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# Spectroscopic properties of mixed Langmuir–Blodgett films of rhodamine dyes and poly(*N*,*N*-diallyl-*N*-octadecylamine-*alt*-maleic acid)

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**Abstract**

Langmuir–Blodgett (LB) films of poly(*N*,*N*-diallyl-*N*-octadecylamine-*alt*-maleic acid) as well as mixed films consisting of polyampholyte and two amphiphilic fluorophores – alkyl substituted rhodamine dyes – were prepared and investigated. The $\pi$–$A$ isotherm of the polyampholyte at air–water interface is typical for monolayer in the liquid state. Mixed monolayers of polyampholyte and rhodamine amphiphiles show improved packing of the hydrophobic chains. Absorption and fluorescence spectroscopic studies of the mixed LB films reveal the aggregation of dyes in the densely packed multilayer films.


*Keywords:* Langmuir–Blodgett films (LB films); Luminescence; Optical spectroscopy; Rhodamine; Polyampholyte

## 1. Introduction

At present thin films of organic materials are widely used in micro- and optoelectronics. There are several examples of obtaining thin-film devices for optical recording and storage [1,2], nonlinear optics [3,4], as sensors [5], etc. One of the most elegant ways of thin organic films assembling is the Langmuir–Blodgett (LB) technique. This method enables to design nanodimensional organic and bioorganic systems on solid substrates. The LB technology allows to assemble the organic dyes that have a high fluorescence quantum yield in the visible region of spectrum and to prepare specific nanostructures with predictable optical properties. Rhodamine dyes which have a high fluorescence quantum yields are good candidates for such purposes. The dyes can be chemically modified by long alkyl chains in order to convert them into amphiphiles and to make them suitable for spreading into insoluble Langmuir monolayers [6–11]. However the most monolayers made of pure amphiphilic rhodamine dyes have a poor stability and are hardly suited for the LB method. Densely packed monolayers with ordered arrangement of molecules can be obtained by co-spreading of amphiphilic dyes with a long chain standard amphiphiles, such as long chain fatty acids [6,7,10,11]. Earlier [10,11] the spectral-luminescent properties of mixed LB films of the amphiphilic rhodamine dyes **R1** and **R2** with stearic acid were investigated. The optical properties of these dyes in LB films were established. It was found that the fluorescence quantum yield can be tuned by either varying the concentration of dye molecules in the monolayer or changing the surface pressure of the monolayer in the course of transfer. In spite of the fact that co-spreading with stearic acid resulted in more densely packed monolayers of the luminophores and enabled the build-up of LB films, the monolayers were still in the liquid state.

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In mixed monolayers due to intermolecular interactions of dye and fatty acid molecules the monolayers are better preserved on the water surface. More densely packed monolayers of the luminophores can be obtained by substitution of low-molecular-weight amphiphiles by polymers [12–14]. Besides, the used polymers considerably improve the thermal and mechanical stability of the LB films. Since the chemical incorporation of dyes into amphiphilic polymers is painstaking and encounters various practical problems, two alternative strategies have been developed. Either the charged amphiphilic dyes are spread on sub-phases containing an oppositely charged polyelectrolyte, forming a polymer complex at the interface [15–19], or amphiphilic dyes are co-spread with polymeric amphiphiles [19,20] analogous to co-spreading with low-molecular-weight amphiphiles. Both strategies provide good results.

In this communication the LB films of regular hydrophobically-modified polyampholyte, namely poly(*N*,*N*-diallyl-*N*-octadecylamine-*alt*-maleic acid) (**PDAOM**), [21–23], as well as the mixed LB films composed of **PDAOM** and two amphiphilic fluorophores **R1** and **R2** are considered. The chemical structures of **PDAOM** and rhodamine dyes **R1**, **R2** are shown in Fig. 1. The **PDAOM** has a regular structure and bears one long alkyl chain at every sixth carbon of the hydrophilic, semi-flexible polymer backbone. There is a good match between the geometric requirements of the hydrophobic chain (minimal diameter in a monolayer is ca. 0.48 nm) and of the polymer backbone (maximal stretched length of the repeated unit is ca. 0.75 nm) in the monolayer for efficient self-organization and packing at air–water interface [12,13,24]. The negatively charged groups of **PDAOM** may provide the electrostatic interactions with cationic groups of **R1** and **R2**. The spectral-luminescent properties of dye molecules in LB films at different concentrations of luminophore and polymer have been studied.

