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Behavior of Liquid Crystalline Side Chain Polymers in an Electric Field

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Liquid crystalline-side chain copolymers are compared with analogous liquid crystalline mixtures. The polymers can be homogeneously orientated by surface effects such as low molar mass mixtures. These substances were investigated using an electric field to study their regularities. The constitution and the concentration of polar guest molecules in copolymers and analogous mixtures were varied. The threshold voltages of polymer and monomer systems altered in the same way and are of the same order of magnitude. If the spacer length of the liquid crystalline-side chain polymer was shortened, an increase in the threshold voltage was observed. Investigations of the threshold voltage indicate that the behavior of the mesogenic side groups of the polymers is similar to that of low molar mass liquid crystals if the spacer is sufficiently long. Optical response times were much greater than those for conventional liquid crystals, due to the high viscosities of the polymers.

I. INTRODUCTION

More than ten years ago, the twisted nematic liquid crystal display (TNLCD = Schadt-Helfrich display) was developed as an electro-optic device.[1] Since then, the commercial interest in low molar mass liquid crystals has increased. Systematically, liquid crystalline substances (LC) have been developed, possessing optimal physical properties for display applications.

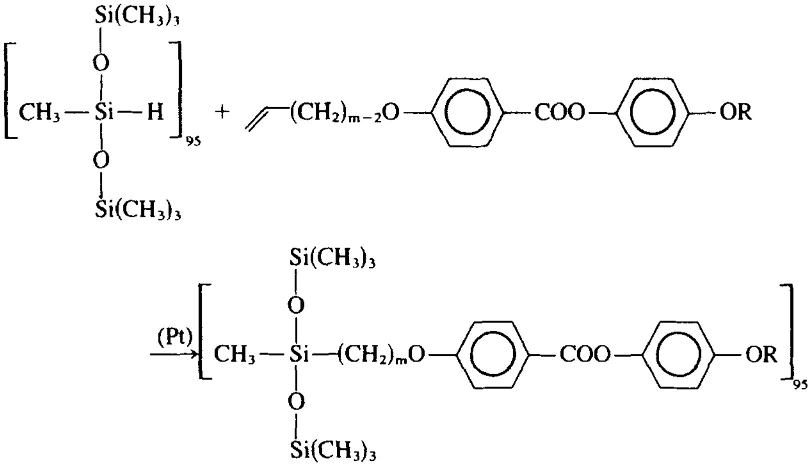
In the last few years LC materials have also become of interest as monomers for the preparation of polymers exhibiting LC properties. For the preparation of these polymers, the mesogenic monomers can be either connected "head by tail" forming "LC-main chain polymers" (Figure 1a) or linked as side chains *via* a flexible spacer to the polymer backbone (Figure 1b) (LC-side chain polymers). The LC-main chain polymers are already commercially used as "high modulus fibers", because the rigid rod-like molecules form highly ordered fibers spun in the anisotropic state. Compared with the conventional low molar mass LC, however, these polymers can hardly be ordered in a magnetic[2] or electric field.[3,4] The LC-side chain polymers, on the other hand, are more similar to the low molar mass LC, because the mesogenic moieties are only tied at one end to the macromolecules *via* the flexible spacer. Actually, recent investigations have shown that the LC-side chain polymers give electro-optical effects very similar to those for low molar mass liquid crystals.[5,6,7] Up to now, however, scant information exists which quantitatively compares the properties of these polymers with those for the corresponding monomers.

Knowledge of the behavior in an electric field is of great interest in view of technological applications and for obtaining theoretical insight into the LC-side chain polymers.

In this paper we will compare the orientation of LC-side chain polymers achieved by surface effects and by electric fields with that obtained for chemically analogous low molar mass LC and with varying chemical constitution of the polymer.

2. MATERIALS

Benzoic acid phenyl esters are used as liquid crystalline materials because they can be easily synthesized.[8,9,10] They also possess excellent properties in LC displays. If these benzoic acid phenyl esters have a vinyl group at the end of one alkyl chain, they can be linked to a siloxane main chain in an addition reaction in the presence of a platinum catalyst:



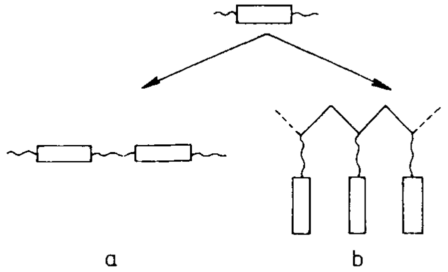


FIGURE 1 Schematic diagram of LC polymers: a) LC-main chain polymers and b) LC-side chain polymers.

