INVESTIGATION OF PHOTOINDUCED ORIENTATION ORDERING IN POLYMETHACRYLATES WITH SIDE-CHAIN AZOBENZENE MOIETIES

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Abstract. The initial and the photoinduced 3D orientational order in the films of two methacrylic polymers containing azobenzene groups with electron-donor or electron-acceptor substituents was studied by using null ellipsometry and UV/Vis absorption spectroscopy. The 3D order was induced by monochromatic polarized light of two different wavelengths corresponding to $\pi\pi^*$ and $n\pi^*$ absorption bands of azochromophores. We found that, depending on the excitation wavelength and terminal substituents of azochromophores, recording kinetics of photoinduced anisotropy in the polymers is dominated by either angular redistribution or angularly selective trans-cis isomerization of the chromophores originally being mainly in the trans-form. Before irradiation, the azobenzene groups in the films of all polymers show preference to the out-of-plane ordering. Scenario of the order transformation under irradiation depends on the prevailing mechanism of the photoinduced anisotropy. In case of angular redistribution, initial positive uniaxial order of azochromophores is transformed into the negative uniaxial order characterized by random orientation of these units in the plane perpendicular to the polarization direction of the exciting light. In turn, in case of photoselection, the chromophores approach a zero-order (spatially isotropic) state due to exhaustion of the anisotropic trans-isomers in all directions. The transient orientational structures in these kinetic processes are biaxial.

Keywords: azopolymer, polymethacrylate, photoinduced orientation.

1. Introduction

In the past years, azo-dye-containing polymer systems have been the subject of intensive researches due to their potential applications in photonics, optoelectronics, and optical signal processing [1, 2]. One of the main interests to these polymer systems is their birefringence property when they are irradiated with a linearly polarized light [3]. This birefringence comes from the ordering of the azobenzene groups through trans-cis-trans isomerization cycles, which leads to an excess of photochromic entities oriented perpendicularly to the light polarization direction. When the light is turned off, a large part of the photoinduced orientation is preserved [4, 5].

The time dependence of the photoinduced order in azobenzene polymers has been explained by Dumont et al. using a theoretical model, which takes into account the population in both the trans- and cis-metastable states [6-8]. This model considers that the polarized light induces a selective optical pumping (“angular hole-burning (AHB)”) and angular reorientations (AR) during the direct trans-cis and reverse cis-trans photoisomerizations and the cis-trans thermal back-relaxation. As a result, two mechanisms are combined, in proportions depending on molecular parameters and on irradiation conditions. The first mechanism is connected with the angular selectivity of the photoexcitation, which produces angular hole burning. This selective depleton of the ground state is common in all photochromic materials. If photoproducts are thermally and photochemically stable, the saturation of hole burning leads to a total depletion of the initial state. The second mechanism occurs when the photochromism is thermally or optically reversible (like trans-cis photoisomerization of the majority of azobenzene derivatives). In that case, molecules undergo a great number of photoisomerization cycles resulting in a kind of mechanical stirring, which induces a random-walk rotation of azochromophores. This process, known as
angular redistribution, leads to the accumulation of molecules perpendicularly to the polarization of the exciting light. More generally, after a great number of photoisomerization cycles, these two processes tend to minimize the probability of optical excitation.

In the past decade, several groups of scientists studied the influence of the molecular architecture on the orientational characteristics of photochromic polymers. In their study, Sekkat et al. [9] have investigated the photoinduced orientation in an azobenzene-containing polyglutamate film. An anisotropy in the cis-isomer orientation has been found when the film was irradiated with a linearly polarized UV light (360 nm), and a theoretical model has described the time dependence of this anisotropy. Furthermore, Natansohn et al. [10, 11] have studied the time dependence of the birefringence of poly[4-[2-(methacryloyloxy)ethyl] azobenzene] film (called pMEA) using polarized UV (360 nm) or visible (514.5 nm) light. A similar birefringence has been measured for the two wavelengths, but its value was lower than those observed in various polymers containing polar azobenzene groups. Pump irradiance and temperature dependences of the photoinduced anisotropy in a pMEA polymer film have also been studied and compared to simple theoretical predictions [12]. Yaroshchuk et al. [13, 14] using irradiation with the polarized light of two different wavelengths have found that the kinetics of photoinduced ordering of azochromophores in the amorphous and liquid crystalline azobenzene polyesters can be dominated by either AR or AHB mechanism depending on the time of thermodynamic and photostimulated relaxation of metastable cis-isomers determined, correspondingly, by the structure of photosensitive side chains and excitation wavelength. Moreover the ordering process was considered in a real 3D space allowing one to identify various spatial distributions of azochromophores. Theoretical consideration of these results was given in [13, 15].

