

Determination of quantum dot size by Fourier transform visible spectroscopy

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Characterization of QD size is important in the process of preparation and their applications. Therefore, it is necessary to be found some quick, efficient, and trusted techniques for determination of the nanoparticle size. This paper proposes a possible investigation method of the size of commercial core-shell CdSe/ZnS quantum dots (QDs) using Fourier transform visible spectroscopy (FTVS). We found some relationships between the given QD sizes and their corresponding fluorescence (average) wavelengths, calculated from each emission peak of the Fourier transform spectra. These results show that FTVS is a novel, simple, fast, and reliable technique to estimate the CdSe/ZnS QD size. This methodology can be easily extrapolated to the size determination of other fluorescent nanoparticles.

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1. Introduction

There has been considerable interest of the world in semiconductor nanocrystals or quantum dots (QDs) over the past years due to their unique optical and electric features [1-5]. Due to their novel electronic, optical, and chemical properties which are strongly dependent on QD size, their study represents a challenge for the current research. Among many other different products of nanotechnology QDs have a great potential to lead to important advances in detection and imaging applications. They are distinguished as a new class of fluorescent markers used in biology and medicine. QDs possess photophysical properties necessary to surmount the limitations of conventional fluorophores.

Unlike classical fluorophores, QDs have unique features that include: wide and continuous absorption spectra, high quantum yields, very large Stokes spectral shifts [6], exceptional photostability and resistance to metabolic degradation [7, 8].

QDs, usually described like “artificial atoms”, present discrete size-dependent energy levels that are the results of the confinement of the charge carriers (electrons, holes) in three dimensions [9-11]. The smaller the size of the dot, the larger the bandgap is, the greater the energy difference between the valence band and the conduction band becomes, which leads to a deeper blue color. In the case of larger QDs, the energy difference between the valence and conduction band is lower, so that the radiation from QDs suffers a red shift. Therefore the bandgap energy of QDs (i.e., the energy difference between excited and ground state) can be accurately modulated by varying the QD size [12]. The capacity to adjust the size of the QDs is important and advantageous for many new applications.

From the quantitative point of view, the bandgap energy that determines the energy of the fluorescent light

is inversely proportional to the size of the QD. In addition, it has been shown that the size of the QDs determines the fluorescence lifetime.

The major advantage of QDs is the possibility of adjusting the absorption and photoluminescence (PL) spectra of QDs by changing their size, based on their strong quantum-size effects. The possibility of size control enables the QDs to be a better choice for some advanced devices that demand broad-band spectra [13-16], in contrast with existing natural elements whose absorption and photoluminescence spectra are fixed.

Characterization of size dependent optical properties of QDs gives the possibility to find a lot of qualitative and quantitative information about them, such as size, shape, quantum yield, and presence of surface defects. Consequently, the characterization of QD dimension is important in their preparation and applications.

Simultaneously with the development of QD applications, the request for QD size control becomes a higher demand. There are some typical methods which are used in order to determine the size of nanoparticles, including: transmission electron microscopy (TEM) [17, 18], size exclusion chromatography (SEC) [19, 20], dynamic light scattering (DLS) [21], capillary electrophoresis (CE) [22-26] and fluorescence correlation spectroscopy (FCS) [27, 28].

In the following sections there are given some information on a few of the conventional techniques for the characterization of the dimension of QDs.

TEM is almost always the first method used to characterize the nanoparticle shape, size and size distribution of nanoparticles. TEM offers plentiful and intuitionistic conclusions. But the process for preparing the specimen in which the sample is deposited on a copper grid is often laborious one. Besides, TEM is very expensive and is a time-consuming process. Another

disadvantage of using this method is the potential occurrence of artifacts from sample preparation [29].

Another well known size separation methods for QDs is SEC. SEC is a chromatographic technique in which nanoparticles, including QDs, are separated into various sizes based on the different hydrodynamic volumes of the nanoparticles. Nanoparticles with various sizes are passed through a column called size exclusion column, composed of a porous material having pores of various sizes in the range of the particles which must be fractionated.

In this separation method, small nanoparticles can enter into pores, but nanoparticles with big size cannot enter into pores they being rapidly eluted through the column in order to generate the chromatographic fractions corresponding to the molecular weight of the nanoparticles [30-32].

