Orientation of Pentyl-Cyanobiphenyl Layers and Variations in Their Luminescence Spectra under UV Irradiation

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Abstract—Polarized fluorescence spectra of layers of a nematic liquid crystal (LC) of *n*-pentyl-*n'*-cyanobiphenyl (5CB), oriented by two different methods, namely, by rubbing the polyimide-coated substrates holding the LC layer and by irradiating the LC layer placed between isotropic fused quartz substrates by polarized UV radiation, are comparatively analyzed. The photoluminescence spectra in the two cases show anisotropy with parameters virtually independent of the orientation method. Contrary to the case of rubbing orientation, the photoluminescence spectrum of the LC layer, oriented by irradiation, substantially differs from the initial one. Possible photochemical processes and mechanisms of photoorientation are discussed. © 2002 MAIK "Nauka/Interperiodica".

INTRODUCTION

Liquid crystals (LC) represent an unique class of anisotropic liquids, layers of which can be rather easily oriented in a prescribed direction and reoriented by weak external fields. This attracts a permanent interest in liquid crystals as active media for displays with low control voltages and power consumption [1]. The majority of currently produced LC displays is based on LC layers with a prescribed orientational configuration. A traditional method of orientation consists in coating the LC cell surfaces by a polymer, most often polyimide, and rubbing the coated surfaces in a prescribed direction. As a result, liquid crystals are oriented, as a rule, in the rubbing direction. Such a method has been the most applicable. However, it does not satisfy many of the requirements of modern technologies of LC displays. The crucial drawbacks of this method are the mechanical contact with the oriented surface (which can damage the surface and form static charges), insufficient orientation uniformity, complicated monitoring of the orientation parameters, and difficult realization of multidomain orientational structures.

An alternative to rubbing, the photoorientation method, was suggested in the early 1990s. This method is based on the photoanisotropy appearing in the layers of some polymer classes under polarized irradiation. Anisotropic polymer layers, in turn, appeared to be excellent orienting coatings for liquid crystals [2, 3]. To the polymers suitable for LC photoorientation, one can assign azopolymers, polymers containing cinnamates or coumarins, and some polyimide classes [4].

A radically new method of photoorientation was recently suggested [5–7]. Its point is to photoorient not the polymer layers, but the LC layer placed between isotropic nonabsorbing substrates. In our experiments, a layer of nematic LC of *n*-pentyl-*n*'-cyanobiphenyl (5CB) placed between two isotropic fused quartz substrates was oriented by polarized UV radiation. The orientation formed by this method had virtually as good a quality as in the case of rubbing. However, the effect of the UV irradiation on the physicochemical characteristics of 5CB LC was left unstudied.

A traditional method of studying photochemical processes in organic compounds is UV spectroscopy. The UV spectra of thin (about 0.3 μ m) oriented 5CB layers were measured in [8]. However, this method gives poor results for micron layers due to the strong LC absorption in the UV region. Keeping in mind the efficient natural fluorescence (FL) of 5CB [9-16], we study the irradiation effect on the 5CB layers by fluorescence methods. Measuring the fluorescence spectra of 5CB layers before and after irradiation, we try to clarify the photochemical processes occurring in the irradiated LC. In addition, we use the dichroism of the polarized fluorescence spectra as a parameter characterizing the induced orientation of the LC layer. This study also compares data obtained for 5CB layers oriented by the offered and traditional methods.

EXPERIMENTAL TECHNIQUE

We studied the 5CB LC produced by "Merck." This liquid crystal has a nematic phase in a temperature range from 18 to 35°C. The 5CB crystallizes below 18°C and changes to the isotropic state at a temperature above $35^{\circ}C$ [17]. We oriented the 5CB LC layers 50 µm thick by two different methods, using two different types of cells. In the first case, the planar orientation of LC layers was formed by polyimide (Du Pont 2555) layers deposited on the fused quartz plates of the cell,



Fig. 1. (1, 5) Stationary and (2, 3) time-resolved fluorescence spectra of 5CB LC (1–4) before and (5) after UV irradiation. (2) $t_d = -4ns$, (3) $t_d = 15 ns$. (4) Difference between (1) and (2) spectra. T = 301 K, $\lambda_E = 337 \text{ nm}$.

which were rubbed in one direction. The quartz substrates of the second cell type were uncoated. The LC layers in this case were oriented by the following technique. The cell filled with a liquid crystal was heated to 50°C and then slowly cooled and simultaneously irradiated by the linearly polarized unfiltered light of a DRSh-250 Hg lamp for one hour. The cell was faced to the radiation and turned over after half of the process. When cooled to the LC phase, the 5CB layer gained an orientation, which was observed with a polarization microscope and proved by FL anisotropy.

