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Main Chain Liquid-Crystalline Elastomers: Swelling Dynamics and Electromechanical Effects

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Swelling behavior and deformations of main chain liquid crystal elastomers (MCLCEs) have been reported. The swelling agent used here is a low molecular weight liquid crystal (LMWLC), 4-n-pentyl-4-cyanobiphenyl (5CB). Monodomain MCLCE swells anisotropically with the swelling degree $q_{\text{mono}} = 11.9$ and polydomain MCLCE swell isotropically in 3D with $q_{\text{poly}} = 12.1$. These values are much larger than observed in swelling side-chain LCEs (SCLCEs). In the stress-strain measurement, the plateau area is found among two steep areas. The electromechanical effects of monodomain MCLCE is also observed, and the results show that the maximum contraction in $\|\hat{n}\|$ is about 7%. The threshold for the onset of the electromechanical effect is about 10 V, which is larger than observed in SCLCEs.

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1. INTRODUCTION

The swelling effect occurs when a dry material is placed in a solvent, in which the volume of the material increases due to absorbing solvent. Swelling properties of isotropic polymer gels in isotropic solvents have attracted much attention and their physical mechanisms are now well understood [1–4]. An isotropic gel can change its shape drastically when it is swollen. This macroscopic shape change arises from the cross-linked polymer networks changes inside the gel by swelling. Conventionally, it is known that the swelling properties and volume phase transitions of swollen gels depend on the environmental factors such as solvent composition, temperature, pH, electric field, light etc. [1–4]. On the other hand, a liquid-crystal elastomer (LCE), an anisotropic gel, has been recently synthesized, for which swelling properties and temperature behavior in an anisotropic solvent such as low molecular weight liquid crystals (LMWLC) are different from the well-known isotropic case [5–10].

In previous studies [6,7] we used the side-chain LCE (SCLCE) with different cross-linking agents, i.e., bifunctionally (8A2) and trifunctionally (V3) cross-linked LCEs; monodomain (LSCE, liquid single crystal elastomers) and polydomain LCEs. They were swollen with a low molecular weight liquid crystals, 4-n-pentyl-4-cyanobiphenyl (5CB), an anisotropic solvent. In particular, we found that the swelling properties and temperature behavior (thermomechanical property) of volume changes were also anisotropic [5–7]. The magnitude of the swelling anisotropy depended on the cross-linking concentration. Three important observations from our previous swelling study are;

1. No dimension changes parallel to the director \mathbf{n} could be observed during the swelling in 8A2 [6]. We believe this is because the cross-linkers in that LCE system order essentially parallel to the tethered mesogenic side chains due to their chemical constitution (the mesogenic units are oriented parallel to the stretching direction, fix a length scale in $\parallel \hat{\mathbf{n}}$), resulting in networks with ‘equasi-smectic’ ordering, i.e., larger frozen-in orientational order (FOO) [6]. In contrast, V3LSCE swells in $\parallel \hat{\mathbf{n}}$ because its cross-linker (trifunctional type) has isotropic-like property which reducing the degree of FOO [7], comparing to the bifunctional cross-linker which has anisotropic property.

2. Side-chain LSCEs swelling is initiated by front propagation when the director reorients entering the LSCE. In V3LSCE, the velocity of front propagation $\parallel \hat{n}$ is twice faster than perpendicular to it which in turn is three times slower than in 8A2 [6,7].
3. We found an intriguing buckling instability in homeotropic 8A2 and V3LSCE when LMWLC propagates isotropically in $2D \perp \hat{n}$. We suggest that this could be a buckling-type transient because 5CB enters the network faster than the network can change to accommodate it. We observed that the transient line patterns were more irregular in the V3LSCE case than they were in 8A2 [6,7].

In this paper we report on a similar study of the swelling behavior as well as the deformation carried out on monodomain and polydomain main chain LCEs (MCLCEs). Most of the experimental works, so far, have focused on SCLCEs because MCLCEs are usually more difficult to synthesize than SCLCEs [11–14]. We have also studied the electromechanical and electrooptical effects of polydomain MCLCE which shows much larger effects [15].