The average degree of polymerization is 95.

The LC-side chain polymers should desirably possess a positive dielectric anisotropy if used in a TNLCD. If the benzoic acid phenyl esters are substituted in the *para*-position by polar substituents, however, *e.g.* R=—Cl, —Br, etc., no nematic, and only smectic homopolymers are obtained. Nematic homopolymers are only observed if no strongly polar substituents are in the *para*-position, *e.g.* R=—OCH_3. The alkyloxy substituted monomers, however, exhibit a dielectric anisotropy which is negative and cannot be used for TNLCD. Therefore, we prepared copolymers, where polar guest monomer units with a positive dielectric anisotropy were added to a nematic host polymer phase of negative dielectric anisotropy.

The phase behavior of the mixtures and copolymers was determined by differential scanning calorimetry (Perkin Elmer DSC II) and by polarizing microscopy (Leitz Ortholux II Pol BK). The phase transitions are listed in Tables I and II.

TABLE I

Phase transition temperatures of low molar mass LC mixtures.

Low molar mass mixtures	Phase transition temperatures in *K*							
M1	*k*	327	*s*	347	*n*	363	*i*
M2	*k*	325	*s*	349	*n*	364	*i*
M3	*k*	324	*s*	351	*n*	364	*i*
M4	*k*	324	*s*	342	*n*	361	*i*
M5	*k*	326	*s*	361	*n*	366	*i*

a) *k* = crystalline; *s* = smectic; *n* = nematic; *i* = isotropic.

TABLE II

Phase transition temperatures of copolymers.

Copolymers	Phase transition temperatures in *K*							
C1	*g*	281	*s*	333	*n*	380	*i*	
C2	*g*	282	*s*	343	*n*	380	*i*	
C3	*g*	283	*s*	352	*n*	380	*i*	
C4	*g*	279	*s*	326	*n*	375	*i*	
C5	*g*	280	*s*	362	*n*	390	*i*	
C6	*g*	290	*s*	355	*n*	391	*i*	
C7	*g*	289	*n*	372	*i*			
C8	*g*	290	*n*	354	*i*			

a) *g* = glassy; *s* = smectic; *n* = nematic; *i* = isotropic.

3. SAMPLE ALIGNMENTS BY SURFACE EFFECTS

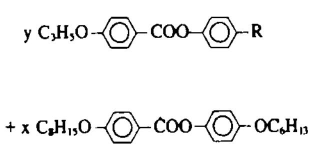
For technical applications, it is very important to have a uniform alignment of the director pattern produced by surface effects. For low molar mass systems, homogeneous alignments can be obtained by several surface treatments. These are, for example, rubbing the glass plates in one direction, evaporation of $(SiO)_x$, and rubbing of polymers anchored to the glass plates. The question arises whether these methods can be applied for LC polymers.

The rubbing of smooth, carefully cleaned glass plates with different materials failed totally for our polymers; no orientation was observed.

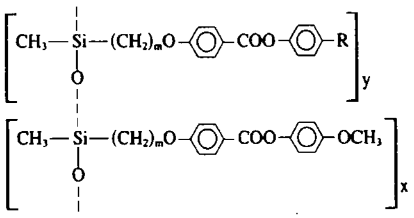
Glass plates, coated with $(SiO)_x$ and evaporated at 60° incidence, led to uniformly aligned samples. Microscopic observations indicate, however, that the director is not parallel to the glass plates, but tilted. Because of this pretilt angle, these preparations were not used for our measurements using electric fields. The best results for orientation of polymers were obtained with glass plates coated with oriented poly(imide). By conoscopic observations using the microscope, no pretilt angle could be observed for the aligned LC-polymers. It must be noted that a useful orientation of the polymers requires an annealing time of several h at temperatures only some degrees below the clearing point, when the polymer exhibits its lowest viscosity in the liquid crystalline state. Furthermore, the annealing time strongly depends on the chemical constitution of the polymer. With increasing length of the flexible spacer, the polymers behave more like low molar mass LC and can be oriented more easily.

