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Dichroic Dyes and Liquid Crystalline Side Chain Polymers**

Anisotropic Glasses
Optical Storage
LC-Displays

By Hans-Werner Schmidt*

1. Introduction

Thermotropic liquid crystalline (LC) side-chain polymers have attracted much attention in the last few years. In LC-side-chain polymers, rod-like mesogenic units are attached as side groups to the polymer backbone. The potential of LC-side-chain polymers is based on the unique combination of specific polymer properties with the anisotropic physical properties of conventional low molar mass liquid crystals. The developments in the area demonstrate the practical interest in this new class of materials, especially for optical and opto-electronic applications. Several review articles and books have been published describing the synthesis, characterization, properties and potential applications of LC side-chain polymers.[1]

The reason for the interest in liquid crystalline systems which contain dissolved or incorporated dichroic dyes is that the anisotropic order of liquid crystalline phases causes the molecular orientation of the guest molecules and the anisotropic properties of these guest molecules can be detected. The interplay of the molecular alignment with the different possibilities for macroscopic orientation is the basis for several spectroscopic methods as well as for commercial display applications. For instance, dichroic dye molecules or fluorescent dyes have been used as probes to study order phenomena in different thermotropic and lyotropic systems.

Mixtures of dichroic dyes, dissolved as guest molecules in low-molar-mass liquid crystals, are used in displays operating on the principle of dichroism. The electro-optical effect, a color intensity change, is based on the cooperative alignment of the dye in the host phase and the dependence of absorption on the macroscopic orientation. This effect is schematically demonstrated in Figure 1 for a nematic guest-host system with positive dielectric anisotropy in a typical Heilmeier cell.[2]

In the off-state (left) the nematic phase and the dye molecules are oriented parallel (homogeneously planar) to the glass plates and parallel to the polarization plane of a polarizer. If the transition moment of the absorption of visible light is in the same direction, maximum absorption occurs. In the on-state (right) the molecules are aligned perpendicular to the glass plates by an electric field. As a result, absorption is low. The areas of the display in the on-state appear colorless, while the ones in the off-state are colored. Suitable dichroic dyes for application should have high order parameters, high solubilities, high absorption coefficients and good photochemical stability. A large number of guest-host mixtures have been investigated and correlations between dye structure and properties have been discussed.[3]

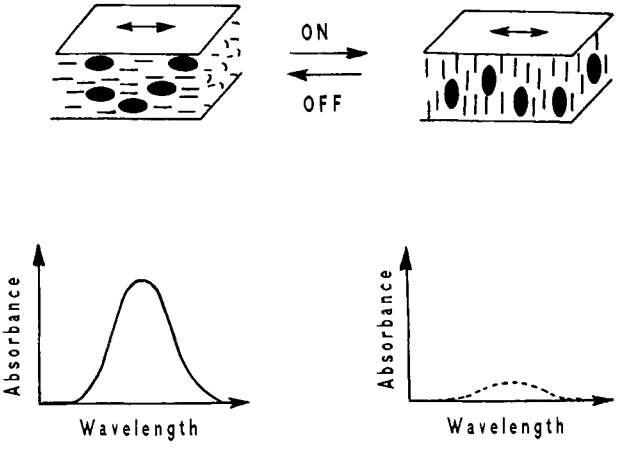

Fig. 1. Schematic representation of the on- and off-states for a guest-host system ($\Delta\varepsilon$ positive) in a Heilmeier cell, and the corresponding absorption spectra. ●: Dichroic dye moiety; ——: mesogenic unit; ↔: polarization plane.

This article focuses on the combination of LC side-chain polymers with dissolved or incorporated dichroic dyes but the results and conclusions are transferable to other types of guest molecules. The structure and properties of different dye containing liquid crystalline systems will be discussed and particular attention is given to copolymers with dichroic dyes and mesogens as side groups. Principal differences to low molar mass guest-host mixtures will be demonstrated and some potential applications of these materials such as dichroic polymer films, media for optical information storage or use as guest components in low molar mass liquid crystals for displays will be discussed.

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2. Dichroic Dyes in Combination with LC-Side-Chain Polymers

Guest-host systems based on LC-side-chain polymers can be classified into three different types. The schematic structures of these types are shown in Figure 2.