2. EXPERIMENTAL METHOD

The polydomain MCLCE was synthesized in a one-step reaction [11–14], by dissolving the monomer 2-ethyl-1,4-phenylene bis [4-[4-(vinylloxy)butoxy] benzoate] ($C_{34}H_{38}O_6$), the chain extender 1,1,3,3-tetramethyldisiloxane ($C_4H_{14}OSi_2$) and 2.5 mol-% of the cross-linking agent pentamethylcyclopentasiloxane ($C_5H_{20}O_5Si_5$) in toluene, adding a Pt catalyst and reacting in a centrifuge cell at 5000 rpm and 70°C for about 4.5 h to form a film (Fig. 1). The polydomain MCLCE was obtained by deswelling the film on a water-surface to avoid any orientation. The crosslinking process was completed at 60°C for 4 days. In order to remove any soluble part, the sample was extracted with toluene. The glass transition T_g is about -10°C and the clearing temperature T_c is about 64°C . The monodomain MCLCE was obtained by mechanical stretching in order to obtain the uniform director orientation, \mathbf{n} , parallel to the stretching direction as described in [11–14].

Stress-strain measurements were performed stepwise with steps ranging from 0.2% up to 3% of deformation referred to the initial sample length with a self-constructed apparatus described elsewhere [18]. After each strain step, the sample was allowed to relax for 1000 sec in the case of the monodomain sample stretched parallel to the director. For monodomain samples deformed perpendicular and the polydomain the relaxation times were 2000 sec before the force was recorded. All measurement were carried out at 30°C in the nematic phase.

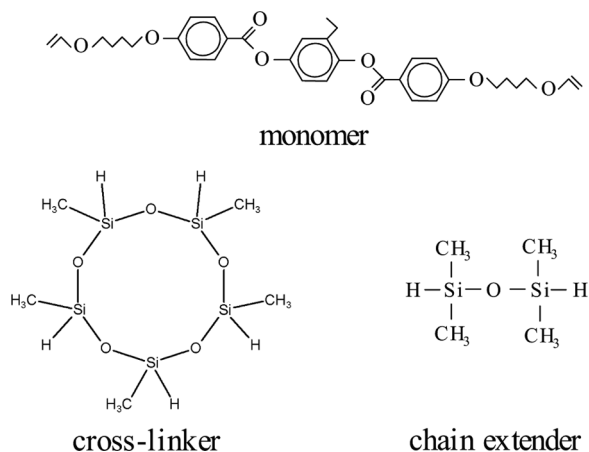


FIGURE 1 The chemical structures of the compounds prepared for the MCLCE.

To check the anisotropic swelling behavior of MCLCE samples, we prepared three types of rectangular MCLCE samples with different bulk director orientations \hat{n} ; MONO-1 is a planar aligned film, MONO-2 is a homeotropically aligned film, and the POLY films have no alignment. The films we prepare are $\sim 300\mu\text{m}$ thick and have an area of $1.0\text{ mm} \times 2.0\text{ mm} \times 0.5\text{ mm} \times 1.0\text{ mm}$. The samples are embedded in LMWLC, 4-n-pentyl-4-cyanobiphenyl (5CB) that was homeotropically aligned between two SiO coated glass plates. The thickness was controlled by a polymer (Mylar) spacer of $\sim 1\text{ mm}$. The swelling behavior was observed in a polarizing microscope (Nikon) equipped with a hot stage (Mettler Toledo FP90 Central Processor) as a temperature controller.

In the electromechanical effects measurement, the monodomain LCE sample with $\sim 20.0 \pm 1.0\mu\text{m}$ thick and with area $\sim 800\mu\text{m} \times 300\mu\text{m}$ is prepared. The sample is embedded in 5CB for swelling between two transparent indium tin oxide (ITO) electrodes with very clean SiO surfaces as in Ref. [19]. The SiO coating here is to avoid complications such as e.g., hydrodynamic effects arising from charge injection and to obtain uniformly homeotropic alignments. The cell gap between both electrodes was controlled by a $100\mu\text{m}$ polymer (Mylar) spacer. An alternating electric field of frequency $f = 100\text{ Hz}$ with rectangular waveform (repetition rate $\sim 1\text{ sec}$) is applied between the electrodes, i.e., $\mathbf{E} = (0, 0, E_z)$ at $T = 26 \pm 0.2^\circ\text{C}$. Measurements of the electromechanical effect were made using a polarizing optical microscope (Nikon E600WPOL) on which a charge-coupled-device camera

(SONY XC-75) was mounted. The mechanical deformation and temporal changes of the electromechanical effect were captured by a computer.

