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Article

Control of Membrane Luminescence Using Colloidal Nanocrystals

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Abstract: The optical properties of the Nafion polymer membrane containing colloidal CdSe/CdS/ZnS nanocrystals embedded by diffusion have been studied. The CdSe/CdS/ZnS nanocrystals have a core/shell/shell appearance. A toluene solution was used to provide mobility of the active sulfone groups of the Nafion membrane and to embed the nanocrystals inside the membrane. The kinetics of the process of nanocrystal embedding into the membrane matrix was studied using luminescence analysis, absorption spectroscopy and laser emission spectroscopy techniques. The diffusion of colloidal CdSe/CdS/ZnS nanocrystals into the Nafion proton exchange membrane resulted in a new molecular complex «Nafion-colloidal CdSe/CdS/ZnS nanocrystals». The presence of new luminescence centers based on CdSe/CdS/ZnS nanocrystals in the membrane was registered using laser emission spectroscopy. Colloidal nanocrystals located in the Nafion matrix represent an analogue of a luminescent transducer. The lines contained in the luminescence spectrum of the membrane, which has been in solution with colloidal nanocrystals for a long time, registered upon its drying, show the kinetics of the formation of the molecular complex «Nafion membrane - nanocrystals». The luminescence spectrum of the membrane with nanocrystals contains additional intensity maxima that do not coincide with the spectral lines of the solvent and Nafion membrane. The intensity of the luminescence spectrum of the membrane with embedded nanocrystals is higher than the intensity of the secondary emission peak of the initial nanocrystals, which is important for the practical use of the complex «Nafion-colloidal nanocrystals» in optical systems.

Keywords: colloidal nanocrystals; Nafion; luminescence; absorption spectroscopy; laser emission spectroscopy; toluene

1. Introduction

Methods of creating light energy converters are urgent practical problems. For example, the process of embedding nanoparticles in optically transparent media, in particular, in polymer films, is used in the production of full-colour displays. This leads to the excitation of spectrally narrow luminescence lines. Determination of the degree of influence of the doping process on the physical characteristics of radiation transducers, in particular, based on polymer matrices is practically important at this stage. Semiconductor crystals implanted in a polymer matrix induce charge carrier transport. As a result, the influence of nanocrystals on spatial charge separation is possible in such systems [1–7]. Nanocrystals embedded in organic semiconductor matrices can act as centers of radiative recombination in the structures of organic light-emitting diodes [1–3].

The emission spectrum of light-emitting systems can be regulated by embedding nanocrystals of different sizes [7]. The embedded nanocrystals exhibit good photostability, high photo-decolourisation threshold and high quantum efficiency. The type of passivating agent of the

nanocrystal surface and the composition of the organic matrix, where these nanobjects are embedded, significantly affects their electrical and luminescent characteristics. For identical nanoparticles whose structure is core/shell/shell CdSe/CdS/ZnS, changing the type of passivator leads to significant differences in the dipole-dipole energy transfer rates.

One of the important applications of semiconductor nanoparticles is diagnostics of various diseases. In this case, nanoparticles play the role of luminescent markers. The use of organic dyes for this purpose is limited as they can be toxic. In this respect, nanoparticles, when properly passivated, have a competitive advantage. However, nanoparticles are quite large entities and are comparable to the pore size of cell membranes. Therefore, it should be clarified whether they can reach the target to be visualized fast enough and whether this will not lead to degradation of the properties of the nanoparticles themselves. This question can be addressed in «in vivo» experiments. However, the answer to this question can be answered «in vitro» by using the Nafion matrix [8], which has already demonstrated its effectiveness as a model of biological tissues.

Nafion (C7HF13O5S C2F4) membrane is produced by copolymerization of perfluorinated vinyl ether monomer with tetrafluoroethylene [9] and consists of perfluorinated vinyl ether groups terminated with tetrafluoroethylene (Teflon) based sulfone groups. Teflon is a highly hydrophobic membrane matrix. The sulfone groups in the membrane are fairly hydrophilic compounds. The Nafion polymer membrane matrix is biocompatible and flexible. The membrane has good mechanical and chemical stability due to the inertness of its fluorocarbon backbone. A large number of works [9–16] are devoted to the study of the effect of impregnation of the Nafion membrane with water [9] and aqueous solutions, for example, methylene blue [17,18], which is accompanied by the manifestation of the effect of forward and reverse micellisation. The inner surface of the membrane channels has negatively charged regions that allow cations to transit [19–21]. These channels have nanometer dimensions and the structure of these channels allows the separation of H^+ and $[OH]^-$ ions on both sides of the membrane. In this regard, the Nafion membrane is actively used in low-temperature hydrogen fuel cells [22].