The stationary and time-resolved spectra and fluorescence kinetics were studied for the two types of cells. The FL spectra were recorded in the polarized light by an SPM-2 monochromator with a photo recording system. The slit spectral width was 0.2–0.4 nm. The fluorescence was excited by a nitrogen laser with a wavelength of 337 nm, pulse duration of 10 ns, pulse repetition rate of 100 Hz, and a pulse power of 5 kW. The stationary polarized FL spectra were also measured when exciting the 5CB LC by the polarized radiation of a mercury lamp at $\lambda_E = 365$ and 405 nm. The 365- and 405-nm lines were separated by interference filters.

The time-resolved FL spectra were measured using a stroboscopic recording system with a 0.1-ns window, which allowed for the recording of the FL kinetics and spectra with a variable delay t_d relative to the laser pulse. The experimental setup was described in our paper [16].

RESULTS AND DISCUSSION

1. Fluorescence Spectra of the Initial 5CB LC

Before studying the fluorescence spectra of UV-irradiated 5CB, let us examine the initial spectra of this liquid crystal. Figure 1 shows the stationary (curve *1*) and time-resolved (curves 2, 3) FL spectra of the initial 5CB LC at 301 K. At a weak UV excitation and short delay times ($t_d = -4$ ns, curve 2), the FL spectrum can be resolved into two bands, 375 and 395 nm. The shape of the spectrum remains almost the same, as the delay time increases from -4 to 0 ns. With the further increase of t_d from 0 to 15 ns (Fig. 1, curve 3), the intensity of the short-wavelength wing decreases, and the spectrum shows peaks at 380 and 405 nm. Such variations in the FL spectrum with t_d is caused by the fact that several monomer and excimer electronic states with various lifetimes are involved in the LC emission. We discussed the nature of these electronic states of the 5CB LC in [16]. It was shown that the differences in the LC emitting states relate to the intramolecular charge transfer in the excited state and to the conformation changes in the 5CB molecule.

The measured kinetics of the 5CB LC luminescence at various wavelengths ($\lambda_{FL} = 350, 375, \text{ and } 410 \text{ nm}$) are shown in Fig. 2. The curves of the FL decay can be approximated by the convolution of the exciting pulse with the sum of exponents $[A_1 \exp(-t/\tau_1)]$ + $A_2 \exp(-t/\tau_2]$, where $\tau_2 > \tau_1$. The time τ_1 is determined from the half-widths of the exciting pulse and the decay curve, and τ_2 is determined from the tail of this curve. The component with the lifetime $\tau_2 = 28$ ns, which can be related to the excimer emission [12, 13, 16], is present in all the cases. As is seen from the curves of FL decay, the relative contribution of this component in the FL spectrum grows with the fluorescence wavelength and dominates at $\lambda_{FL} > 410$ nm (Fig. 2, curve 2). The decay of short-wavelength fluorescence ($\lambda_{FL} = 350$ and 375 nm) is for the most part determined by the monomer components with $\tau_1 = 7.6$ and 10.3 ns, respectively (Fig. 2, curves 4, 5). It was observed, that the FL lifetimes τ_1 and τ_2 depend on the quality of the studied material, method of cell preparation and cell thickness, and the exciting light intensity. In the studied cells, at a weak excitation and T = 301 K, τ_1 and τ_2 of the FL in a range of 350 to 380 nm vary from 5.9 to 10.3 ns and from 22 to 28 ns, respectively. The data given in this paper relate to one of the 5CB LC samples subjected to the whole set of experiments reported below.