For comparison analogous, low molar mass mixtures have been prepared:

a) *low molar mass mixtures*



b) *Copolymers*



Mixtures	x/y	R
M1	0,9	Cl
M2	0,85	Cl
M3	0,8	Cl
M4	0,9	F
M5	0,9	NO_2

Copolymers	x/y[a]	R	m
C1	0,9	Cl	6
C2	0,85	Cl	6
C3	0,8	Cl	6
C4	0,9	F	6
C5	0,9	NO_2	6
C6	0,9	Cl	5
C7	0,9	Cl	4
C8	0,9	Cl	3

a) (x + y = 95)

The guest molecules have strongly polar end groups. These are —F, —Cl and —NO_2. Mesogenic groups substituted with a nitrile group could not be used, because the nitrile group also undergoes an addition reaction with the poly (hydrogensiloxane) giving not linear, but cross-linked polymers. The distribution of the different mesogenic side groups along the siloxane back bone is statistically irregular, because it has to be assumed that the different polar substituents do not influence the reactivity of the vinyl groups in the addition reaction. Therefore, the formation of block copolymers can be excluded. The rigid rod-like side groups are attached *via* flexible alkylene spacers of different length *m* to the polymer back bone.

The preparation with poly(imide) coated glass plates was used for all polymers, giving well defined homogeneous orientations of the samples. These samples only were investigated, using an electric field; a sample thickness of 15 μm was employed.

4. EXPERIMENTAL PART

For the LC monomers, the twisted planar orientation was obtained with smooth glass plates evaporated with $(SiO)_x$ at 60° incidence. These glass plates were coated with a In_2O_3/SnO_2 layer as conductor. Before these glass plates were used, they were dried at 200°C to remove adsorbed water. For the copolymers, glass plates with rubbed poly(imide) were used to induce a homogeneous orientation. Because the polymeric systems possess higher viscosities than low molar mass systems, the devices could not be filled with LC polymers by capillary forces. The copolymers were carefully squeezed between the glass plates. For such a preparation, the temperature of the display was above the clearing temperature of the LC, because of the lower viscosity of the isotropic polymer melt. Thereafter, the sandwiched display was tempered (or annealed) for some hours until textures were obtained without defects. The twisted planar orientation was examined conoscopically with a polarizing microscope (Leitz Ortholux II Pol-BK). Following these observations, the quality of the orientation of low and high molar mass LC did not differ.

The sample thickness was controlled by poly(carbonate) spacers. The display was tempered in a heating stage (Mettler FP 52) and switched by a sinus generator (Schlumbergers solartron 1170 "frequency response analyzer") (at a frequency of 10^3 Hz). The light intensity was measured by a photomultiplier and recorded by a compensation recorder ("RE 541" of Metrawatt).

5. INVESTIGATIONS USING AN ELECTRIC FIELD

To characterize the behavior of the low and the high molar mass systems in an electric field, the TNLCD (Figure 2) was used. In TNLCDs, the director of the LC is aligned by surface effects parallel to the glass plates. The director at the lower glass plate is perpendicular to that at the upper plate, resulting in a twisted nematic structure (Figure 2a). This initial orientation is deformed by an electric field, if the voltage V is larger than the threshold voltage V_{th} (Figure 2b). The threshold voltage for low molar mass nematic LC, in the case of no pretilt, is a function of the dielectric anisotropy $\Delta\varepsilon$ and the splay (k_{11}), twist (k_{22}) and bend (k_{33}) elastic constants:[11]

$$V_{th} = \left(\frac{k_{11} + (k_{33} - 2k_{22})/4}{\varepsilon_0 \Delta\varepsilon}\right)^{0,5} \cdot \pi, \qquad \varepsilon_0 = \text{permittivity of vacuum} \quad (1)$$

If a voltage $V > V_{th}$ is applied to a display, the deformation from the former to the new equilibrium state requires time. These response times depend on characteristic materials' parameters and are given by Eq. (2) and (3)

$$t_{on} = \frac{\eta}{\varepsilon_0 \Delta\varepsilon E^2 - kq^2}, \qquad \text{with } k = k_{11} + (k_{33} - 2k_{22})/4 \quad (2)$$

