

Kinetics of Photoinduced Birefringence in the Guest–Host System of Poly(methyl methacrylate) Doped with Azobenzene-Containing Crown Ethers

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ABSTRACT: The kinetics of the buildup and decay of photoinduced birefringence was examined in a series of host–guest systems: azobenzene-containing crown ethers, differing in the size of the crowns, dissolved in a poly(methyl methacrylate) matrix. In all samples, the kinetics of the buildup of the birefringence was reasonably described by a sum of two exponential functions, the time constants being inversely proportional to the intensity of the pumping light and the magnitudes of the signals at the saturation level depending on the pumping light intensity and sample thickness. The dark decays were best described by the stretched exponential function,

with the characteristic parameters (time constant and stretch coefficient) being practically independent of the type of crown ether. The time constants of the signal decay were orders of magnitude shorter than the respective constants of the dark isomerization of the azo crown ethers, thus indicating that the process controlling the decay was a relaxation of the polymer matrix and/or a rearrangement of the flexible parts of the crowns. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 105: 130–136, 2007

Key words: host–guest systems; optics; photochemistry

INTRODUCTION

Since its discovery, photoinduced birefringence (PIB) has raised much interest, and many papers have been published exploiting this phenomenon and attempting to elucidate its origin. The first reports on the appearance of anisotropy of optical properties of organic systems due to the orientation of molecules induced by light appeared nearly half a century ago.^{1,2} Quite early, a relation was established between the photoisomerization of these molecules and their resulting orientation (e.g., refs. 3 and 4). The appearance of the effect in azobenzene dye-doped polymers and its possible use in information recording were

demonstrated by Todorov and coworkers.^{5,6} Later, the effect was also used in the formation of surface relief gratings,^{7–10} nonlinear optic applications,^{11–13} the photoinduced alignment of liquid crystals,^{14–18} and rewritable memory application,¹⁹ (see also a recent review²⁰ and references therein).

It has been commonly accepted that the phenomenon is linked to the orientation of photochromic molecules due to multiple photoisomerizations under the action of linearly polarized light. The resulting birefringence (Δn) in the sample is probed with a light beam using crossed polarizers, its value being related to the magnitude of the measured signal (I) via the following equation:²¹

$$\frac{I}{I_0} = \sin^2\left(\frac{\pi\Delta nL}{\lambda}\right) \quad (1)$$

where I_0 is the intensity of the incident light (reading beam), λ stands for its wavelength (in the experiments reported in this article, $\lambda = 632.8$ nm), and L is the sample thickness. In the small signal regime, that is, for $\Delta n \ll \frac{\lambda}{\pi L}$, the above equation simplifies to

$$\frac{I}{I_0} \cong \left(\frac{\pi\Delta nL}{\lambda}\right)^2 = \text{const}^* (\Delta n)^2 \quad (2)$$

A kinetic model of the photoinduced orientation of isomerizable molecules was developed by Sekkat

This article is dedicated to the memory of Professor Marian Kryszewski.

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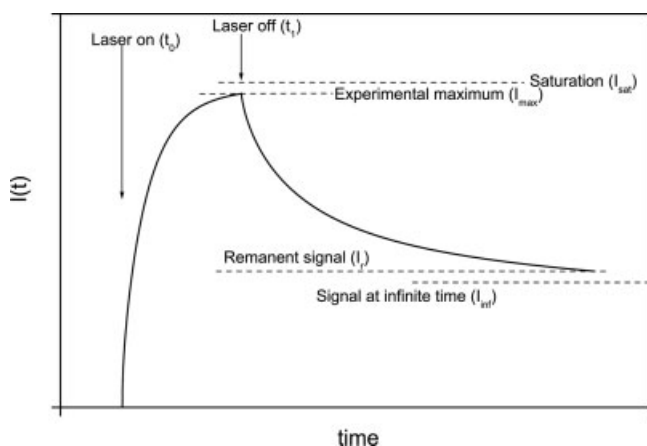


Figure 1 Schematic presentation of the kinetics of the buildup and decay of the PIB signal. The pumping light is switched on at t_0 and switched off at t_1 .

