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Luminescent Properties of Polycarbonate Methacrylates Containing Organic Fluorescent Dyad

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Abstract: The luminescent properties of photocured aliphatic and aromatic network polycarbonate methacrylates, containing organic fluorescent dyad, have been studied. The dyad molecule includes two photosensitive fragments—tetraarylimidazole with a hydroxyl group and azomethinocoumarin—in each of which excited state intramolecular proton transfer (ESIPT) occurs, leading to the appearance of two emissions: blue (450 nm) and green (535 nm). It was established that the ratio of the intensities of these emissions depends very significantly on the excitation wavelength, as well as on the length, flexibility and polarity of the matrix oligomeric bridges.

Keywords: optical materials; oligocarbonate methacrylates; organic dyad; ESIPT; tunable fluorescence



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1. Introduction

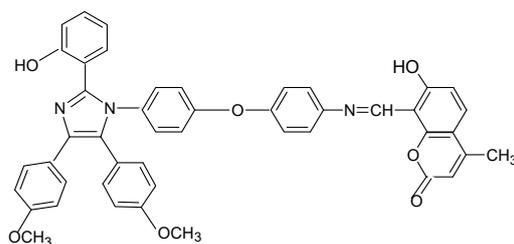
The main directions of oligocarbonate methacrylates studies were initiated by Al. An. Berlin, that were then further successfully elaborated; the formation mechanism of oligocarbonate methacrylates was verified that opened wide the possibilities for the control of the properties of these types of oligomers. Oligocarbonate methacrylates were modified to improve their thermal and thermal-oxidative stability [1–5]. Now, oligocarbonate methacrylates occupy a special place among photopolymerizable acrylic monomers due to their good polymerization ability, lack of coloring, non-toxicity and due to the actual properties of the network polymers formed from them. Their good dielectric characteristics, high impact resistance, low specific gravity, high thermal stability, fire safety, ease of processing, good sound insulation performance, resistance to chemical and mechanical influences and environmental and biological safety determine the wide demand for these polymers in various areas in the production of new materials. They can be used for the fixing, encapsulation and sealing of various products, the assembly of threaded products, sealing of pipelines and connections, and for the encapsulation and moisture protection of the assemblies and modules of different devices in electronic equipment [6].

They are also widely used for the manufacturing of optical products and coatings, especially in radiation conditions [4,7]. Their application in stereolithography is described, for example, in the layer-by-layer manufacture of parts from liquid oligomers, including oligocarbonate methacrylate, using an excimer XeCl laser ($\lambda = 308$ nm) [8]. Now, photopolymerization processes such as direct laser writing, three-dimensional micromachining, optical elements fabrication and data recording and storage are widely distributed, and they all use oligocarbonate methacrylate compositions [9,10]. These functionalized five-member cyclic carbonates can be precursors for a wide variety of polymers [7]. Moreover, recently, a near-infrared photopolymerization, in the depth of photocurable composition, on the

base of oligocarbonate methacrylate and up-conversion nanoparticles, has been developed to produce scaffolds for biomedical applications [11].

At the same time, organic luminophors can be rightfully attributed to a number of important materials in new technologies. They have found application in quantum electronics, fluorescent flaw detection, in the aniline and plastics industry, in biology and medicine and for many other special purposes [12–16].

Previously [17], we synthesized a fluorescent dyad, the molecule of which includes two different photosensitive fragments—tetraarylimidazole with a hydroxyl group and azomethinocoumarin—within each of which intramolecular proton transfer in the excited state (ESIPT) occurs. The photosensitive fragments are connected by a diphenyl oxide bridge, which prevents the energy transfer between them [18]. The formula of the studied dyad is given below.



The spectral-luminescent properties of the dyad in solutions were described in [17]. It was shown that this compound is characterized by dual fluorescence with maxima at 430–450 and 530 nm; the ratio between them depends on the solvent nature and the excitation wavelength. The study of the dyad luminescence in the matrices of linear polyoligoester acrylates with different number of hydroxyl groups [19] demonstrated that the presence of hydroxyl in the polymer matrix has a moderate effect on the relative intensities of the two fluorescence bands. The reason for this effect is the hydrogen bonds' formation between the dyad and polymer.

