PHOTOCHEMISTRY AND MAGNETOCHEMISTRY

Effect of Temperature on the Photoalignment of Azo Dyes in Thin Films

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Abstract—The temperature dependences of the induced dichroic ratios (DRs) of azo dyes after their photoalignment in thin films 80 to 200 nm thick are studied. It is found that the DR values of layers containing dyes of the benzeneazodiphenyl series fall from 6.0 to 1.6 as the temperature rises from 60 to 130°C, respectively. A reduction in induced DR as the temperature rises (from 20 to 100°C) is also observed for the thin films of the dyes of benzeneazo-5,5'-dioxodibenzothiophene group. The absence of induced DR after irradiation with polarized light at 100°C indicates there is no alignment of molecules at this temperature.

Keywords: photoalignment, trans-cis isomerization, azo dye, polarized absorption spectra.

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INTRODUCTION

Various materials such as photo-cross linkable polymers [1, 2], azo polymers [3–5], and azo dyes [6, 7] are used for the photoalignment of liquid crystals (LCs). Azo dyes in particular are thermally and photochemically stable [8] and characterized by high photoinduced anchoring energy of LC [9, 10]. There are a number of azo dyes capable of photoalignment [6, 7, 9]; however, not only the effect their molecular structure has influence on the photoalignment properties but also the conditions for the formation of thin layers and photo exposure must be considered in order to develop new azo dyes of this type.

It was shown in [11] that the rod coating method allows us to produce thin film materials of higher quality than those produced using the spin-coating technique. The use of rod coating results in the formation of more homogeneous amorphous films due to the dye material being applied on the substrate at elevated temperatures, thus increasing the values of induced dichroism of these films at the same dose of exposure. Temperature is another factor that can affect the induced dichroism of thin films of dyes after their irradiation with polarized light. It is known [12, 13] that the disordering of azo dye molecules is observed in the polymer matrix at elevated temperatures because of the relaxation processes in polymers, or when the glass transition temperature of azo polymers is reached. On the other hand, raising the temperature of samples slows the rate of *trans* \rightarrow *cis* \rightarrow trans isomerization [12].

The aim of this work was to examine the effect of the temperature on the rate of photoalignment of the synthesized molecules of azo dyes in thin films, and to determine the temperature that ensures the maximum values of the induced dichroic ratio (DR) at a given dose of exposure.

EXPERIMENTAL

General

¹H and ¹³C NMR spectra were obtained in DMSOd₆ solutions using a Bruker Avance 500 instrument (Germany). IR spectra were recorded with KBr tablets on a Bruker Tensor 27 FT-IR spectrometer (Germany), and an Ocean Optics Maya2000Pro UV–Vis spectrometer (United States) was used to record the absorption spectra.

Azo Dyes of Benzeneazodiphenyl Series

Azo dyes of benzeneazodiphenyl series FbF-2 and GbG-2 (Fig. 1) were synthesized and purified according to the procedure described in [14].

FbF-2. Potassium 4,4'-bis(4-hydroxy-3-carboxylate-6-methylphenylazo)diphenyl. ¹H NMR (500 MHz, DMSO- d_6 , δ): 2.63, s (6H); 6.64, s (2H); 7.94, dd (J = 8.5 Hz, 8H); 8.17, s (2H); 17.80, br. s (OH). ¹³C NMR (125 MHz, DMSO- d_6 , δ): 17.45, 116.77, 117.9, 122.59, 127.50, 140.14, 140.82, 143.41, 152.16, 162.25, 168.51, 170.97. IR (v, cm⁻¹): 3424, 2923, 1637, 1586, 1492, 1460, 1427, 1378, 1265, 1173, 1064, 824.



Fig. 1. Scheme for the synthesis of azo dyes (for denotations, see the text).

GbG-2. Potassium 4,4'-bis(4-hydroxy-3-carboxylate-5-methylphenylazo)diphenyl. ¹H NMR (500 MHz, DMSO- d_6 , δ): 2.19, s (6H); 7.75, s (2H); 7.93, dd (J = 8.5 Hz, 8H); 8.19, s (2H); 17.9, br. s (OH). ¹³C NMR (125 MHz, DMSO- d_6 , δ): 15.85, 118.22, 122.43, 125.25, 125.40, 126.2, 127.44, 140.08, 141.56, 151.71, 168.1, 170.97. IR (v, cm⁻¹): 3428, 2923, 1637, 1466, 1427, 1398, 1302, 1108, 1004, 900, 824, 776.