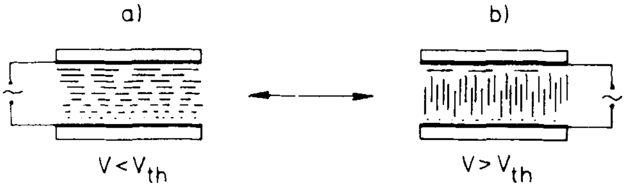


FIGURE 2 Twisted nematic LC display (TNLCD): a) twisted planar alignment ($V < V_{th}$) b) homeotropic alignment ($V \gg V_{th}$).

$$t_{off} = \frac{\eta}{kq^2} \tag{3}$$

For small angles of deformation of the nematic director, these times are a function of the viscosity η, the dielectric anisotropy $\Delta\varepsilon$, the different elastic constants and the wave factor $q = \pi/d$, d being the sample thickness. The turn-on time is additionally a function of the electric field strength E. While the threshold voltage depends only on k and dielectric properties, the response times are directly proportional to viscosity. This is of importance if monomeric LC and polymers are compared. The turn-on time t_{on} [Eq. (2)] is measured as the time which is necessary to obtain 90% of the initial transmission after the driving voltage has been applied. The turn-off time t_{off} [Eq. (3)] is the time required for the transmission to reach 10% of the transmission after the driving voltage has been switched off.

It is of interest whether these regularities for low molar mass systems are transferable to polymer systems. For these investigations, the physical parameters in Eqs. (1)–(3) do not refer to the molecular properties of the entire macromolecule but to the monomer unit of the backbone. In this way, the formalism only refers to the mesogenic moieties and neglects the polymer backbone in the case of the side chain polymers. Therefore, analogous low and high molar mass systems having different polar guest units and polar guest molecules of different concentration have been investigated. Furthermore, copolymers with spacers of varying length have been studied as the host molecules. For these systems, the dielectric anisotropy is expected to be constant. Therefore, any change in the elastic constants should be detected by alterations in the threshold voltages. Furthermore, a copolymer has been investigated with regard to its transient response time.

5.1 Low and high molar mass LC of different dielectric anisotropies

If guest molecules are added to a nematic host phase with different polar substituents but at constant concentration, a change of the dielectric anisotropy $\Delta\varepsilon$ will result for the mixtures. Referring to Eq. (1), different threshold voltages are then expected. Because derivatives of benzoic acid phenylesters containing $-F$, $-Cl$, and $-NO_2$ groups are used as guest molecules, the threshold voltage is expected to decrease with increasing permanent dipole moment. These dipole moments increase from fluorine to nitro derivative.[12]

Figure 3 shows the threshold voltages of the low molar mass mixtures M1, M4, and M5 having 10 mol% of the polar guest molecules.

Comparing the curves at constant reduced temperatures T^+ ($T^+ = T_m/T_c$ where T_m = measuring temperature, T_c = clearing temperature), the mixture M4 possesses the highest threshold voltage, followed by the mixtures M1 and M5. This is in accordance with Eq. (1). The temperature dependence of the threshold voltage of our mixtures is similar to that reported for well known systems;[13,14] the threshold voltage has its lowest value near

