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Original Article

PHOTO-INDUCED ANISOTROPY OF METHYL RED AZO DYE IN POLYMER FILM

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ABSTRACT

A photo-induced anisotropy (PIA) of Methyl Red (MR) azo dye embedded in PMMA polymer solid films was investigated by studying dichroic spectra and photo-induced birefringence. The reversible PIA of MR dye was measured in situ during photoactive irradiation process. Photoinduced order increases with the rise of exposure power. It is shown that PIA dark relaxation slows down at higher exposure energy. The MR rotational diffusion was evaluated at different exposure modes. These data are useful for the characterization of Methyl Red azo dye as a liquid crystal photo-aligning material.

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ФОТОИНДУЦИРОВАННАЯ АНИЗОТРОПИЯ АЗОКРАСИТЕЛЯ МЕТИЛОВОГО КРАСНОГО В ПОЛИМЕРНОЙ ПЛЕНКЕ

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АННОТАЦИЯ

Методом измерения дихроичных спектров и фотоиндуцированного двулучепреломления проведено исследование фотоиндуцированной анизотропии (ФА) метилового красного (МК) красителя, введенного в твердые пленки полимера полиметиметакрилат (ПММА). Обратимую ФА образца МК измеряли во время процесса фотоактивного облучения. Фотоиндуцированный порядок увеличивается с увеличением мощности экспозиции. Показано замедление темновой релаксации ФА при более высоких воздействиях энергии облучения. Выполнена оценка вращательной диффузии МК при различных режимах воздействия. Эти данные важны для характеристики азокрасителя метилового красного в качестве материала для фотоориентации жидких кристаллов и современных жидкокристаллических системах отображения информации.

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Introduction

Photo-induced optical anisotropy (PIA) in organic dyes' films became an important effect for many applications in optical engineering. One of azo dyes (Methyl Red or MR) is a model object in investigations of PIA kinetics as a medium for optical information recording and processing as well as a material for liquid crystals (LC) photoalignment. The PIA in Methyl Red azo dye embedded into a polymer matrix (poly(vinyl alcohol), poly(methyl methacrylate) (PMMA) or poly(vinyl acetate)) [1–3] was used in experiments for optical data processing [4–6] and anisotropic planar waveguides [7, 8]. This azo dye was used for the forced Rayleigh scattering (FRS) study of probe diffusion in polymer solutions [9]. The FRS method is helpful to determine the electric field dependence of thermal and mass diffusion [10–12] and electrohydrodynamics of MR dye molecules in liquid crystals [13]. A MR-doped LC was used as a high-resolution recording holographic medium [14–16]. The reference [17] describes the method of holographic grating formation in MR-doped surface-stabilized liquid crystal film. Such gratings can be formed in typical nematic LC as well as in chiral mesophases, such as blue phases [18].

The MR molecules can be also used for ferroelectric LC photoalignment [19–21]. The photo-induced optical anisotropy of photoalignment layer strongly effects the performance of LC devices [22]. The MR dye can be also used in composite LC materials with different nanoparticles [23, 24]. The material dielectric anisotropy is strongly affected by doping ratio. The anisotropy value decreases with increasing dye concentration.

The paper presents the kinetics of reversible photo-induced birefringence of MR azo dye layers, embedded into PMMA polymer.

Experimental

Sample preparation

The azo-dye 2-[4-(dimethylamino)phenylazo]benzoic acid (Methyl Red, MR) (melting point 179–182 °C) was used in our experiments. The formula of MR azo dye is shown in Fig. 1. The MR free acid form is taken from Dainippon Ink & Chemicals Inc. In our experiment we used the substance with carboxylic group in the *orto*-position.

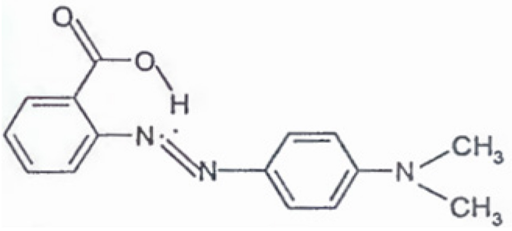


Fig. 1. Methyl Red azo dye

In view of conformations, Methyl red can exist in one of three isomeric forms – *ortho*, *meta*, and *para* [25]. The orientation of LC on the films made of different MR isomers is described in [25]. There was an evidence of weak photoinduced adsorption of *meta*- and *para*-methyl red to the during photoexcitation. The role of the MR photoisomerization in the actual molecules' reorientation process is considered in [26]. The extraordinarily large nonlinear response is obtained that is accompanied by modification of the charge density on the irradiated surface.