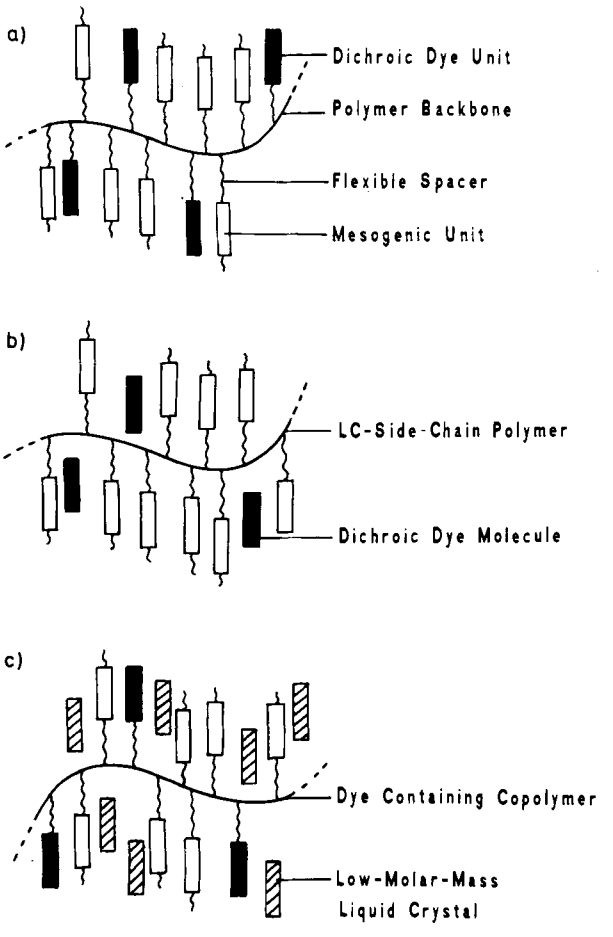


Fig. 2. Schematic structures of host-guest systems in combination with LC-side-chain polymers. a) LC copolymers with dichroic dyes and mesogens as side groups. b) Dichroic dyes dissolved as guests in LC-side-chain polymers. c) Dye containing copolymers as guests in low molar mass LC hosts.

A polymeric guest-host system is a copolymer with dichroic dyes and mesogens as side groups (Fig. 2a). The mesogenic groups as well as the dichroic dyes are covalently attached via flexible spacers to the polymer backbone.[4] This covalent bonding of the mesogenic units and dyes causes significant differences to low-molar mass mixtures. In the case of copolymers, liquid crystalline materials can be prepared with high dye concentrations, but the solubility of dichroic dyes is limited in low molar mass mixtures. In addition, the dye content to the copolymers is independent of the temperature and the type of mesophase. Another important difference is the fact that the order of the liquid crystalline melt does not change for LC-side-chain polymers when they are frozen in the solid state. As a result, the material becomes an anisotropic glass.[5] In contrast to this behavior, the liquid-crystalline order of low molar mass guest-host mixtures is generally destroyed upon cooling by crystallization.

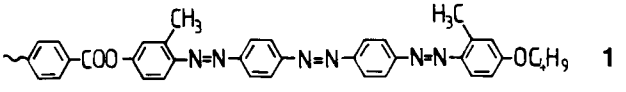
Another approach is the dissolution of dye molecules as guests in LC-side-chain polymers (Fig. 2b).[4d, 5, 6] This approach is limited by the solubility of the dye in the polymer matrix, but is suitable for the use of low dye concentrations. However, it can happen that the dye molecules phase separate and recrystallize above the glass transition temperature.

LC-copolymers with dichroic dyes and mesogens as side groups can be dissolved as guest components in low molar mass liquid crystals (Fig. 2c).[4c] The use of dye containing copolymers in mixtures is a general concept which achieves sufficiently high concentrations of dyes which have otherwise very poor solubility in low molar mass liquid crystals. The mesogenic side groups can be regarded as solubility enhancers for the dye moieties. The properties of such guest-host mixtures with respect to possible display applications are discussed in section 6.