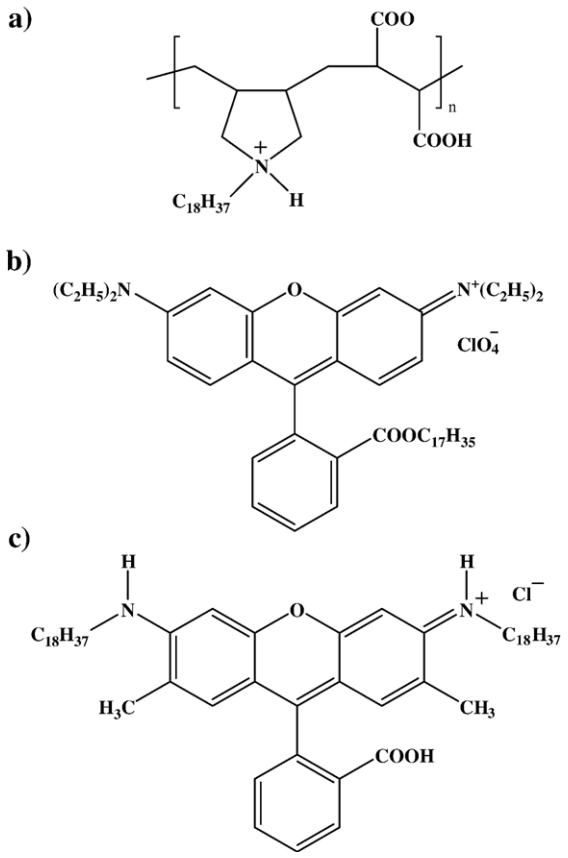


Fig. 1. Structure of polyampholyte **PDAOM** (a) and dyes **R1** (b), **R2** (c).

## 2. Experimental part

Synthetic details and characterization of **PDAOM** are given in Refs. [21–23]. Monolayers were formed at water–air interface using Langmuir trough. The automatized Langmuir device was assembled at the Russian State Scientific Centre "NIOPIK" (Moscow). The device consists of a teflon-coated trough and a control system. The trough has two independent sections which allow to deposit the layers of two systems in one dipping cycle. The trough is equipped with a Wilhelmi pressure pick-up system, a lifting–dipping mechanism, and moving barriers. The compression rate of the polyampholyte monolayer was 0.0012 $nm^2$/s/repeat unit in the course of measuring of $\pi$–$A$ isotherm and transferring of monolayer on substrate.

The surface tension of twice-distilled deionized water was equal to 72.8 mN/m at pH = 5.6 and temperature 17 °C. Fused nonluminous quartz was used as substrate. The polymer was spread on a Langmuir trough from three types of solutions: ethanol, mixtures of ethanol/chloroform (1/4 vol/vol), and ethanol/hexane (1/9 vol/vol). Mixed monolayers of polyampholyte and fluorophores were spread from ethanol/chloroform mixture (1/4 vol/vol). The relative concentrations of **R1** and **R2** in two-component monolayers of polyampholyte and fluorophore were 9, 17, 33, 50, 67 mol%. Monolayers were transferred onto quartz supports by vertical dipping according to Y-type transfer (transfer during downward and upward stroke) for polymer monolayers and according to Z type transfer for mixed dye-polymer monolayers (transfer during the upward stroke only) at surface pressure of 30 mN/m. For pure and mixed dyes monolayers the transfer coefficient during downward stroke (X type) was extremely small.

The dipping speed through the monolayer was 0.02 mm/s. The thickness of the films consisted of 20 monolayers.

Absorption and fluorescence spectra were measured by using a KSVU-23 spectral universal setup. Quantum yields of fluorescence of dye LB films were determined according to Ref. [25], using $10^{-6}$ M solutions of dyes **R1** and **R2** in ethanol as standards.