A photosensitive film based on 5 wt. % of MR azo dye in PMMA was prepared by dissolving in dichlorethane (DCE) and then spin-coating on ITO-covered glass substrate at 3500 rpm during 40 s. The layer thickness was about 0.5 μm. The thick films of 15±0.5 μm were made by dip-coating from a solution of MR and PMMA dissolved in DCE. All films were cured for 40 min at T = 90 °C. As the illumination source, Ar+ laser (λ_{exp} = 457 nm) was used.

The birefringence photo-induced by Ar+ laser was measured *in situ* within the region, where MR/PMMA films were transparent. He-Ne laser (λ = 632.8 nm) was used *in situ* for recording purpose. As the birefringence of thin film photo-aligning layer is small, we constructed a special registration setup [27, 28]. It is based on photo-elastic modulator, allowing to measure small values of photoinduced birefringence with the accuracy of ±0.001 rad [27]. The photo-induced phase retardation was:

$$\Gamma = 2\pi\lambda d\Delta n, \quad (1)$$

where λ is the wavelength of probe light, d is layer thickness, $\delta = d\Delta n$ is the photo-induced birefringence of the azo dye layer. The experiment was performed at a temperature between 20 °C and 22 °C.

The samples demonstrated a good performance stability. Problems of photodegradation and biodegradation of MR are considered in [29–31].

Results and Discussion

PIA determined from dichroic spectra

A typical example of the optically induced dichroism of the absorption spectra of the thin MR/PMMA film before and after exposure to a linear polarized Ar+ laser ($\lambda_{exp} = 457$ nm) light with various power is shown in Fig. 2. According to [32], the order parameter can be estimated as

$$S = \frac{(D_{\parallel} - D_{\perp})}{D_{\parallel} + 2D_{\perp}}. \qquad (2)$$

The order parameter was -0.007 and -0.032 for the illumination energy $H = 0.24$ J/cm^2 and $H = 3.84$ J/cm^2, respectively.

There is no significant shape difference between the five spectra (Fig. 2). Simultaneous decrease of the absorptions $D_{\perp}$ and $D_{\parallel}$ under the influence of activating radiation testifies to the photochemical mechanism of photoinduced anisotropy. Indeed, it is well known that aromatic azo compounds exhibit «*trans*↔*cis*» photoisomerization [33].

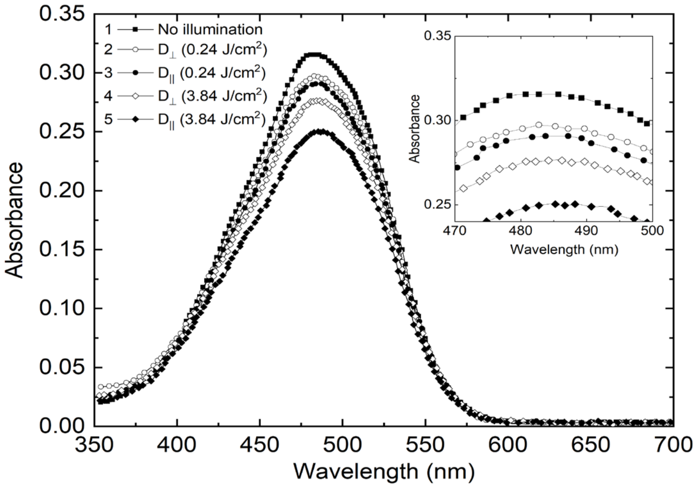


Fig. 2. Dichroic spectra of *o*-MR dye, embedded into PMMA polymer for various exposure energy. The optical density (*D*) without illumination is the curve (1). The curves (2) and (5) are the optical density $D_{\perp}$ and $D_{\parallel}$ for the illumination energy $H = 0.24$ J/cm^2 and $H = 3.84$ J/cm^2, respectively. The symbols ⊥ and ‖ indicate the polarization directions of the probe light perpendicular and parallel to the light polarization of induced illumination

However, the structure of MR azo dye is very similar to azobenzene except for carboxyl group in *ortho* position in one of the benzene rings (Fig. 1). This group is responsible for the shift of strong π–π* transitions into the visible region and formation of intramolecular *H*-bond with the nearest nitrogen atom of the azo group –N=N–. The intramolecular *H*-bond provides the photochemical stability of MR molecule and the quantum yields of «*trans*↔*cis*» photochemical reaction goes to zero.