3. Synthesis and LC-Behavior of Dye Containing Copolymers

LC-copolymers with dichroic dyes as side groups can be obtained using the same synthetic procedures as for LC-side-chain polymers. The majority of published copolymers are polyacrylates, synthesized by a free radical copolymerization of mesogenic monomers and monomeric dyes. Another method is the reaction of mesogenic monomers and dyes containing terminal alkene groups with poly(methyl hydrogen siloxane) resulting in a dye containing copolymers with a siloxane backbone. Some functional groups of the dye moiety cause these procedures to fail and other polymerization techniques such as group transfer polymerization, or polycondensation procedures are necessary.

Azo- (mono, bis and tris)-, stilbene-, anthraquinone-, violanthrone-, and spiropyrane-dyes have been investigated in dye-containing LC side chain copolymers.[4] Of particular interest was the preparation of 'black' copolymers which absorb over the entire spectrum of visible light. Such copolymers can be prepared by the attachment of several dyes to one polymer backbone. Examples of dichroic dyes which are covalently attached in LC-side-chain copolymers are the yellow tris azo dye **1**,[4b] the blue anthraquinone dye **2**[4b] and



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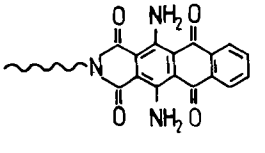

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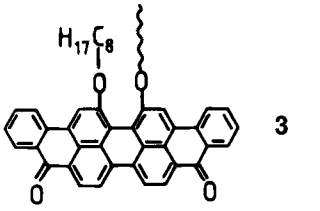
the violanthrone dye **3**.[7] All three dyes are known to have good photochemical stability and high absorption coefficients. *Cabrera* et al. investigated LC-side-chain copolymers with the photochromic spiropyrane dye **4**.[4e,f]



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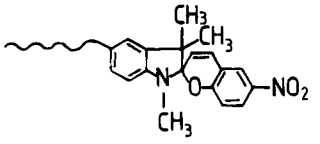

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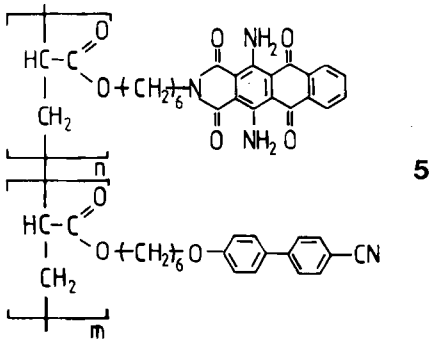
The liquid crystalline behavior of the copolymers depends on the dye content and on the molecular structure of the dyes. It was observed that anthraquinone- and spiropyrane-dyes generally destroy the mesophase. In contrast, the tris azo dye **1** increases the clearing temperatures and strongly broadens the mesophase range as the dye content increases. This behavior was found to be independent of the investigated mesogenic side groups in the copolymer. The liquid crystalline behavior of a polyacrylate copolymer **5** with the blue



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anthraquinone dye **2** and a cyanobiphenyl mesogenic unit as side group will be discussed in more detail. The differential scanning calorimetry (DSC) curve of a copolymer with a dye-content of 2 wt.-% is shown in Figure 3.

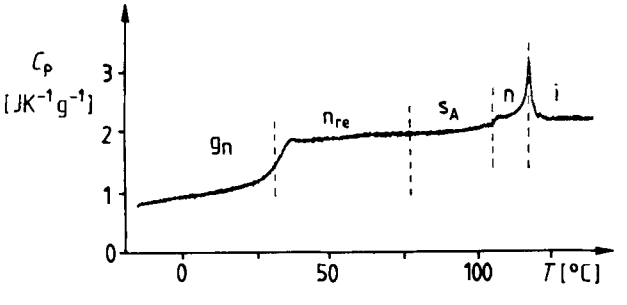

Fig. 3. DSC-heating curve of the anthraquinone-containing LC copolymer **5** (dye content 2 wt.-%). i: isotropic, n: nematic, s_A: smectic-A, n_{re}: reentrant nematic. g_n: nematic glass.