## 3. Results and discussion

The surface pressure–area isotherm of polyampholyte is shown in Fig. 2. Three types of solvents, in particular ethanol, mixtures of ethanol/chloroform (1/4 vol/vol), and ethanol/hexane (1/9 vol/vol), were tested for spreading of polymer on water subphases [26]. The ethanol/chloroform mixture showed the best spreading of film and therefore was used for further experiments. As seen from Fig. 2, the isotherm of **PDAOM** has the typical shape of a monolayer in the liquid state [27,28]. The

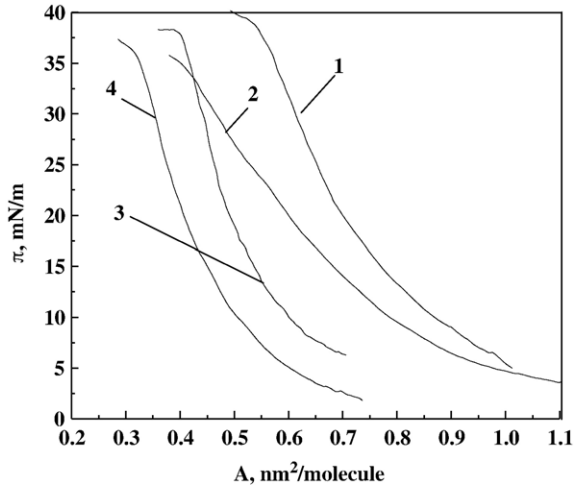


Fig. 2. Surface pressure–area isotherms at 20 °C of polyampholyte **PDAOM** (1), dye **R1** (2), polymer:dye **R1** (50:50 mol%) (3) and polymer:dye **R1** (83:17 mol%) (4).

collapse pressure of polymer monolayer is about 40 mN/m, and the surface area at the collapse is about 0.55 $nm^2$/repeat unit. This value is much higher than the value of 0.20 $nm^2$/repeat unit and is characteristic for a densely packed octadecyl chain [27], supporting the liquid-like nature of the monolayer. At the transfer pressure of 30 mN/m the occupied area was equal to 0.64 $nm^2$/repeat unit.

The stability of polyampholyte monolayers was studied. In the first case the change of monolayer area was studied at constant surface pressure ($\pi$=30 mN/m). During an observation period of 2 h, the area changed only insignificantly within the first 10 min, and then became constant. In the second case, at initial pressure ($\pi$=30 mN/m) the change of surface pressure upon stopping the barrier in the course of compression (i.e., monolayer area $A$ was constant) was observed. The surface pressure decreased only by 2 mN/m within 2 h. These results confirm the high stability of the **PDAOM** monolayer. The analogous experiments were performed for mixed monolayers of **PDAOM**. It was established that the mixed monolayers were stable too.

The shape of the $\pi$–$A$ isotherm of dye **R1** (Fig. 2) is typical for the liquid state [27,28]. Starting from 1.5 $nm^2$/molecule, the surface pressure rises gradually up to 0.42 $nm^2$/molecule with a collapse pressure at 35 mN/m. At transfer pressure of 30 mN/m the limiting area of dye molecule obtained at $\pi \rightarrow 0$ mN/m is 82 $Å^2$.

The $\pi$–$A$ isotherms of the mixed monolayers **R1**–**PDAOM** differ considerably from those of the individual compounds (Fig. 2). The surface pressure begins to rise at much more lower surface areas and the increasing of surface pressure upon compression is much steeper. The collapse area is about 0.42 $nm^2$/molecule with a collapse pressure at 38 mN/m for a molar ratio of 50/50 and 0.33 $nm^2$/molecule with a collapse pressure at 36 mN/m for a molar ratio of 17/83 (dye/polymer repeated unit). The strongly enhanced packing density of both components, e.g. dye **R1** and polymer **PDAOM**, in the mixed monolayers is observed. It might be explained by insertion of hydrophobic parts of dye molecules into the defectively packed octadecyl side chains of the polymer, as well as by conformational rearrangement of the hydrophilic parts of the polymer due to strong electrostatic interactions between the anionic moieties of **PDAOM** and cationic groups of dye. More condensed character of the mixed monolayers of polymer and dye is more suited for LB deposition than pure **R1** monolayer.

