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Ordering phenomena and photoorientation processes in photochromic thin films of LC chiral azobenzene-containing polymer systems

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ABSTRACT

The processes of mesogen ordering in nematic and cholesteric side-chain polymer systems were studied by polarized optical spectroscopy. For this purpose, nematic azobenzene-containing copolyacrylate was synthesized and its cholesteric mixture with chiral dopant was prepared. Thin films of copolymer and mixture were prepared by spin-coating using polyimide-coated and rubbed glass and quartz substrates. Film thickness was comparable or less than helix pitch value. It was found that azobenzene and phenylbenzoate side groups of nematic copolymer are oriented after annealing along a rubbing direction. Cholesteric mixture demonstrates a more complicated behaviour, the direction of orientation of the side-chain groups is rotated at certain angle depending on the film thickness. Dichroism in azobenzene groups' absorbance is lower for cholesteric mixture and decreases with film thickness increasing. A possible mechanism of ordering of the side-chain groups in these thin films was proposed based on the helical order formation. Photooptical switching of the orientation and an influence of the nonpolarized and polarized light on the cholesteric thin films were studied demonstrating a possibility of using such thin films of photochromic cholesterics as promising photoswitchable materials.

1. Introduction

In a present time attention of many scientists specialized in material science is concentrated on the development of new materials for optical data recording and storage. One of the most promising types of materials is the side-chain photoactive polymers [1–7]. A combination of photosensitivity with self-organization of liquid crystalline (LC) side-chain photoactive polymers allows one to obtain materials in the form of the films, or thin coatings with tunable optical properties which could be fixed for a long period of time in the "frozen", glassy state.

Another promising application of photochromic LC copolymers deals with the research field of developing new types of the so-called "command" surfaces [8]. In this case very thin film of photosensitive polymer can change low-molar-mass liquid crystal orientation deposited on glass substrate under the polarized light action.

One of the most interesting types of photoactive LC polymers are the copolymers and composites forming cholesteric mesophase [3]. Helical supramolecular structure has a number of unusual optical properties, one of them is a selective reflection of light. Introduction into cholesteric polymer photoactive groups gives the possibility of the helical structure change by light action affecting optical properties [3]. In a number of papers [9–12] photoorientation processes and holographic recording on cholesteric polymer liquid crystals were studied. A great interest of several research groups is concentrated on induction of a helical cholesteric-like organization in amorphous or LC azobenzene-containing systems without any optically active groups [13–20]. Phenomenon of macroscopic chirality induction was observed many times under irradiation of above-mentioned systems by circularly polarized light and several mechanism types of this effect were proposed.

The most papers in this scientific field were performed using relatively "thick" films with thickness noticeable larger than pitch of the helix. Only few papers [20–22] describe properties of films with thickness lower than the pitch of cholesteric helix. In paper given by Meijer et al. [20] unusual thickness dependence of circular dichroism in thin films of chiral substituted polyfluorene was studied. Authors found the exceptionally high dissymmetry ratios originating on a nanoscopic level and related to the long-range correlation in the polymer chain orientation induced by annealing the film in its liquid crystalline phase.

In our recent papers [21–23] copolymers containing azobenzene photochromic and chiral cholesterol-containing side groups as well as mixtures of copolymers with chiral dopant were studied. It was found that in thin films of such systems the elements of the helical supramolecular structure are presented. Influence of UV, visible

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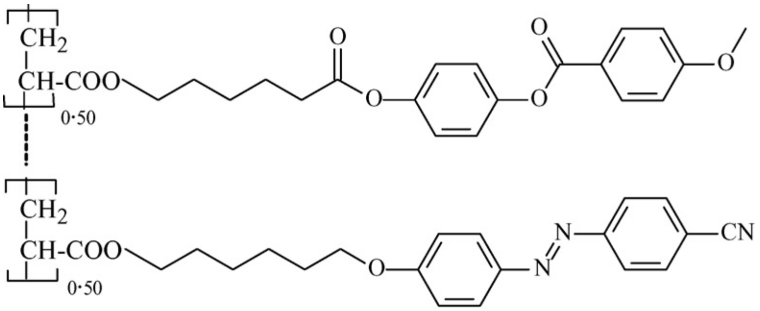
light and temperature actions on structure and orientation of the mesogenic groups in thin films was examined. It was found that an annealing of initially isotropic films of cholesteric copolymers induces more pronounced helical organization as clearly shown using CD spectral measurements. UV light irradiation completely disrupts any helical order due to the E–Z isomerization with high yield of Z-isomer (∼90%) possessing the low anisometry and non-mesogenic character. On the other hand, irradiation with polarized visible light induces photoorientation of azobenzene and mesogenic side groups in the direction perpendicular to the polarization plane.