We revealed a small bleaching of the MR/PMMA film after an hour exposure by the non-filtered radiation of the Hg lamp. This effect occurs practically without variation of the absorption spectrum in the photostationary state. Such deformation is not observed in the case of reversible «*trans*↔*cis*» photoisomerization. Moreover, we observed a very low-intensity band in the long-wave region of the spectrum ($\lambda \approx 560$ nm), which is not typical for the photo-induced «*cis*» isomer of azo dyes [34]. We believe that the effect can take place due to the photo-induced ordering of MR molecules under the action of non-polarized light in such a way that their transition dipole moments become parallel to the direction of light propagation. Similar effect of the photo-induced molecular ordering was found in some photochemically stable azo dyes in polymer matrix [35], amorphous film of azo dyes formed by vacuum evaporation [36] or spin-coating [37] and Langmuir-Blodgett films [38].

PIA determined from photo-induced birefringence

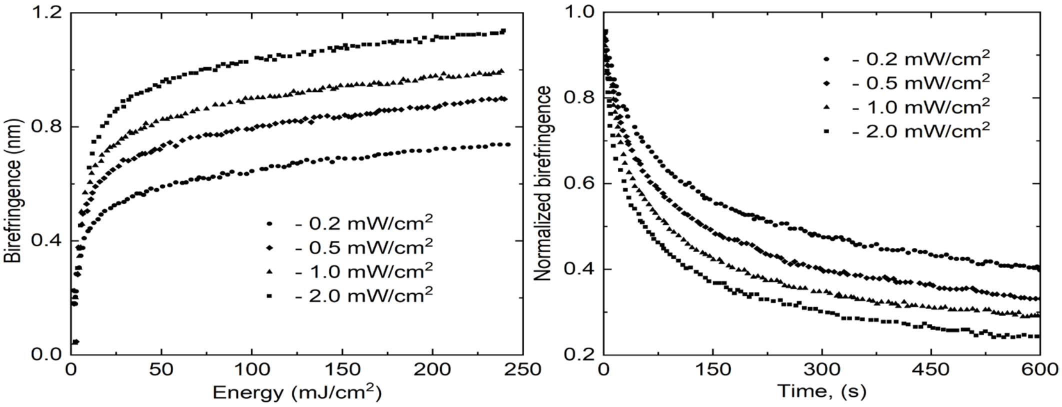
Figure 3, *a* shows the dependence of photo-induced birefringence versus exposure energy, which increases with the exposure time and saturates at some stationary level, getting larger for the higher power of exposure light. This behavior complies with the model of photo-induced anisotropy in rigid molecular structures with rotational Brownian diffusion in the field of polarized light [39].

Dark relaxation of photo induced birefringence

When the activating illumination is switched off, the photo-induced birefringence gradually decreases, and approaches the nonzero steady state level in 10–20 minutes. The value of this level increases for the higher exposure power (Fig. 3, *b*). After that, a certain small optical anisotropy remains for a long time and can be erased by a non-polarized or circularly polarized light of second exposure or by thermal annealing.

The dark relaxation process in *o*-MR/PMMA layer can be explained by two simultaneous processes: dark *cis* isomerization of azo dye molecules [32] and their rotational Brownian diffusion [38]. The kinetics of dark relaxation in Fig. 3, *b* can be described using minimum two exponential functions (Table).

$$\delta = \delta_0 + \delta_1 \exp(-t\tau_1) + \delta_2 \exp(-t\tau_1). \tag{3}$$



a *b*

Fig. 3. The dependence of photo-induced birefringence of MR/PMMA layer on the power density P_{exp}. (*a*). The values of the power density are shown in Fig. 3, *a*. Relaxation the photo-induced phase retardation of MR/PMMA sample after switching off the illumination source with different powers, shown in the Fig. 3, *b*. The exposure energy was 600 mJ/cm²

Table. **Normalized photo-induced birefringence values δ**

P_{exp}, mW/cm²	δ_0	δ_1	τ_1	δ_2	τ_2
2.0	0.238	0.390	17.2	0.332	178.0
1.0	0.287	0.338	17.9	0.364	161.6
0.5	0.320	0.299	21.0	0.374	196.2
0.2	0.375	0.263	30.3	0.345	147

The faster component of the relaxation time τ_1 decreases with the increase of power of illumination P_{exp}. From these results of kinetic characteristics it can be concluded that the photo-induced optical anisotropy in *o*-MR/PMMA layers takes place not only due to the photochromic processes of «*trans*↔*cis*» isomerization in *o*-MR azo dye molecules. The small illumination power promotes the formation of *o*-MR molecular domains of large size, thus hindering the process of dark relaxation. When the power is sufficiently high, the diffusion involves small molecular domains and becomes faster.