The copolymer has a glass transition at 36 °C and shows several additional endothermic transitions. The following LC behavior was observed using a polarizing microscope. On cooling from the isotropic melt, a nematic phase is formed at 120 °C and at 107 °C a transition to a smectic-A phase is observable. On further cooling a reentrant nematic phase is formed at about 75 °C and becomes a nematic glass below the glass transition temperature. The LC homopolymer without dye has the same phase sequence.[8] With increasing dye content the formation of the smectic-A phase is suppressed and copolymers with more than 10 wt.-% anthraquinone dye are only nematic. The phase diagram for copolymers with a dye content of up to 36 wt.-% is shown in Figure 4. The nematic to isotropic transition is lowered and

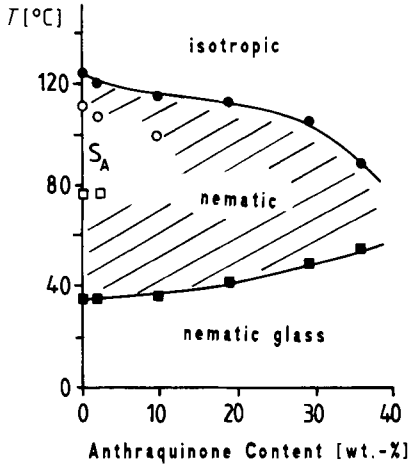

Fig. 4. Influence of the anthraquinone content on the LC behavior of copolymer series **5**. Transition temperatures plotted versus copolymer composition (wt.-% of the dye monomeric unit). ■: glass transition. □: transition reentrant nematic to smectic-A. ○: transition smectic-A to nematic. ●: transition nematic to isotropic.

the glass transition is increased with increasing anthraquinone content. Remarkably the copolymer with 36 wt.-% anthraquinone dye is still nematic. It should be mentioned that similar anthraquinone dyes in low molar mass nematic hosts in general have poor solubilities (below 0.5 wt.-%).

These results demonstrate that it is possible to synthesize liquid crystalline copolymers containing anthraquinone dyes, or other types of dichroic dyes, with very high dye concentrations. These high dye concentrations allow the preparation of thin LC polymer films (down to 1 μm thickness) with adjustable optical densities and absorption properties.

4. Orientational Order in Anisotropic Glasses

The microscopic LC order of side chain polymers can be macroscopically oriented by surface effects, or by magnetic or electric fields in a similar manner to low molar mass liquid crystals. Important is that the macroscopic orientation can

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be transferred to the solid state upon cooling below the glass transition temperature. An optically uniform, transparent, anisotropic glass is obtained. This was demonstrated for nematic as well as smectic glasses. If dye containing LC side-chain copolymers are used, dichroic polymer films are obtained. The oriented films can be prepared between glass plates in a display type cell configuration with a thickness of 0.5 mm to 1 μm (typically 10–20 μm). Uniform films on a substrate are also obtainable by spin coating.

The orientational order of dyes in the anisotropic glasses can be determined from the optical absorbance spectra parallel $A_{\parallel}$ and perpendicular $A_{\perp}$ to the direction of the homogeneous planar orientation (see Fig. 1, on-state). The dichroic ratio R and the order parameter of the dye S_D, characterizing the orientational order of the transition moment of the dyes, are calculated using the following expressions.[3] The order parameters vary from zero for an isotropic system to one for a perfectly aligned system.

$$R = \frac{A_{\parallel}}{A_{\perp}} \qquad S_D = \frac{A_{\parallel} - A_{\perp}}{A_{\parallel} + 2A_{\perp}}$$

As in low molar mass host-guest mixtures the dye structure and type of the liquid crystalline phase have an influence on the order parameter. In Figure 5a the absorption spectra