After the isolation of the nanoparticles in the separation column, the passage of the nanoparticles can be monitored and they can be further characterized using SEC technique together with various detection techniques (SLS – Static Light Scattering or DLS – Dynamic Light Scattering) [32, 33].

Although theoretically SEC appears to be a simple way to separate by size different QDs, the method has several disadvantages. The main one is the unwanted interactions between the column walls and the solute during chromatography leading to a process of irreversible adsorption of nanoparticles onto the size exclusion column [32].

DLS is one of the most popular techniques used to determine the size and size distribution of nanoparticles in solution.

DLS, which is known as "Photon Correlation Spectroscopy" (PCS) or "Quasi-Elastic Light Scattering" (QELS), is based on the irradiation of a solution/suspension of the sample with monochromatic laser light. Its principle involves the analysis of the light scattered by a solution/suspension as a function of time at a certain angle [34].

The type of movement involved in a typical DLS experiment is Brownian motion. The data can be processed to give the nanoparticle size. Specifically, the analysis of scattered light fluctuations allows the finding of the velocity of the Brownian motion and then the particle size using the Stokes – Einstein law. The main disadvantages of DLS are its lack of selectivity and relatively low signal strength. Thus, this fact is true in the case of the analysis of the nanoparticle with size below 10 nm using DLS technique. In this case, due to the uncertainty of the signal which becomes big and also due to the interference from other particles in solutions such as those with a particle size comparable to the particle size under investigation, there may be difficulties in obtaining accurate results [35].

In spite of the existence of these various techniques, there is still a need for simple and rapid methods for QD size determination.

Up to now, researches have demonstrated that group II-VI semiconductor systems, such as CdSe, CdS, ZnS, ZnSe, etc., have shown suitable features for the various

applications like biological probes, lasing media, optical amplifiers and infrared photo-detectors [36]. Besides this, it has been shown that capping those two-element QDs with a passivated layer as a shell, improves luminescence and stability of the QDs. This fact has been successfully proved, e.g. with ZnS capped CdSe core nanocrystals [37, 38]. Due to the high conduction band and low valence band of the core-shell, there is a good adjustment between CdSe and ZnS [36-38].

In this paper, we apply FTVS method to evaluate the diameter of commercially available core-shell CdSe/ZnS QDs. Because of the simplicity and high sensitivity, FTVS has a major advantage over other techniques, in providing quantitative information about QDs, including their sizes.

There are some reports in literature regarding empirical equations which are found by fitting the known size of nanoparticles versus experimental data of the corresponding absorbance wavelength. The empirical formulae proposed by Yu *et al.* [18] describe the relation between the diameter of the QDs and the wavelength of their first absorption peak. These relationships based on the size from TEM are given for CdTe, CdSe, and CdS in equation (1), (2) and (3) respectively:

$$D_{CdTe} = (9.8127 \times 10^{-7})\lambda^3 - (1.7147 \times 10^{-3})\lambda^2 + (1.0064)\lambda - 194.84 \quad (1)$$

$$D_{CdSe} = (1.6122 \times 10^{-7})\lambda^3 - (2.6575 \times 10^{-6})\lambda^2 + (1.6242 \times 10^{-3})\lambda + 41.57 \quad (2)$$

$$D_{CdS} = (-6.6521 \times 10^{-8})\lambda^3 + (1.9577 \times 10^{-4})\lambda^2 - (9.2352 \times 10^{-2})\lambda + 13.29 \quad (3)$$

Here, D (nm) is the average diameter of a given QD sample and λ (nm) is the wavelength of the first excitonic absorption peak of the corresponding QD.

Zhang *et al.* [39] have used fluorescence correlation spectroscopy (FCS) to evaluate the diameter of water-soluble bare and core-shell QDs. The authors also found an empirical equation to provide a quick and convenient method for determination of the hydrodynamic radii of water-soluble QDs. The empirical relationship is:

$$Y_{CdTe} = (-5.6366 \times 10^{-5})X^3 + 0.091345X^2 - 49.249X + 8838.1 \quad (4)$$

where Y (nm) is CdTe QD size and X (nm) is the maximum absorption wavelength.