3. RESULTS AND DISCUSSION

3.1. Thermomechanical Effect of Dry MCLCE

The anisotropic thermo-mechanical effects for planar (MONO-1) MCLCE sample during the heating processes are shown in Figure 2. When increasing the temperature the planar samples shrank parallel to $\hat{n}(\hat{x})$ with a somewhat faster decrease in the vicinity of $T_c \sim 65^\circ\text{C}$ and expand perpendicular to $\hat{n}(\hat{y})$. The relative length changes of MCLCE as a function of temperature $\lambda_i(T)$, as the ratio of expansion/shrinkage length to the initial length in the isotropic phase ($T = 100^\circ\text{C}$), show that recent MCLCE reaches a maximum shrinkage of about 225% and a maximum expansion of about 30%. The shrinkage magnitude is much larger than that of 8A2 and V3LSCE [5,6,20]. This is because the polymer backbone in MCLCEs consists of mesogens, and therefore the mechanical property is more pronounced in MCLCEs than that of SCLCEs.

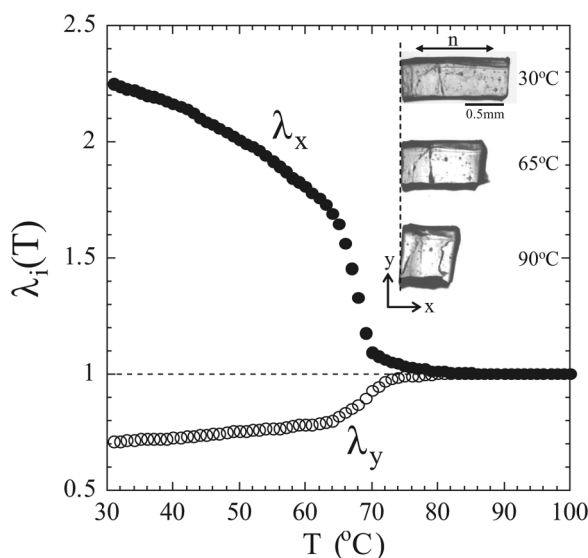


FIGURE 2 Temperature dependence of the relative length changes, $\lambda_i(T)$, of MONO-1 MCLCE during the heating process, $i = x, y$. Drastic shape changes are observed near $T_c \sim 64^\circ\text{C}$.

3.2. Stress-Strain Measurements

Thermo-mechanical measurements show that MCLCEs exhibit a large network anisotropy. Additionally, we performed stress-strain measurements parallel and perpendicular to the director to examine the mechanical anisotropy of MCLCE. As for slightly cross-linked MCLCEs, that are used here, high deformations are possible. We plot the true stress σ_t , which is referred to the actual cross-sectional area of the sample during stretching, against the deformation λ , which is the length of the strained sample divided by the initial sample length (Fig. 3).

Deformation of the monodomain parallel to the director (Fig. 3(a)) shows a linear rubber-like response in the initial part. At higher deformations, the stress is no longer linear but rises exponentially. We were able to fit the stress-strain measurements with the same exponential function described in [21]. Despite the different chemical structure of the network, the behavior is very similar to the one observed previously. The initial slope, which corresponds to the elastic modulus equals 2.2×10^5 Pa, and is therefore a reasonable value for the modulus which is expected from the amount of cross-linker introduced chemically. The exponential factor of the fitting function is 3.5.