The stationary fluorescence spectrum of 5CB (Fig. 1, curve 1) shows bands at 375 and 402 nm. Unlike the instantaneous spectra, the stationary FL spectrum contains the complete set of emission components with various lifetimes. The excimer component with $\tau_2 = 28$ ns dominates in the stationary spectrum of the 5CB LC fluorescence at $\lambda_{FL} > 380$ nm. The spectrum of this component (Fig. 1, curve 4) is plotted by the subtraction of curve 2 from curve 1. The dominant band of this spectrum has a peak at 410 nm. The fluorescence at $\lambda < 380$ nm is identified as the monomer emission. Since the excimer radiative relaxation occurs by transitions to the unstable state, the excimer FL looks like a wide structureless band almost totally over-

lapping the region of the monomer component. The population of monomer and excimer states at room temperature are in thermal equilibrium, which determines the two-component decay of the 5CB LC fluorescence and the presence of the monomer emission with a lifetime close to that of the excimer.

2. FL Spectra of Irradiated 5CB Layers

The structure of the fluorescence spectra of 5CB in the LC and isotropic states strongly depends on the intensity of the exciting UV radiation and is almost not changed under excitation by visible light. The FL spectra in Fig. 1 (curves 1-4) are excited by a defocused laser beam with the intensity $I = 5 \times 10^{-2}$ W/cm². At such intensities, the LC FL spectra barely depend on the excitation duration. A similar stability of the FL spectra is observed under excitation by the monochromatic light of a mercury lamp. At the excitation of a 5CB LC by UV irradiation of high intensity, I = 5 W/cm^2 , which caused the 5CB photoorientation in [5–7], one observes continuous changes in the spectral distribution (Fig. 1, curve 5), quantum yield, and fluorescence kinetics. Therefore, the photoorientational effects in 5CB LC can be related to the photochemical and structural transformations of 5CB molecules in the excited state.

At the LC excitation by the focused laser beam ($\lambda_E = 337$ nm), the intensity of the monomer FL at $\lambda_{FL} < 400$ nm decreases, and the intensity of the 410-nm band grows approximately by a factor of 3 (Fig. 1, curve 5). These changes are nonreversible and are recorded at subsequent excitation of FL by weak UV irradiation. Similar changes in the FL spectra were observed under a long (60 min) irradiation of 5CB in the isotropic or LC states by the unfiltered light of a mercury lamp. No dependence of the 5CB FL spectrum on the irradiation duration was observed under the filtered light of a mercury lamp ($\lambda_E > 400$ nm). Hence, it is believed the photoinduced transformations of the 5CB FL spectrum under the unfiltered light of a mercury lamp are caused mainly by the UV component of light.

The FL spectra of 5CB in the solid crystalline state (at T = 77 and 4.2 K) and in a molecular solution 5CB show no photoinduced changes under efficient UV irradiation. At the same time, the changes photoinduced in the LC or isotropic state, are conserved at low temperatures, when 5CB transforms into solid crystal. This proves that the photoinduced transformations in 5CB are not monomolecular but relate to the interaction of an excited molecule with other molecules in a certain configuration. Such interaction evidently slows down in the solid-crystal phase, which weakens the photostructural transformations.

As we discussed earlier in [16], the majority of 5CB molecules in the LC or isotropic states are in predimer or dimer states. Owing to this, the LC phase of 5CB is centrosymmetrical, in spite of the polarity of separate

molecules. The predimer states, which are characterized by a rather large distance between two interacting molecules, participate in the absorption as monomers, and in the emission, as eximers. Decrease of the monomer radiation intensity and a considerable growth of the 410-nm band (Fig. 1, curve 5) under intensive UV irradiation can indicate that the predimer states transfer to stable dimers, i.e. 5CB photodimerizes. Such dimers obviously have a physical nature, i.e. do not form chemical bonds. This supposition is also confirmed by the absorption spectra of the 5CB LC before and after UV irradiation. The UV irradiation causes an increase in the absorption coefficient in the spectral region from 350 to 410 nm and a shift of the absorption edge to longer wavelengths.

