Cds Quantum Dots Composite Sol-gel Rod for Optical Applications

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Colloidal quantum dots (QDs) based on CdS were directly incorporated into a sol to fabricate solid rods. Various characterization techniques were employed to study the properties of the composite rod. A comparison between the absorption, emission, and excitation spectra of the QDs in the solid rod and in solution revealed their significant and stable nature in the solid state. SEM image shows that QD particles are well interacted with the sol gel environment. The observed particle size of QDs was heteronomous approximately from 5 to 10 nm. The XRD pattern of QDs illustrates their crystalline nature. The significant spontaneous emission (SE) signal was enhanced from QDs composite rod under the excitation of a 355 nm Pico second laser source. These findings suggest that further improvements in the integration of QDs into sol gel rods could be achieved by employing a higher density of QDs in the solid rod. The fabrication of QDs composite rods with a high concentration holds the potential for testing stimulated or amplified spontaneous emission in future studies.

Keywords: CdS QDs composite sol gel, absorption, PL, TEM, spontaneous emission.

1. INTRODUCTION

In recent years, there has been a growing interest in semiconductor nanocrystals known as quantum dots. These quantum dots have unique properties that make them highly attractive, including their ability to absorb light across a wide range of wavelengths [1], their high quantum yield [2] and the size-dependent wavelength of photoluminescence (PL) due to quantum confinement [3 – 6] due to quantum confinement. Among the different quantum dots, CdS quantum dots (QDs) is one of the important II-VI group of semiconductor compounds which possess size dependent PL and is tunable in the visible range [7]. Therefore, QDs have been interested for many optoelectronic applications, such as light emitting diodes [8, 9], lasers [10], telecommunications [11], and solar energy [12]. Enormous studies on different properties of QDs especially in thin films prepared by various techniques [13 – 16] were done for many applications. From the optical application point of view, the most interesting property is the PL stability of QDs in an environment. Currently, sol-gel technology has emerged as a promising matrix for embedding semiconductor nanoparticles [17]. This method allows for low-temperature preparation and enables the use of composite materials for diverse applications. A quantum size effect from the CdS sol-gel composite has been observed [18]. Influence of the Cd/S molar ratio on the properties of nanocrystalline CdS doped sol gel thin films has been reported [19]. QDs embedded in a PMMA composite film demonstrated stable emission bands regardless of variations in excitation energy [20].

CdS QDs doped sol gel matrix resulted in the strong luminescence without any casing [21]. QDs embedded silicate glass as an inorganic color converter was observed with significant stability [22]. Though the significant works of CdS in sol gel matrix have been reported in the literature, most studies of the QDs were either in solutions or composite thin films. In view of the previous studies, the exploration of optical gain from composite QDs is still limited. A research group has successfully fabricated and studied the silicon nanoparticles doped sol-gel rod, ormosil rod [23, 24] and polymer rod [25]. Significant optical stability of the nanoparticles in such matrix rods was observed. Recently, perovskite quantum dots were employed in polymer rods which enhanced the amplified spontaneous emission (ASE) like behavior [26]. In this study, a new approach was adopted in which CdS quantum dots were incorporated into a sol-gel matrix and transformed into solid composite rods. The QDs composite rods were subjected to various optical characterization techniques, and their stimulated emission was tested to evaluate the optical response under a tunable laser system.