(e.g., refs. 22–25). The model predicts, at low intensities of the pumping light, a biexponential time dependence of the buildup of the signal and a nonlinear increase in its saturation amplitude with the intensity of the pumping radiation.

A typical PIB experiment consists of measuring the kinetics of the buildup of the signal upon the exposure of a sample to photoisomerization-triggering radiation and then the decay after the pumping light has been switched off. A schematic picture of the buildup and decay of the signal is shown in Figure 1.

According to eq. (2), changes in Δn can be extracted from $\sqrt{I(t)}$ dependences. For pure convenience, in what follows, the following notation will be used: $\sqrt{I(t)} \equiv Y(t)$. The buildup and decay of the experimental curves can be described by the following equations:

$$Y_{\text{pump}}(t) = Y_{\text{sat}}[1 - \varphi_{\text{pump}}(t)] \quad (3)$$

$$Y_{\text{dec}}(t) = [Y_{\text{max}} - Y_{\infty}]\varphi_{\text{dec}}(t) + Y_{\infty} \quad (4)$$

where Y_{sat} , Y_{max} , and Y_{∞} are related to the magnitudes of the signal of saturation, the signal achieved at the end of the pumping period, and the remanent signal, respectively, and φ_{pump} and φ_{dec} are time-dependent functions. Experimental curves have usually been interpreted, invoking either a biexponential function

$$\varphi = a \exp\left(-\frac{t-t_i}{\tau_1}\right) + b \exp\left(-\frac{t-t_i}{\tau_2}\right), \quad a + b = 1 \quad (5)$$

or a so-called stretched-exponential (Kohlrausch–Williams–Watts) function

$$\varphi = \exp\left(-\frac{(t-t_i)^\beta}{\tau_3}\right) \quad (6)$$

where τ_1 , τ_2 , and τ_3 are the respective time constants; $\beta < 1$ is a time-independent coefficient; and t_i is equal to t_0 for the buildup curves and to t_1 for the decays, t_0

and t_1 determining the moments of switching on and off the pumping laser, respectively (see Fig. 1). In the case of biexponential functions, τ has been sometimes associated with processes occurring in dye molecules and in matrices (usually polymers),²² whereas the choice of the stretched exponential functions suggests rather a distribution of time constants of microscopic processes controlling the macroscopic kinetic process.

Most experiments published to date have been performed on azobenzene-containing materials,^{26–32} less attention being paid to other photochromic systems such as spiropyrans and spiroxazines,^{23,33,34} diarylethenes,²⁴ and bacteriorhodopsin.^{35–38} The most frequently studied azobenzene derivatives have been disperse-type dyes such as the 4-*N*-(2-hydroxyethyl)-*N*-ethyl-amino-4'-nitroazobenzene molecule, which is usually called Disperse Red 1, either dissolved in a polymer matrix (guest–host system) or chemically attached to a polymer chain. Because of the presence of strongly polar groups attached to the azobenzene moiety, the molecule exhibits a fast thermal *cis*–*trans* isomerization. Experiments have been performed, and positive results have been reported also in the case of azobenzenes substituted with nonpolar side groups, for which thermal isomerization is much slower. The latter feature allows one to disconnect (at least in part) effects associated with the relaxation of the matrix from those due to the isomerization of photochromic molecules.