These relationships were developed based on the fact that, in very small volume detection, the nanoparticles diffusion time in solution is correlated with their size. FCS spectroscopy technique involves the recording of the fluorescence intensity, $F(t)$, followed by determination of fluorescence fluctuations, $\delta F(t)$, around the average value. These fluctuations are auto-correlated in time. Essential information regarding the diffusion of fluorescent molecules is provided by the autocorrelation function.

Although FCS is an established tool for many biochemical and microfluidic analyses, it is suitable for fast processes (micro-seconds) and large area diffusion and has some limitations in terms of miscalculations under various environments [27, 28].

2. Analysis of the connection between the QD size and the emission wavelength

In this contribution, we present the size determination of commercial core-shell QDs by FTVS. The various semiconductor nanocrystals like CdSe/ZnS core-shell QDs dispersed in toluene with long chain amine capping agent, used in our studies (both [40] and the present paper), were purchased from EVIDENT Technologies. The dimensions of QDs are in the domain of (3-5) nm and according to this fact, their emission is located in the range (490-600) nm.

FTVS measurements were realized by an ARCSpectroNIR Fourier Transform Spectrometer [40]. The key parts of the experimental device used (described in detail in [40]) to study the fluorescence spectra of QDs are: the sources of excitation (a blue source or a UV source), the sample containing QDs, the optical fibre that transmits the fluorescence signal and the Fourier Transform Spectrometer, acted by a computer.

The spectrometer is based on an interferometer that measures the coherence function of light. At the entrance of the instrument, the light is split into two beams and at the exit, they come together. Interference image is recorded with a CCD array detector and light spectrum is shown by computer using Fourier transform algorithm and calibration tables.

In a previous paper [40] we have studied fluorescence spectra of three kinds of CdSe/ZnS core-shell QDs (0490, 0560, and 0600 Evidot fluids) irradiated by the light from a NdYAG@355 nm laser or from a luminescent diode ($\lambda = 480$ nm). We have reported the average wavelengths calculated from each emission peak of the Fourier

Transform spectra of QDs for those two excitations sources (a UV laser or a blue LED) [40].

For the case of the Fourier Transform spectra of the 0490 Evidot, when we irradiated the probe with the laser light from a NdYAG@355 nm, it was noted that the maximum of the peak intensity corresponds to an average wavelength calculated at 500 nm. When we illuminated the sample with a luminescent diode ($\lambda = 480$ nm), the wavelength fluorescence corresponding to a maximum intensity was 511 nm.

Examining the Fourier spectra of the 0560 Evidot it was found that for UV laser excitation, the average fluorescence wavelength was 572 nm, whereas for the excitation with blue LED light, the corresponding average wavelength of fluorescence was found at 588 nm.

Also, the Fourier Transform spectra of the 0600 Evidot were taken and analyzed. When the sample was illuminated with laser light, an average value of the wavelength of the emitted light was 614 nm and in the case of the blue LED, it was found the value of 634 nm.

We have analyzed the dependence of the size of the QDs and excitation conditions of the maxima of the fluorescence emission by FTVS. The purpose of this analysis was to obtain empirical equations, which fit these dependences in order to develop a simple method for determination the size of QDs, besides classical methods existing at the moment. We developed this method to evaluate the sizes of core-shell CdSe/ZnS QDs by the development of relationships between the dimensions of QDs of a certain type and their fluorescence characteristics represented by the wavelength of each peak of fluorescence.

Table 1 shows the sizes, the emission wavelengths of QDs, as specified in the EVIDENT Technologies catalog and the fluorescence average wavelengths of QDs, according to calculations made in the cases of the two mentioned excitation sources.

Table 1. Catalog specifications of the studied CdSe/ZnS core-shell QDs and calculated values of CdSe/ZnS core-shell QDs parameters obtained using the experimentally determined fluorescence spectra .

Quantum Dot material system	Quantum Dot type	Colour	Catalog crystal diameter* D	Catalog emission peak λ_{e_a}	Calculated emission average wavelengths λ_{e_b} ($\lambda_{ex}^{**} = 355$ nm)	Calculated emission average wavelengths λ_{e_c} ($\lambda_{ex}^{**} = 480$ nm)
CdSe/ZnS	Evidot 490 nm	Lake Placid Blue	3.2 nm	490 nm	500 nm [40]	511 nm [40]
	Evidot 520 nm	Adirondack Green	3.3 nm	520 nm	510 nm ***	525 nm ***
	Evidot 560 nm	Hops Yellow	3.8 nm	560 nm	572 nm [40]	588 nm [40]
	Evidot 600 nm	Fort Orange	5.0 nm	600 nm	614 nm [40]	634 nm [40]

Notes on QD properties

* Includes core-shell diameter.