A totally different mechanical response is observed on deformation perpendicular to the director (Fig. 3(b)). After a threshold deformation of $\lambda = 1.05$, the sample becomes soft and almost no increase of the stress has been found. This mechanical behavior is well known for both side and main chain elastomers. The main difference is the length of the plateau. In the side chain case the end of the plateau, which is defined by an increase of the stress, has been found at deformations of $\lambda = 1.6$ [22]. In contrast we find an almost soft deformation up to $\lambda = 3.05$ for the main chain type. The length of the plateau is directly correlated with the anisotropy of the polymer backbone [22], and therefore a longer plateau monitors a higher backbone anisotropy of MCLCEs. The σ - λ slopes at larger than $\lambda \sim 3.25$ in Figure 3(b) and $\lambda \sim 2.3$ in Figure 3(c) are $E \approx 1 \times 10^5$ Pa and $E \approx 1 \times 10^5$ Pa, respectively. In principle it should be possible to correlate the network anisotropy to the swelling anisotropy discussed later. But when swelling an anisotropic network with an anisotropic solvent, the remaining anisotropy of the polymer backbone in the swollen state is unclear. However, from the thermo-mechanical measurements of swollen MCLCEs, which have not been performed up to now, it should be possible to correlate the data obtained from stress-strain measurements with those of the swelling. This will be analyzed in detail in a forthcoming paper.

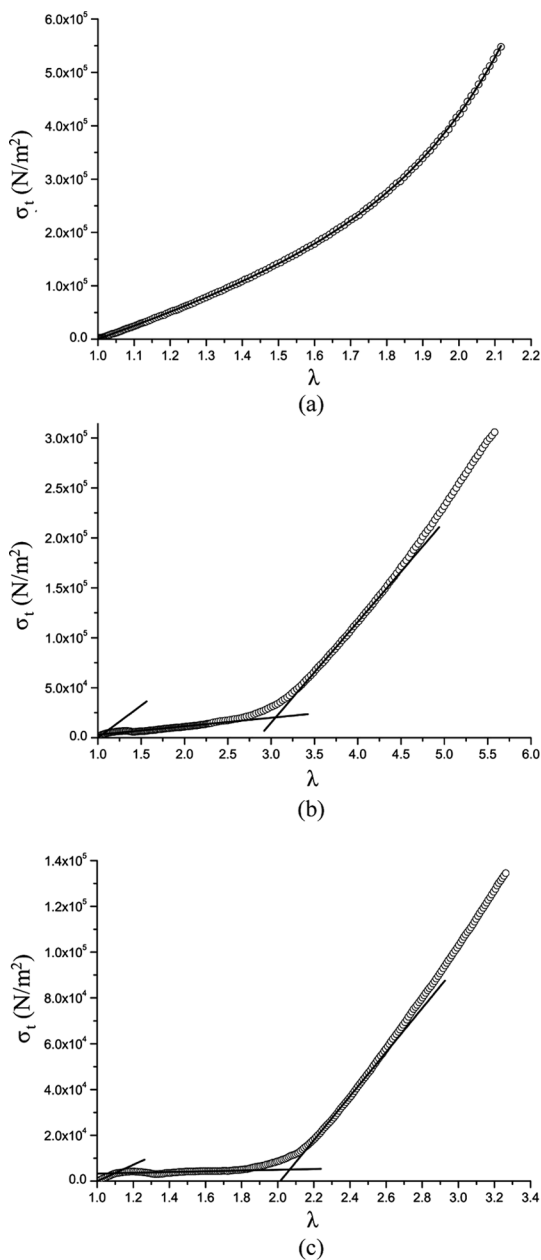


FIGURE 3 Stress-strain curve of the monodomain sample steched (a) parallel, (b) perpendicular to the director and (c) the polydomain sample. λ is the length of the strained sample divided by the initial sample length.

Deformation of the polydomain sample (Fig. 3(c)) reveals a very similar behavior to that of the monodomain perpendicular despite the different mechanism. After a threshold of about $\lambda = 1.08$ a plateau of the stress is observed, which is associated with the rotation of the local director towards the axis of applied stress. In contrast to the deformation of the monodomain sample perpendicular to the director, where a full reorientation of the sample causes a longer plateau, only the misaligned regions have to rotate in the case of a polydomain. Therefore a shorter plateau for polydomain nematic elastomers is observed, being as well determined by the chain anisotropy according to theory [23]. Here we find a plateau up to $\lambda = 2.05$, that exceeds that of side chain elastomers, where the director rotation is completed at $\lambda \approx 1.2$ [24].