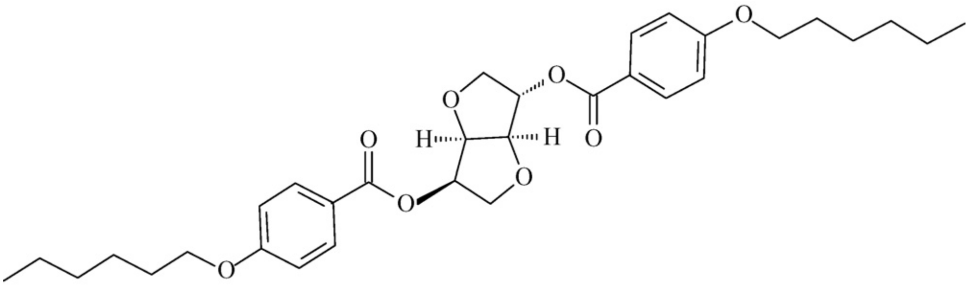
It is noteworthy, that the modern state-of-art in an area of nanotechnology, optical information processing deals mostly with very thin layers, and films of organic or inorganic materials. A large number of research groups are focusing their attention on the preparation and detailed study of the thin layers, films, coatings, etc. prepared by different techniques. Among these techniques one of the most prominent methods for the film preparation is the spin-coating. This method has found a wide application in the microelectronics, optics, display technology and other fields. In this connection a special interest from both fundamental and practical points of view presents a study of influence of the surface and phase boundaries on optical, thermal and other characteristics of thin polymer films. For example, in some papers [24–26] was found that in the case of very thin films (about ∼100 nm and thinner), their glass transition temperature, structure, and molecular mobility markedly depend on the thickness of polymer film.

Considering above-mentioned reasons we have focused our current study on further continuation of previous works [21–23]. In these papers films of cholesteric copolymers were prepared using uncoated quartz or glass plates as substrates, whereas in this paper we have investigated properties of thin spin-coated film of copolymer-based cholesteric mixture placed onto uniaxially rubbed polyimide-coated glass substrates. This very well-known technique allows one to obtain good-oriented thin films of liquid crystals and widely use them in display technology [27].

As objects for investigation nematic photochromic copolymer **PAAzo** and its cholesteric mixture containing chiral dopant **HexSorb** were selected.



PAAzo



HexSorb

Copolymer under study consists of two types of side groups, nematogenic phenylmethoxybenzoate and photosensitive cyanoazobenzene; this sample is characterized by nematic mesophase formation.

The main feature of azobenzene-containing substances is that an action of light on their derivatives induces reversible E–Z isomerization with respect to N=N bond.

It is noteworthy, that E-isomer of azobenzene derivatives has tendency to form LC order due to its rod-like shape. For example, homopolymer of cyanoazobenzene monomer with six-methylene units' spacer (see **PAAzo** structure) forms smectic phase [28]. On the other hand, Z-isomer has a bent shape with low anisometry and, therefore, disrupts the LC order [29,30].

In order to avoid a formation of smectic mesophase which is unconvenient for cholesteric phase induction we have synthesized copolymer containing nematogenic phenylmethoxybenzoate monomer units. Homopolymer of this monomer forms only nematic phase [31].

As low-molar-mass chiral dopant for cholesteric phase induction we have selected isosorbide derivative **HexSorb** (4 wt%) having a very high helical twisting power and good miscibility with polymers [32].

The goal of our investigations concerns spectroscopic study of linearly polarized light absorbance of azobenzene moieties in annealed films of nematic copolymer and cholesteric mixtures placed on uniaxially rubbed polyimide-coated glass substrate. These investigations allow one to find peculiarities of supramolecular structure formation in such systems.

2. Experimental

2.1. Monomers and chiral dopant synthesis

Synthesis of mesogenic phenylbenzoate monomer 4-{[6-(acryloyloxy)hexanoyl]oxy}phenyl 4-methoxybenzoate, low-molar-mass chiral dopant **HexSorb** was performed according to papers [31,32], respectively.

Photochromic monomer was synthesized according to method described in [28]. Yield 69%, m.p. 88 °C.

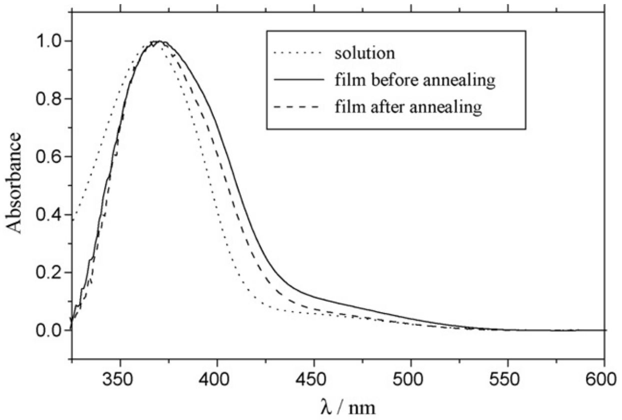


Fig. 1. Normalized absorbance spectra of chlorophorm solution (~10^{-5} mol/L) and film of **copolymer** before and after annealing (60 °C, 6 h).