In this article, we present first results of studies of PIB performed on poly(methyl methacrylate) (PMMA) samples containing dissolved azobenzene-containing crown ethers (cf. Fig. 2), which hereafter are called azo crown ethers for the sake of simplicity. The photochromic properties and, in particular, the kinetics of the isomerization of the same family of crown ethers in the same polymer matrices have been recently studied,^{39–41} and the kinetic parameters have been reliably established, allowing us to compare them with kinetic parameters of PIB. Moreover, the rate constants of the thermally driven *cis*–*trans* isomerization differ by more than 1 order of magnitude between the members of the family,⁴¹ thus making it

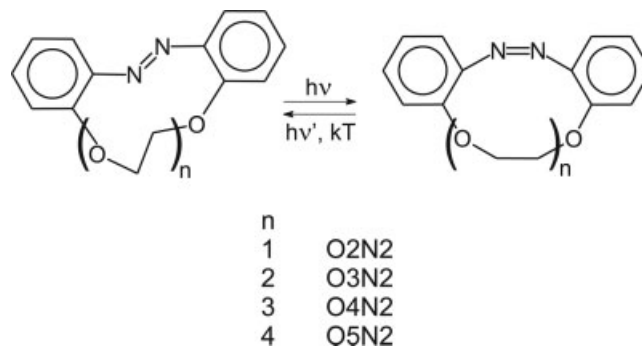


Figure 2 Scheme of the photochromic reaction and structures of the crown ethers under study.

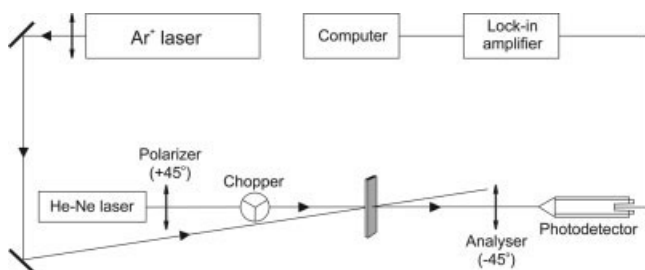


Figure 3 Experimental setup used in this study.

possible to see a difference in the birefringence measurements if any of the processes controlling the kinetics depend on the thermal isomerization kinetics. Finally, the shapes of the molecules and flexibility of the crown ether moieties allowed us to expect that the molecules themselves would create enough free volume for the isomerization to be influenced by the matrix to a minor degree only.

EXPERIMENTAL

Four azo crown ethers, differing in the size of the crown, were studied, their sizes ranging from 10 to 19 members (hereafter called O2N2 through O5N2; cf. Fig. 2). The materials were synthesized and purified as described in refs. 42–47.

The concentrations of the dyes in the PMMA matrices were of the order of 1 wt % (10^{-2} mol/dm³). The polymer films were prepared through the casting of chloroform solutions containing the polymer and the azo crown ether on a horizontally positioned glass plate. After drying (for at least 24 h at room temperature in an atmosphere rich in solvent vapors, allowing for a very slow and nearly uniform drying of the sample bulk), the films were removed from the glass with an ultrasonic bath and left in the ambient atmosphere for several weeks to remove the remaining solvent. Our earlier experiments (e.g., ref. 48) showed no differences between the samples fabricated in this way and those additionally dried at 95°C for several hours. The thicknesses of the films fabricated in such a way ranged from about 50 to 180 μ m.

The absorption spectra of the crown ethers are similar to those of the parent azobenzene and, in the spectral region of interest, contain a relatively weak and broad band peaking around 440 nm (e.g., see Fig. 1 in ref. 41). The absorbances of the samples used in this study did not exceed about 0.2 at the wavelength of the pumping laser (465 nm); those at the wavelength of the reading laser (632.8 nm) were of the order of 0.01.

The measurements of PIB in PMMA samples containing azo crown ethers were performed with the traditional setup described elsewhere⁹ and schematically depicted in Figure 3. The excitation laser inducing the orientation of azo crown ethers molecules (pumping beam) was an Ar⁺-ion laser from Coherent Innova

70C (Santa Clara, CA, maximum power of 50 mW for the wavelength of 465 nm). The light beam came out from the Ar⁺ laser linearly polarized. A light beam from a low-power He–Ne laser (Coherent, Santa Clara, CA, 632.8 nm, 10 mW; reading beam) passing through crossed polarizers and modulated at 270 Hz with a chopper was used to probe the birefringence in the samples. The experimental setup was mounted in such way that the two laser beams crossed the sample almost in a parallel direction. The pumping beam on the sample had a polarization angle of 45° with respect to the polarizer orientation that was placed in the reading beam, whereas the polarizer and the analyzer placed in the reading beam were crossed.