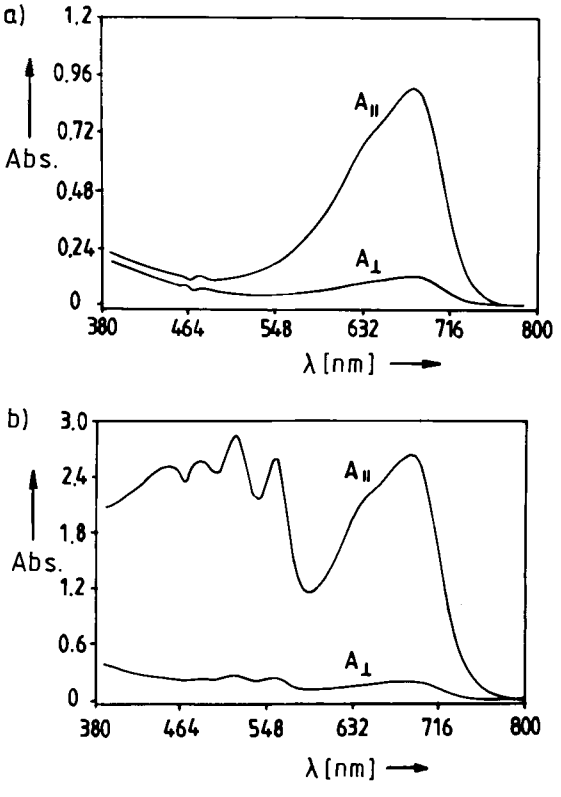

Fig. 5. a) Absorption spectra taken parallel ($A_{\parallel}$) and perpendicular ($A_{\perp}$) to the alignment direction of the anthraquinone containing copolymer **5** in an oriented nematic glass. b) Absorption spectra for a 'black' copolymer with three dyes (yellow tris azo dye, red and blue anthraquinone dyes) in an oriented smectic-A glass.

$A_{\parallel}$ and $A_{\perp}$ for copolymer **5** with the blue anthraquinone dye in the nematic glass are shown. The dichroic ratio is 7.6 and the corresponding order parameter 0.69. The order parameter of the mesogenic side group (0.71) was determined by IR-dichroism measurements, using the vibration of the cyano group attached to the biphenyl unit. The observed S_D values are comparable with those for similar anthraquinone dyes dissolved in low molar mass nematics. Higher dye order parameters are obtainable in smectic glasses, where the host phase has a more ordered structure. The same anthraquinone dye has an order parameter of 0.78 in a smectic-A glass of a polyacrylate with methoxyphenylbenzoate side groups (g 23 s_A 86 n 106 i).

The shape of the dyes and their fixation has also an important influence on the order parameter. The covalently attached rod-like tris azo dye **1** has an order parameter of 0.72 in the nematic glass and 0.84 in the smectic-A glass. For the more plate-like violanthrone dye **3**, which is attached perpendicular to its main molecular axis, lower order parameters were observed as expected. The S_D value in the nematic glass of a polyacrylate with cyanophenylbenzoate side groups (g 32 n 120 i) is only 0.42. In the smectic-A glass (g 30 s_A 93 n 119 i) the order parameter is 0.60.

Figure 5b shows the absorption spectra for a 'black' LC-side-chain copolymer in an oriented smectic-A glass. In this copolymer three dyes are covalently attached to the same polymer backbone: a yellow tris azo dye, and a red and a blue anthraquinone dye. The overall dichroic ratio R for the dyes from 400 to 750 nm is about 10.8.

These examples demonstrate that optically uniform dichroic polymer films, with high dichroic ratios, can be prepared from dyes containing LC-side-chain copolymers. Higher order parameters and dichroic ratios should be obtainable by optimizing the dye structure and the mesogenic side groups.

5. Optical Information Storage with Dye Containing Copolymers

LC-side-chain polymers are considered as one possible organic medium for non-erasable and erasable optical information storage.[9] The interest in this class of materials is based on the various states of order and orientation which they exhibit combined with the possibility of storing defined structures below the glass transition temperature or in highly viscous smectic phases. Storage in low molar mass liquid crystals requires a nematic phase and an additional viscous smectic phase. LC-side-chain polymers allow storage of information in nematic and smectic glasses. Optical storage effects, based on thermal (heat mode)[10] and photochemical (photon mode)[4e,f,11] mechanisms, have been reported for LC-side-chain polymers. Photochemical reactions in cholesteric polysiloxanes doped with benzophenone or carbon black were used for optical write-once storage.[12]

Optical storage based on thermal effects is generally related to a texture change in the LC film. This texture change can be a formation of scattering regions or a defined deformation of the LC-structure with or without the effects of additional external fields. The thermal energy required for a texture change can be generated on the surface or within the active LC film by dye molecules. The latter process has the advantage of higher sensitivity and better resolution. Dye molecules which are either dissolved[10b, 10d] or, like in our work, chemically incorporated into LC-side-chain polymers, can be used for thermal addressing. The covalent fixation of the dye moieties and the possibility of high dye content have additional advantages with respect to the matching of the sensitivity and thermal absorption with the characteristics of the applied laser. This can be done for variable film thicknesses.

