

Layer-by-layer photopolymerization of liquid crystal polymers at low ultraviolet power

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Layer-by-layer photopolymerization of a mixture of liquid crystal diacrylate, methacrylate, and acrylate was studied using IR 651 as a photoinitiator. It was shown that the helical pitch of the cholesteric liquid crystal polymer depends on the concentration of optically active acrylate. With decreasing UV irradiation intensity, a significant change in the concentration of optically active acrylate in various layers of the polymerized composition was observed, both increasing and decreasing from the initial concentration, leading to a broadening of the spectrum of light reflected from the thin polymerized layer. Keywords: cholesteric liquid crystals, liquid crystal polymers, photopolymerization, reflectance spectrum width, ultraviolet radiation.

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Introduction

To design display devices of a new type operating on the basis of the principle of incident light reflection without polarizers, use of thin layers of cholesteric liquid-crystal (LC) polymers is discussed [1,2]. Usage of such materials will considerably cut the costs and decrease their energy consumption in design of display devices, since no lighting is required on the opposite side, and lighting fixtures are excluded. Besides, these materials may also be used to control light in „smart“ windows.

Cholesteric LC-polymers are characterized by a spiral supramolecular structure. The mechanism of spiral formation in cholesteric polymers of diacrylate type (C6M) — chiral additive (EtLact1) — is based on chiral „twisting“ of achiral mesogenic monomer links (C6M) under the action of the optically active chiral additive (EtLact1), which is built into the structure, setting the spiral ordering of the molecules. This causes formation of a chiral non-linear-lamellar structure with a specific helical pitch responsible for the selective reflection of light [1–6]. Therefore, it becomes necessary to control the width of the spectral trail of light reflected from a thin film of a cholesteric LC-polymer.

The results of the studies of the helical pitch dependence on the concentration of the chiral additive conducted in some papers [1–6], showed that the higher is the concentration of chiral fragments in the reaction mixture that contains diacrylate, the more of them will be embedded into a molecular chain, and the stronger the twisting will

be, whereas the helical pitch will be shorter. The maximum broadening of the spectrum, according to papers [1–3], was 220 nm.

To form elastomer coatings Ch-LC, in paper [7] they applied a two-stage thiol-acryl polymerization mechanism after application of the Ch-LC mixture on a rod that provided for even leveling of LC-particles due to shear leveling at room temperature. The authors used mixtures of Ch-LC monomers containing dithiol (20 wt%), photoinitiator BP (1 wt%), catalyst triethylamine (2 wt%), chiral additive (4, 5 or 7 wt%) and LC-monomer diacrylate. At the first stage of mixture polymerization, which is often called the Michael's addition reaction, a linear step polymerization of monomers happens in the presence of a catalyst. At the second stage the produced oligomers are subjected to photopolymerization. The produced radicals cause homopolymerization of acryl end groups, as a result of which acrylates act as crosslinkers. A mesh structure is formed, which „fixes“ the Ch-LC reflection band. The coatings were hardened with an ultraviolet (UV) source Omnicure S2000 at capacity of 30 mW/cm² for 300 s. Application of broadband and ornamental coatings requires an increase in the number of production steps and their duration. For example, the films obtained at the first step were partially hardened with the UV-source Omnicure S2000 at the capacity of 0.4 mW/cm² for 45 s. On the non-hardened sections of the film the Michael's thiol addition reaction continued. In four hours the coating was fully hardened by photopolymerization at the lighting capacity of 30 mW/cm² for 300 s. The authors

of the paper believe that the gradient of the helical pitch in the thickness of the layer of $30\text{ }\mu\text{m}$ was achieved by removal of the Michael's reaction front from the surface, which was illuminated with UV-light at low capacity of emission, into the depth, where the concentration of the chiral additive was lower, and, accordingly, the polymer twisting degree was lower as well.

By changing the composition of the monomer mixture, the duration of the procedure for the formation and hardening of the coating, it was possible to broaden the light reflection band from 120 to 400 nm [7]. Therefore, in papers [1–3,7] the objective to control the width of the spectral band of light reflected from a thin film of a cholesteric LC-polymer was solved in a limited spectral interval.

This paper is also dedicated to the study of the influence of the LC „diacrylate–chiral additive“ system composition at the width of the spectral band of light. The spectrum broadening that we obtained was 500 nm, which is 2.3 times higher than in papers [1–3]. This result was achieved due to the addition of a neutral methacrylic component with the polymerization speed that is much lower than in the main acrylic component [8].