^{1}H NMR ($CDCl_3$): δ 1.4–1.9 (m, 8H, CH_2), 3.9–4.3 (m, 4H, CH_2–O), 5.6 (d, 1H, CH_2=), 6.3 (d, 1H, CH_2=), 6.1 (m, 1H, –CH=), 7.0 (d, 2H, Ph), 7.7–8.0 (m, 6H, Ph).

IR (KBr, cm^{-1}): 2212 (C≡N), 1715 (C=O in RCOOR′), 2942, 2854 (CH_2), 1596 (C–C in Ar), 1250 (COC), 1627 (C=C).

2.2. Copolymer synthesis

Copolymer was synthesised by radical polymerization in benzene solution at 60 °C. As initiator azobisisobutyronitrile (AIBN) was used (2 wt% to monomers mixture). Obtained copolymer was purified from low-molar-mass and oligomeric substances by prolonged treatment by boiling methanol followed by drying in vacuum at 50 °C for 2 h. Composition of the copolymer was determined by UV/visible spectroscopy [33]; according to this method ratio of azobenzene and phenylbenzoate units in copolymer is 1:1, that is correspond to molar ratio in monomer mixture (for acrylic monomers of such type the radical polymerization rates are almost equal and high conversion of polymerization also leads to the same content of monomer units in copolymer as it is in the feed). Degree of polymerization for copolymer is about 15 as measured by GPC method using polystyrene standard.

2.3. Cholesteric mixture preparation

For mixture preparation copolymer **PAAzo** and chiral dopant **HexSorb** (4 wt% to copolymer) were dissolved in chlorophorme followed by slow evaporation at room temperature and drying in vacuum at 50 °C for 2 h.