The response signal associated with the birefringence was measured with a home-made photodetector and a lock-in amplifier (model SR350, Stanford Research Systems, Sunnyvale, CA). The signal amplitude from the analog output of the lock-in was recorded on a computer for further analysis. In our measurements, the beam light of the Ar⁺ laser was defocused to improve the beam uniformity and also to decrease the light intensity that impinged the sample. The intensity of the pumping beam in the sample was varied between 5 and 15 mW/cm².

The samples were fixed in a sample holder that allowed controlling the temperature, and during the measurements, the temperature was fixed at 30°C, about 5°C above room temperature. All measurements were performed in the ambient atmosphere.

RESULTS

The analysis of the experimental curves was aimed at determining the parameters governing the kinetics of

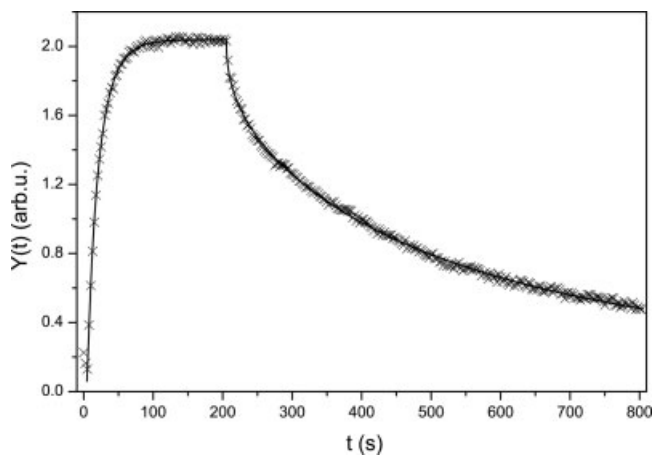


Figure 4 Typical experimental curve for sample O2N2 (0.98 wt %) in PMMA (thickness = 78 μ m, intensity of the pumping laser = 15 mW/cm²). The crosses are experimental points (only every 25th point is marked), and the full lines are fits obtained with the functions given by eqs. (7) and (8). The parameters of the fit are $\tau_1 = 13.9$ s and $\tau_2 = 28.5$ s for the buildup and $\tau_3 = 323.6$ s and $\beta = 0.579$ for the decay.