Currently, using the methodology developed earlier in [13, 14, 16] we study features of the photoinduced ordering of azochromophores in methacrylic azopolymers. The case of ππ* excitation of azochromophores was considered in [17, 18]. In the present paper we compare orientational transformation caused by ππ* and nπ* excitation. It is demonstrated that, just as for azopolymesters, the process of 3D order transformation depends essentially on the relaxation kinetics of the excited cis-isomers. This allows us to conclude that photoinducing regularities established earlier in [13] are common for different classes of azopolymers and thus the theory of 3D ordering [13, 15] predicting these regularities is rather general.

2. Experimental

2.1. Materials Synthesis and Characterization

We used poly[4-(methacryloyloxy)-4'-nitroazobenzene] (P1) and poly[4-(methacryloyloxy)-4'-methoxyazobenzene] (P2) differing by the donor/acceptor nature of end substitutes of the side-chain azochromophore. The general formula of these compounds is presented in Fig. 1. It is evident that the polymers differ only by the end fragment R, which is NO₂ and OCH₃ in polymers P1 and P2, respectively.

![General formula of azopolymers](image)

Fig. 1. General formula of azopolymers

Synthesis of the two polymers was similar, and only the general steps towards making polymers are detailed.

Monomer synthesis. The corresponding monomers were synthesized by general methods. The azocompound (0.06 mol) and triethylamine (9.0 ml) were dissolved in THF (200 ml). The solution was kept in an ice bath for 10 min. A solution of distilled methacryloyl chloride (6.0 ml, 0.06 mol) was added slowly to the above mixture. After the addition of methacryloyl chloride, the resulting mixture was stirred at room temperature overnight. Then the solution was poured into distilled water (1 l) and the obtained residue was filtered and air-dried. Recrystallization of monomers was carried out in ethanol.

4'-methacryloyloxy-4-nitroazobenzene (M1): Orange crystals; yield 69 %; mp 418 K (by DSC). ¹H NMR (CDCl₃), δ (ppm): 8.43 (d, 2H, Ph-ortho to NO₂), 8.07 (d, 2H, Ar), 8.03 (d, 2H, Ar), 7.40 (d, 2H, Ar), 6.34 (s, 1H, =CH₂), 5.91 (s, 1H, =CH₂), 2.05 (s, 3H, -CH₃). UV-vis (Ethanol) λmax: 360, 485 nm. Elem. Anal. Calc. for C₁₈H₁₅O₂N₂: C, 61.74 %, H, 4.18 %; N, 13.50 %. Found: C, 61.70 %, H, 4.16 %; N, 13.52 %.

4'-methacryloyloxy-4-methoxyazobenzene (M2): Yellow crystals; yield 65 %; mp 545 K (by DSC). ¹H NMR (CDCl₃), δ (ppm): 7.85 (d, 2H, Ar), 7.65 (d, 2H, Ar), 7.74 (d, 2H, Ar), 6.91 (d, 2H, Ar), 6.30 (s, 1H, =CH₂), 5.84 (s, 1H, =CH₂), 2.08 (s, 3H, -CH₃).

<table>
<thead>
<tr>
<th>Monomer</th>
<th>Structure</th>
<th>Formula</th>
<th>Color</th>
<th>Yield</th>
<th>Mp (K)</th>
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<tr>
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<td>Yellow</td>
<td>65 %</td>
<td>545</td>
</tr>
</tbody>
</table>
and Illumination

2.2. Film Preparation

The polymers were dissolved in chloroform at the concentration of 2 wt %. The filtered solutions were used to spin coat the polymer films onto quartz slides. The films were kept for 30 min at 353 K to remove any remaining solvent. The film thickness measured with a profilometer was 300–500 nm. These films were of good optical uniformity.