Taking into account the intensive development of luminescent materials, the increase in demand for them and the growth in requirements for their characteristics, the possibility of introducing a luminophore into oligocarbonate methacrylates, and studying the features of the dyad fluorescence manifestation in the network polymers of oligocarbonate dimethacrylates, is of great interest, since they can find practical applications. Therefore, the aim of this study is to elucidate the influence of the polymer matrices' structure and morphology, within a class of polymers without hydroxyl groups, on the luminescent properties of the dyad in order to find its most pronounced effects.

2. Materials and Methods

CH_2Cl_2 (spectroscopic grade) and tetrahydrofuran (THF) were purchased from Acros Organics. Photoinitiator Darokur 4265 was purchased from Aldrich. Ethylene glycol monomethacrylate was purchased from Alfa Aesar and ethylene glycol bischloroformate, diethylene glycol bischloroformate, 2,2-diphenylpropane bischloroformate and resorcinol bischloroformate were purchased from LLC "Korund" (Russia).

2.1. OCM Synthesis

Earlier Soviet scientists developed a method of OCMs synthesis based on single-stage nonequilibrium low-temperature condensation–telomerization between bis- or polychloroformates of glycols or bisphenols (or without them), and mono- (met-)acrylic esters of glycols and bisphenols [1]. The reaction is carried out in a solution of CH_2Cl_2 , or other lower chloralkanes, in the presence of organic bases—acceptors of the released HCl at a temperature from $-10\text{ }^\circ\text{C}$ to $40\text{ }^\circ\text{C}$. In all cases, it is also very important to use completely dry reagents to obtain individual compounds. OCM-1, 2, 5 and 7 were obtained by this method, with yields $\approx 85\%$ – 90% ; their synthesis is described in detail in the patent [20] and the general scheme of the synthesis is presented below.

the organic layer was washed with water until a neutral reaction, and an absence of chlorine ions, were obtained, then dried over calcined sodium sulfate and filtered, after which the solvent was distilled off at a residual pressure of 1–2 mm Hg and a temperature of 25 °C. The residue after distillation of the solvent was an almost colorless transparent product.

Under similar conditions, the remaining oligocarbonate methacrylates were obtained. In the case of OCM-1, ethylene glycol bischloroformate, 2,2-diphenylolpropane bischloroformate and resorcinol bischloroformate were used in the case of OCM-5 and OCM-7, respectively, instead of diethylene glycol bischloroformate.

OCM-2/1 was synthesized according to the method described in [21].

2.3. Detailed Description of OCM-2/1 Synthesis

In a 4-necked flask fitted with a stirrer, a thermometer and a dropping funnel, a solution of 1 mol of ethylene glycol monomethacrylate in 160 mL methylene chloride, 1 mol of ethylene glycol bischloroformate and 0.5 mol of diethylene glycol was placed and cooled to -10 °C. Then, a solution of 82 mL pyridine in 160 mL of methylene chloride was added, dropwise, at such a rate that the temperature of the reaction mass did not rise above -5 °C. Then, the reaction mass was heated to 42 °C and kept at this temperature with stirring for 5–6 h. After cooling to room temperature, the reaction mixture was treated with 1 L of 3% HCl and then washed with distilled water until neutral. The mixture was dried with sodium sulfate and filtered. An amount of 0.03% of the weight of the synthesized oligomer of hydroquinone was added. Then, the solvent was distilled off at room temperature and under reduced pressure.

The study of the obtained OCM-1, 2, 5 and 7 by GPC (gel permeation chromatography) methods showed that they are practically individual compounds, and that their elemental composition, and main physical-chemical characteristics—density, viscosity and n_D^{20} values—coincide with the published data [20,21]. In the case of OCM-2/1, several oligomergomologs are always formed, which are characterized by a certain molecular mass distribution, which depends on the order of the components' addition [21].