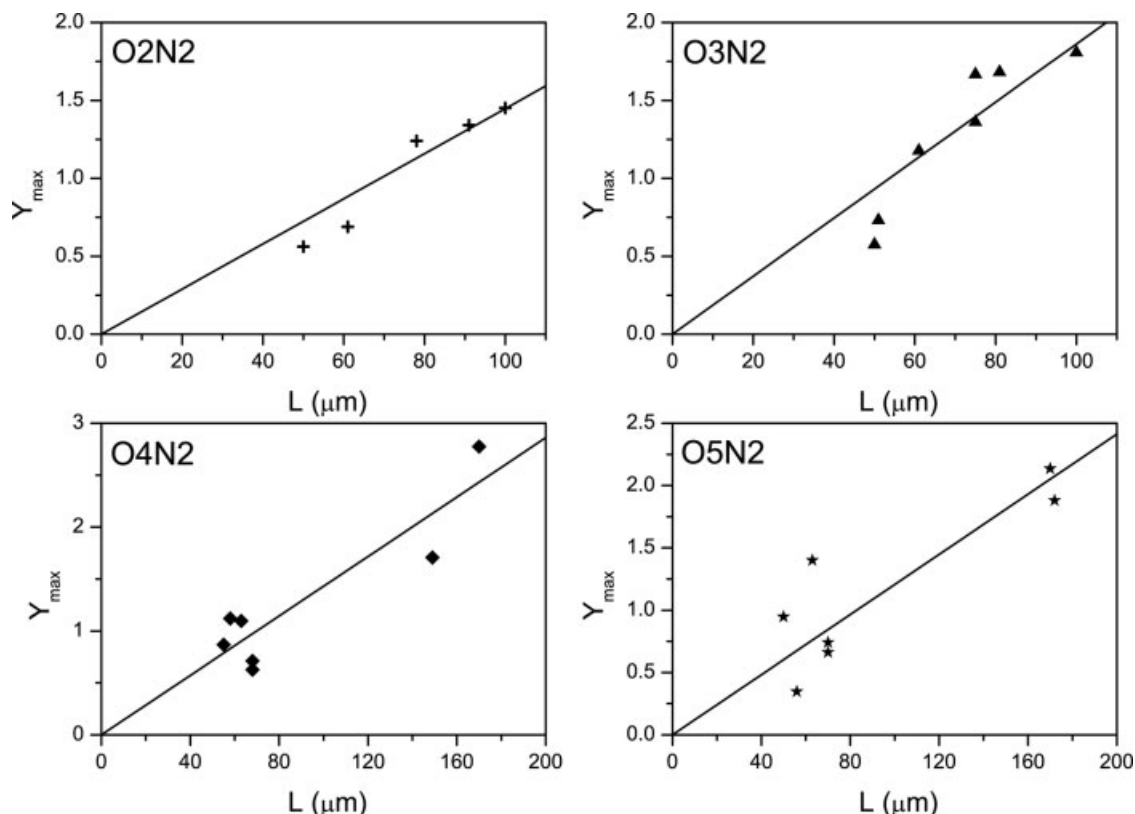


Figure 5 Thickness dependence of the saturation amplitudes in azo crown ethers dissolved in PMMA. The dye concentrations were 0.98 wt % (0.049 mol/dm³) for O2N2, 0.95 wt % (0.040 mol/dm³) for O3N2, 0.98 wt % (0.036 mol/dm³) for O4N2, and 0.85 wt % (0.028 mol/dm³) for O5N2. The pumping laser intensity was 10 mW/cm² in all cases. The lines are the fits taken under the assumption of the proportionality of Y to the sample thickness.

the buildup and decay of the signal—the respective time constants and amplitudes of the saturation and remanent signal—as a function of the pumping laser intensity and the sample thickness. A typical experimental curve is shown in Figure 4.

The saturation amplitude of the signal (for the purpose of this article determined to be $Y_{\max} \equiv \sqrt{I_{\max}}$; cf. Fig. 1) was found to vary with the pumping laser intensity, the azo crown ether concentration in the sample, the temperature, and the sample thickness. To within the accuracy of our measurements, the saturation amplitude increased with the sample thickness, as shown in Figure 5. The dependence of the saturation amplitude on the intensity of the pumping radiation was weaker: within the employed range of intensities (5–15 mW/cm²), the amplitude increased by about 20–40% (cf. Fig. 6).

Several functions were tried to fit the temporal dependences of the experimental curves. The buildup curves were found to be reasonably described by a combination of two exponential functions (see Fig. 4):

$$\varphi_{\text{pump}} = a \exp\left(-\frac{t-t_0}{\tau_1}\right) + b \exp\left(-\frac{t-t_0}{\tau_2}\right), \quad a + b = 1 \quad (7)$$

The accuracy of the fits was always checked: the R^2 factors taken into account in further analysis always exceeded 0.995 (in most cases, $R^2 > 0.997$).

The time constant characterizing the initial (fast) rise of the signal (τ_1) was found to depend on the intensity of the pumping light, as shown in Figure 7. Plotted in the figures are the inverse time constants versus the intensities of the light, and the fits indicate their proportionalities.

The time constant controlling the slower part of the buildup (τ_2) was more difficult to determine reliably, and its values exhibited a more erratic behavior. In general, however, the latter constants were of the order of 10² s, exhibiting a tendency to decrease with the increasing intensity of the pumping light.