This behavior is in agreement with the model of photo-induced anisotropy of rigid molecular structures with rotatory Brownian diffusion in the field of polarized light [38, 39]. Photoinduced birefringence arises is increasing with the increase of power density P_{exp} and under of the coefficient of rotation diffusion of the azo molecule D = const, where

$$D = kT/6\pi\eta a, \quad (4)$$

and k is Boltzmann coefficient, T is absolute temperature, η is macroscopic viscosity of a film, a is the characteristic size of molecule or molecular cluster. The relation (4) states that the coefficient of rotation diffusion is independent on the light illumination power density and energy [38].

From (2), the n_{max} value under λ_{meas} = 632.8 nm for the photosensitive film based on 5 wt. % of *o*-MR / PMMA is 0.02. Really, the birefringence $n(\omega_{meas})$ is related to the dichroism $D = D_{\parallel} - D_{\perp}$, and it can be evaluated using the Kramers-Kronig transformation [39]:

$$n(\omega_{\text{meas}}) = \int \frac{cP}{\pi} \cdot \frac{D(\omega')}{\omega'^2 - \omega_{\text{meas}}^2} d\omega' , \quad (5)$$

where P is the principal value of integral, ω_{meas} is the measurement frequency of light, c is the speed of light, and ω′ represents the whole spectrum.

From the analysis of the values the order parameter S and the birefringence n_{max} under λ_{meas} = 632.8 nm, it is clear that the photoinduced ordering of transparency for activating illumination monomeric fragments of polymeric matrix. Absorption light molecules of oMR is an important contributor to the overall birefringence.

The data in Fig. 3, *b* allow also evaluating the MR rotational diffusion D in PMMA as well its dependence on the illumination intensity P_{exp}.

The orientational relaxation time τ is determined as the time of e^{-1} reduction of photo-induced birefringence, and the rotational diffusion D can be established as τ^{-1}. Evaluation data are presented in Fig. 4. The dependence $D(P_{exp})$ in Fig. 4 can be described by the expression: $D = 1/\tau = D_0 + D_E \cdot pE \cdot E = D_0 + D_E \cdot pE^2$, where D_0 is the rotational diffusion at zero exposure, D_E is a coefficient describing the rotational diffusion acceleration under action of the light wave electric field of strength E, p is the MR molecule's polarizability. The product pE^2 is dimensionless because [p] = $C \cdot m^2 \cdot J^{-1}$, [E] = $V \cdot m^{-1}$.

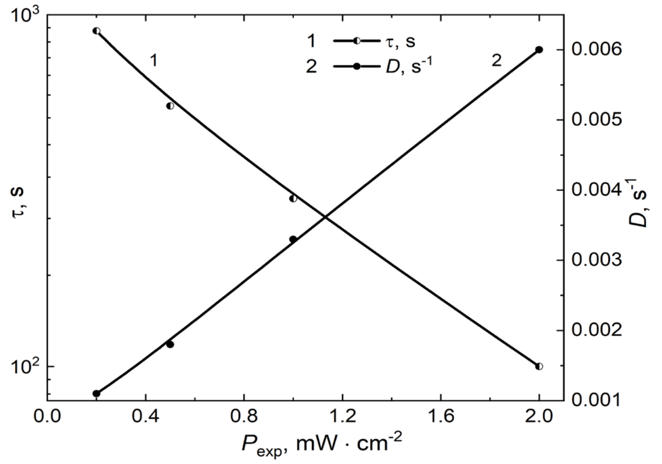


Fig. 4. The orientational relaxation time τ and rotational diffusion D dependences *vs* illumination intensity P_{exp} measured in the MR/PMMA layer

Cyclic forming of PIA

Fig. 5 shows cyclic photoinduction and dark relaxation (birefringence) in thin MR/PMMA film for two values of the power density of activating light illumination. We observe the following: (i) increase of photosensitivity in cyclic process, (ii) slowing down of the rate of dark relaxation and (iii) appearance of a continuously growing background until saturation is eventually reached (Fig. 5). These photokinetic curves correspond to typical dependences obtained for other substances including the MR [25].