The purity of the used oligomers was 97%, all reagents were tested by chromatography before use. To determine the MMR (molecular mass distribution) and MW (molecular weight) of oligomers, a liquid gel chromatograph GPH-200 (Waters), with three styrogel columns with porosities of 100, 500 and 100 Å THF solvent feed rate = 1 mL/min, temperature = 25 °C, was used.

The polymer films under study were prepared by the photopolymerization of monomers in the presence of added luminophore dyad in methyl chloride (followed by its removal) and photoinitiator Darokur 4265. The photocuring was performed upon irradiation by a DRT-1000 UV lamp (Russia) through a PS 13 filter for 3 min, which provided a depth of curing equal to 92%–98% and the preservation of the absorption spectra of the dyad in the monomer. The concentration of the dyad in most of experiments was 10^{-4} mol/L (except those for the relative quantum yield measurement, see below); the concentration of the photoinitiator was 1 weight %. The thickness of the resulting polymer films was 800–900 μm .

A Spectrophotometer Carry 60 «Agilent Technologies» was used for steady-state UV-Vis absorption studies. Steady-state UV-Vis fluorescence was measured using spectrofluorimeter Cary Eclipse (Varian) with optical slit width 5 nm. All measurements were carried out at room temperature using holders for the polymer films. The orientation of the films in the holder for fluorescence measurements was 60° relative to the excitation slit and 30° relative to the registration slit. In order to compare the fluorescence efficiency of the dyad in different films, the relative quantum yields were determined by measuring the areas under the emission spectra at the excitation wavelength 350 nm, where the absorption of the polymer matrix was negligible. The absorption at the excitation wavelength was 0.1 for all samples studied in order to determine the relative quantum yields by comparison of the areas under the emission spectra [17,22].

3. Results and Discussion

All compounds are carbonic acid esters and contain carbonate groups (-O-CO-O-). The presence of two end methacrylic groups in each molecule ensures the formation of a spatial three-dimensional net of polymethacrylate chains, connected by transverse oligomeric blocks, during polymerization. That is, the studied polycarbonate methacrylates differ in the nature, length, flexibility, and polarity of their oligomeric bridges. Polymers obtained from OCM-1, OCM-2 and OCM-2/1 are aliphatic polymers, and polymers obtained from OCM-7 and OCM-5 contain aromatic fragments. In addition, the content of simple ether bonds in the structure of polymers affects the physical properties of the polymers and, as this study has shown, it also affects their fluorescent properties.

The relative quantum yields of fluorescence in these matrices are 1.0, 0.5, 0.5, 0.6 and 0.7 for OCM-1, OCM-2, OCM-7, OCM-5 and OCM-2/1, respectively. These values are practically the same in all these matrices and the larger value is obtained only for OCM-1, characterized by the smallest net size, which probably leads to a decrease in the contribution of radiation-less relaxation within it.

A photostability test was carried out for the dyad in the polymer matrix obtained from OCM-2. No changes were obtained in the absorption spectra after photolysis with a halogen lamp of 50 w/m² intensity through a SS-4 light filter (transmission range 340–470 nm), at a distance of 0.15 cm for 5 h.

Table 1 shows the fluorescent properties of photo-cured oligocarbonate methacrylates containing a luminescent dyad.