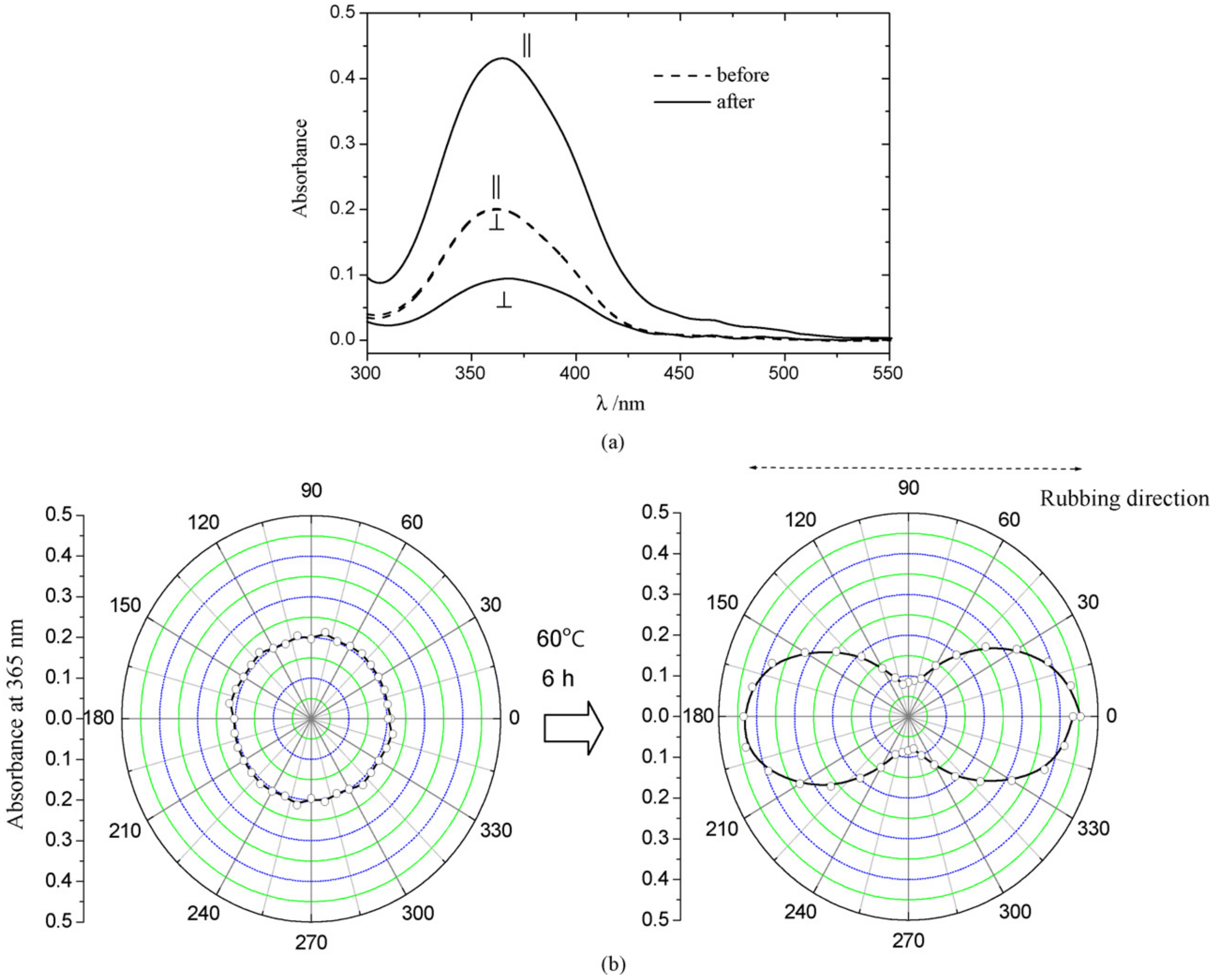


Fig. 2. (a) Polarized absorbance spectra for **copolymer** film before and after annealing; (b) corresponding polar plots of polarized absorbance at 365 nm. The spectra (a) were recorded for probe light that was linearly polarized either in parallel (||) or perpendicular (⊥) to the polarization of the irradiation light. Polar plots (b) were obtained from a number of spectra measured by rotation of polarizer with step 10°.

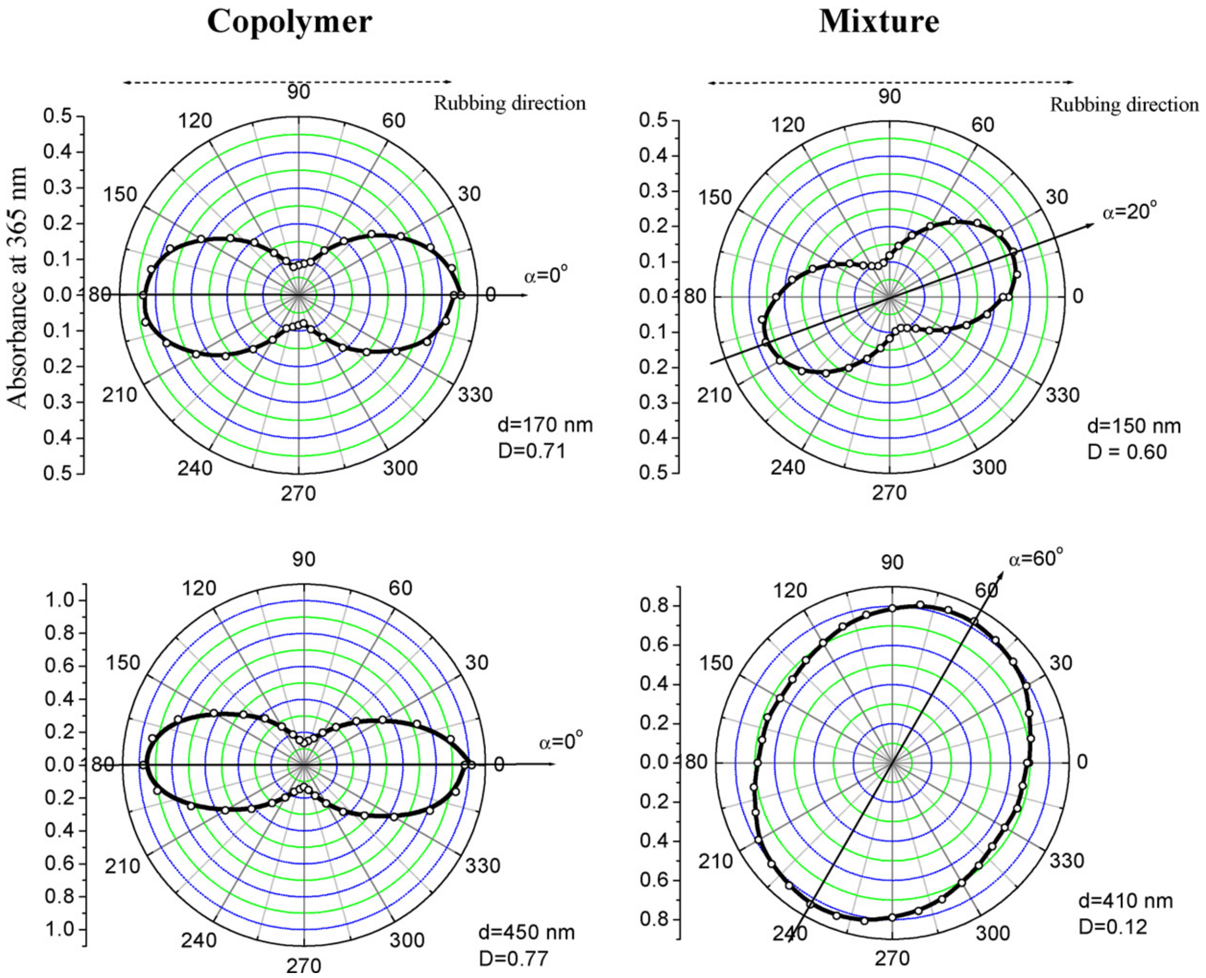


Fig. 3. Polar plot of azobenzene fragments polarized light absorbance at 365 nm obtained for nematic **copolymer** (left) and cholesteric **mixture** (right) at different films thickness (shown in figures).