In the present work, the embedding process of colloidal CdSe/CdS/ZnS nanocrystals into Nafion matrix was studied to investigate the effect of doping on the optical properties of the polymer membrane. The process of nanocrystal embedding was controlled by luminescence, light absorption spectroscopy, and laser emission spectroscopy methods.

2. Materials and Methods

The colloidal nanocrystals (CdSe/CdS/ZnS) represent a core/shell/shell system structurally and have two type I heterojunctions. The nanocrystals used in this work were synthesized using a technique similar to that presented in [23,24]. According to the results of electron microscopy performed on a transmission electron microscope JEOLJSM-7001F using ImageJ program, the sizes of nanoparticles were estimated taking into account the passivation layer: 8.1 ± 0.2 nm for NC1, 8.3 ± 0.2 nm for NC2, 9.1 ± 0.2 nm for NC3. The standard deviation of the nanoparticle sizes was about 1.3 nm [23–25].

The embedding of colloidal nanocrystals (CdSe/CdS/ZnS) from solution into Nafion membrane was carried out by soaking the membrane in toluene solution (C₇H₈) with nanocrystals for 100 minutes and for 30 days. Nafion plate thickness was $d = 175$ μ m. The volume of toluene solution with colloidal nanocrystals was 4 ml. Soaking of the membrane in the solution with nanocrystals was carried out for 100 minutes and for 30 days while the volume was thermostated at $T = 22$ °C. The kinetics of the process of settlement of nanocrystals in the membrane was determined. For this purpose, the membrane was kept in solution and the panoramic absorption spectrum of the membrane was measured at 5 min intervals. Toluene was used as a means of suspending colloidal nanocrystals and their further transport inside the polymer membrane. In doing so, toluene molecules activated the mobility of the sulfone groups of the membrane. The result of nanocrystal embedding was monitored by photoluminescence and absorption spectra of the samples in the visible and UV wavelength ranges, as well as by laser emission spectroscopy.

The luminescence spectra were registered using an experimental setup consisting of a laser radiation source — a neodymium laser on alumina garnet (QX500 SolarRS) — 1 with a fundamental generation line at a wavelength of 1064 nm, a beam splitter — 2, a sample — 3, and a secondary radiation receiver — 4 (Figure 1). The third harmonic of Nd³⁺: YAG laser with wavelength $\lambda = 355$ nm was used to excite luminescence in the studied samples. The laser operated in Q-switching mode and generated pulses with a duration of 10 ns and a repetition rate of 20 Hz. The mini-spectrometer (ASP-100) was a receiver of secondary radiation and allowed the registration of a useful signal in the range of 180-1100 nm with a spectral resolution of ≈ 0.3 nm (Figure 1).

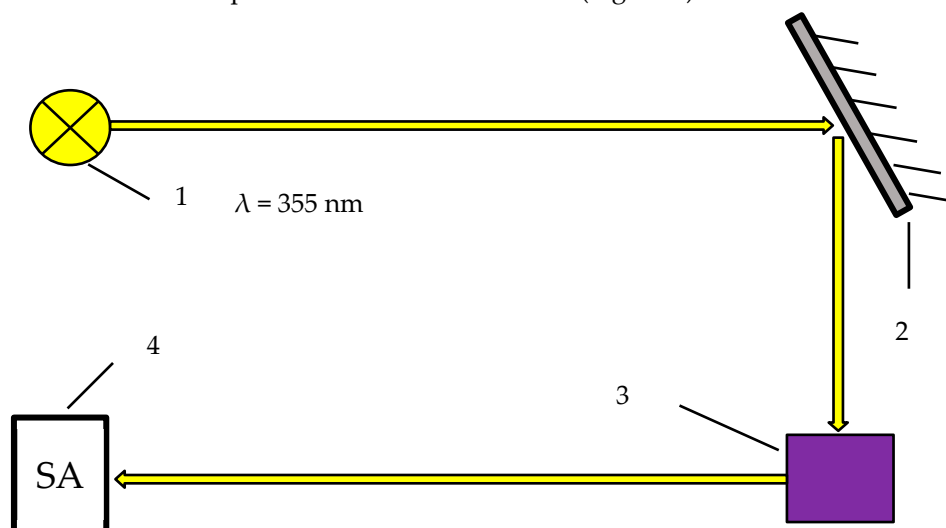


Figure 1. Block diagram of the experimental setup for luminescence registration.