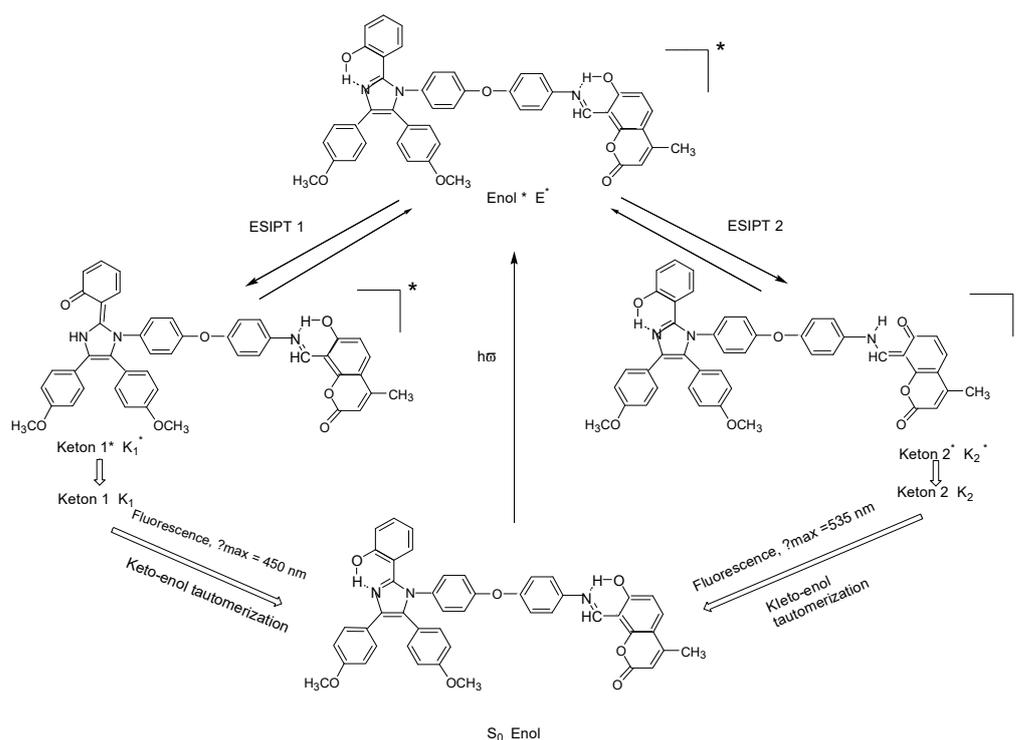
Table 1. The ratio between intensity of dyad fluorescence at 450 nm and 535 nm in oligocarbonate methacrylate polymers at different excitation wavelengths.

OCM	MW	Ratio of Fluorescence Band Intensities with Maxima at 450 nm and 535 nm, $\lambda_{ex} = 330 \text{ nm}$	Ratio of Fluorescence Band Intensities with Maxima at 450 nm and 535 nm, $\lambda_{ex} = 350 \text{ nm}$
OCM-1	374	3.45	2.15
OCM-2	418	1.23	0.73
OCM-7	420	1.05	0.64
OCM-5	540	0.79	0.59
OCM-2/1	683	2.94	1.98

As it can be seen from the data given in Table 1, the fluorescent dyad in almost all of the photo-cured samples of oligocarbonate methacrylates studied is characterized by two emission bands, which can be attributed to proton transfer processes in both luminophore fragments—imidazole (ESIPT band 1, $\lambda_{max} = 450 \text{ nm}$)—and those of coumarin (ESIPT band 2, $\lambda_{max} = 535 \text{ nm}$) (Scheme 1).

Let us start with a consideration of aliphatic polymers' properties. Their fluorescence spectra are shown in Figures 1–3. It should be noted that all spectra are normalized to a 450 nm band.

As can be seen from the data presented in Figure 1, the fluorescence intensity in the aliphatic polymer OCM-1 with the smallest link length depends on the excitation wavelength. The most intense blue band, with a maximum at 450–460 nm under the short wavelength (330–340 nm) excitation, only had an asymmetric long wavelength shoulder, which transformed into a clearly defined green band of 530–550 nm at $\lambda_{ex} = 350\text{--}360 \text{ nm}$.



Scheme 1. Schematic representation of ESIP processes in the dyad. * In the figure indicates the electronically excited state.