The surface pressure–area isotherm of dye **R2** is shown in Fig. 3. In contrast to **R1**, its collapsed area is about 0.5 $nm^2$/molecule and corresponds to collapse pressure of 37 mN/m. This is due to fact that the **R2** bears two octadecyl chains and is more densely packed. The mixed monolayers of **PDAOM** and **R2** exhibit the increased surface pressures only at higher surface concentrations than that of individual components.

Polymer monolayers were transferred onto quartz substrates by vertical dipping showing to Y-type transfer. The transfer coefficient of the monolayer for the downward stroke was 0.82, and for the upward stroke was 1.15. The average transfer coefficient was equal to 0.99. The number of the monolayers in LB films was 20.

The Y-type deposition of the polymer monolayer with a high transfer coefficient is assumed to result in LB films made of centro-symmetrical sandwich bilayers, in which the polar and hydrophobic parts interact with each other (Y-type multilayer). Since the investigated polymer is a polyampholyte [21], the electrostatic interactions between $R_3N^+H$ and $–COO^-$ groups are strong enough to allow multilayer build-up. The higher value of the transfer coefficient of the polymer monolayer for upward stroke than for downward stroke is a frequently observed phenomenon.

The formation of LB films from the pure **PODMA** was confirmed by measuring the electronic absorption spectra of the films in the far UV region. In the spectra we observed only part of absorption band the maximum of which was at 200 nm (Fig. 4). This band is attributed to carbonyl group, being the only chromophore in the polymer.

Mixed dye-polymer LB films were transferred according to Z type transfer. The films of the dyes **R1** and **R2** showed an

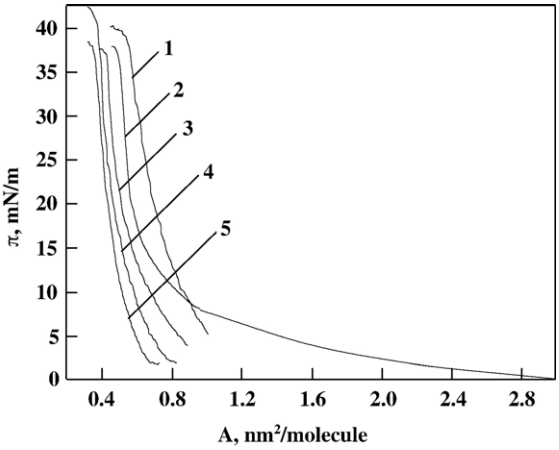


Fig. 3. Surface pressure–area isotherms of polyampholyte monolayer **PDAOM** (1), dye **R2** monolayer (2), polymer:dye **R2** (50:50 mol%) monolayer (3), polymer:dye **R2** (67:33 mol%) monolayer (4), polymer:dye **R2** (83:17 mol%) monolayer (5) (the areas are normalized to the average number of moles dye and polymer repeated unit).

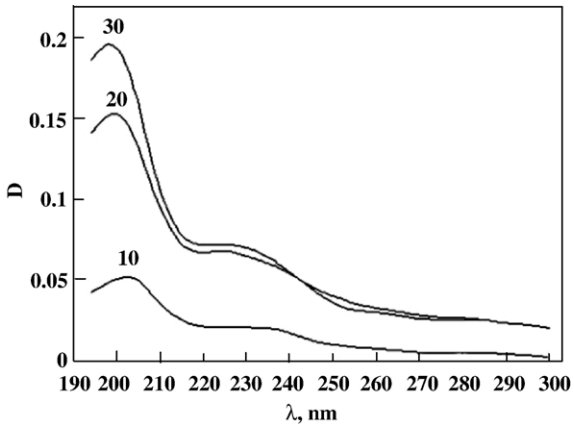


Fig. 4. Electronic absorption spectra of polymer LB films of different thickness (the number of monolayers is shown above the spectral curve).

inhomogeneous discontinuous structure which probably is connected with formation of dye molecules aggregates in the monolayer as observed earlier for similar rhodamine dyes [7].