Experimental materials and method

Materials

The paper used commercial products: optically inactive LC-diacrylate C6M by Merk, optically active chiral dopant Et Lact1 — being a linear LC acrylic monomer, optically inactive methacrylic monomer CP-439, absorbant of exposing radiation coumarin 102, photoinitiator Irgacure 651 by Ciba–Geigy, which is further specified in the article as IR 651.

Units for the physical experiment and radiation of samples

To prepare LC compositions, a quartz plate ($80 \times 20 \times 2$ mm) was prepared with five holes in a row, each with volume of $\sim 2\text{ cm}^3$. To heat and mix powdered components, a glass current-conducting plate was made with the size of $100 \times 100 \times 10$ mm and sputtering of ITO and a thermal controller of heating temperature in the interval of $(20–250) \pm 1^\circ\text{C}$. The manufactured quartz plate for sample exposure was located at the distance of $\sim 1–2$ cm from the heating plate, after which it was radiated with UV-light on a unit for lithography M–6000. To adjust the power of the optical radiation, filters were made on the basis of a metal mesh with cells that have the size from 1 to 0.5 mm. The total capacity of DRT-350 lamp used in the unit was measured when preparing for the experiment. The total capacity of the flow was approximately 30 W of radiation for the entire spectral composition in the focus of the radiation spot. Further the total capacity of the light flow was measured at the wavelength of 365 nm —

absorption wavelength of photoinitiator IR-500. The DRSh-350 lamp, apart from xenon, contains another two primary gases: argon — serves as a buffer gas that facilitates discharge initiation and supports stable arching, mercury vapors — provide for high glow brightness and help to generate a specific spectrum of radiation that is specific for mercury lamps. The 365 nm line in this lamp is the most powerful. Its power was 20 mW in the total area of the light spot with the diameter of 13 cm. In terms of 1 cm^2 this will amount to 0.3 mW/cm^2 . In process of experimental work the absorption spectra were measured on spectrophotometer Lambda 950 by „Perkin Elmer“ USA. Samples of cholesteric polymer films reflect light at the wavelength that corresponds to the helical pitch. Therefore, this light will not penetrate the device. We work on a spectrophotometer for absorption and believe that the measured values of absorbed power are the reflected power.

Experimental results and discussion

Building an experimental chart of dependence of dopant concentration influence on the spectrum of reflection of a thin layer of a liquid crystal mix

Prior to research of the polymerization duration effect at the spectrum expansion, the dependence of dopant concentration effect on the color of the produced mix and wavelength of light reflected from it was investigated. Dependence of the wavelength of the reflected light of the LC-composition on the concentration of optically active monomer EtLact 1 is shown in Fig. 1.

To build the chart, mixes of Et Lact1 and diacrylate C6M were prepared, in which the content of dopant varied from 0.5 to 36 wt%. From the chart it follows that the increase of the concentration of the optically active monomer in the mix decreases the reflected light wavelength. In other words, the higher is the content of dopant in the mix, the deeper goes the spectrum into the UV-area.

All concentration changes of the dopant are described with a chart in Fig. 1. Change of the dopant concentration in the LC-system that changes helical pitch manifests itself in the change of spectral characteristics of the sample, which are written on the spectrometer. Since polymerization happens in layers from the inlet window to the outlet one, it is obvious that in different layers the concentration of dopant will match different color points in Fig. 1. In other words, the chart of dependence of the LC-composition reflected light wavelength on the concentration of the optically active monomer EtLact 1 (Fig. 1) may be used as a calibration chart to determine the concentration of dopant in various layers of the coating.

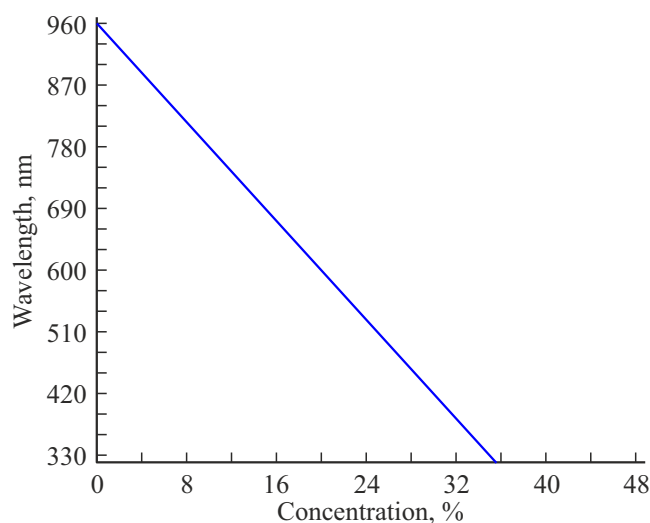


Figure 1. Dependence of the wavelength of the reflected light of the LC-composition on the concentration of optically active monomer EtLact1.