3.3. Swelling Behavior

We define the relative length change, $\lambda_i(t)$, for swelling as the ratio of the swollen length $\ell_i(t)$ to the initial dry length ℓ_i^0 is x , y , or z . Length measurements were made in the middle of the each edge.

The shape changes during swelling of monodomain and polydomain MCLCEs in a homeotropically-oriented LMWLC (5CB) at room temperature are shown in Figure 4. During the swelling process, there are significant shape changes in both planar (MONO-1) and homeotropic (MONO-2) samples $\perp \hat{\mathbf{n}}$ and a smaller change $\parallel \hat{\mathbf{n}}$. Swelling

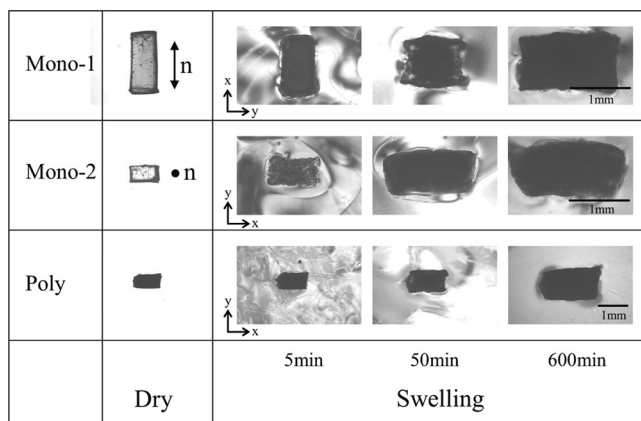


FIGURE 4 Typical swelling dynamics of MCLCEs in 5CB at room temperature. The numbers refer to minutes after the dry MCLCEs are embedded in solvent.

properties of MCLCE in LMWLC are also anisotropic. In our previous results on the swelling dynamics of 8A2 in 5CB, no dimension changes $\parallel \hat{n}$ could be observed [6]. However, V3LSCE samples swell $\parallel \hat{n}$ [7] which the swelling magnitude depends on the cross-linking density. From those studies we conclude that the swelling behavior depends on the cross-linking density and the functionality of the crosslinker, i.e., FOO and elastic interactions [6,7]. In the present study, the dimension change of MONO-1 MCLCE $\parallel \hat{n}$ show the shrinking (in first 10 min) and the elongating processes before it saturates (in 200 min). This is caused by different network properties between the networks parallel and perpendicular to \hat{n} . The networks $\perp \hat{n}$ are more anisotropy and less elasticity than that of $\parallel \hat{n}$ (see Fig. 3).

Figure 5(a) shows the relative length changes λ_x and λ_y , parallel and perpendicular to \hat{n} of swelling planar MONO-1 sample. The speed of the deformation by swelling, $\dot{\lambda}_y(\perp \hat{n})$, shows a maximal value at 10 min. At that time as $\dot{\lambda}_y(\perp \hat{n})$ can not follow $\dot{\lambda}_y$, shrinkage to the network $\parallel \hat{x}$ simultaneously occurs. After 20 min $\dot{\lambda}_y$ becomes small, then $\dot{\lambda}_x$ recovers to positive value and λ_x saturates at 200 min. Here λ_y increase exponentially in time and saturates after 12 hours, much slower than for the 8A2 LSCEs and V3LSCEs [6,7]. The time constant for swelling is $\tau\lambda_y \sim 175$ min. It is larger than the values observed in 8A2 and V3LSCE [6,7] (see Table 1). In saturation, $\lambda_x(t \rightarrow \infty)$ and $\lambda_y(t \rightarrow \infty)$ is ~ 0.93 and ~ 3.60 respectively.