Benzidine Sulfone (3,7-Diamino-5,5'-dioxodibenzothiophene) (Fig. 1) [15]

Eighty milliliters of 20% oleum were placed into a three-necked flask equipped with a mechanical stirrer, thermometer, and air condenser with a calcium chloride drying tube. Twenty grams of benzidine 1 were added in small portions under stirring so that the reaction mixture would not be heated above 60° C. This was followed by heating the mixture in an oil bath at 120°C for 3.5 h. The flask contents were cooled to room temperature under stirring and poured in small portions into 250 g of ice. The resulting precipitate was filtered off, washed with water, transferred to a beaker,

and a solution of 30% NaOH was added in small amounts with cooling and stirring to an alkaline pH of 11. The resulting benzidine sulfone **2** was filtered off, washed with water and 40 mL of ethanol, and dried at room temperature for one day. The product was reprecipitated from the DMF–water mixture and dried in vacuum. The yield of benzidine sulfone **2** was 68.3%. ¹H NMR (500 MHz, DMSO- d_6 , δ): 5.72, s (4H); 6.8, d (J = 8 Hz, 2H); 6.89, s (2H); 7.49, d (J = 8 Hz, 2H). ¹³C NMR (125 MHz, DMSO- d_6 , δ): 105.37, 118.47, 119.41, 121.44, 137.4, 149.23. IR (v, cm⁻¹): 3470, 3371, 1609, 1481, 1351, 1315, 1261, 1308, 1153, 1131, 1031, 824.

Azo Dyes of Benzeneazo-5,5'-dioxodibenzothiophene Series (Fig. 1)

We placed 2.46 g of benzidine sulfone **2**, 4.8 mL (0.06 mol) of concentrated hydrochloric acid and 30 mL of distilled water into a beaker equipped with a mechanical stirrer, thermometer, and dropping funnel. The mixture was heated under stirring at 90°C for



Fig. 2. Experimental setup for irradiating samples at elevated temperatures, where I is a thin film of dye on glass, 2 is the heater, 3 is the velvet, 4 is the temperature controller, 5 is the blue LED, and 6 is the grid polarizer.

15 min. The resulting suspension was cooled to 0° C and a solution of 1.87 g of potassium nitrite in 5 mL of water was added dropwise, followed by stirring the mixture for 1.5 h.

Solution **3** of diazo salt was added dropwise to a solution of 0.022 mol of 4-methylsalicylic acid and 9.66 g (0.07 mol) of potassium carbonate in 50 mL of water at a temperature of 5° C for 45 min, and the mixture was stirred for 5 h and kept overnight. The dye was then isolated and purified according to the known procedure [14].

FtF-2. Potassium 3,7-bis(4-hydroxy-3-carboxylate-6-methylphenylazo)-5,5'-dioxodibenzothiophene (Fig. 1). Yield, 54.0%. ¹H NMR (500 MHz, DMSO d_6 , δ): 2.65, s (6H); 6.65, s (2H); 8.24, m (6H); 8.41, d (J = 8 Hz, 2H). ¹³C NMR (125 MHz, DMSO- d_6 , δ): 17.63, 112.94, 117.37, 117.67, 118.58, 124.11, 129.98, 130.43, 138.89, 140.59, 144.86, 154.17, 170.53, 170.76. IR (v, cm⁻¹): 3424, 2923, 1634, 1582, 1492, 1456, 1373, 1298, 1194, 1062, 830, 741.