Optical spectra in polymerization of liquid crystal light sensitive mix using a non-crosslinking absorber coumarin 102 and a crosslinking metacrylic monomer CP439

For the maximum expansion of the reflection spectrum, two mixes were chosen with the composition C6M(65wt%):Et Lact1 (25 wt%): coumarin 102(9.5 wt%):IR651(0.5 wt%) and C6M (65 wt%):Et Lact1(25 wt%):CP 439(9.5 wt%):IR 651(0.5 wt%), one of which was with a non-crosslinking component — coumarin 102 that absorbed UV-radiation, and the second one — absorbing and crosslinking under the exposure to UV-radiation contained CP 439. Based on these two optimized molten compositions at temperature of 70°C, corresponding to the nematic phase, samples of LC-polymers were prepared, which were aligned using shear method. The presence of the LC-state was controlled visually by color change from glass shear. Then the samples were radiated with UV-light in a unit of EM-6000 grade with adjustment of the exposure power from 0.3 to 0.005 mW/cm². Using mesh filters, emission of the lamp (DRSh-350) may be weakened 10 times (from 0.3 to 0.03 mW/cm²).

Fig. 2 shows the absorption spectra of composition consisting of C6M (65 wt%):Et Lact1(25 wt%): coumarin 102(9.5 wt%): IR 651(0.5 wt%) at radiation power from 0.3 to 0.005 mW/cm². Under gradual decrease in the radiation power from 0.3 to 0.03 mW/cm² the maxima of the absorption spectra of this composition vary from 555 (Fig. 2, *a*) to 785 nm (Fig. 3, *a*), i.e. are displaced to the red area. Under further decrease of power the Et Lact1 acrylate layer, which forms a spiral of a cholesteric LC-composition, is displaced from the system, and the spectrum begins expanding and decreasing by amplitude (Fig. 3, *b* and 4). If the power of radiation decreases down to

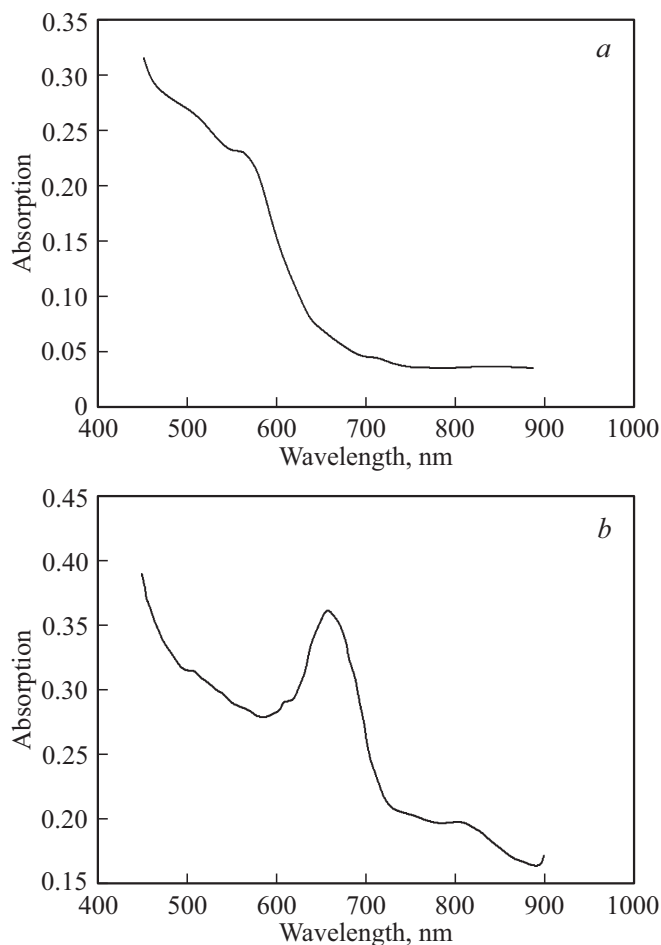


Figure 2. Absorption spectra of composition consisting of C6M (65 wt%):Et Lact1(25 wt%): coumarin 102(9.5 wt%): IR 651(0.5 wt%): (a) at radiation power of 0.3 mW/cm², (b) at radiation power of 0.1 mW/cm².

