of the spectra, with different absorption bands visible, indicating the nanocrystals' monodispersity; and (c) an estimate of the sample's concentration in the solution, taken from the optical density of the first excitonic peak. the absorption and emission spectra of CdSe nanoparticles of varying sizes as synthesised at various times. In the case of an electronic transition, the Stokes shift is the distance, measured in wavelength or frequency units, between the absorption and emission spectra's band maxima. This applies to both fluorescence and Raman spectra, among others. George G. Stokes, an Irish scientist, was its namesake.

Growth time	max Abs	max Em	Stokes shift	FWHM
·	507		20	24
2 min	507 nm	537 nm	30 nm	26
5 min	537 nm	565 nm	28 nm	25
7 min	552 nm	579 nm	27nm	25
10 min	569 nm Broad	595 nm	26 nm	24
15 min	580 nm Broad	605 nm	25 nm	24

Table (1): Shows the absorption, emis	sion, stock shift, and FWHM	of as-prepared CdSe quantum
dots	at different time intervals	

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Fig. (1): Histogram of the as prepared CdSe QDs

The TEM and HR-TEM images, as well as the related histograms revealed that the CdSe QDs were uniformly shaped, monodispersed, and had an average size of about 4.2 nm. The particles had generally good morphology, and the largest one measured 7.2 nm. Based on the XRD pattern of the as-prepared CdSe QDs and the data from the JCPDS-XRD Cards (no: 04-014-0286), we can see that the particles have a Zinc Blend (ZB) cubic structure. This is because of three distinct features: the first, at $2\theta = 25^{\circ}$, is caused by the reflection of planes at (111) angle, and the two broad features, at $2 \setminus = 42^{\circ}$ and 50° , are caused by the reflection of planes at (220) angle and (311) angle, respectively. The shoulder can be attributed to the (200) planes at $2\theta = 30^{\circ}$. The XRD pattern of the as-prepared CdSe QDs sample displays all of these characteristics. Furthermore, the XRD pattern reveals that the ZB with the cubic structure is the most common, as indicated by the (102) reflection at $2\theta = 35^{\circ}$, which is characteristic of the wurtzite (Hexagonal) lattice structure.

The as-prepared CdSe QDs have had their capping agent identified using Fourier transform infrared spectroscopy. the FT-IR spectra of the pure Oleyl amine "reference" show the wave numbers that correspond to the vibrational modes of the functional groups of O.Am, TOP, and CdSe QDs, where v represents stretching modes and δ deformation modes, and subscripts "s" and "a" denote symmetric and asymmetric vibration modes, respectively. The two strong peaks at 2922 cm-1 and 2855 cm-1 are associated with the vas asymmetric and vs symmetric C-H stretching vibrations of the CH2 group, respectively. The band at 3010 cm-1 is attributed to the C-H stretching vibration in the C=C-H cisdouble bond. The absorption maxima at 1458 cm-1 and 721 cm-1 correspond to the scissoring of CH2 deformation and the rocking of numerous (-(CH2)n-, n≥4) methylene groups, respectively. The presence of the Oleyl group's C=C stretching vibrations is shown by the faint signal at 1630 Cm-1. This is the assignment of the infrared absorption peaks of pure Oleyl amine, which exhibits properties typical of an amine group. The stretching mode of N-H is indicated by the absorption band at 3375 cm-1, while the deformation modes of the NH2 amine group, specifically scissorsing and out of plane, are indicated by the

bands at 1616 Cm-1 and 789 Cm-1, respectively. Furthermore, the C-N stretching mode is correlated with the faint peak seen at 1071 cm-1.

The Fourier transform infrared spectra of the pure trioctylphosphene "reference" uncovered two strong peaks at 2924 cm-1 and 2855 cm-1, which are the C-H stretching vibrations of the CH2 group at vs symmetric and vas asymmetric, respectively. The CH2 deformation scissoring absorption peak is at 1461 cm-1, while the rocking absorption peak of numerous (-(CH2) n-, n \ge 4) methylene groups is at 720 cm-1. This is the assignment of the absorption peaks in the infrared spectra of pure trioctylphosphene that exhibit properties of the C-P bond. The TOP C-P stretching modes were observed at 1195 cm-1, 1113 cm-1, and 1022 cm-1, in that order. The FT-IR spectra of the CdSe quantum dots that were passivated with oleyl amine (O.Am) and trioctylphosphene (TOP) as they were prepared show the CH2 stretching mode of the O.Am and TOP at approximately 2924 Cm-1 and 2856 Cm-1, respectively. The absorption peaks at 1458 cm-1 and 720 cm-1 for TOP and O.Am, respectively, are related to the CH2 deformation scissoring and rocking of multiple (-(CH2) n-, n \ge 4) methylene groups. Furthermore, at 3010 Cm-1, the O.Am cis-double bond's C=C-H stretching vibration was detected. It appears that the O.Am and TOP maintain their original shape after being capped with CdSe QDs, as these band locations are nearly identical to those of the pure O.Am and TOP. In contrast to pure O.Am and TOP, there are a number of notable alterations brought about by the interaction of the nanoparticles and capping layer.

The main distinction between the infrared spectra of uncoated TOP and coated QDs is the loss of two peaks at 1113 cm-1 and 1022 cm-1, which represent the two C-P stretching modes. Additionally, the third C-P stretching mode moves from its original location at 1195 cm-1 to a lower wave number of approximately 1150 cm-1, and its intensity decreases as a result. Based on these findings, it may be inferred that the TOP caps the CdSe quantum dots. In addition, the main distinction between the infrared spectra of pure O.Am and coated QDs is the broadening and shifting of the N-H stretching mode from its original position at 3375 cm-1 to a higher wave number of approximately 3417 cm-1. The scissoring and out of plane NH2 deformation modes are shifted from their original positions at 1616 Cm-1 and 789 Cm-1 to wave numbers of about 1576 Cm-1 and 876 Cm-1, respectively, with a decrease in the intensity of the scissors mode. Additionally, the weak peak of the C-N stretching mode is shifted from its original position at 1071 Cm-1 to a lower wave number of about 1033 cm-1. Based on these findings, we can infer that the O.Am caps the CdSe QDs, that the C=C stretching mode remains at its initial location of around 1630 Cm-1 without any discernible impact, and that the Oleyl amine binds to the CdSe QDs entirely via the amine group. It is clear from these results that the O.Am and TOP were effectively capped on the CdSeQDs.

CONCLUSION

Synthesis and optical characterisation of quantum dot semiconductor nanoparticles are crucial to nanotechnology's advancement in optoelectronics, bioimaging, and photovoltaics. High-purity precursors like cadmium oxide (CdO), sulphur, and selenium, combined with stabilising agents such oleyl amine, oleic acid, trioctylphosphine (TOP), and TOPO, allow exact particle size, content, and crystallinity control. When particle size is reduced to the nanoscale, the quantum confinement effect causes tunable photoluminescence and higher fluorescence efficiency. Advanced optical characterisation methods include UV-Vis, photoluminescence (PL), and time-resolved fluorescence studies reveal quantum dot absorption, emission, and carrier recombination dynamics. XRD and TEM confirm their crystalline structure and

shape. Quantum dots can be used in cutting-edge technologies like LEDs, quantum dot solar cells, and medical imaging systems because they can alter their bandgap energy by manipulating synthesis settings. Surface passivation improves quantum dot stability and luminescence efficiency, reducing nonradiative recombination and improving performance. As quantum dots are synthesised and characterised, these semiconductor nanoparticles will influence next-generation nanomaterials and alter scientific and industrial domains.

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