The relaxations in O2N2, O3N2, and O4N2 were best described by a single stretched exponential:

$$\varphi_{\text{dec}} = \exp\left[-\left(\frac{t-t_1}{\tau_3}\right)^\beta\right] \quad (8)$$

The behavior of the samples containing O5N2 was somewhat different: immediately after the laser was switched off, rapid decays were observed, their time constants being of the order of 10⁰–10¹ s, and only

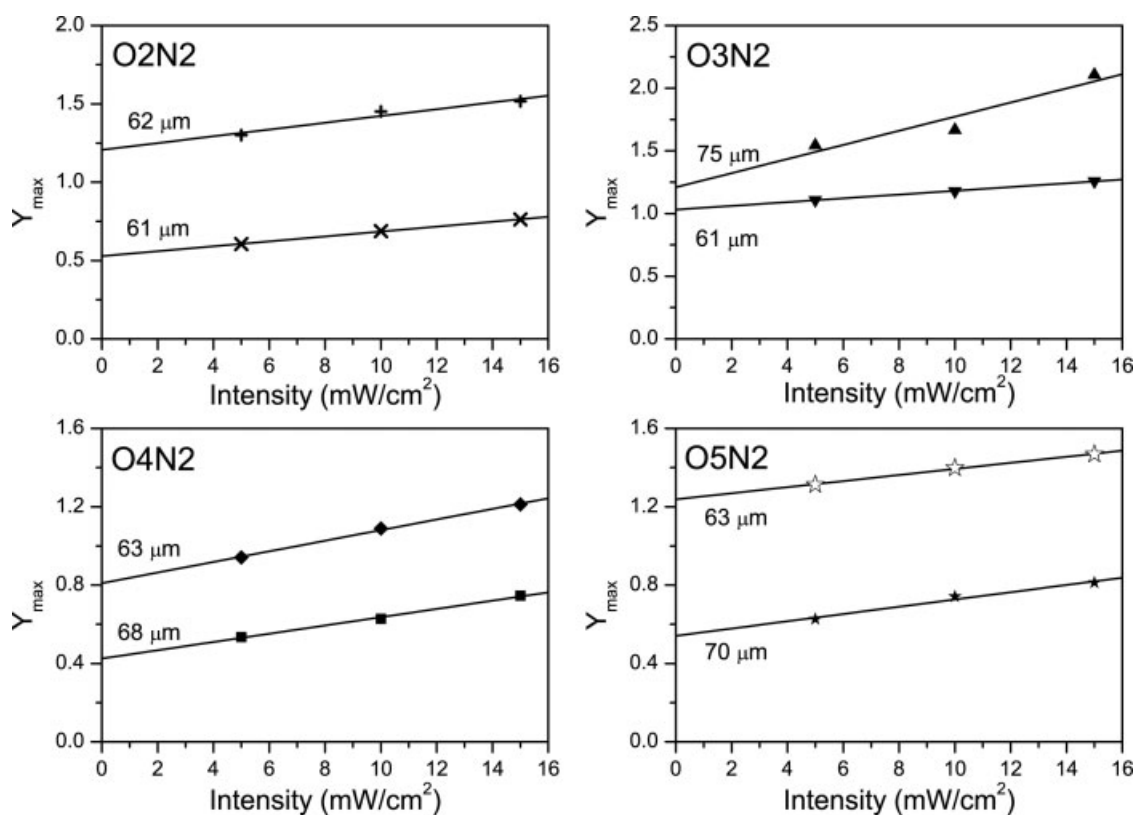


Figure 6 Intensity dependence of the saturation amplitudes in the azo crown ethers dissolved in PMMA. The sample thicknesses are indicated.