2. EXPERIMENTAL DETAILS

A solution of CdS quantum dots (QDs) dispersed in methanol at a concentration of 10 mg/L was obtained from MK Impex Corp., Canada. The QDs were 98 % pure and had a size range of 5 – 10 nm (APS). The QDs solution was doped in sol gel matrix. In the procedure of sol-gel preparation, the inorganic precursor tetraethylorthosilane (TEOS; Aldrich, 98%) and methanol (Riedel-deHaen) was reacted to form silica sol. Formamide and nitric acid was involved as drying control chemical additive (DCCA) and catalyst respectively. The molar ratio of the sol composition was [1:1.5:0.5:1:0.01], TEOS: methanol: formamide: water: HNO_3 acid respectively. The final sol was further stirred during which the QDs solution was directly encapsulated dropwise. Then the QDs composite sol was poured into some polystyrene test tubes and quartz cuvettes and placed in an oven at 50 °C. The samples slowly became a gel and formed crack free solid in a few

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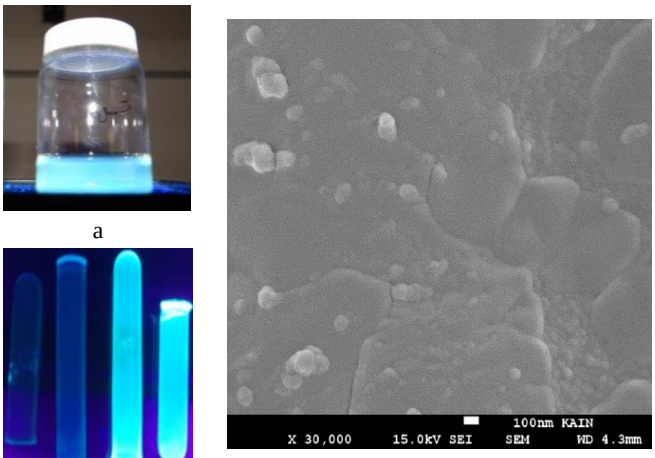
weeks. The final product of QDs composite rods was studied using different characterization techniques.

Absorption and emission spectra of the QDs were recorded using a UV–Visible-NIR spectrophotometer (Jasco V-670) and fluorescence spectrometer (Lumina, Thermo) respectively. The crystal structure of the QDs was inspected using XRD equipment PANanalytical X'Pert with Cu-Kα radiation (λ = 0.154 nm). The size and morphological structure of the QDs was examined by HRTEM (JEOL, Model-JEM2100F) and FESEM (JEOL, Model-JSM-6380LA) respectively. The spontaneous emission spectra of the QDs in solution and composites rod were tested by 355 nm laser pulse at the repetition rate of 15 Hz from Q-switched Nd:YAG picosecond tunable laser system (LS-2151, Lotti III) through an optical fiber attached with a spectrograph (QE65 Pro, Ocean Optics). A UV lamp (model XX15NF, Spectroline-USA) was used in this case to obtain images of samples with luminescent emission of QDs.

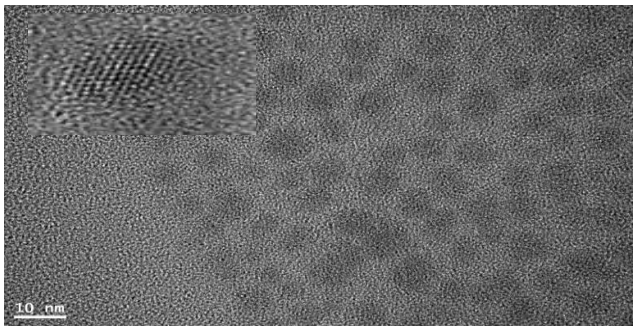
3. RESULTS AND DISCUSSION

The digital image of the light emitting QDs solution, undoped and doped sol-gel rods under the exposure of UV lamp is displayed in (Fig. 1 a and b).



a b c

Fig. 1. CdS QDs: a – solution; b – undoped and doped sol gel rods under UV exposure; c – SEM image



a

Fig. 2. TEM image of QDs: a – composite sol-gel; b – EDX spectrum

The emission of light from the QDs composite rods indicates a well-distributed dispersion of QD particles within the matrix, demonstrating their stability during the sol gel process.

The noticeable inhabitant of QD particles inside the rod environment was confirmed by the image of Scanning Electron Microscopy (SEM) as shown in (Fig. 1 c).