The photoordering processes were initiated by irradiation with polarized light directed normally to the films. Two different wavelengths were used to pump the samples:

\[ \lambda_{ex1} = 365 \text{ nm, from a mercury lamp, selected by an interference filter and polarized with a Glan prism.} \]

The light intensity \( I \) was varied in the range of 4–10 mW/cm\(^2\).

\[ \lambda_{ex2} = 480 \text{ nm from an Ar+ laser (} I = 20 \text{ mW/cm}^2 \). \]

As it will be shown below, the lines \( \lambda_{ex1} = 365 \text{ nm and } \lambda_{ex2} = 480 \text{ nm correspond to } \pi \rightarrow \pi^* \text{ and } n \rightarrow \pi^* \text{ excitation, respectively. In all cases the polarization of exciting light (} E_{ex} \text{) was chosen along the } x \text{ axis of the Cartesian coordinate system with } x \text{ and } y \text{ axis parallel to the verges of the rectangular polymer film and } z \text{ axis normal to this film (Fig. 2).} \]

2.3. Methods of Studying of 3D Orientational Order

In these studies the transmission null ellipsometry (TNE) method dealing with refractive indices has been adapted to azopolymers. The optical scheme of the method is presented in Fig. 2. The probe beam (628 nm) is linearly polarized at 45° with respect to the in-plane main axes of the sample. The elliptically polarized transmitted beam is converted into a linearly polarized beam by a quarter wave plate (axis parallel to that of the polarizer). The angle \( \varphi \) of the output polarization, determined by rotation of a linear analyzer, gives the in-plane retardation \((n_r-n_i)\delta\). Then the sample is rotated around the \( x \) axis vertically aligned and the polarization angle \( \varphi \) is measured as a function of the incidence angle \( \theta \). The out-of-plane retardation \((n_z-n_i)\delta\) is determined by fitting a theoretical expression of \( \varphi(\theta) \). The TNE was used to study 3D orientation of azochromophores in both polymers, before irradiation and after successive irradiation steps with \( \lambda_{ex1} = 365 \text{ nm and } \lambda_{ex2} = 480 \text{ nm exciting light.} \)

As a complementary characterization method we also perform 2D-dichroizm measurement in the UV/visible spectral range. The UV/vis absorption measurements were carried out using a S2000 diode array spectrometer from Ocean Optics Co. The samples were set normally to the testing light from a low intensity deuterium lamp. A Glan-Thomson prism was used to polarize the probe beam.

The optical densities \((D_x \text{ and } D_y)\) corresponding to \( x \) and \( y \) in-plane polarizations, are measured with a probe beam propagating perpendicularly to the sample. The third component \((D_z)\) is estimated by the total absorption (TA) method, which presumes the conservation of the total absorption \( D_{tot} = D_x + D_y + D_z \). The \( D_{tot} \) can be easily obtained, if at some instant of time \( t_0 \) the sample is uniaxial, with an in-plane orientation of the axis of anisotropy, say \( x \). Then:

\[ D_{tot} \equiv D_x(t_0) + D_y(t_0) + D_z(t_0) = D_x(t_0) + 2D_y(t_0) \]

(1)
If the number of azobenzene units in trans configuration remains constant at each instant of time \( t \), \( D_z \) can be estimated as:

\[
D_z(t) = D_{tot} - D_x(t) - D_y(t)
\]

where \( D_x(t) \) and \( D_y(t) \) are experimentally measured. Then, the diagonal terms of the tensor of orientational order \( S_{ij} \) can be estimated. For example:

\[
S_{xx} = \frac{D_x - \frac{1}{2}(D_y + D_z)}{D_x + D_y + D_z}
\]

The components \( S_{yy} \) and \( S_{zz} \) can be obtained by cyclic permutation in expression (3).

The total absorption method can be applied in some experimental situations described below.