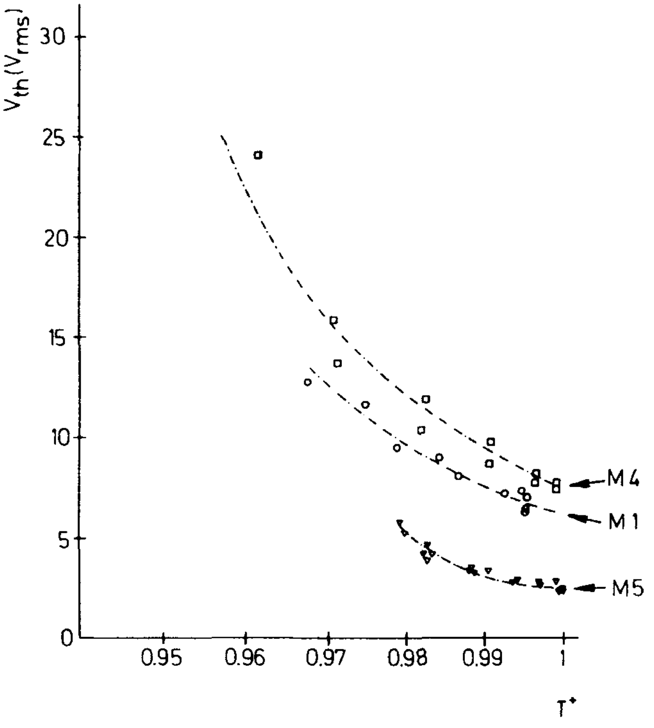


FIGURE 3 Temperature dependence of the threshold voltage V_{th} of mixtures M1, M4, and M5 ($T^+ = T_m/T_c$; T_m = measuring temperature, T_c = phase transition temperature nematic to isotropic). V_{rms} = effective voltage for alternating current.

the clearing point and increases with decreasing temperature. At low temperatures, a phase transition nematic-smectic is indicated by a strong increase in the threshold voltage.

For the chemically analogous poly(siloxanes), the results are shown in Figure 4. It is obvious that the curves for the temperature dependence of the threshold voltage for the polymers are very similar to the curves for the low molar mass mixtures. The copolymer C5 with the highest dielectric anisotropy possesses the lowest threshold voltage, and, as expected, copolymers C1 and C4 have higher threshold voltages at the same reduced temperature. The temperature dependences of the threshold voltages are lower for the copolymers than for the analogous mixtures. This is due to the broader range of the nematic phase of the polymers (see Table II). In the case of the polymers, the low temperature smectic phase has a smaller influence on the threshold voltage.

If analogous low and high molar mass systems are compared at a defined reduced temperature, it is noted that the threshold voltages are always lower

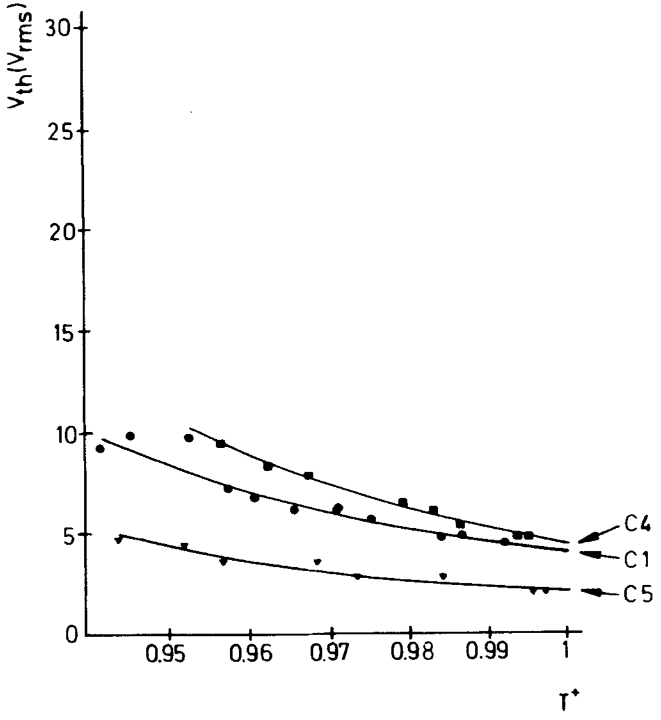


FIGURE 4 Temperature dependence of the threshold voltage V_{th} of copolymers C1, C4, and C5.

for the copolymers than for the corresponding mixtures. This can be explained by the following considerations.

The only constitutional difference between low and high molar mass systems is the polymer backbone. Its polarizability, as well as its permanent dipole moments, can be neglected, compared with the strong dielectric anisotropies of the mesogenic side chains. Therefore, following Eq. (1), differences in the threshold voltages of low and high molar mass systems have to be attributed to different effective elastic constants. This is due to the polymer fixation of the mesogenic side groups. In view of the magnitude of the absolute value of V_{th}, the results indicate that the mesogenic groups, which are linked as side chains to the main chain, behave very similarly to the monomeric systems. Another way to change the dielectric anisotropy of mixtures and copolymers is to alter the concentration of the polar guest molecules. If their concentration increases, the dielectric anisotropy becomes larger and the threshold voltage decreases [Eq. (1)]. To investigate this influence, the host phases were doped with 10, 15, and 20 mol% of the chloro derivative. The results for the threshold voltages are given in Figure 5 for monomers and polymers. As expected, the threshold voltage decreases with increasing concentration of the polar guest molecules. This is valid for low and high molar mass systems. For all measurements, the threshold voltages of the mixtures are higher than the values for the corresponding polymers.