The change in the 410-nm FL kinetics proves that, after UV irradiation, this band is no longer the excimer radiation. Kinetics of the monomer (λ_{FL} < 390 nm) and excimer ($\lambda_{FL} > 400$ nm) fluorescence of the UV-irradiated 5CB LC measured at 301 K, are shown in Fig. 2, curves 3, 6, and 7. It is seen that, after UV irradiation, the lifetime of the fluorescence at $\lambda_{FL} < 390$ nm increases from 10.3 to 13.4 ns (curve 3), and the intensity of the excimer fluorescence with the lifetime τ_2 = 28 ns decreases (curves 6, 7). In addition, one can also see a weak emission with $\tau = 14.5$ ns. As the temperature increases and 5CB transforms into the isotropic phase, the lifetime of the fluorescence in the longwavelength region ($\lambda_{FL} > 400$ nm) further decreases, and the lifetime of the short-wavelength FL (λ_{FL} < 390 nm) increases approximately by a factor of 1.5 as the temperature increases from 301 to 335 K. A decrease in the excimer FL lifetime testifies to the fact that the radiative transition becomes allowed after the UV irradiation due to an increase in the oscillator strength of the transition to the ground state. Such



Fig. 2. Kinetics of the 5CB LC fluorescence at λ_{FL} =

(2, 6, 7) 410, (3, 4) 375, and (5) 350 nm, (2, 4, 5) before and (3, 6) after irradiation during $t_{ir} = (1-6)$ 1 and (7) 2 h.

(1) The laser pulse shape, T = 301 K.



Fig. 3. Stationary spectra of polarized fluorescence of 5CB LC oriented by UV irradiation for the excitation wavelengths $\lambda_{\rm E} = (1-4)$ 365 and (5–8) 405 nm and geometries (1, 5) I_{zy} , (3, 7) I_{zy} (4, 8) I_{yy} , and (2, 6) I_{yz} .

changes can be caused by the transformation of some predimer pairs with the excited-state lifetime $\tau_2 = 28$ ns into stable dimer pairs with a lower intermolecular distance and an excited-state lifetime of 6 ns.

Thus, analysis of the kinetic processes leads us to the same assumption that we used when analyzing the FL spectra of irradiated samples. The monomer FL component with a lifetime longer than that before irradiation, appeared in the spectral region with $\lambda_{FL} < 390$ nm under irradiation, and an increase of its lifetime with growing temperature indicates that the photo-dimerization under UV irradiation is accompanied by a reverse process of dimer and predimer decay to monomers.

From the fluorescence spectra and decay kinetics, one can conclude that there are other products of 5CB LC phototransformations under intensive UV irradiation. One such product can cause the peak at 540 nm (Fig. 3, curves 5-8) which appears at the excitation of the irradiated LC layer by 405-nm radiation. The FL lifetime in this region is 14.5 ns. Additional experiments have shown that this FL component makes an especially significant contribution to the FL spectra of thin irradiated 5CB LC layers.

We also suggest that one of the triple bonds in the cyanide groups is broken under UV radiation and then the neighboring molecules are cross-linked, as was described in [18] for the ciano-containing molecules in polyacrylonitrile. The activation energy of this process is about 4 eV, which is comparable with the energy of the 5CB LC electronic excitation, i.e., in view of the energy, such a mechanism is possible. Thus, the intensive UV irradiation of 5CB LC layers causes complicated photochemical processes, which can result in the formation of stable physical and chemical dimers. Such photoproducts may be responsible for an increased

absorption at $\lambda > 350$ nm and for the slight yellow color of the irradiated samples.

3. Dichroism of FL Spectra of Oriented 5CB LC Layers

Once the effect of the intensive unpolarized UV irradiation on the 5CB FL spectra is considered, we come to the case of polarized UV radiation. At the long irradiation by the polarized UV light of a mercury lamp, we observed changes in the FL spectra similar to those caused by unpolarized light. In addition, the FL spectrum became polarized, which pointed to the orientation of the 5CB LC layer. This orientation of the irradiated LC layer was also observed with a polarization microscope.