As an example, the generation of optical scattering regions in optically clear macroscopically oriented films will be discussed. One possible write-store-erase cycle is schematically shown in Figure 6 for a dye-containing copolymer with a

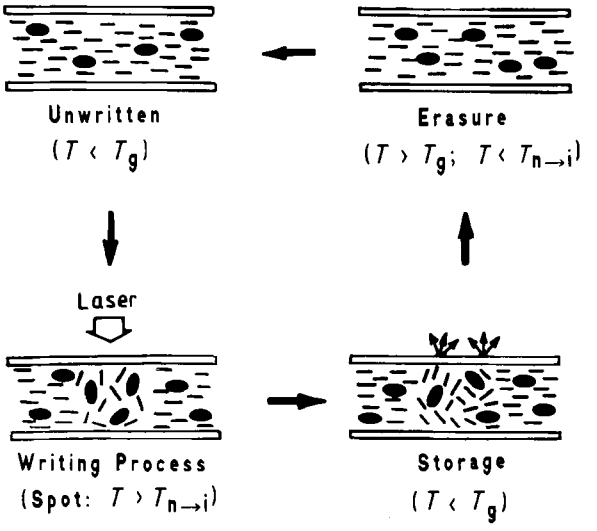

Fig. 6. Schematic representation of a write-store-erase cycle starting from a homogeneous planar oriented nematic glass of a dye-containing copolymer.

nematic phase. The unwritten state is an optically clear, nematic glass with homogeneous planar orientation. The writing process is based on local heating with a focused laser beam above the clearing temperature. On cooling, unoriented light scattering regions can be formed and stored below the glass transition temperature. Erasure is accomplished by heating the material into the nematic phase. The same write-store-erase cycle is possible with copolymers exhibiting a nematic and a smectic-A phase although in this case the information is stored in a smectic glass.

Figure 7 shows laser written scattering lines (width 2–3 mm) written with a He-Ne-laser (4 mW, 633 nm) between crossed polarizers within the macroscopically oriented glass of an anthraquinone dye-containing LC-copolymer.

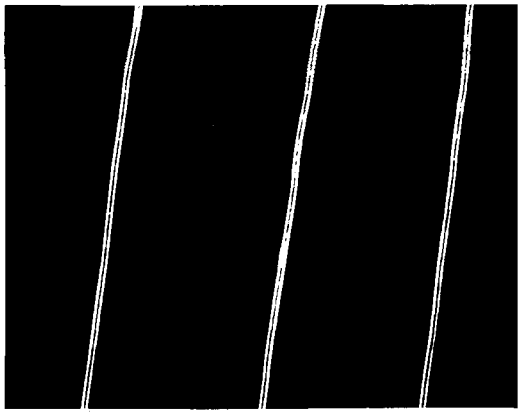

Fig. 7. Scattering lines (width: 2–3 μm) within a homogeneous planar oriented nematic glass (thickness 5 μm) of copolymer **5** (dye content: 10 wt.-%), written with a He-Ne laser (4 mW) at 633 nm. Photomicrograph between crossed polarizers and parallel to the director orientation (IAF, Freiburg).

The series of photographs in Figure 8 demonstrates the storage and erasure process. Figure 8a shows optically isotropic spots with a diameter of 60 μm written with a He-Ne laser in the homogeneous planar orientation of an anthraquinone dye containing copolymer. The information is stored below the glass transition temperature. If the sample is heated for one hour at 50 °C, slightly above the glass transition temperature, reorientation begins and the spot become smaller (Fig. 8b). After heating for an additional hour at 80 °C the spots are completely erased and the original homogeneous planar orientation is again obtained (Fig. 8c).