2.4. *Films preparation*

A number of samples of **copolymer** and **mixture** with different thickness were prepared by spin-coating method. As substrates optical-quality glass plates with rubbed polyimide layer (ZLI-2650, Merck) were used. Thickness of the films was controlled by changing the solution concentration (from 6 to 50 mg/mL), whereas the rate of rotation was the same (3000 min^{-1}). Determination of the film thickness (l) was performed by a standard method [34] using values of isotropic extinction coefficient of azobenzene groups ($\varepsilon_i^{365} = 1.4 \times 10^4$ L/mol cm) and absorbance value of film at the same wavelength (A_i^{365}). Taking into account concentration of azobenzene groups in copolymer film (1.26 mol/L), the films thickness (in nanometers) was calculated using Bouguer–Lambert–Beer law: $l = A_i^{365} \times 10^7/(1.4 \times 10^4 \times 1.26)$.

2.5. *Physico-chemical and photooptical investigations*

The phase transitions of the **copolymer** and **mixture** were studied by differential scanning calorimetry (DSC) with a Perkin Elmer DSC-7 thermal analyzer (a scanning rate of 10 K/min).

The polarizing microscope investigations were performed using a Mettler TA-400 thermal analyzer and a LOMO P-112 polarizing microscope.

^{1}H NMR spectra were recorded using spectrometer MSL-300 "Bruker", in deuterochlorophorm.

The photochemical properties were studied using a special instrument described in [35] and equipped with a DRSh-250 ultra-high pressure mercury lamp with 365 nm interference filter and KLM-473/h-150 diode laser (473 nm). To prevent heating of the samples due to IR irradiation of the lamp, water filter was used. To obtain plane-parallel light beam, quartz lens was used. The intensity of light was equal to 1.0 mW/cm^2 (365 nm) and ~2 W/cm^2 for laser, as measured by LaserMate-Q (Coherent) intensity meter. Spectral measurements were performed using Unicam UV-500 spectrophotometer.

The orientational order was studied using polarized UV/visible spectroscopy, because the transition moment of the E-isomer of azobenzene moiety is directed along the long axis of this group. For this purpose the angular dependence of the polarized light absorbance was measured using a photodiode array UV/visible spectrometer (J&M) with a step-width of 10°.

The dichroism values were calculated from the spectra using the following equation:

$$D = \frac{A_{\parallel} - A_{\perp}}{A_{\parallel} + A_{\perp}} \tag{1}$$

where $A_{\parallel}$ is the polarized light absorbance at the preferred direction of chromophore orientation; $A_{\perp}$ is the absorbance perpendicular to this direction. It is noteworthy, that $A_{\parallel}$ in some cases does not coincide with value of absorbance in rubbing direction.

3. Results and discussion

3.1. Optical properties of thin films of nematic copolymer and cholesteric mixture

According to polarizing optical microscopy and DSC **copolymer** and **mixture** form nematic and cholesteric mesophases, respectively. Thermal properties of **copolymer** and **mixture** are presented below:

Copolymer: g 24 °C N 120 °C I.
Mixture: g 24 °C N* 108 °C I.

Planarly oriented films of **mixture** with thickness of several microns and annealed at temperatures higher T_g demonstrate selective light reflection in the near-infrared spectral range, close to the visible one ($\lambda_{max} \sim 730$ nm).

Let us consider spectra of copolymer **PAAzo** in solution and thin films before and after annealing at the temperature higher than glass transition. As clearly seen from Fig. 1 shape of absorbance peak, as well as maximum position are practically the same that can be explained by absence of aggregation phenomena in the films of copolymer (aggregation phenomena in the case of similar azobenzene-containing polymer system leads to more significant spectral changes) [36,37]. Evidently, the introduction of phenylbenzoate side groups in polymer system prevents possible aggregation of photochromic moiety due to a dilution effect. In addition, a weak donor–acceptor interaction between electron-withdrawing cyanoazobenzene groups and electron–donor phenylbenzoate fragments is also possible, that prevents an aggregation [38].

Fig. 2a and b shows polarized absorbance spectra and polar plots of absorbance at 365 nm (corresponding to a maximum of π–π^* electronic transition) before and after annealing. If freshly prepared films are completely isotropic, the annealing above T_g induces an anisotropy of absorbance associated with cooperative orientation of azobenzene and phenylbenzoate side groups. For the **copolymer** films direction of this orientation is independent on the film thickness and completely coincides with rubbing direction of polyimide layer (Fig. 3).