### 3. Results and Discussion

#### 3.1. Electronic Spectra

In our study we perform polymers containing azobenzene groups with electron-donor and electron-acceptor substituents, which strongly absorb in the visible region of the electronic spectrum. Fig. 3 shows the absorption spectrum in the 300–600 nm regions of a P1 and P2 polymer films before irradiation. These UV-vis spectra display high-intensity \( \pi\pi^* \) bands in the UV (at about 350 nm) and low-intensity \( n\pi^* \) bands in the visible region (at about 450 nm) [19]. Considering the absorption spectra of trans- and cis-azobenzene [18], the band at nearly 350 nm corresponds essentially to the absorption of the trans-isomers while the band at about 450 nm is mainly due to the absorption of the cis-isomers. The exact positions of the maximum of the \( \pi\pi^* \) absorption bands, \( \lambda_{max} \), are presented in Table 1. A red shift of P1 spectrum is explained by stretching of \( \pi \) electronic system of azochromophore due to the presence of electron-donor NO\(_2\) group.

#### 3.2. Photoinduced Ordering: Case of \( \lambda_{ex} = 365 \) nm Irradiation

At first we consider polymer P1 containing push-pull chromophore. The values of the in-plane, \( (n_x-n_y)d \), and the out-of-plane \( (n_x-n_z)d \) retardation measured for the film of this polymer after successive exposure steps are presented in Fig. 4a. It is evident that before irradiation azochromophores demonstrate slight preference to the out-of-plane orientation \( n_z > n_x = n_y \), Fig. 4a). This prolate uniaxial order is caused by a selforganization in the P1 film. In the stationary state of irradiation, \( n_x < n_y = n_z \). This implies that the UV exposure results in transformation of the initial prolate order with \( z \) axis into the oblate uniaxial order with \( x \) axis determined by the symmetry of light field. The ratio \( n_x < n_y = n_z \) implies that the azochromophores are randomly distributed in the plane perpendicular to the light polarization direction. The transient orientational structures are biaxial \( (n_x \neq n_y \neq n_z) \).

The in-plane components of optical density, \( D_x \) and \( D_y \), of the same P1 film are presented in Fig. 4b.
Initially, $D_x=D_y$, which reflects in-plane isotropy of azochromophores. In a course of irradiation, $D_x$ increases while $D_z$ decreases. This suggests that azochromophores are partially redistributed from $x$ to $y$ direction, i.e., perpendicularly to $E_{ex}$. These changes correspond to reorientation mechanism [6-8] implying that concentration of cis-isomers after irradiation steps is negligible. In this case, total absorption approach can be applied. $D_{tot}$ can be easily obtained, because, according to Fig. 4a, $D_z=D_y$ in the photostationary state. Then $D_z$ after each step of exposure can be estimated by using Eq. (2). These data are presented in Fig. 5a too.

The values of $D_x$, $D_y$, and $D_z$ are used to calculate order parameters as described above in the Experimental section. The oblate initial order is described with the parameters $S \equiv S_z = 0.08$ and $S_{xx} = S_{yy} = -S_z/2 = -0.04$ (positive uniaxial order along $z$ axis). At saturation, $S \equiv S_z = -0.145$ and $S_{xx} = S_{yy} = -S_z/2 = 0.073$ (uniaxial negative order along $x$ axis). The transient order is biaxial, i.e., $S_{xx} \neq S_{yy} \neq S_{zz}$.

The retardation curves for the P2 film are shown in Fig. 5a. Similarly to P1, the initial state of this film is characterized with the prolate uniaxial order along $z$ axis. The UV light transforms this order to biaxial ($n_x \neq n_y \neq n_z$). At long exposures, the film approaches spatially isotropic state where ratio $n_z=n_x=n_y$ is fulfilled.

The $D_x(t)$ and $D_z(t)$ curves for the P2 film are presented in Fig. 5b. In contrast to the case of P1, both $D_x$ and $D_z$ values decrease with irradiation. This indicates that the azobenzene chromophore order due to angular photoselection mechanism [6-8]. The conversion of trans-isomers in the long living cis-form first in the direction of light polarization $E_{ex}$ and then in all other spatial directions may explain isotropic order realized in P2 film at high irradiation doses. Because of growing concentration of cis-isomers with exposure time, the total absorption is not preserved and so $D_z$ component cannot be estimated. In turn, it does not allow to calculate the order parameter components for the initial and transient stages. In the photosaturation state, $S_{xx} = S_{yy} = S_{zz}$.