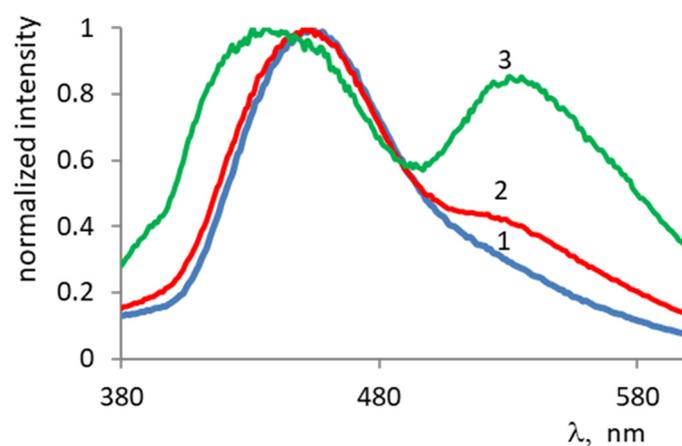


Figure 1. Emission spectra of the dyad in the polymer films obtained by photo-curing of OCM-1, at $\lambda_{ex} = 330$ nm (1), 350 nm (2) and 360 nm (3).

The increase in the length of the oligomeric bridge from $-\text{CH}_2\text{CH}_2-$ in the polymeric films obtained from OCM-1—to $\text{CH}_2\text{CH}_2\text{-O-CH}_2\text{CH}_2$ in the polymeric films obtained from OCM-2, leads to a larger globule size and an increase in the flexibility of the OCM-2 [23] film that, in turn, results in more effective ESIP in the azomethinocoumarin fragment and is accompanied by a change in the luminescence spectrum (Figure 2).

In this case, the fluorescence of the dyad in the OCM-2 polymer film is characterized by the presence of two emission bands with maxima at 450 and 550 nm at all λ_{ex} from 330 to 440 nm. Here, the fluorescence intensity is noticeably less than in the more rigid OCM-1 polymer film. It should be noted that an increase in the excitation wavelength leads to an increase in the long-wavelength emission.

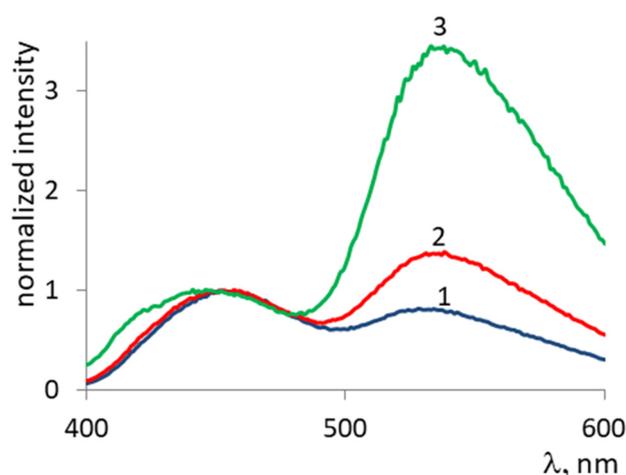


Figure 2. Emission spectra of dyad in the polymer films obtained by photo curing of OCM-2, at $\lambda_{\text{ex}} = 330$ nm (1), 350 nm (2) and 360 nm (3).

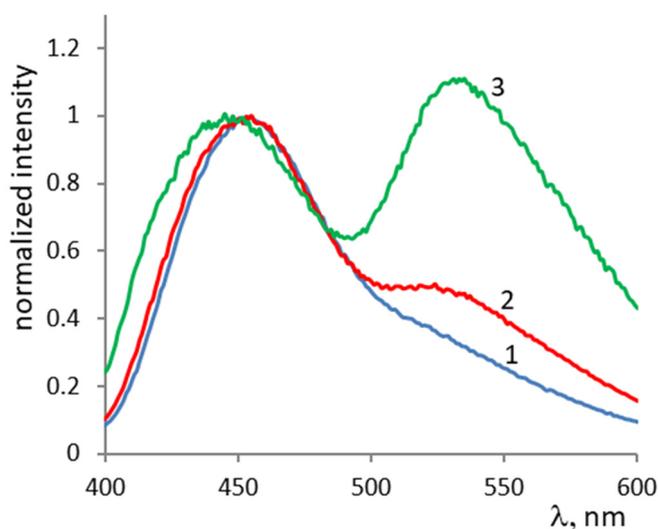


Figure 3. Emission spectra of dyad in the polymer films obtained by photo curing of OCM-2/1 at $\lambda_{\text{ex}} = 330$ nm (1), 350 nm (2) and 360 nm (3).