We have performed the comparative study of polarized absorbance spectra for nematic **copolymer** and **mixture** films of the different thickness. As shown in Figs. 2b and 3 the direction of maximal absorbance for the **copolymer** is always coincides with the rubbing axis whereas for the **mixture** this direction is rotated to the same side and to different angle α (between rubbing axis and maximal polarized absorbance direction) depending on film thickness. Dichroism value calculated using Eq. (1) is also strongly depends on the films thickness (Fig. 3).

Thickness dependences of dichroism and rotation angle α for the copolymer and the mixture were investigated (Fig. 4). As clearly seen from these dependences for the **mixture** the dichroism values gradually decrease and the α values increase with thickness increasing, whereas for the **copolymer** angle $\alpha \sim 0$ at any thickness.

The same character of dependences was qualitatively observed for mesogenic phenylbenzoate side groups, i.e. thermally induced orientation has cooperative character. (Cooperative character of the orientation in copolymers was investigated in detail by a number of scientists [39–44] and we do not concentrate our attention on this topic.)

It is very important to compare the values of thickness of films studied in our work with the values of pitch of supramolecular helix realized in relatively thick films (several microns) of cholesteric mixture. According to the spectral studies of the annealed films with 10 μm thickness a selective light reflection peak is observed at $\lambda_{max} \sim 730$ nm. Corresponding pitch of the helix is inversely proportional to average refractive index **n** (in our case **n** ~ 1.6) and λ_{max}, therefore $P \sim 450$ nm. According to these calculations the thickness of our films obtained by spin-coating method is comparable or slightly less than the pitch of the undeformed cholesteric helix.

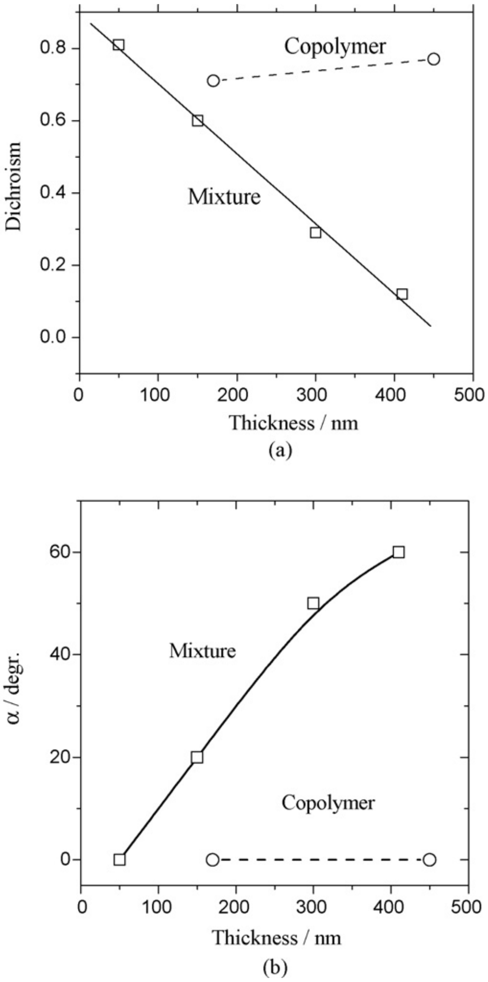


Fig. 4. Dependencies of dichroism at 365 nm (a) and angle α (b) on films thickness for **copolymer** and **mixture**.

According to the experimental results described above we proposed the following model of side groups' orientation in the thin films of our systems. Fig. 5 shows the possible idealized scheme of side group packing in the films of nematic copolymer **PAAzo** (Fig. 5a), cholesteric mixture films with high thickness (Fig. 5b), and with thickness comparable or less then the helix pitch (Fig. 5c).

Appearance of linear dichroism for side groups observed in this work is associated with the low thickness of films prepared by spin-coating and also with complete or partial untwisting of cholesteric helix (layer 2 in Fig. 5c) due to the interface boundaries. On polyimide-coated substrate the mesogenic and photochromic groups have tendency to be oriented along the rubbing direction (layer 1 in Fig. 5c). At the interface polymer-air mesogenic groups in order to minimize free energy have tendency to be oriented homeotropically, i.e. along normal to the film plane (Fig. 5a, layer 3 in Fig. 5c). The similar tendency in nematic low-molar-mass liquid crystals was described in several papers [45–47].