In the homeotropic MONO-2 sample (Fig. 5(b)), both λ_x and λ_y increase exponentially in time and saturates after 3 hours. For the homeotropic sample, the time constant for swelling is $\tau\lambda_X \sim 39$ min and $\tau\lambda_y \sim 33$ min, and the saturation values of λ_x and λ_y are ~ 3.67 and ~ 3.31 respectively. These time constants are shorter than that of planar ones, as well as in swelling SCLCEs (see Table 1). These are due to reorientation process of the 5CB-director and elastic anisotropy.

The polydomain MCLCE swell isotropically similar to the polydomain 8A2 [6]. λ_x and λ_y , as well as λ_z , increase in time with the swelling time constant $\tau_{poly} \sim 192$ min and saturate at about $\lambda_{x,y} \sim 2.3$ (Fig. 5(c)). The time constant for swelling in polydomain MCLCE is longer than that of monodomain MCLCE because there are many domain walls where reorientation of the 5CB director may be required. The degree of swelling, $q = \lambda_{\parallel} \lambda_{\perp}^2$ of monodomain and polydomain MCLCEs is $q_{mono} = 11.9$ and $q_{poly} = 12.1$, respectively. These values are larger than in swelling SCLCEs [6,7] (see Table 1).

The swelling quickly proceeds in the plateau area which shows extremely small Young's modulus and saturates at $\lambda_y \sim 3.4 \pm 0.1$ for monodomain and $\lambda_{x,y} \sim 2.25 \pm 0.05$ for polydomain where the slope of

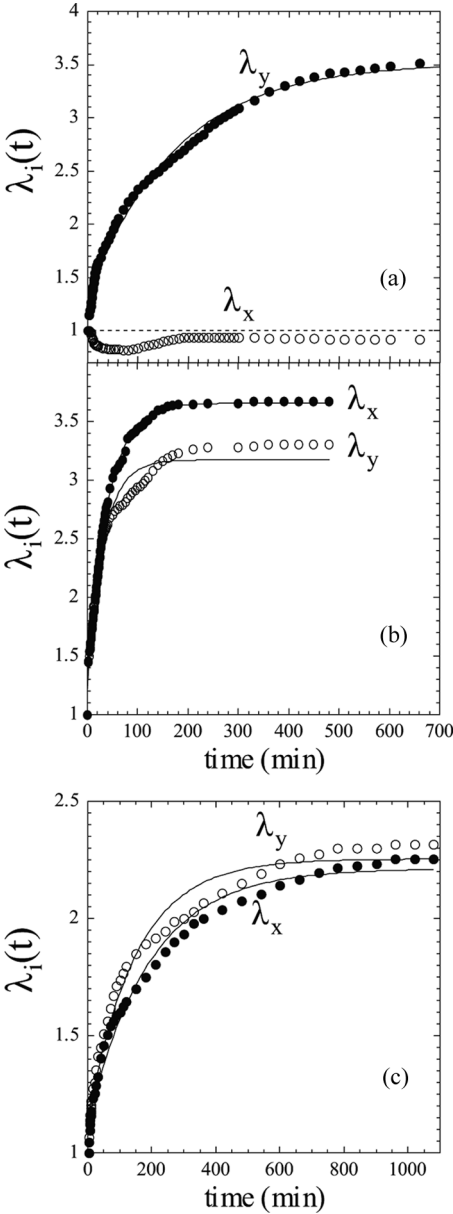


FIGURE 5 Relative length change, $\lambda_i(t)$, of planar monodomain (MONO-1) (a), homeotropic mon-odomain (MONO-2) (b) and polydomain (c) MCLCEs during the swelling process in 5CB. The solid lines represent fits to simple exponential behavior.