Research of the optical membranes properties was carried out using spectrophotometer PB2201 (SOLAR) by recording the absorption spectra of the samples in the wavelength range of 190-900 nm.

Laser emission spectroscopy was performed on the LAES MATRIX SPECTROMETER. The LAES series instruments use a two-pulse solid-state laser as an excitation source (Nd³⁺ — laser, generated radiation wavelength: 1.064 μ m; pulse repetition rate: 1-10 Hz; pulse repetition rate: 1-10 Hz; radiation pulse energy: at least 100 mJ/pulse) with Q-switching, which reduces the limiting concentrations of detection of elements and allows excitation of chemical elements with high ionisation energy.

3. Results

1. Research of luminescence spectra of samples

Figure 2a (dependence 1) shows the luminescence spectrum of the original Nafion polymer membrane. The secondary emission maxima during radiation excitation of the untreated membrane are observed at wavelengths $\lambda_{\text{Naf}} = 538, 584, 705$ nm. The intensity maxima in the luminescence spectrum of toluene, which was used as a base for the preparation of the solution with colloidal nanocrystals, are observed at wavelengths 497 and 541 nm (Figure 2a, dependence 2).

The secondary emission spectrum of the membrane soaked in toluene is shown in Figure 2a, dependence 3. The luminescence maxima of the Nafion membrane soaked in toluene for a long time are observed at wavelengths — 502, 538, 584, 705 nm. From the comparison of the spectral distribution of luminescence intensity maxima of the original sample (Figure 2a, dependence 1) and the Nafion membrane soaked in toluene (Figure 2a, dependence 3), it follows that the luminescence lines became more intense, but the positions of the spectral maxima remained practically unchanged.

Afterwards, the seeding of nanocrystals into the membrane was carried out. The process of introduction of nanocrystals into the Nafion membrane was carried out by placing the plate in a solution with colloidal nanocrystals suspended in toluene. Figure 2b, dependence 1 shows the luminescence spectrum of a solution of colloidal nanocrystals in toluene. The maximum of secondary emission of quantum dots is detected at a wavelength of 634 nm (Figure 2b, dependence 1). At the

same time, the most intense spectral maximum of toluene (497 nm), which is shown in Figure 2a, dependence 2, was not actively manifested in the system «toluene-nanocrystals». The intensity of all luminescence maxima of the toluene-nanocrystals system is greater than that of the initial toluene solution with nanocrystals.