![Fig. 4](image1.png)

**Fig. 4.** The in-plane and out-of-plane phase retardations (a) and the principal optical density components $D_x$, $D_y$, and $D_z$ (b) for the film of polymer P1 as functions of exposure time. The light parameters are $\lambda_{ex} = 365$ nm, $I = 5$ mW/cm$^2$, linear polarization along the $x$ axis

![Fig. 5](image2.png)

**Fig. 5.** The in-plane and out-of-plane phase retardations (a) and the principal optical density components $D_x$, $D_y$, and $D_z$ (b) for the film of polymer P2 as functions of exposure time. The light parameters are $\lambda_{ex} = 365$ nm, $I = 5$ mW/cm$^2$, linear polarization along the $x$ axis
3.2. Photoinduced Ordering: case of $\lambda_{ex} = 480$ nm Irradiation

The phase retardation $(n_y-n_d)d$ and $(n_z-n_d)d$ vs. irradiation time curves for P1 and P2 films are presented in Figs. 6a and 7a, respectively. One can see that in both cases, similarly to $\lambda_{ex} = 365$ nm irradiation of P1 film, initial upright prolate uniaxial order is continuously transformed through the number of biaxial structures in the oblate uniaxial order with the ordering axis lying in plane of the film parallel to the light polarization direction ($x$ axis). The transient orientational structures are biaxial ($n_x \neq n_y \neq n_z$). Similar kinetics of phase retardation suggests similar photoordering mechanisms.

The absorption vs. exposure time curves for P1 and P2 are presented in Figs. 6b and 7b, respectively. The behavior of $D_y(t)$ and $D_z(t)$ curves for P1 film is similar to that for $\lambda_{ex} = 365$ nm irradiation and implies reorientation mechanism of photoordering. Interestingly, similar trend is observed for P2 that contrasts well with the case of $\lambda_{ex} = 365$ nm irradiation of this polymer. The ordering according to reorientation mechanism suggests that concentration of cis-isomers in both polymers at $\lambda_{ex} = 480$ nm irradiation is small. For P1 this is primarily due to short lifetime of cis-isomers, while in case of P2 this is caused by high excitation probability of cis-isomers and their forced transition to the initial trans-form. As a result, azochromophores undergo many cycles of trans-cis-trans isomerization and eventually accumulate in the directions perpendicular to $E_{ex}$ [14]. A small concentration of cis-form allows one to apply a total absorption approach for calculation of $D_x$. The calculated $D_x(t)$ curves are added to the $D_y(t)$ and $D_z(t)$ plots in Figs. 6b and 7b.

The values of $D_x$, $D_y$, and $D_z$ were used to estimate order parameters for various states. Below only uniaxial structures are characterized. The upright prolate order before irradiation is described with the parameters $S \equiv S_{xx} = 0.084$ and $S_{yz} = S_{zy} = -S_{yz}/2 = 0.042$ for P1 and $S = S_{xx} = 0.09$ and $S_{yz} = S_{zy} = -S_{yz}/2 = 0.045$ for P2. One can conclude that the spontaneous ordering in the initial state is rather weak and it slightly enhances with adding hydrophobic tails to azobenzene side chains. The oblate uniaxial distribution of azochromophores with the $x$ ordering axis, realized in the stationary state of irradiation, is characterized by the scalar $S \equiv S_{xx} = -0.13$ for P1 and $S = S_{xx} = -0.11$ for P2.

The values of photoinduced in-plane birefringence and order parameter corresponding to saturation state of irradiation are gathered in Table 2. The birefringence values were extracted from the values of phase retardation by measuring film thickness $d$ with a profilometer. It is noteworthy that the value of the photoinduced birefringence of about 0.15 is attained. This makes the studied polymers attractive for various photonic applications, such as information processing and storage, holography, waveguides, etc.