Absorption and fluorescence spectra of the **R1**, **R2** LB films in the visible region are broadened and shifted in comparison with ethanol solutions. This indicates on aggregation of dye molecules leading to densely packed films. According to the exciton model of the molecular aggregates [7,8,29], the splitting of the $S_1$-electronic excited level in dimers occurs on two sublevels, respectively with higher and lower energies. The short-wave transition band is related to "parallel" dimers ("sandwich" dimers), whereas the long-wave transition band is related to "plane" dimers. Importantly, only "plane" dimers are able to fluoresce [30].

Absorption and fluorescence spectra of mixed LB films of the **R1** at concentrations of 17 and 50 mol% are shown in Fig. 5. There are two bands in absorption spectra. The band with highest intensity ($\lambda_{max}$=565 nm for the film with dye concentration of 50 mol%) is red shifted in comparison with the absorption band of the **R1** in ethanol solution ($\lambda_{max}$=546 nm). The low-intensive absorption band ($\lambda_{max}$=528 nm for the film with dye concentration 50 mol%) observed as shoulder is shifted hypsochromically compared to the monomer in ethanol solution. When the

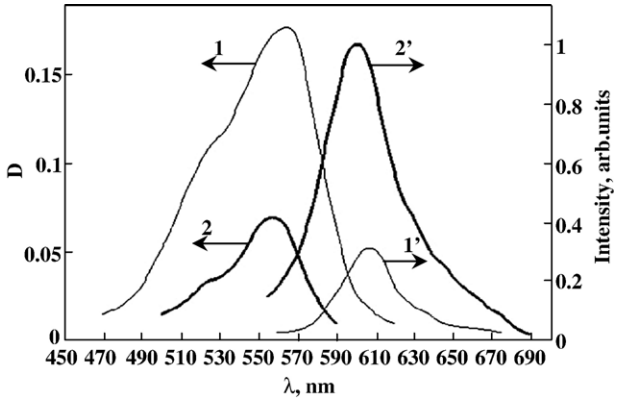


Fig. 5. Absorption (1,2) and fluorescence (1′,2′) spectra of the mixed LB films of dye **R1**–polymer **PDAOM** at dye concentration of 50 mol% (1, 1′), and 17 mol% (2, 2′).

concentration of **R1** decreases in the film, the maxima of the long-wave band and the shoulder shift hypsochromically. Moreover, the ratio of long-wave and short-wave band intensities increases. For example, at concentration of dye 17 mol%, the bands are shifted to the blue region by 5–7 nm compared to the concentration of 50 mol%. The ratios between the band intensities for concentrations of 17 and 50 mol% are 0.50 and 0.66 respectively.

Fluorescence excitation of LB films of **R1** was done at 540 nm. The obtained fluorescence spectra are red shifted compared to those of the dye in ethanol ($\lambda^{fl}_{max}$=582 nm). In addition, when the dye concentration increases in the film, significant quenching of the fluorescence is observed (Fig. 5). LB films which have the ratio of dye and polymer 17:83 mol% exhibit the highest fluorescence intensity and quantum yield (Table 1). Besides, increasing of the dye concentration results in a small red shift of the maximum of the fluorescence spectrum. The emission maxima are 600 and 607 nm for the concentrations of 17 and 50 mol% respectively.

The shape and position of the absorption bands of the **R1** LB films reveal the formation of mainly "plane" dimers with a small number of "parallel" dimers. Along with these types of dye aggregates, the existing of monomers is also probable. Their absorption band overlaps with the aggregate bands. Decreasing of dye content in films leads to decreasing of the probability of both "plane" and "parallel" dimer formation. As a result, a blue shift and a decreasing of the intensity of shoulder in absorption spectra take place. The changes observed in the fluorescence spectra confirm these assumptions. The red shifted fluorescence band corresponds to a radiative transition from the lower exciton level of "plane" dimers which are able to fluoresce. The observed fluorescence spectra probably consist of monomer and "plane" dimer bands, where the dimer fluorescence is predominant. It is well known that the aggregation processes result in effective quenching of dye fluorescence [31]. It can be caused, in particular, by inactive absorption of aggregates which do not fluoresce and by nonradiative transfer of electron excitation energy from monomers to nonluminescent aggregates which serve as energy traps. Besides, even dimers, for which radiation transitions are allowed, have a very high probability of intersystem crossing because of approaching singlet and triplet levels [29]. This nonradiative process competes with fluorescence. The fluorescence quantum yield of the LB films with dye **R1** decreases (Table 1).