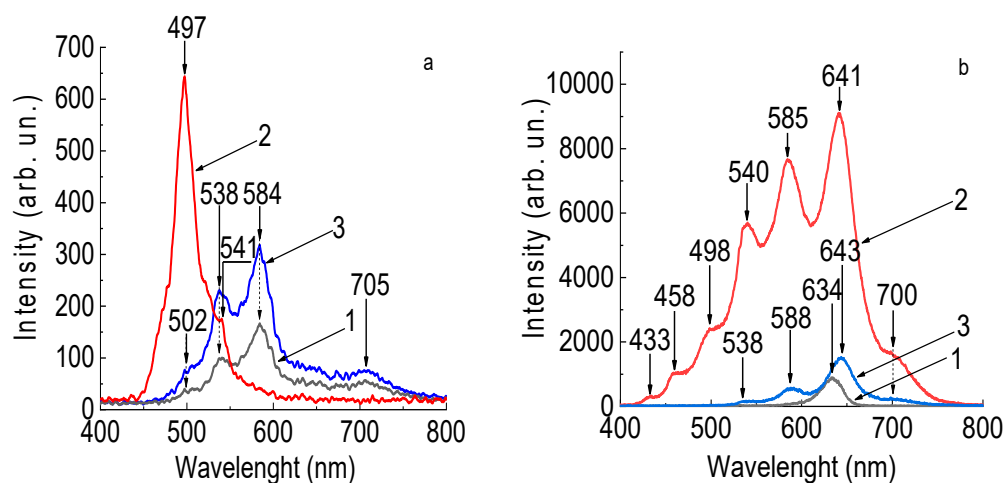


Figure 2. Luminescence spectra of the samples: (a) 1 — Nafion membrane; 2 — toluene; 3 — Nafion membrane soaked in toluene; (b) 1 — CdSe/CdS/ZnS nanocrystals; 2 — Nafion membrane with embedded nanocrystals in the wet state of the membrane; 3 — Nafion membrane with embedded nanocrystals in the dried membrane.

The luminescence spectrum of the membrane, which was soaked for 30 days in the colloidal nanocrystal solution, is shown in Figure 2b (dependence 2). The spectrum was measured immediately after the sample was extracted from the solution and the membrane was in a wet state when the secondary emission was recorded. Then the membrane with embedded nanocrystals was dried in air and the luminescence spectrum was measured again in the same spectral range (Figure 2b, dependence 3). The luminescence spectrum of Nafion membrane with introduced nanocrystals after its drying shows intensity maxima at wavelengths of 538, 588, 643 and 700 nm. Importantly, an additional maximum appears in the secondary emission spectrum of the dried membrane, which is observed at a wavelength of 643 nm. The luminescence maximum observed in the spectrum of CdSe/CdS/ZnS nanocrystals falls at a wavelength of 634 nm (Figure 2b, dependence 1). In the dried membrane with embedded nanocrystals, this maximum is observed at a wavelength of 643 nm (Figure 2b, dependence 3). Thus, the wavelength at which the luminescence maximum of the formed molecular structure obtained by embedding CdSe/CdS/ZnS nanocrystals into the Nafion membrane was observed shifts in wavelength by about 10 nm. The 643 nm photoluminescence line does not appear in the spectra of the solution components.

2. Research of optical properties of samples.

The kinetics of nanocrystal embedding into the membrane was investigated by measuring the membrane absorption spectra as a function of membrane exposure time in solution with colloidal nanocrystals. The absorption spectrum of the membrane after the introduction of colloidal nanocrystals into it changed significantly. Figure 3 shows panoramic absorption spectra of the membrane with embedded nanocrystals in the range of 250-800 nm and with time intervals of 5, 10, 25, 30, and 35 from the beginning of nanocrystal embedding. The ordinate axis indicates the relative units of radiation absorption coefficient. At wavelengths of 424 nm and 490 nm, two new absorption maxima appeared. Investigations of radiation absorption by the membrane in the range of 250...800 nm indicate an increase in the absorption coefficient of the membrane with embedded nanocrystals in relation to the original membrane.

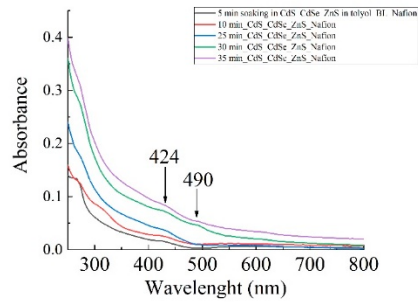


Figure 3. Absorption spectrum of the membrane.

Figure 4 shows the time dependence of the membrane absorption coefficient at a wavelength of 300 nm. The process of CdSe/CdS/ZnS nanocrystals embedding can be considered as completed after 35 minutes, so the kinetic curve reaches its maximum. The relative radiation absorption intensity of the membrane with colloidal nanocrystals at 300 nm wavelength can be approximated as $0,23 \cdot e^{\frac{t}{57,36}} - 0,20$. The embedding rate of CdSe/CdS/ZnS nanocrystals into the Nafion polymer membrane was approximately $4 \cdot 10^{-3} \text{ s}^{-1}$.

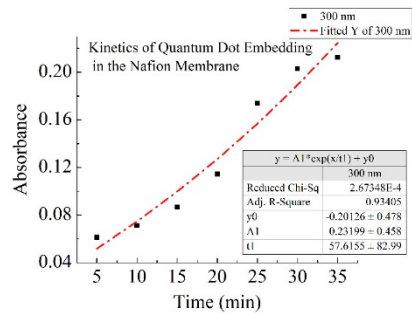


Figure 4. Time dependence of the in the process of nanocrystal embedding, membrane absorption coefficient at a wavelength of 300 nm.