Many QD particles are scattered inside the surface layer of sol-gel matrix. The composite surface shows that QD particles are well intermingled with the sol-gel environment. The good interaction of the QDs with the matrix environment could yield a good stable composite material. The size of QD particles was inspected by TEM. Fig. 2 a shows the image of QDs distribution of different size in sol gel environment. The majority of QD particles are well spreading and heteronomous sizes of approximately from 5 to 10 nm. The crystalline nature of the QD particle is seen in (Fig. 2 a-inset). It indicates that there is no agglomeration of particles within the solid environment. In addition, the presence of QD particles in the sol gel is confirmed by EDX spectrum. Cadmium rich with sulfide is seen in the EDX spectrum as displayed in (Fig. 2 b). The typical crystalline nature of QDs in the TEM image is supported by the XRD pattern as shown in (Fig. 3).

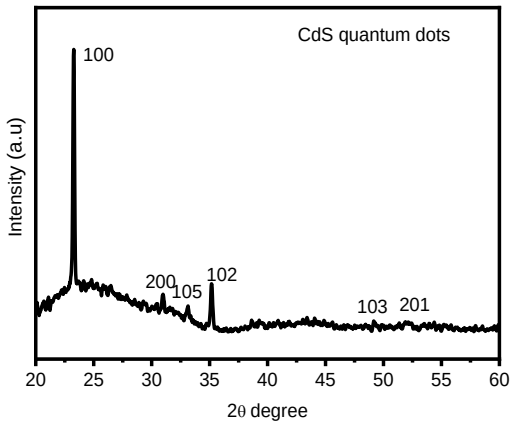
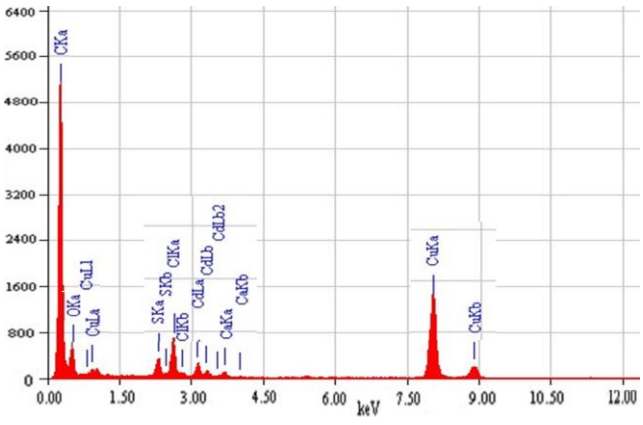


Fig. 3. XRD spectrum of CdS quantum dots

The XRD peaks of QDs indicate crystalline nature.



b

Prominent peaks and few cubic phase of QDs structure are observed at 23.30, 35.12, 49.9, 52.2 deg. corresponding to lattice planes (100), (102), (103) and (201) which correspond to the data from JCPDS file no. 42-1411. The XRD peak at around 31 deg. of CdS QDs shows the presence of cubic phase and corresponds to plane (200) according to the result work [27]. The peak at around 33 deg. may correspond to diffraction plane 105 of the hexagonal phase [28].

To observe the higher absorbance of quantum dots (QDs) in both solution and sol-gel solid rod, the absorption spectra were measured using high (10 mg/L) and low (5 mg/L) concentrations. The results are depicted in (Fig. 4). The enhancement of absorbance with a concentration of QDs is observed in solution and sol-gel rod. The differences in absorbance of QDs between the solvent and sol-gel rod are shown in (Fig. 4). The absorption peak of QDs solution is observed with a shoulder peak at around 315 nm which is supported by the observed absorption peak value at 320 nm of 2.5 nm size [29].