0.005 mW/cm² the absorption spectrum maximum disappears (Fig. 4). The optically active acrylate will not enter the copolymerization reaction. In the second mix of composition C6M(65 wt%):EtLact1(25 wt%):CP 439(9.5 wt%):IR 651(0.5 wt%) metacrylate was chosen instead of coumarin as an absorber, and metacrylate may be copolymerized with diacrylate C6M and acrylate EtLact1. The speed of crosslinking and accordingly its concentration in various layers of the composition in layerwise photopolymerization will differ [8].

Fig. 5–8 shows the absorption spectra of polymerized films with composition of C6M(74.7 wt%):EtLact1(22 wt%):CP 439(2.9 wt%):IR 651(0.4 wt%), at radiation powers of 0.1, 0.3 and 0.03 mW/cm² and exposure time 1–20 min.

The initial wavelength of the maximum of reflected light with composition of C6M(74.7 wt%):EtLact1(22 wt%):CP 439(2.9 wt%):IR 651(0.4 wt%) prior to polymerization is 610 nm. The absorption spectrum of polymerized LC-layers of this composition expands with decrease in the radiation power and increase of exposure time (Fig. 6). A similar picture is observed in the absorption spec-

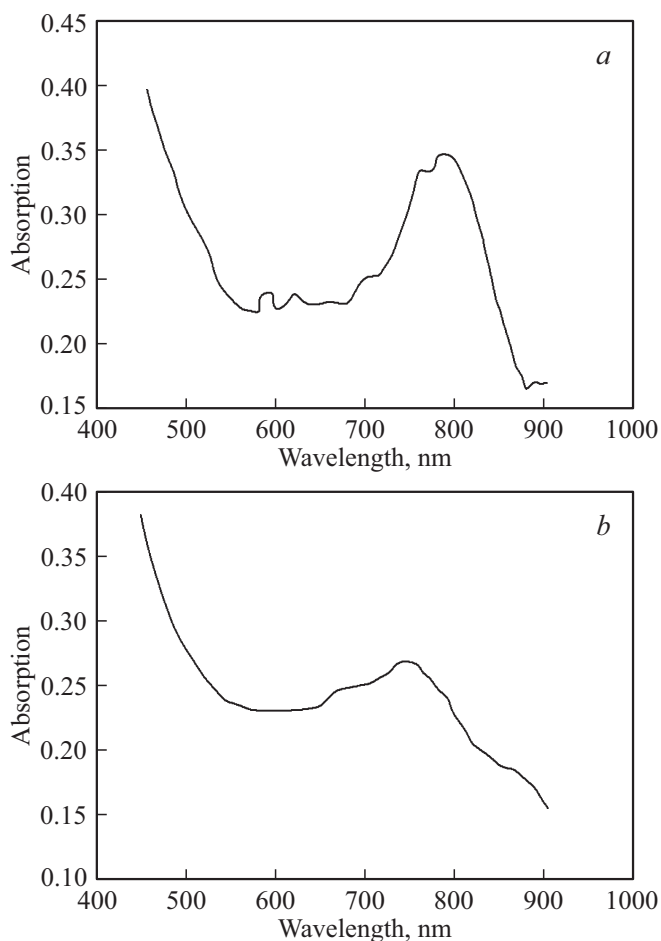


Figure 3. Absorption spectra of composition consisting of C6M (65 wt%):Et Lact1 (25 wt%): coumarin 102 (9.5 wt%): IR 651 (0.5 wt%): (a) at radiation power of 0.03 mW/cm², (b) at radiation power 0.01 mW/cm².

tra of polymerized LC-layers of the same composition C6M(74.7 wt%):EtLact1(22 wt%):CP 439(2.9 wt%):IR 651(0.4 wt%), but differing with duration of preparation (Fig. 7).

Therefore, for the samples of the same composition the absorption spectrum expands (Fig. 6, 7) with the decrease of the radiation power and increase of the exposure time.