A direct and independent confirmation of the diffuse penetration of CdSe/CdS/ZnS nanocrystals into the membrane are the results of laser emission spectroscopy experiments obtained on the LAES MATRIX SPECTROMETER. Atoms of the following chemical elements, Cd, Zn and C, were found to be present in the Nafion membrane (Figure 5).

Three spectral lines for — Zn (202.5 nm, 206.2 nm, 213.8 nm), Cd (214.4 nm, 226.5 nm), as well as carbon — C (248 nm) were detected (Figure 5). The method of obtaining the registered radiation in the process of laser emission spectroscopy and processing of the obtained lines do not allow us to determine fluorine (F) and sulphur (S) present in the polymer membrane fibers.

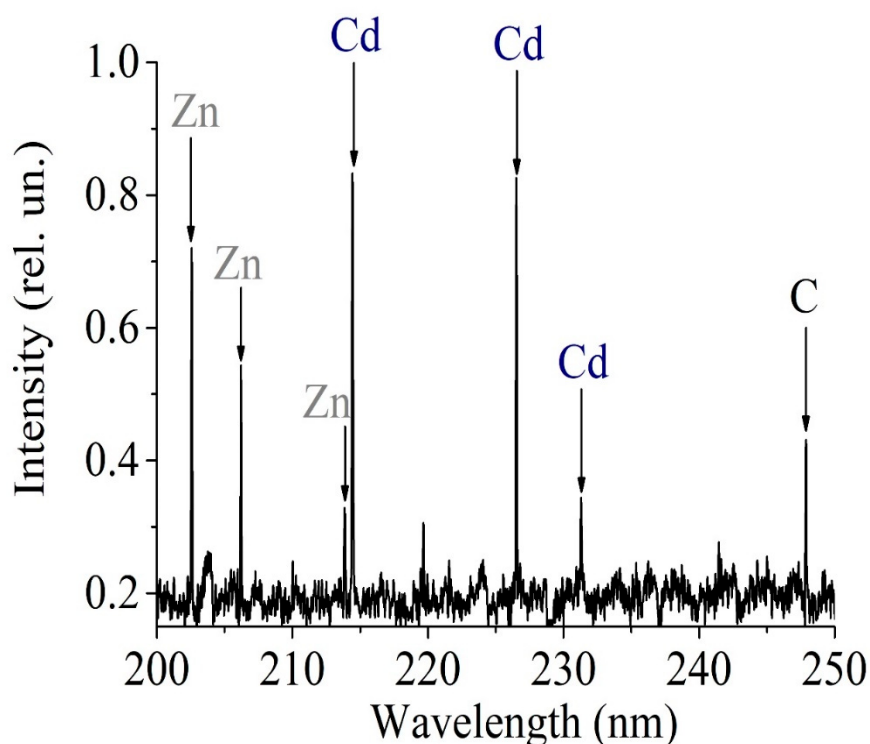


Figure 5. Emission spectrum of the membrane with CdSe/CdS/ZnS.

4. Discussion

Seven strongly expressed maxima are observed in the luminescence spectrum of the Nafion membrane with embedded nanocrystals (Figure 2b, dependence 2). An additional high-intensity spectral component of luminescence appears at a wavelength of 641 nm. It can be noticed that in the dried membrane with introduced nanocrystals the observed intensity maximum is shifted 2 nm to the long-wave region of the spectrum (Figure 2b, dependence 3). From the luminescence spectrum of nanocrystals in toluene in Figure 2b (dependence 1), it follows that the corresponding luminescence maximum is observed at a wavelength of 634 nm. The intensity of the luminescence spectrum increases significantly compared to the secondary emission intensity of the initial solution components.