** Values of the wavelengths for those two excitation sources that were used: a laser (NdYAG@355 nm) and a blue LED.

*** For 0520 QDs fluid obtained in this paper.

In the fourth and fifth columns from Table 1 there are the sizes of QDs and the emission wavelengths of the same QDs, which are provided by the EVIDENT Technologies catalog specifications.

In addition to the results mentioned in [40], we have obtained the fluorescence average wavelengths of 0520 Evidot fluid using the same experimental arrangement. Our experimental data set of four types of CdSe/ZnS core-shell QDs are shown in Table 1 (the sixth and seventh columns).

The analysis of wavelength corresponding to the measured maximum of fluorescence, for QDs of the same type but different sizes, is signaling the increasing of the value of the wavelength with the increasing of the QD size.

By using the Mathematica numerical analysis software, we found the fitting functions of the curves (a), (b) and (c) from the Fig. 1.

The method chose by us involves the interpolating polynomials, which can be used as an aid for data visualization, to deduce values of a function where no data are available and to summarize the connections among more variables.

The interpolating polynomials used by Mathematica software are based on Lagrange interpolation method. This polynomial model is among the most frequently used empirical technique for curve fitting. The polynomial approximation relies on the uniqueness theorem of the polynomials: "A polynomial of degree "n" that passes exactly through "n+1" points (or more general, through "n+1" constraints) is unique".

In other words, the polynomial that passes through a specific lot of points can take different forms, but all these forms are equivalent. Any form can be written into another form by simple algebraic rearrangement.

In accordance with the above affirmations, we chose a third degree polynomial which exactly fitted four points. These data points, given in Table 1, are used to construct the fitting functions (5), (6) and (7), which are exhibited below.

It must be pointed out, that the use of the Lagrange polynomial model has the following advantages:

- it has a simple form;
- the Lagrange polynomial is a well-behaved function and is computationally easy to use;
- in terms of adaptability, a certain form exhibits a moderate degree a flexibility;
- the data should not be ordered ascending or descending, according to the abscissa, x ;
- the Lagrange polynomial model can be used when the data points are unequally distanced.

We want to mention that an interpolation of high degree polynomial may be an unsatisfying approximation of the function between points, even though the accurateness at the data points will be "perfect".

In other words, the more data points are used in interpolation, the higher the degree of the resulting polynomial, thus the bigger oscillation will be shown

between the exact-fit values. This fact can result in highly unstable models. For this reason, it is indicated to choose a degree as low as possible for an exact match on all constraints, and as the case may be, an even lower degree, if an approximate fit is acceptable.

A triplet of the fitting curves of the QD size with their emission wavelength is shown in Fig. 1.

The catalog diameter values of QDs are plotted against their catalog values of fluorescence wavelengths, as shown in curve (a) of the Fig. 1 (the symbols used for points are circles). The curves (b) and (c) of the Fig. 1 illustrate the dependence between the known dimensions of the QDs and the calculated values of the emission average wavelengths [in the case of the curve (b), when the QDs were irradiated by the laser light from a NdYAG@355 nm, the symbols used for points are squares and in the case of the curve (c), when the QDs were irradiated by the blue LED, the symbols used for points are rhombs].

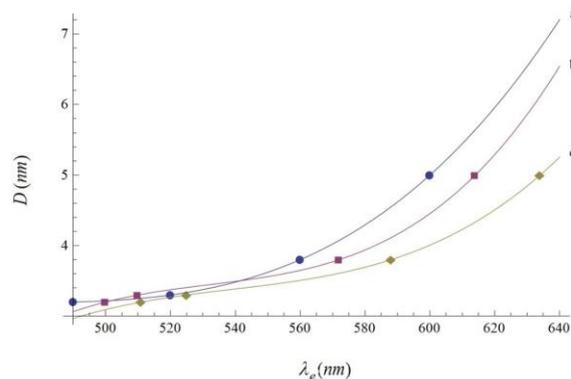


Fig. 1. Sizing curves for CdSe/ZnS core-shell QDs. Solid lines a, b, c represent the fit to corresponding equations (5), (6) and (7), while the symbols used for points (circles, squares, rhombs) on curves correspond to the catalog data (curve a) and experimental data (curves b and c).