Therefore, for the thin films of our cholesteric **mixture** partial untwisting is realized. Schematic representation of top projection of mesogenic and azobenzene groups in these thin films is presented in Fig. 5d. Partial untwisting of the helical structure results not only in appearance of linear dichroism but also in noticeable rotation of direction of polarized absorbance maximum in respect to the rubbing direction. Angle of such rotation α becomes larger with

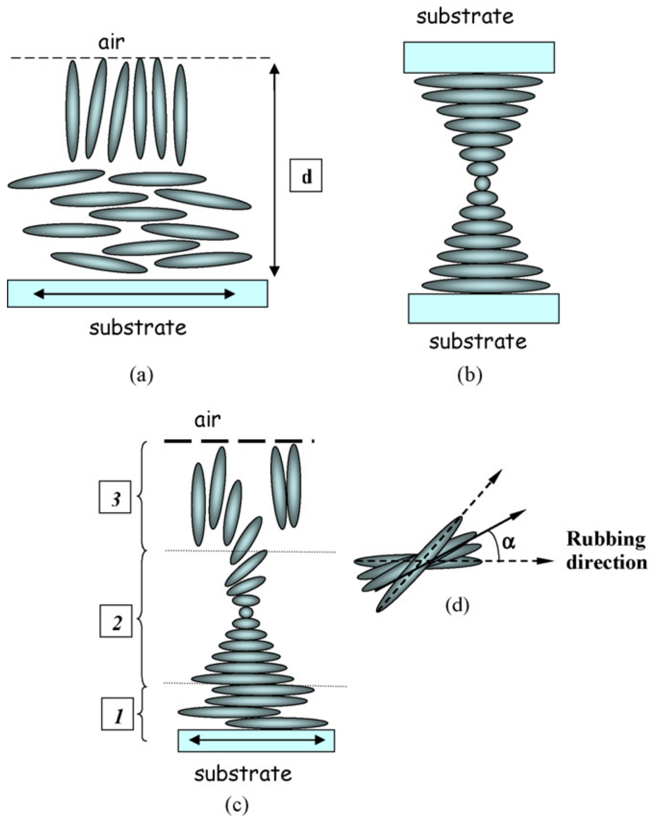


Fig. 5. Schematic representation of chromophore mesogenic groups orientation in different types of films: (a) thin film of nematic liquid crystal on uniaxially rubbed substrate; (b) planarly oriented cholesteric liquid crystal between two glass substrates (**d** > **P**); (c) thin film of cholesteric liquid crystal on uniaxially rubbed substrate (**d** ≤ **P**); (d) projection of partially untwisted helical structure along the film normal. (Main-chains and spacers are omitted for clarity.)

thickness increasing, whereas dichroism value is decreased. It is very intriguingly, that both, dichroism and angle, dependences are almost linear (Fig. 4). This fact can be considered as additional clear evidence for the mechanism discussed above.

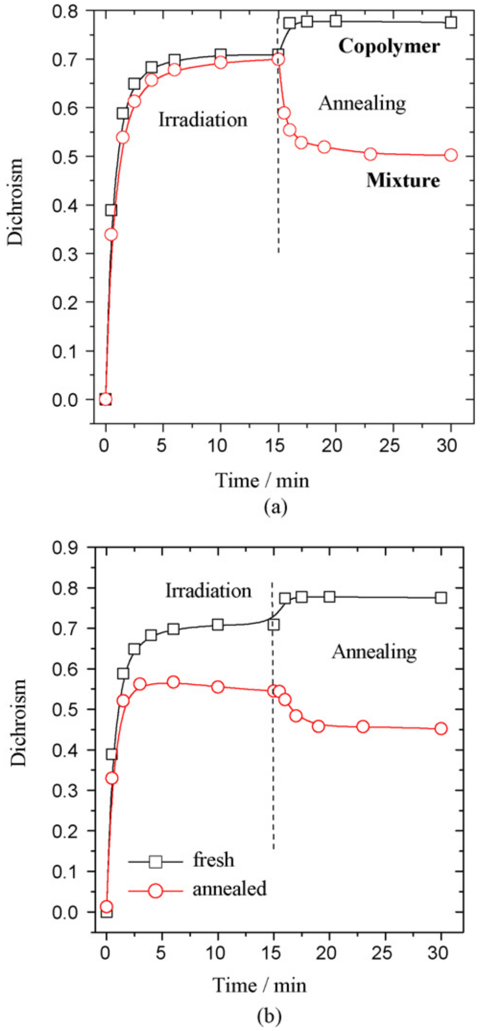


Fig. 6. Kinetics of dichroism changes during irradiation with polarized light (473 nm) followed by annealing at 50 °C for (a) fresh amorphous films of **copolymer** and **mixture**; (b) for film of **copolymer** annealed before irradiation at 50 °C for 30 min.

3.2. *Photooptical properties of **mixture** and **copolymer** films*

Irradiation of the annealed **mixture** and **copolymer** films by UV light (365 nm) induces E–Z isomerization of azobenzene groups with high yield of the bent-shaped Z-isomer and, thus, diminishes the dichroism in absorbance of both, azobenzene and phenylbenzoate side groups, i.e. completely destroys orientation and the films become isotropic. Subsequent annealing results in complete back thermal Z–E isomerization and recovers the chromophore orientation, dichroism, and α values.