FtF-1. Sodium 3,7-bis(4-hydroxy-3-carboxylate-6-methylphenylazo)-5,5'-dioxodibenzothiophene. The compound was synthesized and isolated via the same procedures, but sodium salts of the reagents were used. Yield, 50%. ¹H NMR (500 MHz, DMSO-*d*₆, δ): 2.65, s (6H); 6.65, s (2H); 8.24, m (6H); 8.41, d (*J* = 8.5 Hz, 2H); 17.98, br. s (OH). ¹³C NMR (125 MHz, DMSO*d*₆, δ): 17.63, 113.06, 117.39, 117.68, 118.55, 124.14, 129.96, 130.47, 138.9, 140.70, 144.86, 154.15, 170.24, 170.91. IR (v, cm⁻¹): 3424, 2922, 1634, 1582, 1532, 1446, 1370, 1298, 1254, 1053, 836, 795, 731.

Preparing Surfaces and Samples

Glass substrates (alkaline float glass 2 mm thick and 50 × 25 mm in size) were successively purified in an ultrasonic bath with distilled water and isopropanol, dried at 110°C for 5 min and additionally UVpurified with a Photo Surface PL 16-110D processor (SEN Lights Corp., Japan). Thin films of azo dyes were applied from their 2% DMF solutions onto the glass substrates via the rod coating technique at a linear translation rate of heating plate 1 cm s⁻¹ and a temperature of 55°C. The resulting samples were dried at 100°C for 5 min.

Studying Thin Films of Azo Dyes

The setup shown in Fig. 2 was used for the simultaneous heating and irradiation of thin films of dyes.

Sample *1* was placed onto heater *2* covered with black matte velvet *3* to prevent reflection and the scattering of light from the surface of heater *2*. The temperature of heater *2* was then set, and the substrate with applied azo dye was thermostated for 10 min. After measuring the temperature of sample *1* with a pyrometer; the former was irradiated with blue light ($\lambda_{max} = 457$ nm) generated by LED *5* through polarizer *6* over a certain time interval. The sample was then cooled and the polarized absorption spectra (S- and P-polarization) of the sample were recorded.

The obtained polarized absorption spectra were used to determine the DR of each sample according to the formula

$$DR = \frac{A_{\perp}}{A_{\parallel}},\tag{1}$$

where A_{\parallel} and A_{\perp} are the absorption of the linearly polarized light of the probe radiation in the directions parallel and perpendicular to that of the polarization of the irradiating light, respectively.

RESULTS AND DISCUSSION

The dependences of induced DRs on the substrate temperature show a reduction of DRs in thin films of GbG-2, FbF-2, FtF-1, and FtF-2 azo dyes at temperatures above 60°C (Figs. 3a, 3b). For FbF-2 and GbG-2 dyes, raising the temperature from 20 to 60°C causes the induced DRs to grow from 3.5 to 4.5 and 4.6 to 5.8, respectively, while a reduction in induced DR is observed at higher temperatures (down to 1.6 at 130°C) (Fig. 3a). With thin films of FtF-1 and FtF-2 azo dyes irradiated at a constant exposure, the value of induced DR falls as the temperature of the sample rises. Thus, while DR = 6 is induced at room temperature (20°C), the DR is equal to 1.2 at a temperature of 103°C (and the same dose of radiation); i.e., there is a



Fig. 3. Temperature dependences of the induced DRs of our (a) GbG-2 and FbF-2 and (b) FtF-1 and FtF-2 samples.

drop to almost DR = 1 when there is no photoalignment (Fig. 3b). To explain the obtained results, we must determine what influences the magnitude of the induced DR.

The diffusion model and the reversible intermolecular bonding model consider the realignment of molecules via their spatial rotation; however, the effect of possible *cis*-*trans* isomerization of azo dyes is not taken into account [16, 17], and the *trans*-isomer, energetically advantageous under normal conditions, serves as a molecular rotor [18, 19].

On the other hand, the excitation of dye induced by photon absorption results in the reversible *trans* \rightarrow *cis* \rightarrow *trans* isomerization [20]. It was reported in [21] that no *cis*-*trans* transitions were observed in the thin film state after irradiation with polarized light, due to the high rate of isomerization.

The observed dependences of induced DR on temperature (Figs. 3a, 3b) indicate effects (a drop in the DR as the temperature of the sample rises) that cannot be described by the diffusion model. From the viewpoint of the diffusion model, an increase in temperature raises the average kinetic energy of the molecules. This should result in an accelerated rate of molecule realignment, due to an increase in the rate of rotational diffusion [22].