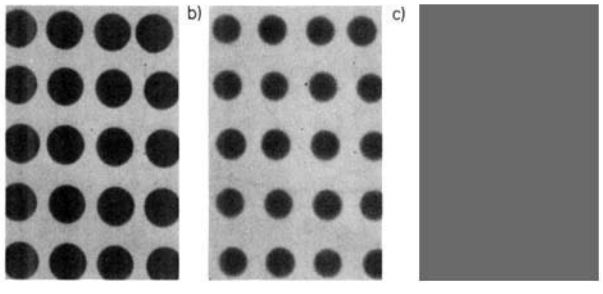

Fig. 8. Photomicrographs between crossed polarizers (45° relative to the director of the macroscopic orientation) of laser written spots on an anthraquinone dye-containing LC-side-chain copolymer (BASF, Ludwigshafen). a) information stored at room temperature. b) sample heated for one hour at 50 °C. c) sample heated for an additional hour at 80 °C.

As mentioned earlier, the writing process is also possible by laser addressing via a conducting IR-absorbing surface. Figure 9 shows a photograph of a cell drawn with a scanning Ne-YAG-laser at room temperature on a homogeneous planar aligned sample of LC-homopolymer with cyanobiphenylene side groups (**5** without the anthraquinone dye). Today, after more than three years, the information is still stable.

Photochemical storage effects in dyes contaning LC-polymers based on the cis-trans isomerization of the azobenzene side groups have been demonstrated by *Eich* et al.[11] and used for reversible digital and holographic optical storage.

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Fig. 9. Outlines of the Federal Republic of Germany and the German Democratic Republic drawn by a Ne-YAG-laser onto a homogeneous aligned nematic LC-homopolymer. Photomicrograph between crossed polarizers and parallel to the orientation of the director. (Cell: 4 cm × 3 cm; thickness: 10 μm; line width: 20–30 μm, IAF, Freiburg).

The photochromic and thermochromic behavior of spiropyrane dyes **4** in liquid crystalline polyacrylates and polysiloxanes have been recently described by *Cabrera* et al.[4e,f] The yellow spiropyrane dye was converted by UV-irradiation into its blue merocyanine form. Heating above the glass transition temperature caused the merocyanine side groups to form red dimers. The reverse photoconversion from the merocyanine structures occurred on irradiation with visible light.

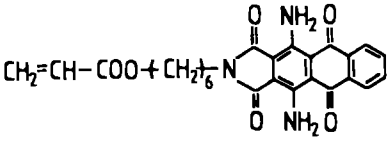
The examples discussed above demonstrate that dye-containing LC side chain copolymers can be used in principle as a medium for different modes of reversible optical storage. Studies have to be done on the different systems to further investigate application criteria such as speed, sensitivity, resolution, image density, storage stability, read-out stability, erasability and rewritability.

6. Copolymers as Guests in Low Molar Mass LC-Hosts

Nematic dye-containing copolymers in displays show the same orientation behavior and electrooptical effects as low molar mass host-guest mixtures.[4a] Due to the higher bulk viscosity of the polymers, the response times are longer under comparable conditions. The switching times can be shortened by raising the temperature, applying higher voltages and reducing the cell thickness. Recently, *Kiefer*[13] demonstrated that it is possible to achieve rise times in the order of 25 msec for dye-containing copolymers in thin cells (typically 2 μm) with an applied voltage of 16 V.

A different approach to shortening the switching times is the reduction of the bulk viscosity by adding low molar mass liquid crystals. LC-side-chain polymers are in general miscible with low molar mass liquid crystals if a) the chemical structure of the liquid crystal is similar to the mesogenic side groups and b) if both components have the same mesophase.[14] In the same manner, dye-containing copolymers can be dissolved in low molar mass liquid crystals. The mesogenic side group in the copolymer can be regarded as a solubility enhancer for the dye molecules. The use of dye-containing copolymers as guests is a general concept and is an alternative to the chemical modification of low molar mass dichroic dyes. Several types of dye-containing copolymers were investigated in different nematic low molar mass hosts with respect to the dye solubility, the order parameter S_D and the switching behavior in displays.[4c,15]

As an example, a mixture of anthraquinone copolymer **5** in a binary nematic host consisting of cyanobiphenyls **7** and **8** (40 mol-% **7**; 60 mol-% **8**) will be discussed. Mixtures with



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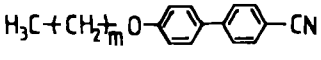
a dye concentration of 1.5wt.-% were prepared. The total polymer concentration in the mixtures can be adjusted by using copolymers with different dye contents. It should be noted that 1.5 wt.-% is not a maximum value, but is already three times higher than the achievable dye concentration of the corresponding dye monomer **6** in the binary host. Dye-containing copolymers can also be dissolved in multicomponent commercial mixtures, which are currently used in LC displays.