Referring to Eq. (1), under these conditions, the threshold voltage is proportional to $\Delta\varepsilon^{0.5}$. To a first approximation, $\Delta\varepsilon$ should be nearly a linear function of the concentration of the polar guest molecules, and a change of the threshold voltage can then be related to a change in the concentration of the polar guests. Comparing the ratio of the different threshold voltages for monomers and polymers involving chloro derivatives, the same ratio of V_{th} is found by varying $\Delta\varepsilon$ (Table III). The measurements indicate that relation (1) for low molar mass LC is also valid for the polymers.

5.2 Variation of the spacer length

The chemical construction of LC-side chain polymers is based on the linkage of the mesogenic side groups to the polymer main chain *via* a flexible spacer (Figure 1). A direct linkage of the rigid, rod-like side groups to the polymer backbone results in a strong hindrance with the consequence that no LC phase occurs. If the spacer is sufficiently long and therefore rather flexible, one can expect that the motions of the side groups and of the polymer main chain are decoupled. Therefore, the physical properties,

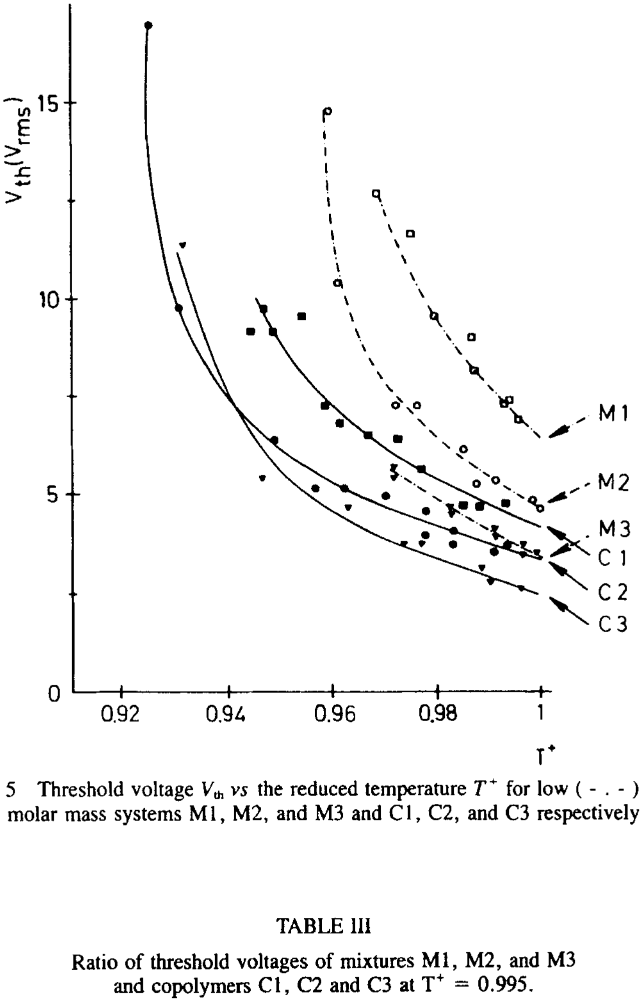


FIGURE 5 Threshold voltage V_{th} *vs* the reduced temperature T^+ for low (- . . -) and high (———) molar mass systems M1, M2, and M3 and C1, C2, and C3 respectively.

TABLE III

Ratio of threshold voltages of mixtures M1, M2, and M3 and copolymers C1, C2 and C3 at $T^+ = 0.995$.