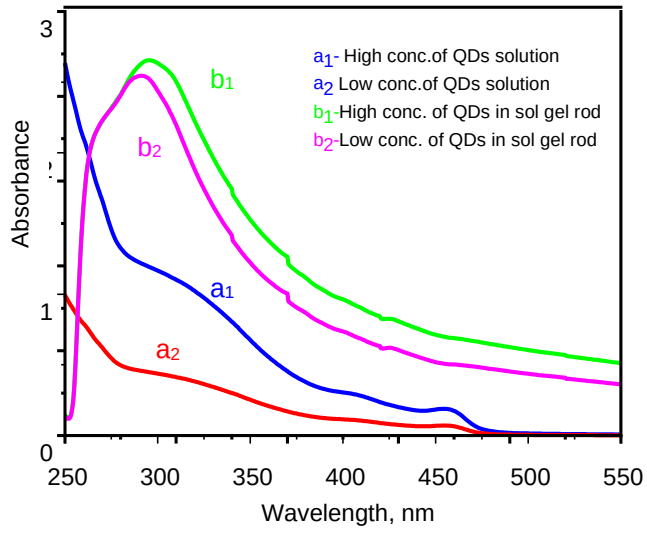
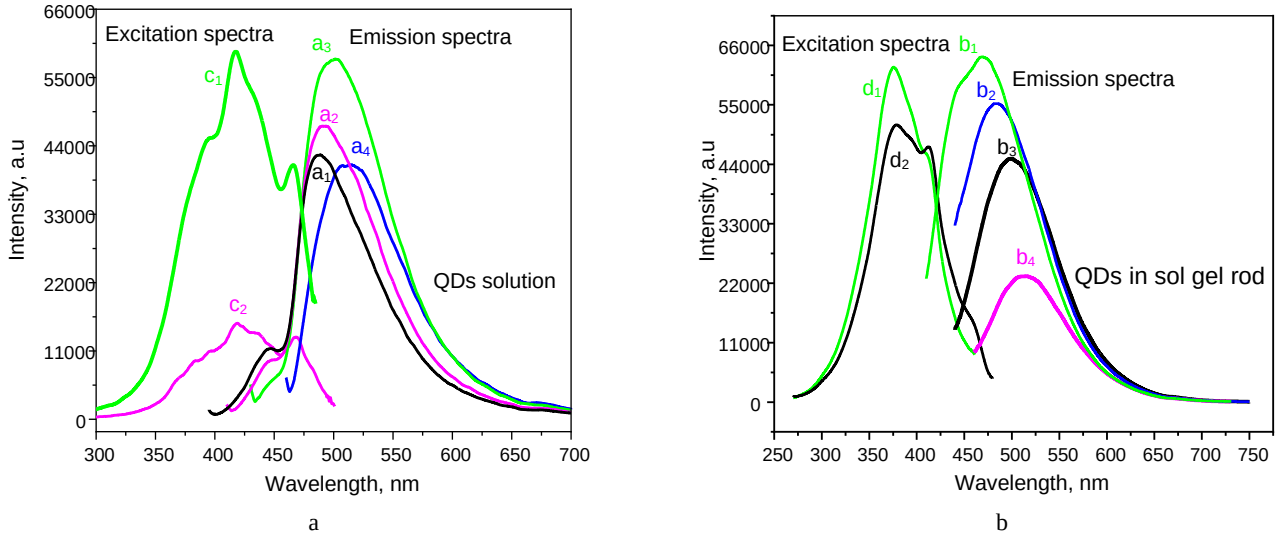