Increase in content of EtLact 1 from 22 to 24 wt% and absorbing component from 4 to 10 wt% in the sample causes expansion of the absorption spectrum 2.3 times (Fig. 8). At the same time the time of radiation increases from 1 to 50 min. From Fig. 8 you can see that the absorption spectrum of mixture C6M(65.6 wt%):EtLact1(24 wt%):CP 439(10 wt%):IR 651(0.4 wt%) is practically a straight horizontal line from 400 to 880 nm with a sharp rise at 380 nm.

According to authors [1,2], in process of radiation with UV-light ($\lambda = 365$ nm) over a thin layer of the aligned above LC-composition, the diffusion of the chiral component starts towards the increase in the light intensity, i.e. towards the inlet window. At the same time the concentration of the LC-

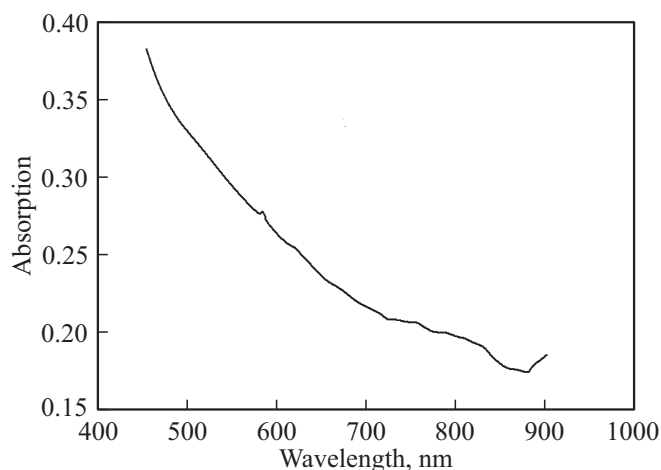


Figure 4. Absorption spectrum of composition consisting of C6M (65 wt%):Et Lact1 (25 wt%): coumarin 102 (9.5 wt%): IR 651 (0.5 wt%) at radiation power of 0.005 mW/cm².

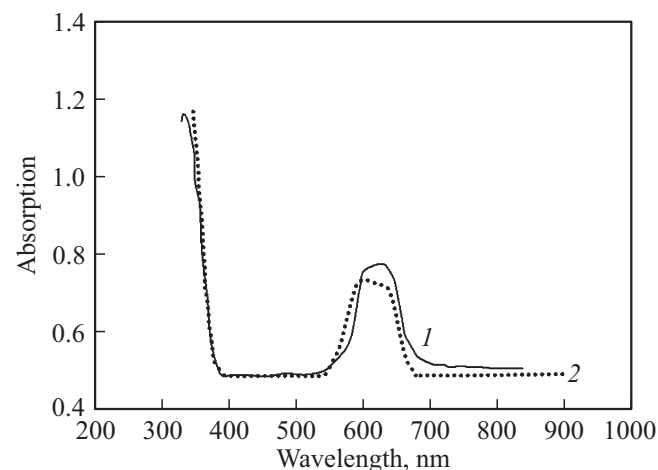


Figure 5. Absorption spectra of polymerized LC-layers with composition C6M(74.7 wt%): EtLact1(22 wt%):CP 439(2.9 wt%):IR 651(0.4 wt%): polymerization time 2 (1), 1 min (2); radiation power 0.1 (1), 0.3 mW/cm² (2).

mix from the front plane of the cell to the rear one changes. In accordance with the distribution of concentration, each layer within the cell has a certain helical pitch. Amplitude of the reflected light for a certain wavelength will depend on the thickness of the LC-layer and helical pitch. In other words, the LC-cell with the cholesteric structure may be seen as a multi-layer mirror, where the reflection coefficient of each layer is equal to the full reflection coefficient divided into the number of layers.

Therefore, knowing the full thickness of the LC-cell, you can estimate the reflection coefficient of one layer, and by intensity of the reflected light you can estimate the number of layers in the multi-layer mirror, which makes it possible by speeds of increase and decrease of spectral characteristics to define the distribution of chiral dopant concentration upon the end of the diffusion process and completion of