7: m = 4 **8**: m = 6

The mixtures have relatively low viscosities. Therefore commercial displays can be easily filled by capillary action and the LC mixture macroscopically aligned into a homogeneous planar orientation by surface effects. The order parameter of the dichroic dye moieties can be determined in the same manner as described in section 4. Figure 10 compares the order parameters S_D of a mixture of copolymer **5** (anthraquinone content 19 wt.-%) dissolved in the binary cyanobiphenyl host, with a mixture of the monomeric dye **6** in the same host. The order parameters S_D are plotted versus the reduced temperature (T_{red}). The reduced temperature is defined by the ratio of actual temperature (K) to the clearing

temperature (K). The value of S_D and the temperature dependence are almost identical for both systems and are not influenced by the polymer fixation of the dye.

Displays filled with copolymer containing host-guest mixtures can be switched by an electric field as shown in Fig-

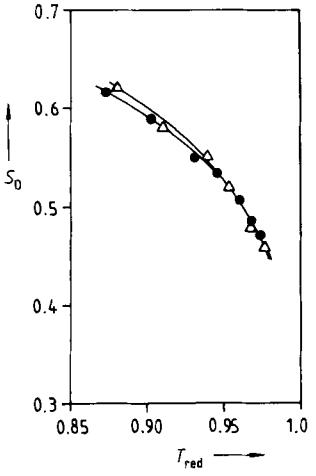


Fig. 10. Order parameter S_D versus T_{red} for the anthraquinone monomer **6** (△) and copolymer **5** (●) in the binary nematic biphenyl host of **7** and **8** (Compositions see [16]).

ure 1. As an example, switching conditions and switching times for a mixture of 14.1 wt.-% of an anthraquinone copolymer (dye content 13 wt.-%) in the commercial nematic phase ZLI 1840 (Merck, Darmstadt) are given. The rise time is about 60 msec and the decay time 350 msec in a display (thickness 8.0 μm) at 20 °C and an applied voltage of 7 V. The response times are longer by a factor of 3.8 (rise) and 5.5 (decay) than those measured for host phase ZLI 1840 under the same conditions. It should be noted that a dissolved low molar mass dichroic dye also increases the response times. Additional switching experiments have to be carried out in order to study the influence of the polymer content and of the molecular weight of the copolymers. The results have to be compared with low molar mass host-guest mixtures.

7. Summary and Outlook

Functional guest molecules incorporated into LC-side-chain polymers lead to polymeric materials which combine the physical and optical anisotropic properties of liquid crystals with the special functions of the guest components. This contribution focused on dichroic dyes, but the results are transferable to other guest moieties. The covalent fixation of the dye to the polymer backbone affords several advantages. Liquid crystalline materials with high dye concentrations were obtained opening the way to the preparation of highly ordered, optical uniaxial films with adjustable thickness and optical density. Thin well aligned LC-polymer films can in principle be used as an active medium for reversible optical information storage and mixtures of dye-containing copolymers in low molar mass liquid crystals can be used in displays. Current developments in the area of ferroelectric chiral smectic-C side-chain polymers will further enhance interest in this area.

Only a small part of the rapidly growing field of LC-polymer materials with specific optical and electrooptical properties has been discussed in this contribution. Combinations of novel polymeric LC-structures with or without special functional guest moieties will certainly lead to a variety of advanced polymeric materials with enormous potential in the areas of integrated optics, non-linear optics, optoelectronics, display and information storage technology.

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[16] Binary host: 40 mol-% **7** and 60 mol-% **8**; dye monomer **6**: 0.5 wt.-% dissolved in binary host; copolymer **5** (19 wt.-% dye) 7.8 wt.-% dissolved in binary host, resulting dye content 1.5 wt.-%.