<table>
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<tr>
<th>Polymer</th>
<th>Irradiation with polarized UV light ($\lambda_{ex} = 365$ nm)</th>
<th>Irradiation with polarized visible light ($\lambda_{ex} = 480$ nm)</th>
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<tr>
<td>P1</td>
<td>$S_{xx}$ 0.15, $n_y-n_d$ 0.13, $S_{yz}$ 0.13, $n_z-n_d$ 0.07</td>
<td></td>
</tr>
<tr>
<td>P2</td>
<td>Isotropic 0.11, $n_y-n_d$ 0.02</td>
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Table 2 The values of the photoinduced order parameter $|S_{xx}|$ and in-plane birefringence $n_y-n_d$ for the films of azopolymers in the saturation state

Fig. 6. The in-plane and out-of-plane phase retardations (a) and the principal optical density components $D_x$, $D_y$, and $D_z$ (b) for the film of polymer P1 as functions of exposure time. The light parameters are $\lambda_{ex} = 480$ nm, $I = 15$ mW/cm², linear polarization along the $x$ axis.
4. Conclusions

We studied the photoinduced ordering processes in polymethacrylates with side-chain chromophores. The results show that the photoinduced order strongly depends on the structure of chromophores as well as on the spectral composition of light, as described below. The molecular structure determines the photoorientation properties of azochromophores by modifying their photochemical properties and intrinsic self-organization. Irradiation of azochromophores by modifying their photochemical mechanism, usually called angular redistribution, is rather probable of trans-cis-trans isomerization cycles occur during irradiation. These excitations are accompanied by rotation of azochromophores and their accumulation in the direction perpendicular to the exciting light polarization \( E_{\text{ex}} \). A similar ordering process takes place in the film of P2 at \( \lambda_{\text{ex}} = 480 \text{ nm} \), when excitation probability of cis-isomers is higher or comparable with that of trans-isomers. In this case the multiple cycles of trans-cis-trans-isomerization occur due to photoexcitation of both trans- and cis-isomers. This mechanism, usually called angular redistribution, is rather typical for azopolymers [5-9]. In contrast to case of \( \lambda_{\text{ex}} = 480 \text{ nm} \), at \( \lambda_{\text{ex}} = 365 \text{ nm} \) the long living azochromophores of P2 do not undergo multiple excitations and thus ordering is induced due to angular hole burning.

The 3D orientational order is observed in the non-irradiated films as well as the films after irradiation. Before irradiation, the azochromophores of both polymers show preference to the out-of-plane ordering (upright prolate uniaxial order). The transformation of this order under irradiation depends on the prevailing mechanism of the photoinduced anisotropy. In case of AR mechanism, the initial order is transformed into the oblate uniaxial order with the ordering axis coinciding with a light polarization direction \( E_{\text{ex}} \). It is characterized by random orientation of azochromophores in the plane perpendicular to \( E_{\text{ex}} \). In turn, in case of AHB, the chromophores approach the spatially isotropic state due to exhaustion of trans-isomers in all directions. The transient orientational structures in these kinetic processes are biaxial.

The regularities we observed for the studied polymers are in good agreement with the regularities earlier observed for azobenzene polyesters [14] as well as for the polymers with photosensitive cinnamoyl groups [20]. Therefore, the regularities established may be the general rules, which can be used to predict orientational order in other types of polymers.

References


ДОСЛІДЖЕННЯ ФОТОІНДУКОВАНОЇ ОРІЄНТАЦІЇ В ПОЛІМЕТАКРИЛАТАХ З БІЧ-НИМИ АЗОБЕНЗОЛЬНИМИ ФРАГМЕНТАМИ

Анотація. Вивчено кінетику фотоіндукованого впорядкування двох метакрилових полімерів, що містять азобензольні групи з електрон-донорними або електрон-акцепторними замісниками, при їх опроміненні за різних довжин хвиль. Експериментальні дослідження було проведено з використанням методів нуль-еліпсометрії та спектроскопії. Показано, що азофрагменти орієнтуються в залежності від довжини хвилі за механізмом фотоорієнтації або фотоселекції. За механізмом фотоорієнтації азокоморфі спонтанно розміщуються в площині, перпендикулярні вектору поляризації світла $E_{ex}$. У випадку, коли переважає механізм фотоселекції, 3-мірний розподіл азокоморфів у стані насичення є ізотропним завдяки суттєвому вичерпанню анеізотропних транс-ізомерів.

Ключові слова: азополімер; поліметакрилат, фотоіндукована орієнтація.