The use of oligocarbonate methacrylate OCM-2/1 as a matrix for the introduction of the dyad is characterized by an elongation of the oligomeric bridge in the polymer molecule due to an increase in the number of ethylene–ether bonds and carbonate groups. This leads to a change in the fluorescence spectrum of the dyad (Figure 3), which becomes similar to the spectrum in the most rigid polymer, OCM-1 (Figure 1).

It can be seen from the spectrum that the fluorescence of the dyad in the polymer obtained from OCM-2/1 is characterized by a sufficiently intense blue band, with a maximum at 450 nm, and a less intense long-wavelength band, with a maximum at 530 nm. The intensity of the bands depends on the excitation wavelength. In the polymer matrices with the longest flexible oligomeric bridge, OCM-2/1, the imidazole luminescence band predominates again. This is probably due to the intermolecular interaction of long oligomeric blocks in network polymers, leading to the formation of associates capable of tightening the polymer, as described in [24]. From the data obtained for the studied aliphatic polycarbonate methacrylates, it can be seen that OCM-2 is the most suitable for the observation of double fluorescence.

Next, we considered the luminescent properties of the dyad in polymer matrices of OCM containing aromatic fragments in an oligomeric polymer block. Figure 4 shows the luminescence spectra of the dyad in the polymer films of aromatic oligocarbonate methacry-

lates, OCM-5 and OCM-7. It can be seen that two emissions are observed in both of these matrices, the ratio between the intensities of which depends on the excitation wavelength.

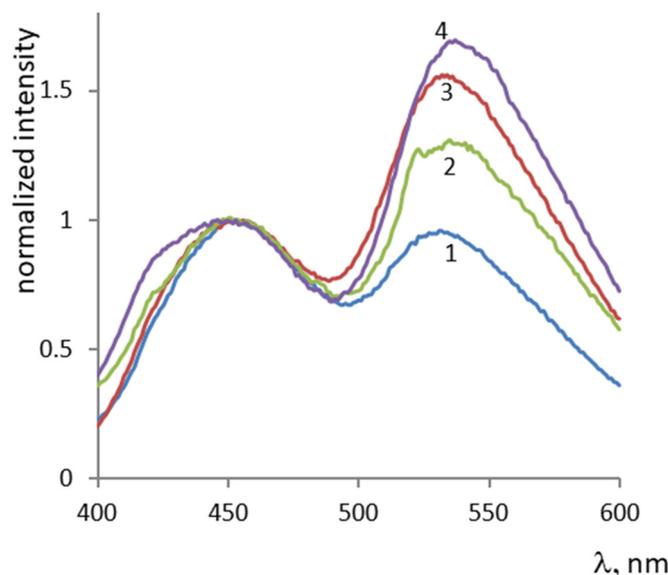
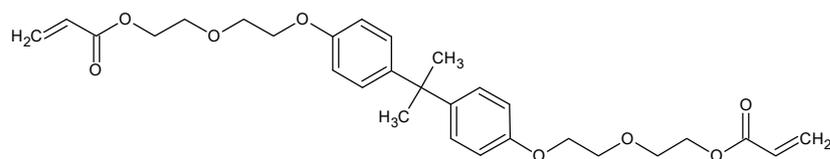


Figure 4. Emission spectra of the dyad in the polymer films obtained by photo curing of OCM-7 (1,3) and OCM-5 (2,4) at $\lambda_{\text{ex}} = 330$ nm (1,2) and 350 nm (3,4).