As follows from [23], the size of CdSe/CdS/ZnS nanocrystals is approximately 8.1 nm. Research of the internal structure of the Nafion membrane shows that the polymer base (hydrophobic phase) consists of fluorocarbon and ether chains. Functional sulfogroups are grouped inside spherical cavities with a diameter of about 40 Å. Figure 6 shows the scheme of arrangement and possible interaction of membrane links and colloidal CdSe/CdS/ZnS nanocrystals. The hydrophilic phase of the membrane is represented by a system of cavities connected by narrow channels, usually containing hydrated cations [8], the structure of which can accommodate CdSe/CdS/ZnS nanocrystals (Figure 6). The change in the position of the luminescence maximum of the secondary emission of the nanocrystals introduced into the membrane, which is 9 nm (Figure 2b, dependences 1, 3), means a change in the height of the potential well relative to the surroundings, as well as a change in the effective size of the nanocrystals in the surroundings of the massive membrane molecular complex. According to the assessment, the diameter of CdSe and CdS nanocrystals has increased. This leads to the fact that the exciton localization region in nanoparticles increases [24]. Thus, the Nafion polymer membrane acts as a matrix, which together with nanocrystals forms an electronic structure different from the electronic structure of colloidal nanocrystals.

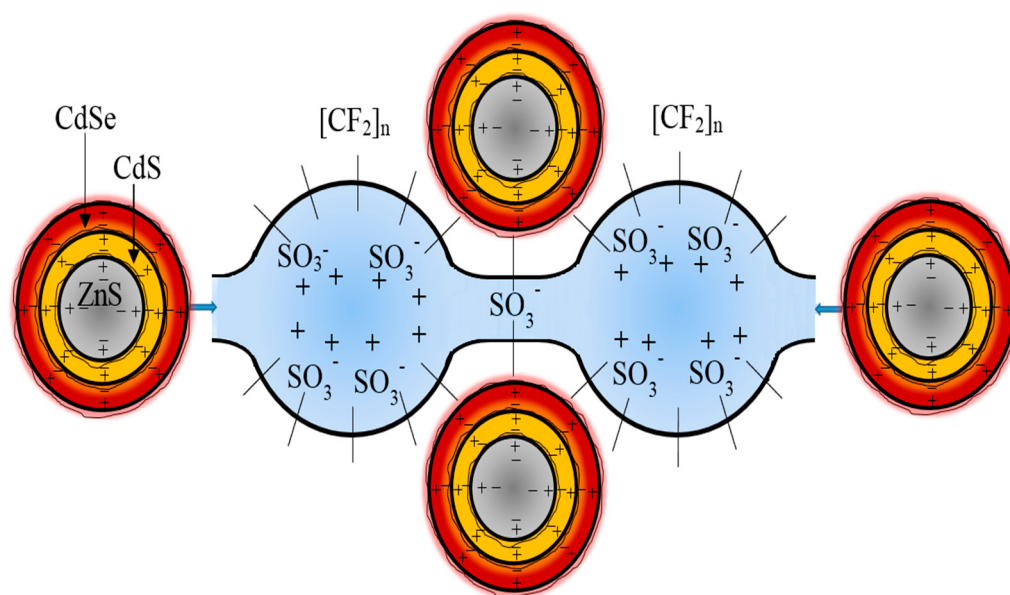


Figure 6. Schematic diagram of Nafion membrane links and colloidal CdSe/CdS/ZnS nanocrystals.