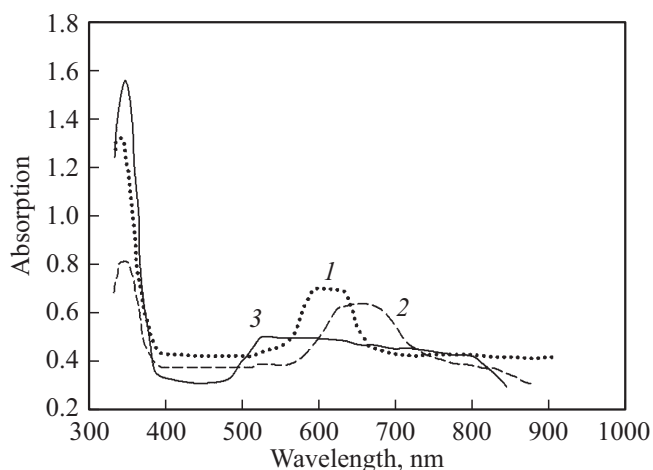


Figure 6. Absorption spectra of polymerized LC-layers with composition of C6M(74.7 wt%):EtLact1(22 wt%):CP 439(2.9 wt%):IR 651(0.4 wt%): polymerization time 2 (1), 5 (2), 20 min (3); radiation power 0.3 (1), 0.1 (2), 0.03 mW/cm² (3).

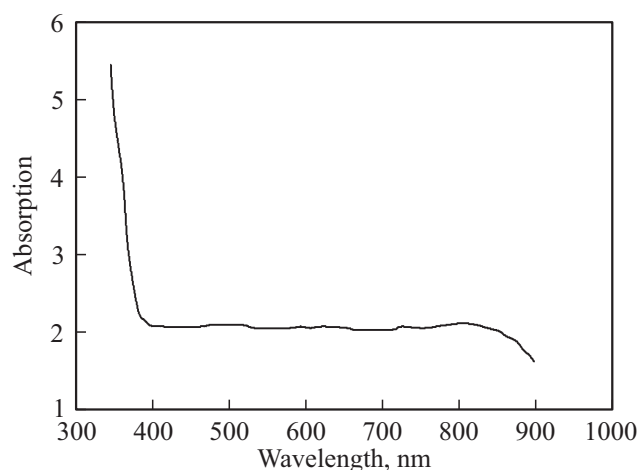


Figure 8. Absorption spectrum of polymerized LC-mix C6M(65.6 wt%):EtLact1(24 wt%):CP 439(10 wt%):IR 651(0.4 wt%), polymerization time 50 min.

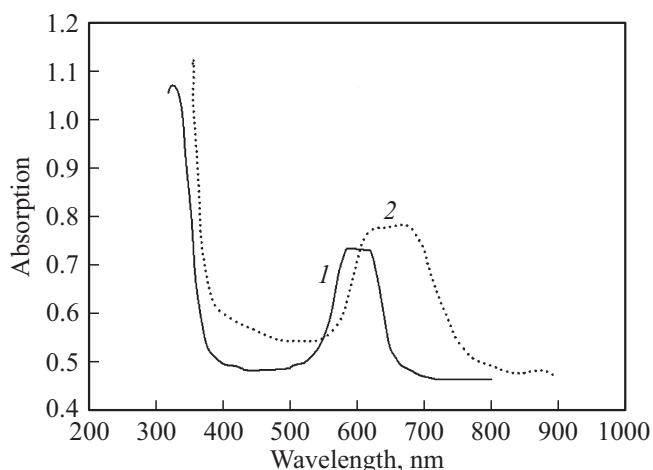


Figure 7. Absorption spectra of polymerized LC-layers with the composition C6M(74.7 wt%):EtLact1(22 wt%):CP 439(2.9 wt%):IR 651(0.4 wt%): polymerization time 2 (1), 20 min (2); radiation power 0.3 (1), 0.03 mW/cm² (2).

the photopolymerization process. The found initial and final values of transverse concentrations of the layer will make it possible in the further studies to build a diffusion equation passing in the process of exposure.

In early papers [1,2] it was not possible to expand the spectral range of LC-compositions of the studied composition by more than 220 nm since the concentration of dopant in the system during mixture polarization changed insufficiently for the required spectral expansion. To overcome the above faults, we introduced the third neutral component CP 439 into the LC-composition, and this component provided for the distribution of chiral active component concentration with spectrum broadening of 500 nm (Fig. 8). If in the described system the change of concentration from 20 to 30 wt% leads to reflection

spectrum change in the range from 380 to 680 nm, then to expand the spectral range 2 times, it is necessary to add 10 wt% of the neutral component into the system.