From the viewpoint of the reversible intermolecular bonding model, molecules are realigned due to the dissociation of intermolecular coordination bonds, which requires an energy of approximately 40-60 kJ mol⁻¹. A change in the temperature of the sample within the studied range cannot lead to the breaking of intermolecular bonds, since the energy of thermal motion $kT = 2.5 \text{ kJ mol}^{-1}$ at 293 K is less than the energy of the bonds, and a rise in temperature to 600 K leading to $kT = 5 \text{ kJ mol}^{-1}$ is also not enough to disorder the molecules [17]. Meanwhile, the breaking of bonds and a change in the alignment of azo dye molecules can occur only after the absorption of a photon with $\lambda = 450$ nm and an energy of approximately 266 kJ mol⁻¹, which is enough to break the intermolecular coordination bonds. For all molecules within the mean field, the self-consistency condition is satisfied after the breaking of coordination bonds; i.e., the S order parameter of a single molecule, determined from the average deviation from the equilibrium direction, is equal to the S order parameter found by averaging over all the molecules [23]. The mean-field potential depends on the order parameter because the equilibrium ordering of molecules changes depending on the temperature, since an increase in the latter reduces the equilibrium S order parameter. The disordering of dye molecules, and hence the values of photoinduced dichroism, should thus be observed in any model, including the reversible intermolecular bonding model, after simultaneously irradiating and heating the azo dye thin films if the mean-field potential is used to describe the realignment of molecules.

The disordering of azobenzene moieties is observed in thin films of azo polymers at elevated temperatures [24-26]. This effect leads to the disappearance of anisotropic properties, due apparently to the low glass transition temperature [24], relaxation processes in the polymer layer [25], and the influence of the mean field [26]. The same phenomena could occur in thin films of azo dyes, thus leading to the absence of photoalignment at elevated temperatures (>100°C).

To test this hypothesis, a thin film of FtF-2 dye on glass was irradiated with polarized light at room temperature for 6 min, and the polarized absorption spectra were recorded. The sample was then rotated by 90° relative to the irradiating light, thermostated at 103°C, and again irradiated with polarized light for 6 min. The polarized absorption spectra were recorded after cooling and rotating the sample to its initial position, followed by calculating the induced DR.

DR = 4.2 was induced after irradiating the thin film of FtF-2 at room temperature. After rotating the substrate by 90° and exposing the sample at 103°C, the



Fig. 4. Absorption spectra of FtF-2 recorded at 103°C before and after irradiation with (a) polarized and (b) unpolarized light or (c) at room temperature before and after irradiation with polarized light, where A_{\parallel} is the absorbance before irradiation at coinciding directions of the probe and exposure radiations, A_{\perp} is the absorbance before irradiation with the perpendicular directions of the radiation, and $A_{\parallel ex}$ and $A_{\perp ex}$ are the abovementioned absorbance obtained after irradiation.

DR value fell to 3.5 (Fig. 4a). This likely indicates a slight photoalignment of azo dye molecules in the thin layer (approximately 150 nm).

On the other hand, raising the temperature results in an increase in molecular diffusion and possibly to an accelerated rate of realignment after irradiation with polarized light, or to disordering after exposure to unpolarized light. The FtF-2 sample was therefore irradiated with unpolarized light at 103°C for 6 min, and the polarized absorption spectra were recorded (Fig. 4b).

The DR value fell from 3.5 to 3.36 after exposing the thin film of FtF-2 dye. This suggests a slight disordering of the azo dye molecules in the layer or their partial vertical alignment after exposure.

The same sample was then rotated by 90° relative to the direction of irradiation and exposed to polarized light at room temperature for 12 min; i.e., the already aligned molecules were realigned, followed by rotating the sample to its initial position and recording the absorption spectra. The increase in the absorption of the polarized light of the probe radiation when it coincided with the polarization of the irradiating light and its reduction when the direction was perpendicular indicate the photoalignment of molecules (Fig. 4c).

The photochemical and thermal stability of thin layers of FtF-2 azo dye was confirmed by the photoalignment of azo dye molecules in the thin film after its repeated irradiation at room temperature, with the sample being previously exposed to polarized and unpolarized light at 103° C (Figs. 4a–4c).