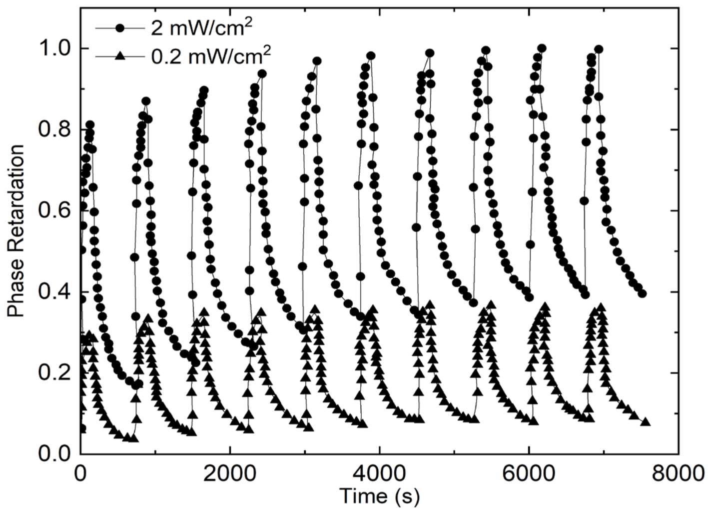


Fig. 5. Optical induced and dark relaxed phase retardation of MR/PMMA film in a cyclic process. A thin layer of the MR/PMMA was illuminated by the activating light having two values of power (0.2 and 2.0 mW/cm^2). The exposure and relaxation time in each cycle was 125 and 625 s accordingly

These experimental data contradict with Todorov et al. [40], where the «recording-self-erasure» cycles in MR/polymer matrix were perfectly reproducible. We have also shown more complicated behavior of dark relaxation in MR/PMMA film (Fig. 6). After partial erasure of the photo-induced birefringence during the secondary illumination with the light of orthogonal polarization, the birefringence level can be decreased or even increased depending on the birefringence initial value. The effect can be explained by the photo-physical process of photo-orientation and the consequent dark self-ordering of MR molecules in polymer matrix [28, 41].

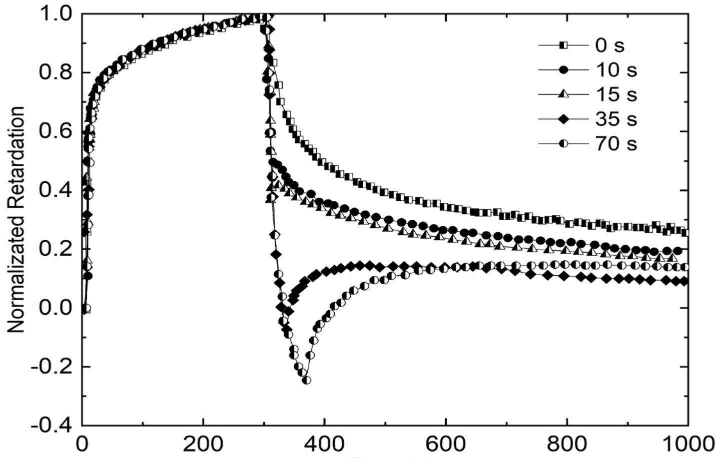


Fig. 6. Optical writing-partially optical erasure and subsequent dark relaxation processes of the photo-induced birefringence in thin MR/PMMA film for different times of optical erasure

Similar effect was observed in [15]. The diffraction efficiency enhancing up to 11.5 % was obtained in Methyl Red doped liquid crystal grating by re-illumination of the sample. From the change of the phase retardation presented in Fig. 6, we can evaluate the MR rotational diffusion in every cycle. It equals to 0.002 s^{-1}. This value is a few orders higher than for liquid crystals (10^8 s^{-1} [42]).

Conclusion

The photo-induced anisotropy (PIA) of Methyl Red embedded into PMMA polymer (MR/PMMA) solid films was investigated by studying the dichroic spectra and photo-induced birefringence. The MR order parameter calculated from the dichroic spectra is small, but it becomes higher for the larger illumination power. The photo-induced birefringence increases with the power density of activated illumination. When the source is switched off, the birefringence value relaxes to some saturated non-zero value. The characteristic relaxation time decreases with the illumination energy. The MR rotational diffusion is 0.001–0.002 s^{-1}. It increases proportionally to the writing light intensity.

The photo-induced optical anisotropy in MR/PMMA solid film can be explained mainly by photo-physical process of active photo-ordering and photoorientation of MR molecules and its surroundings. These data are important for the characterization of Methyl Red azo dye as the material for LC photo-aligning or LC holography elements.

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