Diffusion under layerwise photopolymerization

Let us consider in detail the process of layerwise polymerization in a triple system: LC-diacrylate C6M: linear acrylic LC chiral monomer EtLact1: neutral component (linear metacrylic monomer CP439 or coumarin). Neutral component (linear metacrylic monomer CP439 or coumarin) creates an illumination gradient. Diffusion processes during photopolymerization of LC-mixtures presented in this paper to some extent remind of the process of polymerization and microrelief development in the liquid photopolymerizing compositions (LPPC), when the decrease in the power of radiation of the LPPC layers caused significant increase in the produced microrelief [9–15]. When the light falls onto the front plane of the cell (Fig. 9), the inlet light flow fades by amplitude.

When radiated with UV-light upon the end of the induction period, layered polymerization of the triple system starts. Diacrylate is copolymerized with acrylic monomer, and coumarin 102 creates a light field gradient. In other words, the process of polymer mesh formation starts, with the maximum density at the inlet face. Mesh density decreases in direction towards the rear face gradually. Besides, because of consumption of both components, the difference of concentrations between the layers occurs. We believe that in the layer that is closer to the inlet face, the concentration of monomers will be lower due to their consumption in process of polymerization and therefore withdrawal from the liquid mobile system. Therefore, both diacrylate and linear acrylate will diffuse towards the inlet face. The third neutral component will be displaced

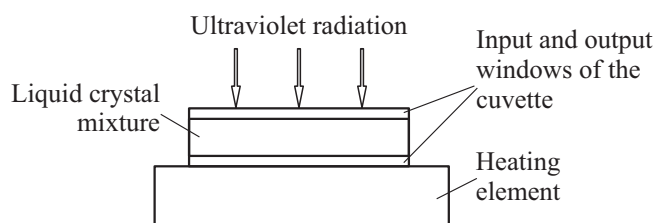


Figure 9. Schematic diagram of liquid-crystal samples photopolymerization unit.

from the mesh being formed to the opposite side, and its concentration will be increasing gradually towards the rear window of the cell.

Therefore, we will have two diffuse flows to different sides. First flow — diacrylate C6M and acrylate-chiral additive EtLact1, going towards the inlet window, second flow — metacrylic monomer CP439 (or coumarin 102), displaced from the system, since it is replaced with an active flow of acrylates due to constantly maintained difference of concentrations in process of photopolymerization. Reactive monomers of acrylates, displacing non-reactive components, are simultaneously fixed on the surface of glasses forming a cell. The difference between CP439 and coumarin 102 consists in the fact that coumarin 102 does not interact with the components of the mix and may not be fixed in the system, therefore, in the end it is fully displaced from it. Monomer CP439, passing through the system of acrylates, is crosslinked with them. The main task is to select the crosslinking speed, i.e. the photopolymerization reaction speed so that CP439 is not fully displaced beyond the system, when the concentration of C6M and Et Lact1 acrylates is low within the rear window. This is necessary to form a cholesteric mixture of low concentration.

Besides, it is assumed that reactive monomers pass through a non-reactive monomer, and the length of this passage will linearly increase with the reaction and movement of the polymerization front towards the rear wall. As the reactive monomers travel through the reaction zone of the neutral component, a barrier is observed, through which the linear and bifunctional monomers must travel, whose diffusion coefficients are determined by the degree of relationship to it and may differ significantly. Further, as the concentration of the neutral component increases towards the outlet window, relative decrease of EtLact concentration happens in the system, which displaces the length of the reflected wave to the red area (Fig. 1). As you can see from Fig. 2, *a*, the maximum of the absorption spectrum of mix C6M (65 wt%):Et Lact1(25 wt%): coumarin 102(9.5 wt%): IR 651(0.5 wt%) is observed at 555 nm, radiation power of 0.3 mW/cm². As the radiation power decreases and the time of exposure increases, the maximum of the absorption spectrum shifts to the red area (655 nm, Fig. 2, *b*; 785 nm, Fig. 3, *a*), in Fig. 3, *b* the peak starts collapsing due to coumarin 102 displacement towards

the rear wall of the cuvette. The absorption maximum of coumarin 102 disappears (Fig. 4).

Therefore, addition of a neutral component coumarin 102(9.5%) to the mix of C6M (65 wt%):Et Lact1(25wt%): IR 651(0.5 wt%) caused expansion of the sample absorption spectrum into the long wavelength area. The neutral component coumarin 102 is fully displaced from the system at low power of UV-radiation (Fig. 4). In this case the LC-mix absorption spectrum broadening is not observed.