The processes that occur in thin films of dyes likely explain the observed dependences. The molecules can undergo reversible *trans* \rightarrow *cis* isomerization under their exposure [27], with the reverse process (dark relaxation) of a *cis*-isomer transition to the *trans*-form also being possible [28]. However, a rise in the sample temperature is known [29] to result in a drop in the fraction of *cis*-isomer [30] due to a reduction of the isomerization quantum yield [28]. At high temperatures, there are almost no *trans* \rightarrow *cis* transitions, and the *trans*-isomers are stabilized [31].

A reduction of induced DR during the irradiation of thin films of dyes is observed at temperatures above 70°C. This could be due to the stabilization of the *trans*-isomers. On the other hand, the maximum of the induced DR of layers of azo dyes of such benzeneazodiphenyl series as FbF-2 and GbG-2, observed in the temperature dependences of induced DR at approximately 60°C (Fig. 2a), could indicate an accelerated rate of realignment of the azo dye molecules as the temperature rises.

The differences between the temperature dependences of the induced DRs of the pairs of FtF-1 and FtF-2 or FbF-2 and GbG-2 dyes (Figs. 2a, 2b) seem to be due to the structures of the dyes. The central cores of azo dyes are thus different, and it is their structure that determines the free volume of film and the ability of molecules to realign [32]. The presence of electron withdrawing sulfone groups in the structures of the FtF-1 and FtF-2 dyes (Fig. 1) could also affect the values of the induced DRs. The free volume of a thin film is known [33] to significantly influence the *cis-trans* isomerization of molecules, and the larger the free volume, the higher the quantum yield of the process. Substituents affect the free volume, in



Fig. 5. Polarized absorption spectra of (a) FtF-2 and (b) FbF-2, before and after irradiation with UV light.

turn influencing both the lifetimes of *cis*-isomers and the rate of isomerization [34].

Azo dyes are characterized by different rates of $cis \rightarrow trans$ isomerization, depending on their structure [35]. A combination of electron-donor and electron-acceptor substituents in the structures of dye molecules accelerates this process considerably [36]. Indeed, there are electron-acceptor sulfone groups in the structures of FtF-1 and FtF-2, and electrondonor phenolic hydroxyl groups in the para-position to azo group of the side moieties of these molecules, which can accelerate photoisomerization. With FbF-2 and GbG-2 azo dyes, there are no electron-acceptor groups, and the rates of $cis \rightarrow trans$ isomerization should be lower. The probability of the photon absorption is also known [37, 38] to depend on the wavelength of the incident radiation and the position of the absorption maximum of a dye; the closer the maximum to the wavelength of the irradiating light, the higher the probability of absorbing, and hence the photosensitivity of material. With FbF-2 and GbG-2, $\lambda_{max} \approx 390$ nm; for FtF-1 and FtF-2, $\lambda_{max} \approx 420$ nm. As the absorption maximum of the second pair of dyes is closer to the wavelength of the irradiating light ($\lambda =$ 457 nm), the second pair is characterized by higher probabilities of photon absorption, and therefore higher rates of the photoalignment of molecules of azo dyes than in the first pair of dyes.

At the same time, the rate of the *cis*-isomer transition to the *trans*- form grows (dark relaxation is accelerated) in the films of FbF-2 and GbG-2 azo dyes (which are characterized by a relatively slow *cis* \rightarrow *trans* transition) as the temperature rises to 60°C [39], thus enabling us to perform a greater number of cycles of reversible photoisomerization in the same time; i.e., the DR value increases. A further rise in temperature leads to the stabilization of the dye's *trans*-isomer and lowers the probability of its transitioning to the *cis*form [40], resulting in a reduction of the induced DR. The same effect is also observed for the FtF-1 and FtF-2 dyes.

From the viewpoint of the reversible intermolecular bonding model [17], the photodissociation (breaking) of the intermolecular coordination bonds that ensure the long-term stability of the photoinduced alignment of molecules in thin films is determined by the possibility of *trans-cis* isomerization.