It is well-known that an irradiation of azobenzene-containing side-chain copolymers by the polarized light induces cyclic E–Z–E isomerization process in azobenzene fragments that leads to light-induced cooperative orientation of photochromic and non-photochromic side groups [3,4]. This effect leads to an appearance of photoinduced dichroism and birefringence. For copolymer **PAAzo8** (with eight methylene units in spacer) and its mixture with **Hex-Sorb** photoorientation phenomenon was studied previously [22] using polarized UV (365 nm) and blue light (436 nm).

In this paper we have studied the similar phenomena by using more powerful light source with longer wavelength, diode laser with emitting wavelength 473 nm (170 mW). Fig. 6 shows kinetic curves of dichroism growth under irradiation of **copolymer** and **mixture** films with thickness of 160 nm. As clearly seen from figure, rate of photoorientation as well as maximal values of dichroism are almost equal for both systems. From the other hand, annealing of irradiated samples results in quite different effects. For nematic **copolymers** an increase of dichroism takes place (Fig. 6a), whereas for cholesteric **mixture** degree of orientation decreases achieving approximately the same value as for film prepared on polyimide-coated substrate, ~0.5 (see Fig. 4a). Direction of orientation did not change under annealing. Increase in dichroism for nematic **copolymer** is associated with so-called “gain effect” [4,37,48] and relates to uniaxial ordering enhancing of chromophores and mesogens under annealing. Small decrease of orientation degree in cholesteric **mixture** films evidently is related to the partial helical ordering as it was discussed above.

An annealing of **copolymer** and **mixture** films before irradiation reduces values of dichroism on 10–15% (Fig. 6b). Formation of LC multidomain state prevents only in small extent possibility of reorientation. It is noteworthy that for annealed **copolymer** film subsequent thermal relaxation of a photoinduced order appears as small decrease in dichroism. Probably this effect is associated with the “memory” effect: chromophores and mesogens “remember” a little bit initial orientation in the randomly oriented multidomains of the annealed film. For cholesteric **mixture** such “memory” effect has not been found and annealing results in approaching of the same dichroism value as for the non-annealed films.

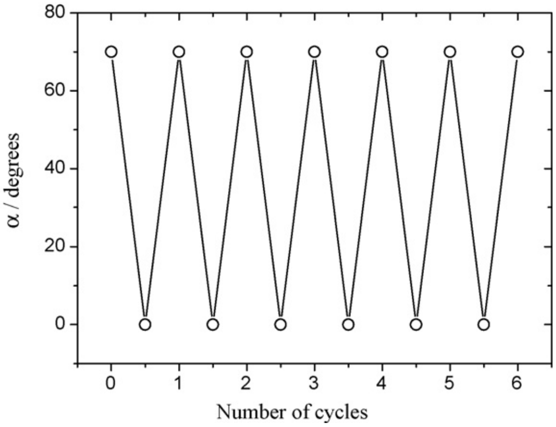


Fig. 7. Changes of angle α during cycles of irradiation and annealing. Irradiation was performed by 473 nm laser during followed by an annealing at 50 °C. Irradiation and annealing time was 15 min.

In the present work we would like also raise the question as to whether there exists a possibility of photoinduced *re*-orientation of side groups by irradiation with light polarized in different directions. Film of the mixture with thickness of ca. 500 nm was irradiated with blue laser light polarized in direction perpendicular to rubbing axis. After irradiation, the chromophores (azobenzene and phenylbenzoate) are re-oriented from $\alpha \sim 70^\circ$ to 0°, i.e. along to rubbing axis. An annealing at 50 °C recovers initial orientation and the dichroism and α approach the values measured before irradiation. We have realized several cycles of irradiation and annealing and found that reorientation can be observed without noticeable fatigue. Fig. 7 shows the changes of characteristic angle α during seven cycles of irradiation-annealing. It is noteworthy that kinetics and fatigue resistance for dichroism and angle α are similar for any direction of laser light polarization with respect to rubbing axis.

In conclusion, thermally induced orientation processes on uniaxially rubbed polyimide-coated substrates were studied for nematic copolymer and cholesteric mixture obtained on its base. Possible mechanisms of uniaxial (for nematic **copolymer**) and partial helical organization (in the case of cholesteric **mixture**) were suggested. Results on influence of nonpolarized and polarized light on cholesteric thin films were obtained. It is shown that UV light action completely destroys any ordering in films whereas action of polarized blue light results in reversible reorientation of azobenzene and phenylbenzoate side groups. These effects allow one to consider such thin films as promising materials for photo- and thermo-optical switching.

Acknowledgements

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