To measure polarized spectra and determine the anisotropy parameters, we use a traditional method [19–24], as is described below. The FL spectra of oriented samples are measured in four geometries shown in Fig. 4. The first subscript in the notation of the FL intensities corresponds to the exciting light polarization, and the second denotes the emission polarization. To characterize the dichroism of the FL spectrum, we introduce anisotropy parameters, which are the ratios of the emission components to the total FL intensity,

$$R_{1} = (I_{zz} - I_{zy})/(I_{zz} + 2I_{zy}),$$

$$R_{2} = (I_{yy} - I_{yz})/(I_{yy} + 2I_{yz}).$$
(1)

Figure 5 shows the spectra of the polarized FL, excited by the polarized radiation at $\lambda_{\rm E} = 337$ nm, for two types of LC cells. Curves 1-4 correspond to the LC layers oriented by polyimide substrates, and curves 5-8 represent the polarized spectra of the LC layer oriented directly by the linearly polarized UV radiation of a mercury lamp. As the z axis, we choose, for the first case, the direction of rubbing of the substrates and, for the second case, the direction perpendicular to the orienting radiation vector E. One can see from Fig. 5 that the maximum FL signal is recorded when the exciting light polarization is parallel to the z axis. This indicates that the LC layer is oriented in the rubbing direction, when oriented by rubbing, or in the direction perpendicular to the orienting light polarization, when oriented by irradiation.

Figure 5 also demonstrates that the polarized FL spectra of the UV-irradiated layers are shifted to longer wavelengths. The fluorescence peaks of the irradiated samples lie around 410 nm. The relative quantum yield grows under UV irradiation approximately by a factor of 3. In addition, a splitting of the I_{zz} and I_{yy} FL components is observed in both cases. The peak of I_{zz} fluorescence is shifted by about 260 cm⁻¹ to longer wavelengths with respect to the I_{yy} peak. At the 365- and 405-nm excitation (Fig. 3, curves I-4 and 5-8, respectively), this splitting is a little larger and equal to 290 cm⁻¹. The polarized FL spectra given in Figs. 3 and

5 are normalized to the peaks of the most intense I_{zz} components.

From the polarized FL spectra shown in Figs. 3 and 5, the anisotropy parameters are calculated for various excitation and fluorescence wavelengths. The parameters R_1 and R_2 virtually do not depend on the fluorescence wavelength at a given λ_E . The anisotropy parameters for the excitation wavelengths $\lambda_E = 337$, 365, and 405 nm are listed in the table. The table demonstrates that the R_1 and R_2 parameters have close values for the nonirradiated and irradiated LC layers in spite of the difference in the FL spectra. With the growing excitation wavelength, the R_1 parameter increases from 0.35 to 0.65 both for the nonirradiated and irradiated and irradiated layer. The R_2 parameter of the FL anisotropy is negative, and its dependence on the excitation wavelength is insignificant (it is equal to approximately –0.35).

The polarized FL spectra of the nematic and isotropic phases of 5CB LC were measured at various temperatures. These spectra served as a base for the calculation of temperature dependences of the anisotropy parameters, which are given in Fig. 6 for $\lambda_E = 365$ nm. One can see that R_1 and R_2 differ from each other in the nematic phase and coincide in the isotropic phase. Both parameters crucially vary in the vicinity of the nematic–isotropic phase transition.

Let us compare the data obtained in this study with the conclusions of the theory of polarized fluorescence considered, for example, in [24]. If the LC layer is perfectly oriented in the z axis direction, and the moments of the absorption and radiative transitions of its molecules are oriented along the long molecular axis, one would have $I_{yz} = 0$ and $R_1 = 1$. In the considered case, R_1 for $\lambda_E = 365$ nm is close to 0.6 in a wide spectral region of the fluorescence. This can point both to an imperfect ordering of molecules and to the existence of moments of absorption and radiative transitions oriented along the short molecular axis. It is well known that the LC ordering is imperfect, and the parameter of the orientation order far away from the phase transition to the isotropic liquid is 0.4–0.6 [24]. On the other hand, the emission anisotropy also can be far from ideal. This conclusion can be made when considering another assumption. For the isotropic distribution of molecules with absorption and emission dipoles oriented along the long molecular axis, the minimum R_1 and R_2 should be equal to 0.4 if the molecule is not reoriented during the emission. According to [14], deep in the isotropic 5CB phase, the reorientation and emission times are comparable, i.e. the assumption should still work. In the experiment we conducted, $R_1 = R_2 =$ 0.24 in the isotropic phase. This may testify to the fact that the real emitting centers in 5CB have several differently oriented dipole radiative transitions. This may be caused by the predimer and dimer complexes, which comprise a great part of 5CB emitting centers. The FL anisotropy of such molecular complexes can be notice-

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Fig. 4. Scheme of the polarized FL excitation and recording.