Fig. 4. Absorption spectra of QDs: a_1, a_2 – solution; b_1, b_2 – sol-gel rod

The two weak absorption peaks of QDs in solution at around 412 nm and 460 nm appeared. The absorbance of the QDs composite sol-gel rod is higher than the solution. But the absorption peak becomes broad with a slightly blue-shift when QDs doped in sol-gel rod as shown in (Fig. 4). There are two absorption peaks observed at around 370 and 420 nm in the sol gel matrix which is fundamentally weak. The effect of absorption spectra is due to the solid environment of the matrix. In addition, the nature of the distribution of QD particles in sol-gel may affect the absorption property. It is also possible to cause the absorption property of QDs during the transition of phase from sol to solid [25]. Present absorption spectra of QD in sol-gel rod are comparable to the observed feature of CdS particles in xerogel state [30] and hybrid sol gel matrix [21]. The broad particle size distribution within the matrix [30] likely contributes to the effect of QDs in the matrix. In the present study, the sample with high concentration was only employed. The main motive of this work is to understand the PL and SE property of QDs in sol gel rod that could be employed in testing the optical gain. The excitation wavelengths for emission and excitation spectra of QDs were evaluated from 450 – 650 nm and 300 – 500 nm respectively. The excitation peaks of the QDs in solution were observed at 395 nm, 418 nm and 465 nm as seen in C_1 and C_2 spectra of (Fig. 5 a).

The excitation wavelengths of the QDs for a solution and sol-gel rod were selected as 355 nm, 395 nm, 418 nm, 465 nm and 355 nm, 378 nm, 418 nm, 460 nm respectively as listed in Table 1. Fig. 5 a, the peaks a_1, a_2, a_3 and a_4, (Fig. 5 b), peaks b_1, b_2, b_3 and b_4 indicate that the emission peak and relative intensity of QDs solution and sol-gel rod are influenced by the excitation wavelengths respectively. This suggests that varying the excitation energy has an impact on the relative intensity of the QDs in both samples. But the change in emission peak of the QDs solution and sol-gel rod by different excitation is not clear but it may be possible to the presence of a large number of heterogeneous QDs size. Moreover, the emission peaks and relative intensity of QDs sol gel rod is considerably changed from the peaks of QDs solution.



a b

Fig. 5. The emission and excitation spectra of QDs: a – solution; b – sol-gel solid

Table 1. Comparison of peaks between the QDs in solution and sol-gel rod

Samples	Absorption peak, nm	Excitation peaks, nm	Emission peaks, nm	Spontaneous emission peaks, nm
QDs Solution	315 412 460	395 418 465	a_1 – 485 at λ_{exc} 355 a_2 – 492 at λ_{exc} 395 a_3 – 501 at λ_{exc} 418 a_4 – 510 at λ_{exc} 465	506
QDs sol-gel rod	298 370 420	378 412 460	a_1 – 470 at λ_{exc} 355 a_2 – 483 at λ_{exc} 378 a_3 – 498 at λ_{exc} 418 a_4 – 512 at λ_{exc} 460	500

For instance, the peak observed at 485 nm of QDs solution was excited by the 355 nm wavelength observed at 470 nm in sol-gel rod. It means that emission peaks and intensity of QDs are quite caused and enhanced by sol gel solid medium. Similarly, the excitation spectra of the QDs in solution are affected when they are embedded in the sol gel rod. The influences on the emission peaks and band shape of the QDs in the sol gel may be attributed to the solid environment, suggesting that the presence of the sol-gel matrix alters the optical properties of the QDs.

The absorbance and emission intensity of quantum dots (QDs) solution exhibit remarkable stability in comparison to the absorption and emission of QDs in solid rod, as demonstrated in (Fig. 4 and Fig. 6).

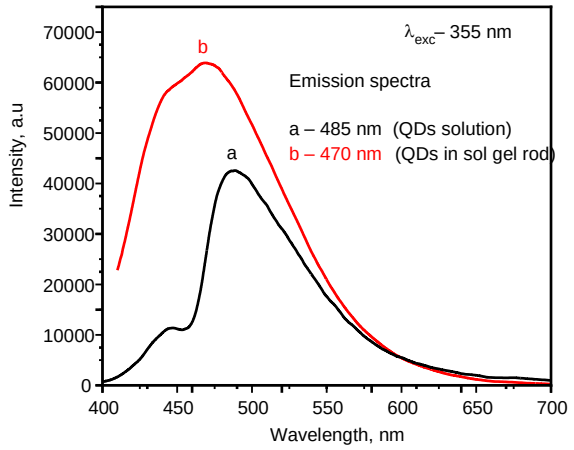


Fig. 6. The comparison between the emission spectra of QDs solution and sol-gel solid at 355 nm excitation wavelength