Ratio of the threshold voltages of	
Low molar mass mixtures	Copolymers
$\frac{V_{th(M1)}}{V_{th(M2)}} = 1.4$	$\frac{V_{th(C1)}}{V_{th(C2)}} = 1.3$
$\frac{V_{th(M2)}}{V_{th(M3)}} = 1.4$	$\frac{V_{th(C2)}}{V_{th(C3)}} = 1.4$
$\frac{V_{th(M1)}}{V_{th(M3)}} = 1.9$	$\frac{V_{th(C1)}}{V_{th(C3)}} = 1.8$

for example the elastic constants, should vary with the spacer length. The properties of the polymers with long spacers should be more similar to the properties of low molar mass systems, whereas polymers with short spacers should be strongly influenced by the polymer main chain. Optical investigations of chiral nematic copolymers support these considerations.[15]

To investigate the influence of spacer length on the behavior of the polymers in an electric field, polymers were studied, which differ only in the length of the spacer ($m = 6, 5, 4$, and 3). This variation does not influence the dielectric anisotropy of the systems to a first approximation, but changes the elastic constants [Eq. (1)]. The results are shown in Figure 6. With falling spacer length, a strong increase in the threshold voltage is observed. If the length is only shortened by one methylene group, the threshold voltage changes from $V_{th} = 4\ V_{rms}$ for $m = 6$ to $V_{th} = 7{,}3\ V_{rms}$* for $m = 5$, indicating an increasing influence of the main chain. For $m = 4$, we found a threshold voltage $V_{th} > 50\ V_{rms}$ and for $m = 3$ no field effect could be observed under the experimental conditions ($T^+ = 0.995$). The measurements clearly confirm the consideration that the length of the flexible spacer directly influences the elastic constants of the system. If the spacer length is long enough to decouple the motions of the mesogenic groups from the motion of the polymer main chain, the elastic properties of the polymers tend to the elastic properties of the analogous low molar mass LC. This also confirms the result of the previous optical investigation.[15]

5.3 Optical transmission and transient response times

The electro-optic transmission characteristic is given by the change in intensity of light normal to the cell surface with altering voltage. It has been shown[16] that the slope of the optical transmission curve depends on the ratio of the bend and splay constants k_{33}/k_{11}. As our measurements indicate, the effective constants change from monomer to polymer system; therefore, it is of interest to examine whether the slope of the optical transmission curve is influenced. Under conditions with the same initial boundary orientations, typical transmission curves for a mixture M1 and a copolymer C1 are shown in Figure 7 for normal incident light and crossed polarizers.

For all polymers, a steeper slope in the curves of the transmission characteristic was found. According to Baur,[16] these results probably indicate that the quotient k_{33}/k_{11} is smaller for the polymeric systems than for the mixtures. Detailed measurements have to prove this assumption. These investigations can also be of technological interest, *e.g.* for adding poly-

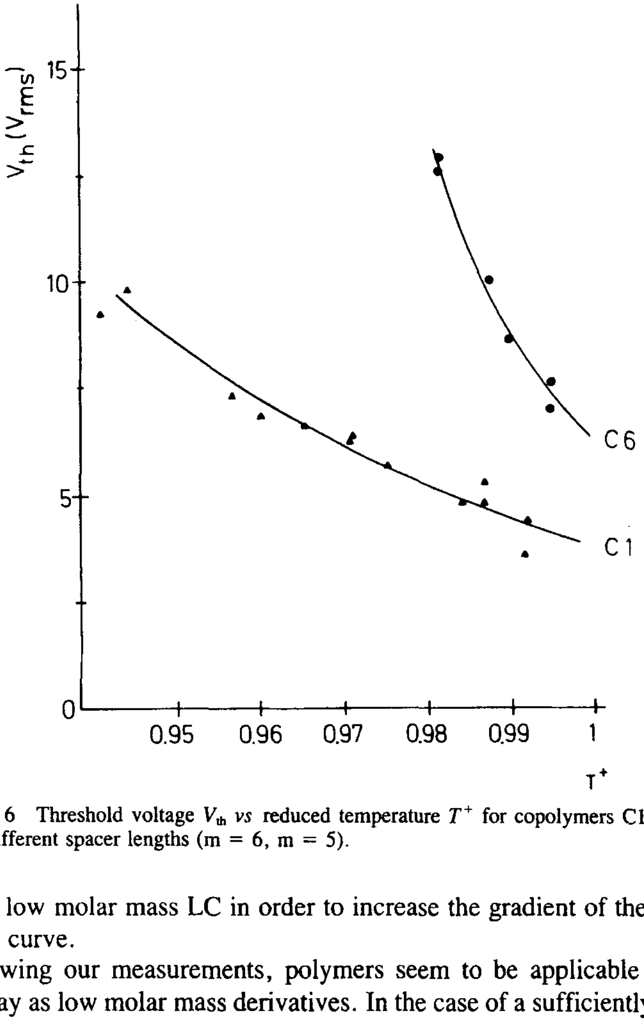


FIGURE 6 Threshold voltage V_{th} *vs* reduced temperature T^+ for copolymers C1 and C6 having different spacer lengths (m = 6, m = 5).

mers to low molar mass LC in order to increase the gradient of the transmission curve.