Irradiation at 540 nm excites "parallel" dimers and monomers. But because of the dense packing of dye molecules in film, the excited "parallel" dimers and monomers lose their energy

Table 1
Spectral-luminescence characteristics of dye **R1** in mixed LB films at different dye concentrations

| Dye concentration, (mol% per repeated unit) | Peak optical density | Fluorescence quantum yield |
|---|---|---|
| 50 | 0.18 | 0.03 |
| 33 | 0.11 | 0.08 |
| 17 | 0.07 | 0.30 |
| 9 | 0.06 | 0.19 |

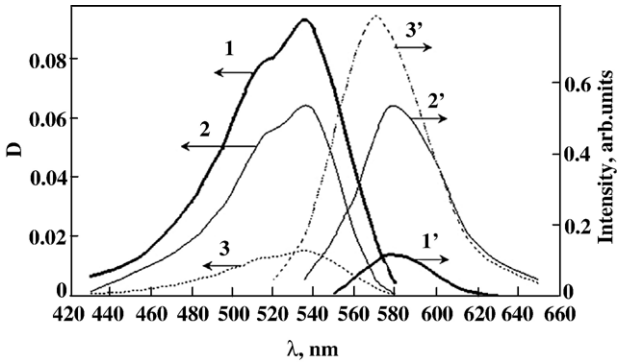


Fig. 6. Absorption (1,2,3) and fluorescence (1′,2′,3′) spectra of the mixed dye **R2**–polymer LB films at dye concentration of 50 mol% (1,1′), 33 mol% (2,2′), 17 mol% (3,3′).

through energy migration process to the lower exciton level of the "parallel" and "plane" dimers due to dipole–dipole energy transfer. As mentioned above, the "plane" dimers of the dye **R1** give fluorescence maxima at about 600–610 nm, whereas "parallel" dimers lose their energy by nonradiative decay. As seen from the ratio of shoulder and long-wave band, with decreasing of dye concentration in films the number of "parallel" dimers (energy traps) decreases faster than that of "plane" dimers. Therefore the fluorescence intensity increases.

Absorption and fluorescence spectra of mixed LB films of **R2** and **PDOAM** are shown in Fig. 6. The absorption and emission bands are deformed in comparison with spectra of dye itself in dilute ethanol solution, where the dye molecules are molecularly dispersed. This suggests a strong aggregation of the fluorophores. The absorption spectra display two maxima at shorter ($\lambda_{max}$=512 nm for the film with dye concentration of 50 mol%) and longer wavelengths ($\lambda_{max}$=535 nm for the film with dye concentration of 50 mol%) with respect to fluorophore in ethanol solution ($\lambda_{max}$=525 nm). The ratio between the intensities of the short-wave and long-wave absorbance maxima ($\alpha$) of **R2** LB films is higher than that of **R1** LB films (for the dye concentration of 50 mol% the **R1** has $\alpha$=0.66 and the **R2** has $\alpha$=0.85). Moreover in comparison with the **R1**, the maximum of the long-wave band of **R2** is less red shifted with respect to the dye band in ethanol solution. The fluorescence spectra of **R2** in LB films show a slightly broadened band with similar shape and maximum position compared to ethanol solution. For example, the half-widths of the fluorescence bands in solution and in the LB film at dye concentration of 17 mol% are 43 nm and 52 nm, respectively. The fluorescence spectra are blue shifted with decreasing of **R2** concentration in the film as in the case of **R1**. For example, the fluorescence maximum of the LB film at dye concentration of 17 mol% is blue shifted by 10 nm compared to corresponding value at dye concentration of 50 mol%. Moreover, **R2** fluorescence intensity decreases significantly with increasing of dye concentration in the film (Fig. 6).