When the QDs solution is directly incorporated into a sol and converted into a solid rod, the optical properties of the QDs in the solid rod remain largely unaffected. The comparison of emission spectra between QDs solution and sol-gel rod is displayed in (Fig. 6). No degradation on the emission intensity of QDs in sol-gel rod is observed since the scattering and reflection of light are highly possible within the solid environment. The emission and excitation peak positions of QDs in solution and solid sol-gel are listed in Table 1. There is clearly a shift in PL features by different excitation wavelengths which may correspond to excitation energy and the presence of quantum size effects [12]. With the consideration of optical gain, we investigated the spontaneous emission spectra (SE) of Quantum Dots (QDs) in both solution and solid sol-gel rods. A high-power picosecond laser system with a 355 nm laser source was utilized for excitation. The energy pumped ranged from 50 µJ to 1.5 mJ was used to prevent photobleaching of the solid sol-gel rod by the 355 nm laser source. Fig. 7 display the SE spectra of QDs in solution and sol-gel rod, respectively, under various excitation energies. As the excitation energy increases, the intensity of QDs in both solution and sol-gel rod also increases. However, the SE intensity of QDs in the sol-gel rod is more pronounced compared to the solution when pumped with the same excitation energy, as depicted in (Fig. 7).

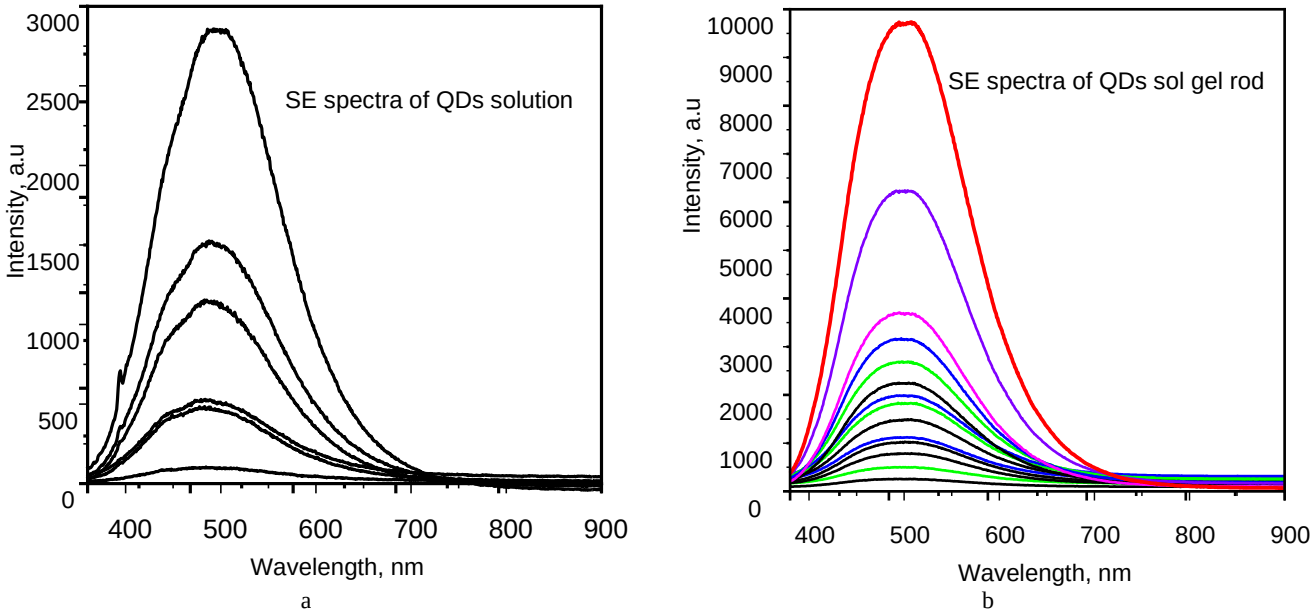


Fig. 7. Spontaneous emission spectra of QDs: a – solution; b – sol gel rod

Additionally, there is a slight shift in the peak position of QDs in the solid sol-gel rod compared to the peak of QDs in the solution. For example, the spontaneous peak observed at 506 nm in the QDs solution of (Fig. 7 a) shifts to 500 nm in the solid sol-gel of (Fig. 7 b). The spontaneous emission of QDs in the solid sol-gel exhibits significant improvement under different excitation energies. The SE peak positions of the QDs in the composite sol-gel rod and solution are listed in Table 1.