Figure 7 schematically shows possible electronic transitions observed during luminescence: a) for CdSe/CdS/ZnS nanocrystals; b) for the initial Nafion membrane; c) for the Nafion membrane with introduced CdSe/CdS/ZnS nanocrystals after membrane drying. In Figure 8 shows possible radiative transitions from excited electronic levels for the luminescence spectrum of wet Nafion membrane with introduced nanocrystals. It can be noted that due to the change in the mass of the formed molecular complex and the change in the size of nanocrystals, there was a shift in the luminescence lines compared to Figure 2a. The luminescence spectrum of the wet membrane shows additional intensity maxima at wavelengths of 433 nm and 458 nm, which were not observed before. The appearance of the luminescence band in Figure 2b (dependence 2) in the wavelength range 490 - 590 nm is similar to the intensity distribution of the original and soaked Nafion (Figure 2a, dependences 1,3). By the appearance of the observed dependence in Figure 2b (dependence 2), it is natural to assume that the new spectral components characterized by a monotonic decrease in intensity relative to the longer wavelength peak in the region of 433 nm and 458 nm are related to radiative transitions from excited electronic levels of the membrane. The sharp increase in the luminescence intensity of the wet membrane with introduced nanocrystals relative to the dried sample and the appearance of new spectral components can be related to the achievement of a higher power density of excitation radiation in a more optically dense medium due to the content of toluene in the membrane, the refractive index of which is 1.49. Due to a larger number of excitation photons, the occupancy of shorter-wavelength energy levels increases, which is manifested as low-intensity satellites at 433 nm and 458 nm. The secondary emission intensity of the membrane with introduced nanocrystals is greater than that of the original membrane and the nanocrystals themselves (Figure 2). The luminescence line width for the colloidal solution of nanocrystals in toluene is twice as large compared to the luminescence line width of the «Nafion membrane-nanocrystals» system. The increase in the intensity of secondary emission can also be observed as a result of resonance excitation of the substance's own exciton level [26] or the impurity center of the complex under investigation [27]. In this case, the luminescence spectrum takes the form of a broad band consisting of a large number of equidistant intensity maxima, which correspond to the manifestation of optical phonons of the substance.

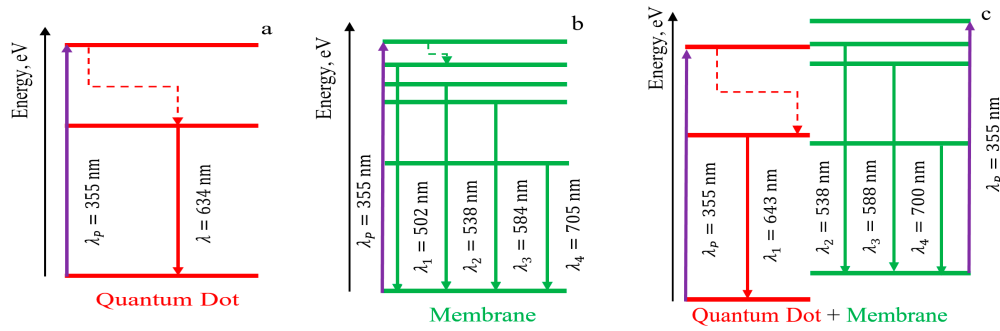


Figure 7. Structure of energy levels of the investigated systems: (a) *CdSe/CdS/ZnS* nanocrystals, (b) Nafion membrane, (c) dried Nafion membrane with introduced *CdSe/CdS/ZnS* nanocrystals.