The absorption spectra of **R2** LB films can be interpreted as superposition of monomer and dimers spectra of two types. According to the exciton model, the presence of an intensive short-wave shoulder ($\lambda_{max}$=512 nm) and a long-wave band ($\lambda_{max}$=535 nm) in the LB film indicates on the formation of "parallel" and "plain" dimers, respectively. The values of $\alpha$ show that the number of "parallel" dimers formed in **R2** LB films is higher than that of LB films with **R1**. As seen from $\pi$–$A$ isotherm and conformational analysis of **R2**, the parallel configuration of dimers should have lower energy, because the interactions between the hydrophobic chains probably stabilize this configuration. The close packing of molecules in the LB film also results in the formation of a small number of "plain" dimers. Fluorescence excitation of **R2** LB films was carried out from the short-wave side of the absorption curve by irradiating at 520 nm. As in case of **R1** both "parallel" dimers and monomers of **R2** were excited.

As a result of inactive "parallel" dimers absorption and migration of electron excitation energy from monomer to nonluminescent dimers, the fluorescence quantum yields of the **R2** LB films are low. Because of the small number of "plain" dimers in LB films with **R2**, no individual band corresponding to dimers in the absorption spectrum is observed. The spectrum belongs to monomers mainly. Absorption of the "plain" dimers results in only a little broadening of the spectra. With decreasing of molar concentration of dye in the film, the number of aggregated molecules, particularly "plain" dimers, decreases, resulting in a blue shift of the absorption spectra.

Fatty acids are often used for obtaining a stable condensed monolayers and LB films in which the organic dyes are incorporated. But most of them have low melting temperatures and thermally are unstable. Therefore, the thermal stability of the LB films based on **PDOAM** was investigated. Two types of LB films containing **R1** were prepared and compared. For the first type of LB film the **PDOAM** was used, while stearic acid was selected for the second type of LB film as reference. Both LB films were heated up to 200 °C under the same conditions and kept at this temperature for 30 min at atmospheric pressure. After heating the optical density ($D$) of polymer LB film with $\lambda_{max}$=200 nm decreased by a factor of 1.3 compared to initial value. For LB film on the basis of stearic acid the $D$ decreased at $\lambda_{max}$=200 nm by a factor of 2. For the dye absorption peak ($\lambda_{max}$=565 nm), the $D$ decreased by a factor of 1.5 and 2 for LB films on the basis of the polymer and stearic acid, respectively.

In order to increase the mechanical and thermal stability of multilayers the "sandwich-like" LB films were prepared. Their structure consisted of 10 layers of **PDOAM**, 20 mixed layers of polymer and dye **R1** (17:83 mol%), and 10 layers of **PDOAM**. The electronic absorption spectra in the visible region and the fluorescence spectra of the "sandwich-like" films coincided well with the spectra of LB films made from 20 layers with the same ratio of dye **R1** and polymer. The "sandwich-like" multilayer structure was annealed at 80 °C under atmospheric pressure for 4 h. In the course of such procedure the electronic absorption spectrum of sample was not changed, however the fluorescence intensity increased eight times. The drastic increase of fluorescence can be explained by defect healing, which takes place during the annealing of the film. Such defects could act as electron excitation energy traps. This assumption is reasonable because the subsequent annealing of the sample does not give any change of the fluorescence intensity.

## 4. Conclusions

Molecular monolayers of an amphiphilic polyampholyte and amphiphilic rhodamines **R1** and **R2** form stable monolayers. These mixed monolayers are well suited for transfer onto solid substrates by the LB method. The mixed multilayer films are thermally and mechanically more stable than analogous mixed films made of long chain fatty acids and dyes. The absorbance and fluorescence spectra of the mixed polyampholyte-dye multilayers depend on the amount of amphiphilic dye incorporated, as well as on the number of deposited monolayers. For the fixed molar ratio of dye and polyampholyte (17:83 mol%), the highest fluorescence quantum yield and the most effective emitting molecular systems are observed.

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