Following our measurements, polymers seem to be applicable in the same way as low molar mass derivatives. In the case of a sufficiently long, flexible spacer, the threshold voltages of the polymers have the same magnitude as those for the corresponding monomers. So far, however, dynamic effects have not been considered. Referring to the response times [Eqs. (2) and (3)], the response times of the polymers and mixtures must differ considerably, because these effects are determined by the viscosity of the LC. In any case, the viscosity of a polymer differs by orders of magnitude from that for the corresponding low molar mass system, especially if the temperature of the polymeric system is located near the glass transition.

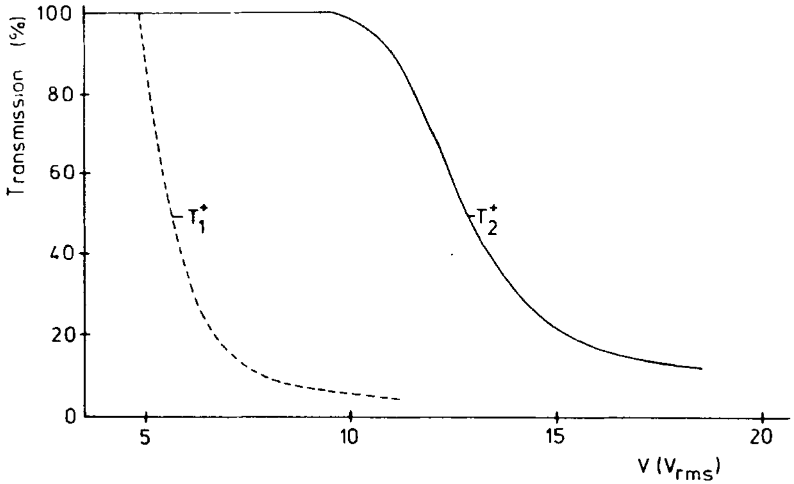


FIGURE 7 Optical transmission of low (———) and high (------) molar mass systems M1 and C1 at the reduced temperatures: T_1^+ = 0.987 and T_2^+ = 0.983.

In Table IV a typical example is given for the response times of the polymeric system C1, which is most similar to the monomers. It is obvious that the response times are larger by several orders than those for low molar mass systems.

Without further detailed information, we have to conclude that polymeric systems exhibit larger response times than monomeric systems principally because of their high viscosities.[6,17,18] Therefore, the application of pure polymers seems to be impractical in fast switching LC displays. On the other hand, the viscosity of the polymers can be widely influenced, *e.g.* by copolymerization or by softening effects using low molar mass LC, and this can lower the response times and improve the slope of the transmission curves (see Figure 7 and Table IV).

TABLE IV

Transient response times of copolymer C1 at normal incidence in a TNLCD for different reduced temperatures T^+, a sample thickness of 15 μm and a reduced voltage V/V_{th} = 10.

T^+	$t_{on}(s)$	$t_{off}(s)$
0,988	70	190
0,972	75	220
0,952	80	390

6. CONCLUSION

Comparing LC-side chain polymers with chemically analogous low molar mass systems in an electric field, the behavior of the polymers depends very sensitively on the chemical constitution of the linkage of the rigid mesogenic moiety to the polymer backbone. Only in case of a sufficiently flexible linkage, the polymers are very similar to the monomers. Under these conditions, the threshold voltage, and therefore the elastic constants, are of the same magnitude. However, due to the high viscosity, the pure polymers always exhibit very long response times, which make their use in conventional display technology less practicable. On the other hand, LC polymers are significant because of their glass transition, at which a LC polymer phase is converted into the glassy state. This freezing-in process does not affect the LC structure. Therefore, LC side chain polymers, *e.g.* a LC polymer film, can be used as storage material. The information, which is inserted in the LC state by the electric field can be stored durably in the glassy state of the material.

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