In the fluorescence spectrum of the dyad in a polymer obtained from OCM-7, characterized by the presence of one aromatic fragment in an oligomeric block, in contrast to the spectra observed in the aliphatic polymer matrices described above in the case of $\lambda_{\text{ex}} = 330$ nm (Figure 4, spectrum 1), the emission intensity in both bands is almost the same, and, at $\lambda_{\text{ex}} = 350$ nm (Figure 4, spectrum 3), the long-wavelength emission band dominates. In the fluorescence spectrum of the dyad in the polymer matrix OCM-5, the oligomeric block of which is characterized by the presence of two aromatic fragments, there is a completely clear predominance of the long-wavelength emission band. Thus, it can be seen that an increase in the number of aromatic fragments in the polymer chain promotes the ESIPT process in the azomethinocoumarin fragment.

The polymers obtained from OCM-1 and OCM-7 have the shortest oligomeric blocks in the structure of aliphatic and aromatic polycarbonate methacrylates, which causes the most rigid structures in the considered series. Figure 5 shows a comparison of the fluorescence spectra of the dyad in these two matrices.

To clarify the influence of the polymer matrix nature on the luminescent properties, it is also interesting to compare the fluorescence of the dyad in OCM-5, and the previously studied PhEA (Figure 6)—2,2-bis[4-(acryloyloxy-diethoxy)phenyl]propane, the formula for which is given below:



PhEA

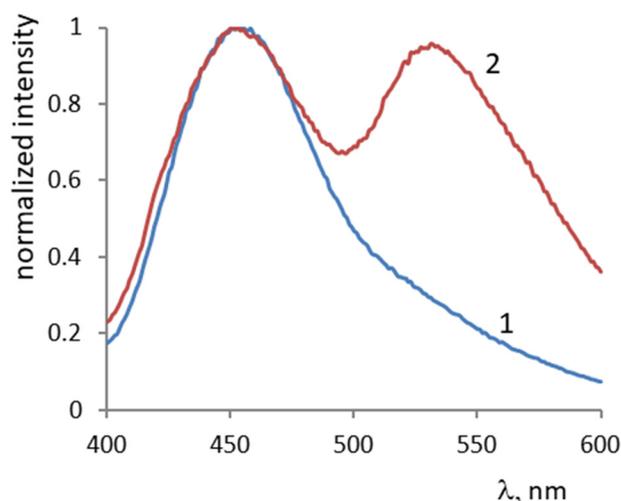


Figure 5. Emission spectra of the dyad in the polymer films obtained by photo-curing of aliphatic OCM-1 (1) and aromatic OCM-7 (2) at $\lambda_{ex} = 330$ nm.

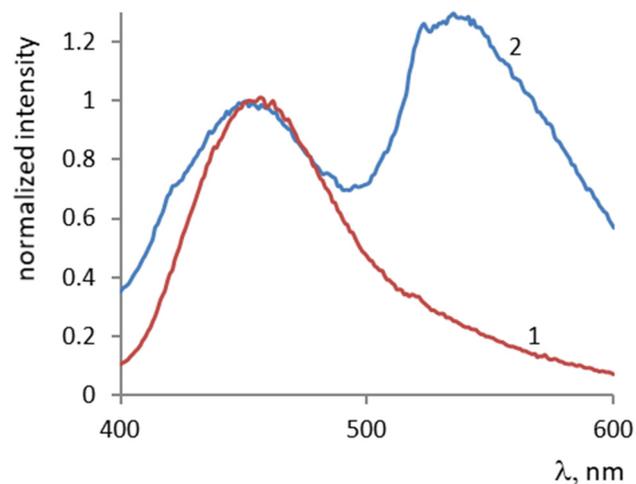


Figure 6. Emission spectra of the dyad in the polymer films obtained by photo-curing of OCM-5 (1) and aromatic PhEA (2) at $\lambda_{ex} = 330$ nm.