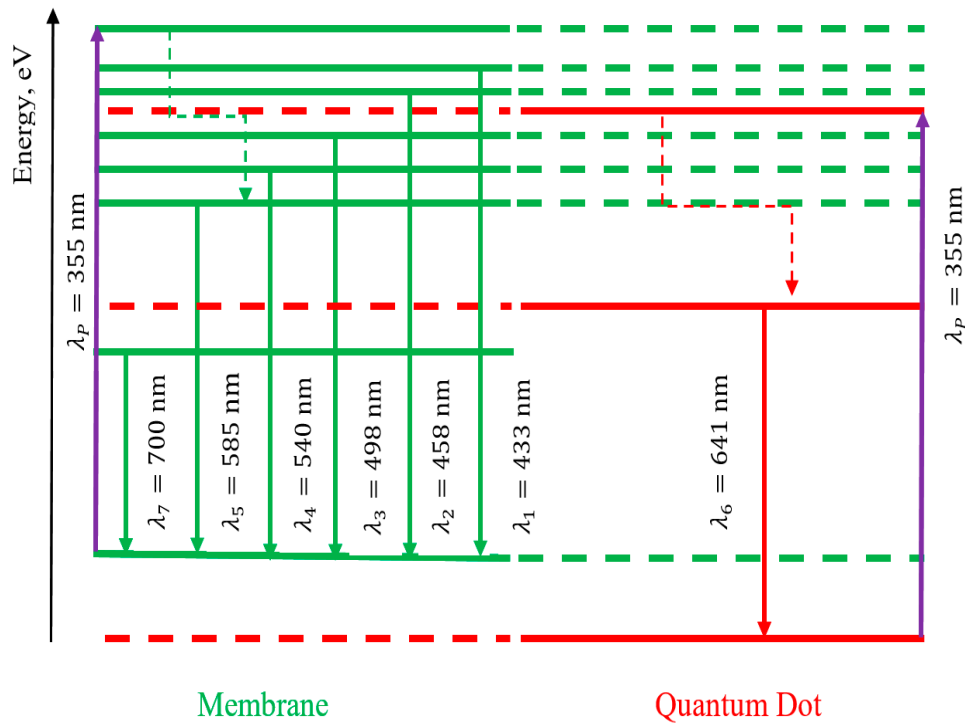


Figure 8. Schematic of energy levels of the wet Nafion membrane with introduced *CdSe/CdS/ZnS* nanocrystals: solid arrows indicate radiative transitions, dashed — non-radiative transitions.

To estimate the penetration depth of colloidal *CdSe/CdS/ZnS* nanocrystals into the Nafion membrane, we used the diffusion theory [28–30]:

$$\frac{1}{D_v} \frac{\partial C_v}{\partial t} = \Delta C_v \quad (1)$$

The interaction time of nanocrystals with the membrane exceeds the characteristic time $\tau = \frac{R^2}{D_v}$, which is of the order of tens of microseconds. It is taken into account here that the diameter of quantum dots in Nafion is 16.20 nm and the diffusion coefficient $D_v \approx 10^{-12} \frac{m^2}{s}$. The process of keeping the membrane in solution with colloidal nanocrystals was not time-limited. Therefore, if the condition $t \gg \tau$ is fulfilled, we can assume that $\frac{1}{D_v} \frac{\partial C_v}{\partial t} = 0$. For the one-dimensional case with a plate of thickness d , from the equation $\Delta C_v = 0$, the concentration distribution of colloidal nanocrystals will be defined as:

$$C(x) = C_{max} \left(1 - \frac{2}{d}x\right) \quad (2)$$

Unfortunately, the depth of nanocrystals penetration into the membrane could not be experimentally established. However, luminescence and laser emission spectroscopy experiments confirm a rather active interaction between the sulfone groups of the membrane and nanocrystals.

5. Conclusions

The results of luminescence, absorption and emission spectroscopy experiments evidence the creation of a molecular complex based on the proton exchange membrane Nafion and colloidal nanocrystals of CdSe/CdS/ZnS species. The characteristics of the luminescence spectrum of the membrane with nanocrystals differ significantly from the spectrum of the original membrane, solvent and embedded CdSe/CdS/ZnS nanocrystals. In our case, the pump radiation was 355 nm, and the spectral maximum of luminescence of the membrane with nanocrystals, which has the maximum intensity was observed at a wavelength of 643 nm. The absorption of optical radiation of the membrane with embedded nanocrystals in the range of 190...900 nm increases (Figure 3), the formation of new spectral lines of radiation absorption is observed. In fact, the polymer proton exchange membrane with embedded nanocrystals is a luminescent transducer, which is able to convert UV radiation into visible radiation. Such a converter can be used in the creation of luminescent balneological dressing in medicine or in plant cultivation and storage systems as a covering material. In our case, the pump radiation was 355 nm, and the spectral maximum of luminescence of the membrane with nanocrystals, which has the maximum intensity was observed at a wavelength of 643 nm.

Embedding of CdSe/CdS/ZnS nanocrystals into Nafion membrane will allow to create conversion and correction light filters of new generation. Polymer membrane with luminescence centers in the form of CdSe/CdS/ZnS nanocrystals is an element of optical system, which can be used to increase the coefficient of conversion of light energy into electrical energy by photocathodes and photodiodes, change the resistance of photoresistors. When solar cells and conversion light filters converting UV radiation into visible and diffused IR radiation are used together, it is possible to achieve a reduction in the heating of solar cells and an increase in the efficiency of solar cells.

Author Contributions: “Conceptualization, S.L.T.; methodology, S.L.T., A.V.S; investigation, E.N.Z., N.A.Z., A.V.S, S.L.T., E.A.Sh.; writing—original draft preparation, S.L.T; writing—review and editing, S.L.T., A.V.S., S.A.A.; visualization, A.V.S. All authors have read and agreed to the published version of the manuscript.

Institutional Review Board Statement: This study did not require ethical approval.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

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Conflicts of Interest: The authors declare no conflict of interest.

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