Introduction of a slowly crosslinking agent, metacrylate CP439, into the LC-system made it possible to maintain the decrease of concentration under opposite diffusion and the expansion of the absorption spectrum into the long wavelength area in the long wavelength area for the mix consisting of C6M (65 w%):Et Lact1 (25 wt%):CP439 (9.5 wt%): IR 651(0.5 wt%). Metacrylate is polymerized 10–50 times slower than acrylate [8]. When moving opposite, while it is displaced towards the rear wall, it is crosslinked with the main components of the composition C6M and EtLact1 and therefore is partially left on the displacement path, decreasing the concentration and therefore expanding the spectrum into the long wavelength area.

From the above it follows that in the specified triple system the spectrum expansion is provided for by the following processes of layered photopolymerization.

- 1) Increase of the concentration of chiral dopant EtLact1 in the area of the inlet window of UV-light happens due to opposite diffusion towards the inlet window under layered photopolymerization, which provides for the spectrum broadening into a short wavelength area of 380 nm.

- 2) Decrease of the concentration of the chiral dopant EtLact1 in the area of the rear window happens due to its pumping from the area of the rear window to the area of the front one with simultaneous mixing with the neutral component CP-439 or coumarin 102 and gradual diffusion displacement towards the rear window, which provides for the spectrum expansion into the long wavelength area of the spectrum of 880 nm.

- 3) Neutral coumarin 102 does not bind with the system components and is displaced under slow polymerization towards the rear wall.

- 4) The optical spectrum of incident light reflection turned out to be even and wide due to the introduction of the optically inactive metacrylic monomer CP-439 into the system with strong absorption at the wavelength of 365 nm, which was crosslinked in the entire diffusion length and was not displaced from the system.

- 5) Introduction of a metacrylic monomer into the system, whose polymerization speed is lower than in acrylates, will make it possible, when necessary, to expand the spectrum of the incident light reflection even more. With this purpose, it is necessary to increase the concentration of metacrylic monomer and select the concentrations of other components in the LC-system.

Conclusions

Adding to the system containing C6M(65.6 wt%), EtLact1(24. wt%), IR 651(0.4 wt%), a linear metacrylic monomer CP-439 in the quantity of 10 wt% at power decrease down to 0.03 mW/cm² and increase of polymerization time up to 60 min causes production of a sample, where concentration of EtLact1 changes in layers from 4 to 32 wt%. As a result, the sample after polymerization absorbs emission in the range from 380 to 880 nm, whereas prior to polymerization it absorbed emission of 520 nm and had spectrum width of 50 nm.

Aligned samples with the thickness of layer from 10 to 20 μm, illuminated with white light, reflect emission from 380 to 880 nm (from purple to infrared). Maximum broadening is 500 nm, which is 2.3 times higher than in papers [1–3]. This effect was achieved through adding to the LC system „diacrylate—chiral additive“ a neutral component, such as a metacrylic component, whose polymerization speed is much lower than that of the main acrylic component.

Use of the Michael's addition reaction catalyzed by triethylamine to produce elastomer coatings Ch-LC in paper [7] enabled the authors to expand the light reflection band from 120 to 400 nm. It should be noted that the duration of formation and illumination of broadband coatings is more than 4 h. Using layered radical polymerization of a mix of LC-diacrylate, metacrylic and acrylic monomers in our paper in the presence of IR 651 as a photoinitiator both considerably decreases the duration of coating production and expands the light reflection band from 50 to 500 nm.

The proposed method makes it possible to create optical filters of the necessary width in various parts of the spectral range. The width and position of the spectral characteristic in the frequency axis may be adjusted both with the help of changing the concentrations of dopant and neutral component, and with the help of changing the radiation power. Having imposed the optical wedge under exposure on a sample with an LC-composition, a linear wedge may be obtained with different spectral width of the reflected emission in each point, and moving such wedge of a thin beam incident on a sample, an instrument may be obtained with the adjustable spectral width of the reflected beam. Having broken the images of any picture into different templates, you can obtain a colorful image of this picture, illuminating a sample with an LC-composition for a different period of time through every template. For photonics devised with waveguide systems, it is possible to create photonic band gaps and to use polymer LC-compositions for the creation of the waveguide layers in a certain spectral range.

Study financing

This study was completed in a proactive manner.

Conflict of interest

The authors declare no conflict of interest.

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