As can be seen from the PhEA formula, the oligomeric bridges in the polymers PhEA and OCM-5 are identical. The difference lies in the presence of carbonate groups —O—C(=O)—O— and methacrylic groups $\text{H}_3\text{C—C(=CH}_2\text{)—}$ in OCM-5, and the absence of carbonate groups and the presence of acrylic groups $\text{—C(=CH}_2\text{)—}$ in PhEA. Consequently, the presence of carbonate groups in the polymer matrix promotes the ESIPT process in the azomethinocoumarin fragment of the dyad.

The data shown in Figures 5 and 6 indicate the influence of aromatics in combination with a carbonate group in a polymer medium on the manifestation of a long-wavelength luminescence band. The results shown in Figure 7 indicate the influence of the oligomer bridge’s nature in network polymers on the peculiarities of the luminescent properties as manifested in the dyad introduced into the polymer.

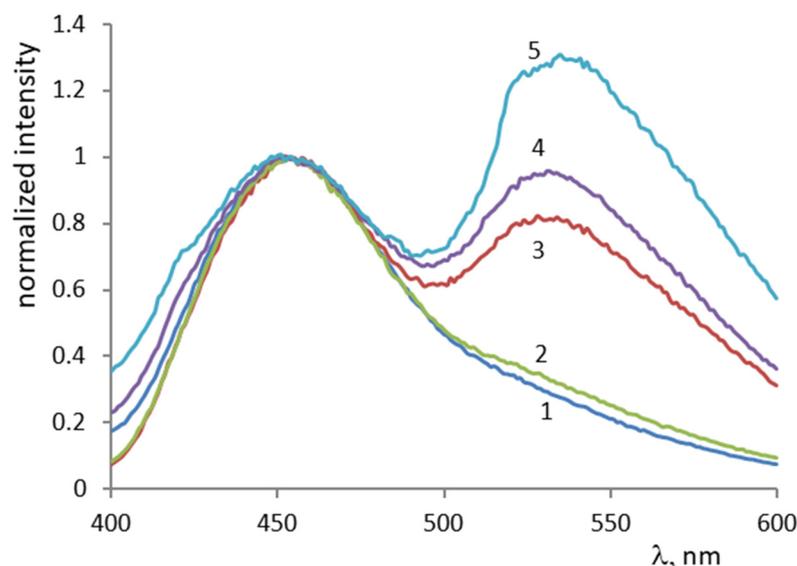


Figure 7. Emission spectra of the dyad in the polymer films obtained by photo curing of OCM-1 (1), OCM-2/1 (2), OCM-2 (3), OCM-7 (4) and OCM-5 (5) at $\lambda_{\text{ex}} = 330$ nm.

A comparison of the dyad fluorescent properties in the matrices of OCM-2, characterized by the presence of a ethyleneglicol ether bond, and OKM-5, containing two aromatic fragments (Figure 7), once again indicates the feature we observed earlier—in the first instance, the intensity of the short-wavelength emission band, with a maximum at 450 nm, increases, and the combination of aromatics with a carbonate group leads to the growth of the long-wavelength emission at 550 nm.

4. Conclusions

It has been found that, in all studied polycarbonate methacrylates, two emissions are observed, which are caused by excited state intramolecular proton transfer (ESIPT) in both luminophore fragments—imidazole (450 nm band) and coumarin (535 nm band). It has been shown that the ratio of emission intensities very significantly depends on the nature of the polymer matrix and the excitation light wavelength. It has been found that, in aliphatic polycarbonate methacrylates, the fluorescence intensity of the imidazole fragment increases and the ESIPT efficiency in the azomethinocoumarin fragment (which needs space for the molecule to rotate) decreases with increasing polymer stiffness. It has been found that, in aliphatic polycarbonate methacrylate, with increasing polymer stiffness, the fluorescence intensity of the imidazole fragment increases and the ESIPT efficiency in the azomethinocoumarin fragment decreases. In addition, it has been shown that, in the matrices containing ethyleneglicol ether bonds, the intensity of short-wave emission increases while, in the matrixes containing aromatic fragments in combination with carbonate groups, the intensity of the long-wave fluorescence band increases.

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