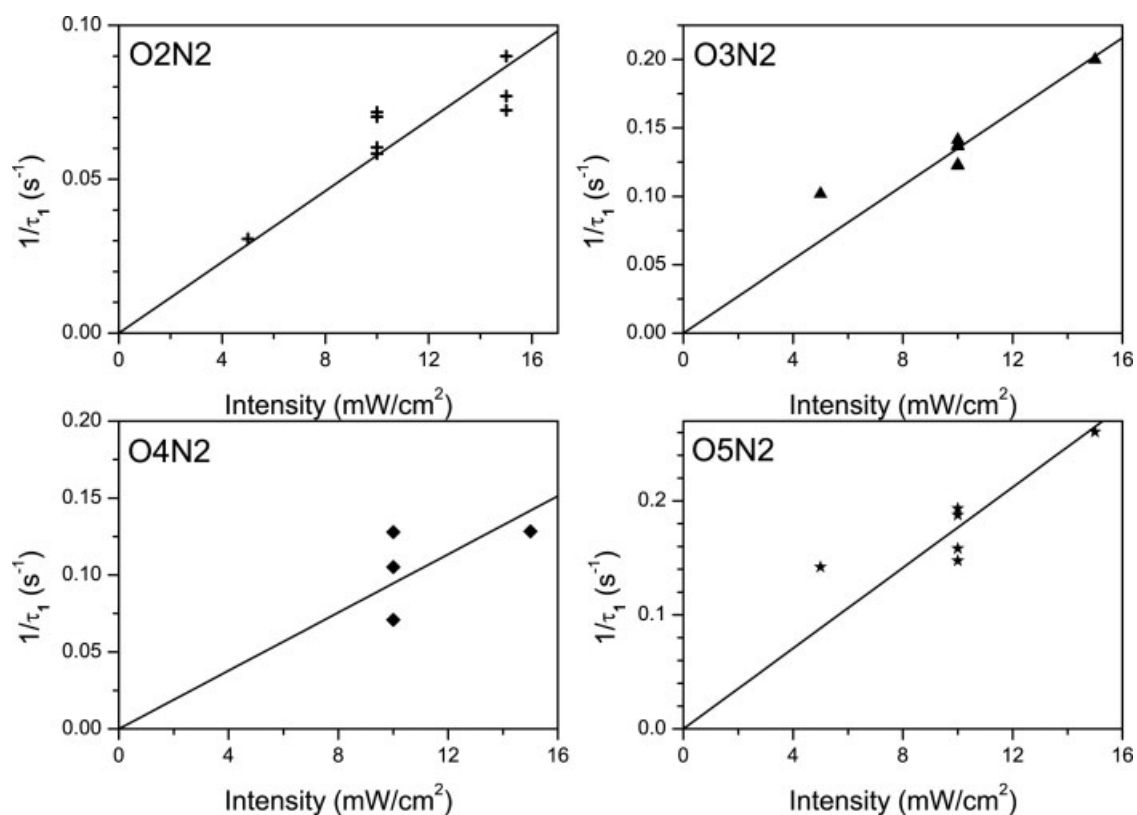


Figure 7 Intensity dependence of $1/\tau_1$ on the pumping light intensity. The lines are the fits taken under the assumption of $1/\tau_1$ being proportional to the intensity of the pumping light.

TABLE I
Average Values of τ_3 and β

Molecule	$1/\tau_3$ (s ⁻¹)	β
O2N2	$(2.5 \pm 0.7) \times 10^{-3}$	0.56 ± 0.04
O3N2	$(2 \pm 1) \times 10^{-3}$	0.59 ± 0.05
O4N2	$(4 \pm 2) \times 10^{-3}$	0.59 ± 0.05
O5N2	$(1.4 \pm 0.5) \times 10^{-3}$	0.52 ± 0.06

after a few seconds, the decays became similar to those observed in other materials. In the case of O5N2, time constant τ_3 was determined for the second section of the curves. To within the accuracy of our experiments, τ_3 and β did not exhibit any noticeable dependence on the sample thickness or on the intensity of the pumping light. Their average values are given in Table I. It is important to note that in nearly all cases, the fits to the decay curves with eq. (8) yielded quite low values of the remanent birefringence [Y_∞ in eq.(4)] close to zero. For a practical evaluation, we determined the ratios of the birefringence remaining after 600 s of decay to that at the beginning of the decay (Y_r/Y_{\max}): typically, they amounted to about 20–30%.