Fig. 5. Time-resolved spectra of the polarized fluorescence of (1-4) nonirradiated and (5-8) UV-irradiated 5CB LC for $(1, 5) I_{zz}$, $(3, 6) I_{zy}$, $(4, 8) I_{yy}$, and $(2, 7) I_{yz}$. T = 301 K, $t_d = 0$ ns, $\lambda_E = 337$ nm.



Fig. 6. Temperature dependence of the anisotropy parameters of a 5CB LC layer oriented by UV irradiation, $\lambda_{\rm E} = 365$ nm. $T_{\rm NI}$ is the temperature of the nematic–isotropic liquid transition.

R	$\lambda_{\rm E},{ m nm}$		
	337	365	405
LC layer oriented by rubbing			
R_1	0.35	0.6	0.6
R_2	-0.35	-0.35	-0.35
LC layer oriented by irradiation			
R_1	0.35	0.6	0.65
R_2	-0.35	-0.35	-0.35

Parameters of FL anisotropy at different excitation wavelengths (T = 301 K)

ably weaker than that of separate molecules. Taking into account that the FL anisotropy of 5CB emitting centers is not completely studied, we believe that the calculation of the p_2 and p_4 order parameters on the basis of the intrinsic polarized fluorescence of 5CB LC would be incorrect, since the calculation formulas are based on the ideal absorption and emission anisotropy [19, 20]. Hence, we restrict ourselves to the consideration of the anisotropy parameters.

In closing, we dwell briefly on the LC photoorientation mechanism. Possible photoinduced molecular processes were previously discussed in [5-7]. It was assumed that light orients the LC layers contacting with the substrates, and deeper LC layers are oriented due to the long correlation length of the orientation. As possible mechanisms, the photoselection processes were considered due to the photoinduced adsorption and desorption of 5CB molecules at the substrate surface, and the process of reorientation of the 5CB molecules near the surface in a direction perpendicular to the light polarization plane. The photoselection and reorientation were theoretically studied in [26] and demonstrated by the example of azopolymers. It is reasonable to suppose that the formation of dimer pairs in UV-irradiated LC is involved in the LC photoorientation. Since the formed pairs are stable, their appearance under polarized irradiation can lead to anisotropy through the photoselection mechanism. On the other hand, photodimers absorbing at longer wavelengths can be more efficiently excited by a mercury lamp. Hence, they can have an efficient mechanism of reorientation. Finally, efficiency of the adsorption and desorption of the photoproducts can essentially differ from those of monomer molecules. This difference may also cause surface anisotropy under polarized irradiation.

CONCLUSION

Thus, irreversible phototransformations occur in the photooriented LC layer, which manifest themselves in the absorption and fluorescence spectra. Since the changes observed in the mesophase and LC phase of 5CB are absent in 5CB molecular solutions, it is supposed that the photoprocesses are at least bimolecular. As a possible process, we consider the photodimerization of 5CB molecules in predimer states. Dimer pairs can participate in absorption and fluorescence as a single center. Changes in spectral and kinetic FL characteristics under irradiation can be described within this supposition.

Formation of photoproducts can play a key role in molecular photoorientation. Earlier it was thought that the orientation of LC layers under irradiation occurs due to the photoordering of 5CB molecules contacting with the substrate. However, it is not improbable that the photoproducts, which have the absorption spectrum shifted to longer wavelengths, are those centers that define the orientation order, as occurs at the photoorientation of LC doped with dyes [27]. To clarify this question, we will conduct additional investigations of the 5CB photoorientation mechanism.

The anisotropy parameters of the polarized FL spectra of LC layers oriented by the rubbing of substrates and by direct irradiation are close to each other, in spite of a considerable difference in the FL spectra themselves. This points to the comparable efficiency of both methods.

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