TABLE 1 Swelling and Electromechanical Properties of SCLCEs and MCLCEs. Here, (\perp L) is for the MCLCE Director and the 5CB are Perpendicular Each Other (Swelling Planar), and (\parallel) is for the MCLCE Director and the 5CB are Parallel Each Other (Swelling Homeotropic)

Sample	τ (min)	q	V_{th} (volt)	Max. contraction (%)
8A2 Mono ^{6,19}	15.9 (\perp L), 7.22(\parallel)	~ 3.2	~ 0.5	14
8A2 Poly ^{6,19}	20.7	~ 5.8	~ 0.5	14
V3 Mono(3V3) ⁷	123 (\perp), 41(\parallel)	~ 7.5	~ 1.0	<5
MCLCE Mono	175 (\perp), 39(\parallel)	~ 11.9	~ 10	7
MCLCE Poly	192	~ 12.1	$\sim 10^{15}$	19 ^[15]

$\sigma(\lambda)$ curve becomes larger beyond the end of the plateau (compare to Fig. 3). This means that the swelling process is limited by network elasticity, i.e., the elastic-stress-limited swelling (ESLS).

The voltage dependence of the shape changes, $\lambda V = L(V)/L_0$, i.e., the ratio of the length with the applied voltage in the direction $\parallel \hat{n}$ to the initial length, observed in swollen monodomain MCLCE is shown in Fig. 6. No shape change observed in $\perp \hat{n}$. The maximum contraction is about 7%. This value is smaller than observed in polydomain MCLCE (Table 1) [15].

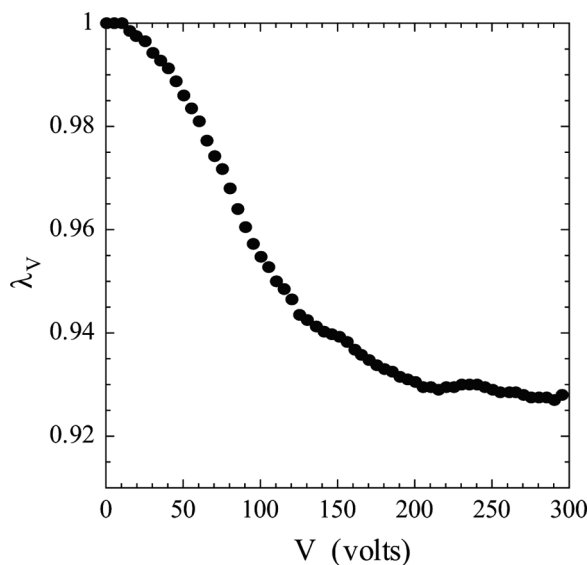


FIGURE 6 Voltage dependence of shape changes, $\lambda V = L(V)/L_0$, observed in swollen monodomain MCLCE.

This is because monodomain MCLCEs are crosslinked under a strong elastic tension with uni-direction. The threshold for the onset of the electromechanical effect for both monodomain and polydomain MCLCEs is about 10 V which is much larger than observed in 8A2 [19] and V3LSCE [7] (Table 1). That is, MCLCEs need larger electric field for electromechanical effects because of embedding the mesogenic unit itself in the polymeric backbone as composed parts, which requires stronger external force againsts strong elasticity of the backbone. Due to these strong elastic couplings the swelling time of 5CB increase a lot comparing to SCLCEs (see also Table 1). No electro-optical effects however can be observed in the swollen monodomain MCLCE, because of no transition between polydomain and monodomain unlike polydomain MCLCE [15].

4. SUMMARY AND CONCLUSION

We studied the swelling dynamics of monodomain and polydomain MCLCEs swollen with LMWLC (5CB) and comparing their swelling, elasticity and electromechanical properties to SCLCEs as summarized in Table 1. Good agreement of the swelling process of MCLCEs with the stress-strain data indicates the limitation of swelling by elastic properties of the networks i.e., ESLS. Faster swelling is observed in the plateau area of $\sigma(\lambda)$ curve which shows extremely smaller values of Young's modulus. Larger swelling times for MCLCEs in 5CB are due to suppression of the swelling process by strong elastic force of the MCLCE networks. The detail will be given elsewhere [25]. The degree of swelling of monodomain and polydomain MCLCEs is $q_{mono} = 11.9$ and $q_{poly} = 12.1$. Slightly more 5CB (1.5 times more) is absorbed in the MCLCE networks because of better affinity. For the swollen MCLCE, thus, network interactions with mesogens are much stronger in SCLCE. Therefore the threshold for the electromechanical effects of MCLCE, as shown in Table 1, is much larger than that of SCLCE.

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