DISCUSSION

As mentioned previously, the experimental results (kinetics of the rise and decay of the birefringence and its dependence on the pumping light intensity, temperature, thickness, etc.) are often analyzed under the assumption of a direct relation between the parameters characterizing these dependences and the kinetics of the photochromic reaction. Moreover, one should expect a link between some of these parameters and the relaxation of the polymer matrix.

The results put forward in this article confirm the buildup of PIB in the azo crown ether samples, it being directly related to the photoisomerization of the azobenzene moiety. Indeed, the dependences shown in Figure 7 fit perfectly into the picture: the photoisomerization rate constant of azobenzene derivatives has been known to be a linear function of the exciting light intensity.⁴⁹ It should be pointed out, however, that the rate constants ($1/\tau_1$) determined from the experiments reported in this article do not have to be identified with the rate constants of the photoisomerization: a sufficient condition is their mutual proportionality.

It has been demonstrated in this article that the buildups cannot be described by monoexponential curves. Thus, one may infer that at least two processes are operative, one of them being related to the photoisomerization of the dyes. It would be tempting to link the other process with motions of the polymer matrix, adapting itself to new orientations of photoaligned azo crown ether molecules. We cannot, however, offer

any strong support to this hypothesis. Equally possible seems a distribution of rate constants associated with a distribution of microenvironments of photooriented molecules.²⁹

The dependence of the birefringence on the sample thickness (see Fig. 5) can be easily rationalized if we take into account the fact that, for a weakly absorbed light, the magnitude of the effect should be proportional to the number of active molecules, that is, to the product of the concentration times the thickness. Because the concentrations of the azo crown ethers are constant, the signal should be proportional to the thickness. Within the experimental uncertainty, our experiments follow this dependence.

The birefringence was found to be a reasonably weak function of the pumping light intensity (cf. Fig. 6). The result is in qualitative agreement with the model put forward by Sekkat and coworkers.^{22,24,25} It should be noted that the amplitudes depend on the nature of the dye (also after normalization to identical concentrations), reflecting different efficiencies of the photoalignment process.

The relaxation of the signal in the dark is due to a process that seems to be completely disconnected from the thermal isomerization of the azo moiety in the azo crown ethers. The kinetic parameters of the process were found to be practically independent of the nature of the dye, being of the order of 10^2 – 10^3 s, whereas the thermal isomerization was shown to be strongly material-dependent: at the temperature of our experiments, the time constants of the thermal cis–trans isomerization range between 4.5×10^9 s for O2N2 and about 10^6 s for O4N2 and O5N2.⁴¹ In other words, on the timescale of the experiments reported in this article, the thermal isomerization of the dyes is negligibly slow. The decay of the birefringence may therefore be due to either a relaxation of the matrix and/or a rearrangement of the shapes of the flexible oligo(oxyethylene) parts of the molecules, both effects resulting in a disordering of the azobenzene moieties.

CONCLUSIONS

We have demonstrated the existence of PIB in a class of photoactive macrocyclic molecules consisting of the photoisomerizable azobenzene moiety and flexible oligo(oxyethylene) fragments of various lengths. Such a shape of the molecules was expected to ensure the relative freedom of their reorientation upon photoisomerization, regardless of the matrix. The buildup of the signal was shown to be related to the photoisomerization rate, whereas its decay was independent of the rate of thermal isomerization and apparently associated with the relaxation of the polymer matrix and/or a rearrangement of the shapes of the crowns. At present, we cannot decide which mechanism prevails.

The flexibility of the molecules may also be responsible for the low values of the remanent birefringence: the extrapolated values are often close to zero, and even after 600 s, the samples retain no more than some 20–30% of the maximum values.

Finally, the relatively great scatter of the results should also be mentioned. One should realize that the time constants are extracted from nonlinear fits involving several adjustable parameters. Thus, apart from obvious experimental errors, an uncertainty due to the existence of local minima should be accepted.

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