Azo dyes are commonly classified into three groups, depending on their photochemical behavior according to Rau [41]. The first group is azobenzenes, which are characterized by low energy $\pi - \pi^*$ and $n - \pi^*$ transitions and a relatively long lifetime of their *cis*-isomers, in some cases allowing the isolation of the pure *cis*-form of azo dyes. The $\pi - \pi^*$ and $n - \pi$ levels of the second aminoazobenzene group are close or considerably overlap, and the lifetimes of their *cis*-isomers are shorter. The third group, e.g., pseudo-stilbenes containing electron-donor and electron-acceptor groups in their structures and characterized by long-wavelength $\pi - \pi^*$ transitions and a sequence of (n, π^*) and (π, π^*) states inverted on the energy scale exhibits the shortest lifetimes of *cis*-isomers [42].

Irradiating aminoazobenzenes and pseudo-stilbenes with the light inducing *trans* \rightarrow *cis* isomerization leads to the *cis* \rightarrow *trans* transition, thus accelerating photoisomerization. However, this process proceeds more efficiently in the second group. The *trans* \rightarrow *cis* transitions of azo dyes of aminoazobenzene and pseudo-stilbene groups [42] can occur after irradiating with both UV light ($\lambda_{max} = 365$ nm) and light with longer wavelengths ($\lambda_{max} = 450$ nm), with the quantum yields of the reaction depending on the structure of the dye.

Thin films of FtF-2 and FbF-2 azo dyes were irradiated with a UV LED (367 nm; power, 100 mV cm⁻²) for 5 min to confirm the possibility of their photoalignment under UV light. Induced anisotropy was observed in recording the polarized absorption spectra after exposing the samples. This was confirmed by an increase in the maximum of the absorption band at a perpendicular orientation of the probe radiation relative to the irradiating light and its reduction when the directions of the radiation coincided (Figs. 5a, 5b).

The realignment of molecules, apparent in the increased absorption of the polarized probe radiation when its direction perpendicular to that of the polarization of the irradiating light and the reduced absorption for the coincident direction, was observed after irradiation with UV light (Figs. 5a, 5b). The presence of realigned FtF-2 and FbF-2 molecules in the thin film after irradiation with polarized UV (Figs. 5a, 5b) and visible (Figs. 3a, 3b) light suggests that these dyes can be classified as belonging to the aminoazobenzene and pseudo-stilbene groups, respectively.

The temperature dependences of the induced DRs of different series of azo dyes varied (Figs. 3a, 3b). For example, an increase in the induced DRs as the temperature rose to 60°C was typical of the thin films of FbF-2 and GbG-2 dyes. This could have been due to an accelerated rate of the transition of the *cis*-isomer to the *trans*-form [43]. A reduction of the photoin-duced DRs in the thin films of FtF-1 and FtF-2 azo dyes and those of FbF-2 and GbG-2 (at temperatures above 60°C with the latter pair) could have been due to the stabilization of the *trans*-forms of the dyes.

There were thus two processes determining the rate of realignment of molecules of the studied azo dyes in films after their irradiation: the $cis \rightarrow trans$ isomerization of azo dyes upon irradiation with light,

$$trans \xrightarrow{hV} cis$$

and the dark relaxation of *cis*-isomers to *trans*-forms under heating [44, 45],

$$cis \xrightarrow{T} trans.$$

CONCLUSIONS

It was shown that heating samples of thin films of azo dyes above 100°C during their exposure at a constant dose reduces the induced DRs (the DR values of FbF-2 and GbG-2 were 1.6, while those of FtF-1 and FtF-2 were 1.2), due to the stabilization of *trans*-isomers at a temperature of 103°C. With dyes FbF-2 and GbG-2, the induced DR grew from 3.5 to 4.5 and from 4.6 to 5.8, respectively, as the temperature rose from 20 to 60°C, due to the accelerated rate of the reversible *cis*-*trans* isomerization. Raising the sample temperature from 20 to 103°C reduced the values of the induced DRs of thin films of FtF-1 and FtF-2 azo dyes from 5.8 to 1.2 and from 5.3 to 1.2, respectively. The rate of the reversible *trans*-*cis*-*trans* isomerization and the changes in this rate depending on temperature determined the values of the induced DRs of thin films after their exposure to polarized light.

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