Increasing of emission intensity of QDs in the rod with the variation of pump energy shows good stability of QD particles in a solid environment. Moreover, the uniformity of inhabitant and distribution of QDs particles in solid medium could produce a stable PL. The comparison of emission and SE peak of QDs in solution and solid sol-gel at excitation of 355 nm is shown in (Fig. 8).

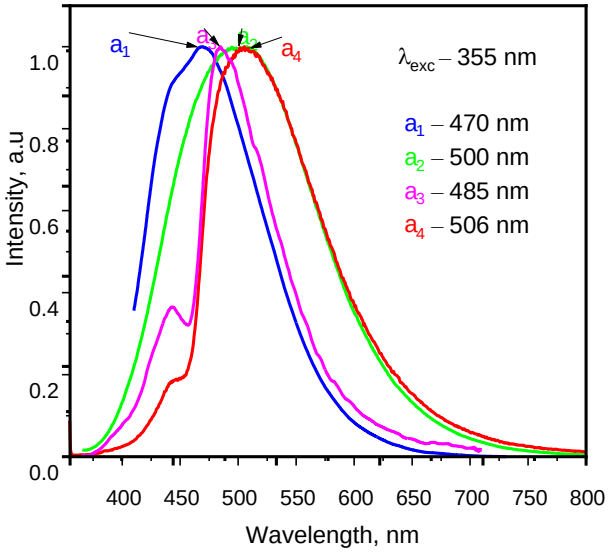


Fig. 8. Comparison of normalized emission and spontaneous emission peaks between QDs in solution and sol gel rod at 355 nm excitation wavelength

In Fig. 8, the emission peaks of QDs in sol-gel rod observed at $a_1 - 470$ nm and $a_3 - 485$ in solution. The SE peak at $a_2 - 500$ nm and $a_4 - 506$ nm of QDs are observed in sol-gel rod and solution respectively. No significant effect on SE peaks between QDs sol-gel solid and solution which is more or less 6 nm. Overall, the observed SE peak of QDs in solid rod upon the laser pump energy is quite stable. Although the current SE peak does not satisfy the optical gain condition, it holds potential for future improvements by utilizing a higher density of QDs solution within such a solid rod. These findings suggest that a high-density QDs composite sol-gel rod could be a promising material for future studies in optical applications, potentially leading to stimulated emission or amplified spontaneous emission (ASE).

4. CONCLUSIONS

The CdS quantum dots composite rods were successfully prepared. Optical stability of QDs in solid sol gel rod was observed. PL property of the QDs composite rod was quite stable. The emission peaks and relative intensity of the QDs in both solution and sol gel rod were found to be influenced by the excitation wavelengths. No aggregate of QD particles in the solid environment was observed. The significant spontaneous emission from QDs composite rod was exhibited under the laser pump energy. Present results revealed that addition of quantum dots in solid rod is quite possible without any degradation of optical property. It is highly expected that such QDs composite rod may be used as active media for the further testing of ASE if it is employs a highly dense packed of QDs nanocomposites rod.

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