The fitting function of the curve (a) is determined relating the known diameters, D , of CdSe/ZnS QDs to the values of emission wavelengths, λ_{e_a} , which are specified in the Evident Technologies catalog (Table 1). So, for this case, the fitting result is:

$$D_{CdSe/ZnS} = (7.9816 \times 10^{-7}) \lambda_{e_a}^3 - (1.12216 \times 10^{-3}) \lambda_{e_a}^2 + (5.25882 \times 10^{-1}) \lambda_{e_a} - 78.9545 \quad (5)$$

In order to obtain the empirical fitting functions of the curves (b) and (c) shown in Figure 1, we have used our experimental results from [40] and Table 1. The fluorescence wavelengths were all originally determined using FTVS for various CdSe/ZnS QDs irradiated by 355 nm laser radiation (curve b) or by 480 nm luminescent diode (curve c). The corresponding empirical relationships of the curves (b) and (c), obtained by fitting the known

sizes of QDs versus experimental data of emission wavelengths, are provided as follows:

$$D_{CdSe/ZnS} = (1.96547 \times 10^{-6}) \lambda_{e_b}^3 - (3.13626 \times 10^{-3}) \lambda_{e_b}^2 + (1.67384) \lambda_{e_b} - (295.338) \quad (6)$$

$$D_{CdSe/ZnS} = (1.27001 \times 10^{-6}) \lambda_{e_c}^3 - (2.05218 \times 10^{-3}) \lambda_{e_c}^2 + (1.11082) \lambda_{e_c} - (198.023) \quad (7)$$

In the above equations, (6) and (7), $D_{CdSe/ZnS}$ (nm) is the diameter of the core-shell QDs and λ_{e_b} , λ_{e_c} (nm) are the calculated values of the average wavelengths from each emission peak of the corresponding sample spectrum for those two excitation sources ([40], Table 1).

It is necessary to mention that the functions indicated above are only polynomial fitting functions of the experimental data. These functions may become invalid in the size ranges of QDs which are not covered by our experimental data.

The equations (6) and (7) represent a fast and effective way to evaluate the core-shell QD diameter.

We observe that contours of the sizing curves (b) and (c) are analogous with those of Yu *et al.* [18]. It must be pointed out that our results are based on the whole core-shell QD diameter (thus including both the thickness of CdSe hard-core and the thickness of ZnS shell), in contrast with the results given by Yu, which are dependent on only the hard-core diameter of QDs.

The use of the Fourier spectroscopy technique in visible range, for the determination of QD size like CdSe/ZnS core-shell QDs, has not been reported until now. This approach can be extended to other types of QDs. In conclusion, these results allow the finding of the total size (including the shell) of the QDs.

3. Conclusions

In this paper we used the fluorescence emission characteristics of commercial QDs core-shell, CdSe/ZnS, with dimensions of 3.2 nm, 3.3 nm, 3.8 nm, and 5.0 nm, depending on the conditions of irradiation.

As sources of irradiation were used a pulsed laser, with pulse duration of (5-6) ns and a wavelength of 355 nm and a broadband diode with peak emission at 480 nm. For detection, we used a Fourier transform spectroscopic system, able to evaluate the optical properties of QD core-shell CdSe/ZnS including fluorescence emission spectral characterization of type ARCSpectro HT-HR (ARCOPTIX S.A. Switzerland).

This work describes the size determination of core-shell QDs with a rapid and efficient technique, namely FTVS. We discussed here, the relationship between the QD size and its calculated fluorescence average wavelength of corresponding Fourier transform spectrum. Comparing with other studies, our empirical formulae included both the thickness of the core and the thickness of

the QD shell. However, the use of FTVS for determination of the CdSe/ZnS core-shell QD size has not been reported. This approach can be actually extended to other types of QDs. In conclusion, our results allow the finding of the total QD size (including its shell). This fact can provide experimental conditions that give the possibility of the nanoparticles size control. Therefore, our study represents a helpful reference on the research of QDs based on FTVS.

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